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FORUM FOR THEORY AND PRACTICE OF REPRESENTATIVE SAMPLING

IPGSA
INTERNATIONAL PIERRE GY SAMPLING ASSOCIATION

SST

SAMPLING SCIENCE & TECHNOLOGY

Development and Evaluation of Efficient Field Deployable Sample Preparation

Fit-For-Purpose Representative pXRF

Heterogeneity Tests Briefly Reviewed
Estimating the FSE Variance

Optimizing the Sample Mass
A Simpler and Cheaper Way

Theory of Sampling (TOS)
Up For Debate?

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CONTENTS

Cover Photo

On-site deployment of Block10 field sample preparation equipment, Victorian Goldfields, Australia.
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BLOCK 10

We are most grateful to Block 10 Pty Ltd for sponsorship of this issue.

Ambitions for SST Are (Sky) High

By Kim H. Esbensen (Editor)

DOI: 10.62178/sst.002.001

It is a pleasure to welcome readers to the second issue of Sampling Science and Technology. There is a lot to be satisfied with – first of all, a steady influx of high-quality manuscripts covering a wide field of topics.

- This issue begins with a down-to-earth example of practical applied sampling – literally down on the earth and in the field – in this issue's feature article "Development and evaluation of efficient field deployable sampling preparation: fit-for-purpose representative pXRF".

- Theoretical reflections re. TOS, in this case related to how to conduct heterogeneity testing, an opening salvo deliberately intended to start a debate. There are three articles collected around this topic, one in which is presented a phenomenological whifferdill regarding the 'liberation factor' (is it needed or not) as well as an immediate response hereto. If these three articles do not succeed starting a debate, the editor does not know what will! Readers are welcome to weigh in …

- Alan Rawle, sampling historian *extraordinaire* continues his erudite series on "Sampling Giants", this time featuring D.W. Brunton, an early sampling legend.

- The sampling standard "Representative Sampling – Horisontal Standard", DS3077, has been launched in an augmented 3rd revision (Oct. 2024). Even better, it is already commencing a journey with the aim to become an ISO standard. This has been a goal for IPGSA since 2008; today everybody rejoices.

International Pierre Gy Sampling Association (IPGSA). In this issue readers will find a comprehensive report on the 11th World Conference on Sampling and Blending (WCSB11), complete with the Pierre Gy Sampling Gold Medal committee's justification for the two 2024 awardees (the number breaking with a long tradition) along with IPGSA President Ralph Holmes' justification for IPGSA's first ever Distinguished Service Award.

From our own little world. SST#2 is the first issue produced by our now complete editorial team: editor, editorial assistant and publisher (see the journal 'Imprint'). In the same context, we are finally able to present SST's inaugural **Editorial Board**, which commences activities as soon as this issue has been released. SST is proud of this highly experienced and impeccably competent collegium of sampling experts from industry, academe, consultancies – and beyond. The seven board members are presented on page 51. With this scientific sounding board in place, SST has very high ambitions to become a premier scientific magazine. **Readers are encouraged to contribute to this development!**

Finally, readers will undoubtedly appreciate early insight regarding the venue and dates for **WCSB12** (2026), at the Camborne School of Mines, University of Exeter, Penryn Campus, Cornwall, UK (why not plan a family holiday in Cornwall as well?)

Editor's scope. Since 2017 the IPGSA takes care of the world sampling community with organisational efficiency and elan. While the 'Sampling Column' in Spectroscopy Europe/world (2015–2023) is now defunct (see SST#1), as is the erstwhile TOS Forum, our community is now in the satisfactory position to enjoy continuation of both endeavours in the form of 'Sampling Science and Technology'. The path forward for IPGSA, the WCSB conferences and our new scientific magazine is on the absolute right track – and all have (sky) high ambitions for the development of our science!

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Development and Evaluation of Efficient Field Deployable Sample Preparation: Fit-For-Purpose Representative pXRF

By Steven Russell[1], Ross Cunningham[1], Chris O'Haire[1] and Harrison Martin[1]

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ABSTRACT

Since acquiring the designs of the REFLEX™ Instruments Sample Preparation range in early 2024, Block10 has embarked on an ambitious redesign, updating the high-quality crusher, mill and press equipment to meet the evolving and growing interest for small, portable, field deployable analysis solutions in the mining and exploration industries. This article outlines the features and benefits of this expanding product range, presenting Replication Experiments (TOS) performance evaluation and other test-work to highlight the high precision, low bias, highly effective safe operating potential of Block10 preparation equipment for pXRF and other analytical modes.

1. Introduction

When analysing geological samples three things matter: quality, cost and turn-around-time. High quality central laboratories are located in most developed resource regions in the world, and on-site at larger mining operations. These laboratories are capital intensive, with high investment required for permanent equipment, personnel and processes. Such operations require large sample quantities and a continuity of work in order to achieve a reasonable unit cost.

The scale of these laboratories, often processing thousands of samples daily, means that they are invariably located in the larger and more established locations. Those working further afield – the explorers, drilling sites and junior operations - must send their samples over great distances (and at great expense) and wait weeks or even months for assay results. The cost of shipping and submitting samples, whilst significant, is often dwarfed in comparison to the opportunity cost of slow results; processes run sub-optimally for longer, and sampling resources cannot be targeted to greatest effect. But results need not be "online" or "real-time" in most instances; the value is unlocked in having confidence in turn-around-times measured in hours, not months.

There are many examples of mobile sample laboratories being successfully deployed into remote locations to combat this tyranny of distance, but the scale of these "mobile" solutions is usually based on shipping container multiples, and that is for the sample preparation equipment alone.

The introduction and widespread availability of portable XRF analysis (pXRF) instruments has opened new opportunities to address these challenges. By 2001 the technology had advanced to a level to make lightweight, safe, reliable, accurate instruments a reality, and since then, their application and use has proliferated, with multielement analysis on a sample possible in minutes. Most recently, the patented detectORE™ process (Portable PPB, 2024) has expanded the applicability of pXRF to attain low level gold results down to low parts per billion (ppb) levels. Portable-XRF has also proved popular within the larger laboratories, as an efficient, cost-effective multielement analysis solution and for initial screening purposes.

Recognising the opportunity for pXRF, and seeing very limited solutions in the market, REFLEX™ Instruments, a leading IMDEX brand, developed a crusher, disc mill and sample puck press for the optimal preparation of samples for pXRF analysis.

1 Block 10 Mining Pty Ltd, Australia. www.block10.com.au

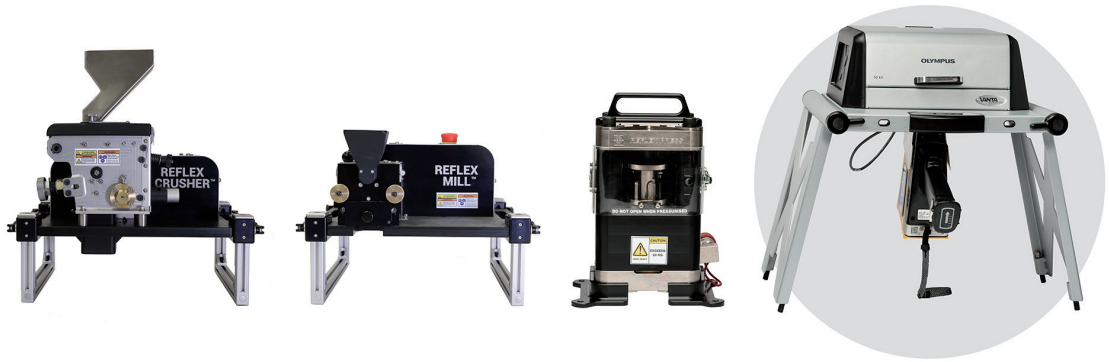


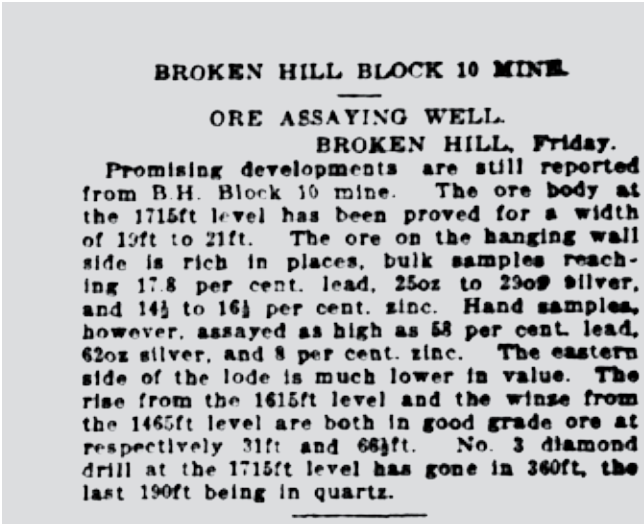
Figure 1: The range of REFLEX Instruments.

The attributes of reliable operation, compact size and high-quality output make this product suite highly applicable to other analytical techniques too; their use is by no means limited to pXRF.

In early 2024, Block10, a company specialising in the development of automation, sensing and mechanical solutions for the mining industry, acquired the designs as the new vendor of the former REFLEX-Crusher, REFLEC-Mill and REFLEX-Press in-field preparation tools (IMDEX, 2024). In consultation with existing clients and users, a design review and update has been undertaken, culminating in the release of the new BX-C Crusher and BX-M Mill.

Leveraging the latest in motor and manufacturing advancements, these new releases offer significant equipment weight and size reduction, improved safety, many optional customisations, and high-quality results. The product range has also been expanded to include the BX-R Riffle Splitter, and further developments are ongoing to meet the specific requirements of the industry into the future.

A comprehensive evaluation of the Block10 equipment performance has been undertaken, with results highlighting the powerful sample preparation potential, whether in-field, remote, or at central laboratories.



BROKEN HILL BLOCK 10 MINE.

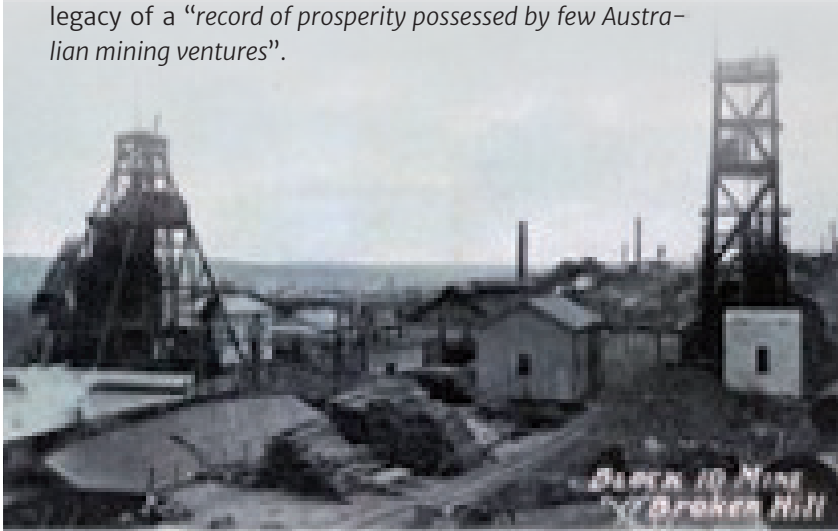
ORE ASSAYING WELL.

BROKEN HILL, Friday.

Promising developments are still reported from B.H. Block 10 mine. The ore body at the 1715ft level has been proved for a width of 19ft to 21ft. The ore on the hanging wall side is rich in places, bulk samples reaching 17.8 per cent. lead, 25oz to 29oz silver, and 14½ to 16½ per cent. zinc. Hand samples, however, assayed as high as 58 per cent. lead, 62oz silver, and 8 per cent. zinc. The eastern side of the lode is much lower in value. The rise from the 1615ft level and the winze from the 1465ft level are both in good grade ore at respectively 31ft and 68½ft. No. 3 diamond drill at the 1715ft level has gone in 360ft, the last 190ft being in quartz.

The BHP Block 10 Co. Ltd was floated on March 14, 1888 and struck ore in December 1889. The mine went on to produce over 40 million oz of Silver, 400,000 tons of Lead and 400,000 tons of Zinc over a 35 year life. With declining grades and a collapse in metal prices following the end of World War I, Block 10 was liquidated in 1924, and its mine purchased by the Broken Hill Proprietory Company Ltd (BHP).

A century later in 2022, Block 10 Pty Ltd was established by decendents of these early mining pioneers, with the spirit of innovation and enginuity continuing the legacy of a "*record of prosperity possessed by few Australian mining ventures*".



2. Block10 Sample Preparation Methodology

Although extending its involvement upstream to sample collection on specific projects, the Block10 Sample Preparation Methodology typically begins with a sample, extracted previously by others and presented in a calico bag or similar.

The specific primary sampling method – reverse circulation (RC) drill chips, diamond core, manual or mechanical grab or auger samples, cross-belt or falling-stream process samples – and the relative merits of these methods (Theory of Sampling (TOS)), is not considered here; the focus is on accurately preparing and representing these samples for analysis in the field.
Optimal presentation usually involves pressing a finely milled sub-sample into a 30mm puck, with the high hydraulic force and quality dies resulting in a smooth surfaced, well-mixed sample. Whereas higher homogeneity is possible by fusing a bead (for example, with an xrFuse Electric Fusion Machine (XRF Scientific, 2024)), the requirements for high temperature (1200°C) operation, expensive platinum crucibles, and additional flux dosing means that a pressed puck can reasonably offer an optimal balance between portability, performance and cost. Studies (Rohiman & Arifin, 2020) have also validated that pressed samples are superior for trace element (<100ppm) analysis, where fused beads are impacted by high (flux) dilution.

The Block10 general methodology for sample preparation for multi-element analysis can be summarised as:

a) Obtaining a representative sample (typically >=500g), with particle size <30mm (TOS to the fore).
b) Crushing to <2mm in the BX-C crusher.
c) Dividing to 250g, then 125g in the BX-R riffle splitter.
d) Milling to <100µm in a BX-M mill.
e) Riffle splitting again to (a nominal) 62.5g.
f) Scooping 10mL into a sample die. Some samples may require the addition of a binder.
g) Pressing the sample in the BX-P hydraulic sample press.

The samples may then be analysed with a pXRF instrument, or other techniques.

Many variations to this standard methodology exist, and in each application the steps, settings and processes can be adapted to best suit the specific requirements.

For example:

- Some fine samples may not require crushing and can be milled directly.
- Some samples containing heavier elements at macro levels may be analysed as a milled powder rather than a pressed puck.
- Different sample materials may require crushing & grinding at smaller size to achieve homogeneity or may tolerate a wider size for faster operation.

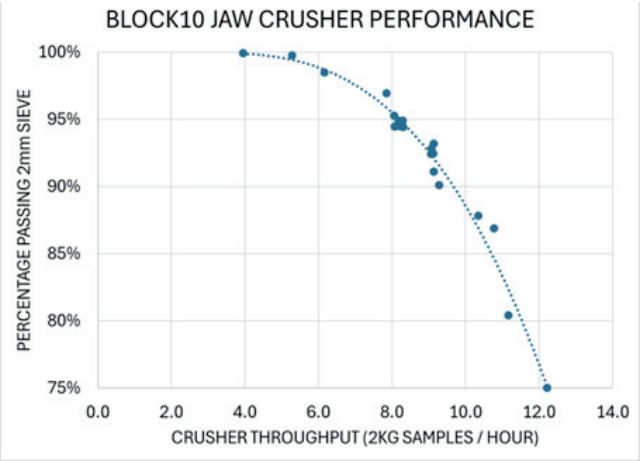
Credit: Block10; used with permission.

Figure 2: The updated Block10 BX-C Crusher and BX-M Mill

3. Crushing and Milling Specifications

Regarding crushing and milling specifications, typical targets are 90% passing 2mm and 90% passing 100µm respectively. These are the settings used for the testwork included in this article. Adjusting these settings is a quick process using a supplied hand tool to turn an adjusting screw.

In selecting an appropriate crushing specification, the throughput of the machine should be considered as one of the competing objectives: finer crushing to smaller particles (higher percentage passing 2mm) vs faster processing. Several samples of "bluemetal" (<20mm screened basalt) were tested with varied jaw gap adjustments, to plot the curve in Fig. 3, showing a highly productive ~10 samples / hour if processing 2kg samples at a 90% passing 2mm specification. These performance curves are dependent on material type and can also be impacted by other factors (e.g. sample moisture content), so results will vary for different sample types.



Credit: Block10; used with permission.

Figure 3: Block10 crusher throughput performance

Sizing specifications require routine validation as part of the QA/QC process, most simply and reliably achieved by sieving a sub-sample in the applicable sieve size. For crushed size specification, dry sieving suffices, unless there are high concentrations of agglomerating fines present, whereas wet sieving is required to accurately determine the mill specification. There are other alternatives such as laser or vision-based particle size analysers.

The crushing stage takes the sample to a 2mm top-size. Portable XRF analysers typically have a FoV spot size between 3mm and 8mm (MicroXRF, 2024), so in most cases it is best practice to further reduce the particle size before analysis to present a better mixed sample.

Samples are typically milled to a range between <50µm and <200µm specification, with ~75-100µm preferred.

- If particles are too large, they may not bind together properly when pressed, and pucks simply crumble.
- Larger particles closer to the sample surface can also "shadow" smaller particles behind them which may then not be quantified properly.
- Lighter elements (e.g. Na, Mg, Al, Si) are only detectable at shallow depths with low energy X-rays"; Na for example can only be analysed in the first ~10µm of sample. When analysing for these lighter elements the impact of surface roughness and particle size is much more pronounced than for heavier elements (e.g. Fe, Cu), where larger penetration depths make for less susceptibility to particle size influence.

4. Machine Safety

Electrical equipment (e.g., the crusher and mill) are fitted with emergency stop circuits, and sensor interlocks to prevent operation whilst the covers are open, or sample trays/chutes are missing. The electrical enclosure is fitted with tamper resistant fasteners.

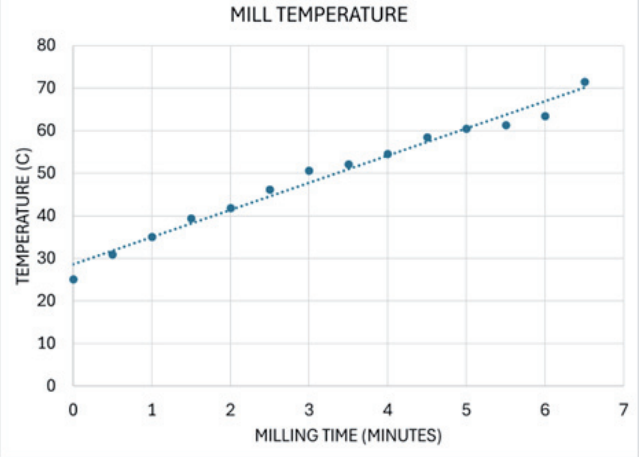
The updated BX-C and BX-M equipment features dual redundancy safety, to give improved safeguarding of the machinery to SIL 2 (IEC 61508) / Category 3 (IEC 60204-1). The use of a safety relay allows detection of short circuits and any lack of simultaneity between the two channels trips the emergency stop.

5. Milling Specification – Thermal Impacts

Whilst a lower milling specification (in terms of output particle size) is often desirable, it requires longer milling times, and the corresponding increase in duty cycle causes an increase in temperature.

Unlike a conventional ring or puck mill, where the bowl is enclosed and vibrating, the Block10 BX-M mill, with its rotating disc design allows much better temperature dissipation, via aluminium heatsinks that draw heat away from the milling disks. Fig. 4 shows a typical temperature gradient for a milled sample.

Geological samples are typically dried at 105°C, so should suffer no ill effects even if milled for extended times at temperatures below this, however if temperature sensitive samples are processed, consideration should be given to detecting and limiting the temperature.



Credit: Block10; used with permission.

Figure 4: Milling temperature.

Block10 offers several upgrade options including:

- Temperature sensor for monitoring,
- Temperature controls, to regulate operation and inhibit milling if temperatures exceed a threshold,
- Water Cooling.

Ultimately, the best solution is to limit the heat generation in the first place, either by a relaxed milling specification or smaller samples (both reduce the milling time and duty cycle), or with multiple mills if one unit is being over-utilised.

6. Dust Control

Dust control (or lack thereof) has dual impacts on personnel safety and sample quality.

From a quality perspective, higher extraction airflow is not necessarily better, as it removes sample material (particularly fines) and biases the result. However, too low an airflow increases the likelihood that material will build up and carry over between uses, contaminating subsequent samples.

The key to optimal extraction is to focus on fugitive dust only, leaving particles that may have become airborne but still within the sample chute / jaws / trays every opportunity to remain as part of the sample flow. Only once dust exits the vessel should it be extracted away (as this sample material was lost to the process anyway, so dust extraction does not further bias the results).

The Block10 BX-D dust extraction system is configured to give operational flexibility with one, two or three machines and includes a HEPA-14 filtration system, reducing airborne contamination and improving cleanliness.

The HEPA-14 filter (to EN 1822:2019) captures at least 99.995% of particles 0.3µm or larger. It does not remove the need for personnel to wear appropriate PPE (including respiratory protection), in accordance with local requirements, but it does give added protection and reduced contamination.

Samples by definition contain unknown components, and with an ever-increasing understanding of the risk of dust exposure (silica, asbestos) it is becoming increasingly critical to manage and suppress dust generation in all workplaces. Whilst central laboratories typically have ducted dust extraction systems, the remote/field operations often lack this infrastructure, so the BX-D equipment offers an efficient, lightweight and portable means to address dust.

The inclusion of a HEPA-14 filter ensures that harmful particles are efficiently and safely captured - rather than being recirculated into the environment as is often the case with other vacuum systems.

7. Contamination

The typical sample loss in the Block10 crushers are <0.5% by mass, with effective dust control in place. Only a portion of this is carried over to the next sample, with the balance extracted or escaping as airborne dust. The crusher infeed chute can be removed via a quick release to give quick access to the crusher jaws between samples for visual inspection, and if necessary, an additional vacuum or brush if there is visible carryover material remaining in the jaws.

The Block10 mills are also readily cleanable and can be opened in seconds without tools. Best practice is to open the top of the mill every cycle and brush any residue into the outfeed bin to ensure it remains with the sample. Once the sample bin is removed, a more aggressive suction and brushing, including for the inside of the milling disks, ensures any contamination to the next sample is minimised. Following this 'good laboratory practice', with dust extraction, milling losses are typically <1%.

Figure 5: Cleaning the crusher between samples

The other potential source of contamination is from the active wear parts – the jaw plates in the crusher and the discs in the mill. These parts wear with use, and the lost material ends up as a contaminant in one and more samples.

Block10 crushers and mills feature full tungsten carbide wear parts, for ultimate hard-wearing performance. Hardened steel wear parts are available if tungsten (W) is an analyte of interest.

Most soft(er) samples may not perceptibly wear the tungsten surface, however tests milling a highly abrasive, high surface area graded silica sand (0.6–0.8mm) did indicate W contamination, at low but detectable levels. Such results were not observed when milling other reference materials. In the vast majority of cases, W contamination is either negligible or of no significant interest, so tungsten carbide offers best performance, with optimal lifetime.

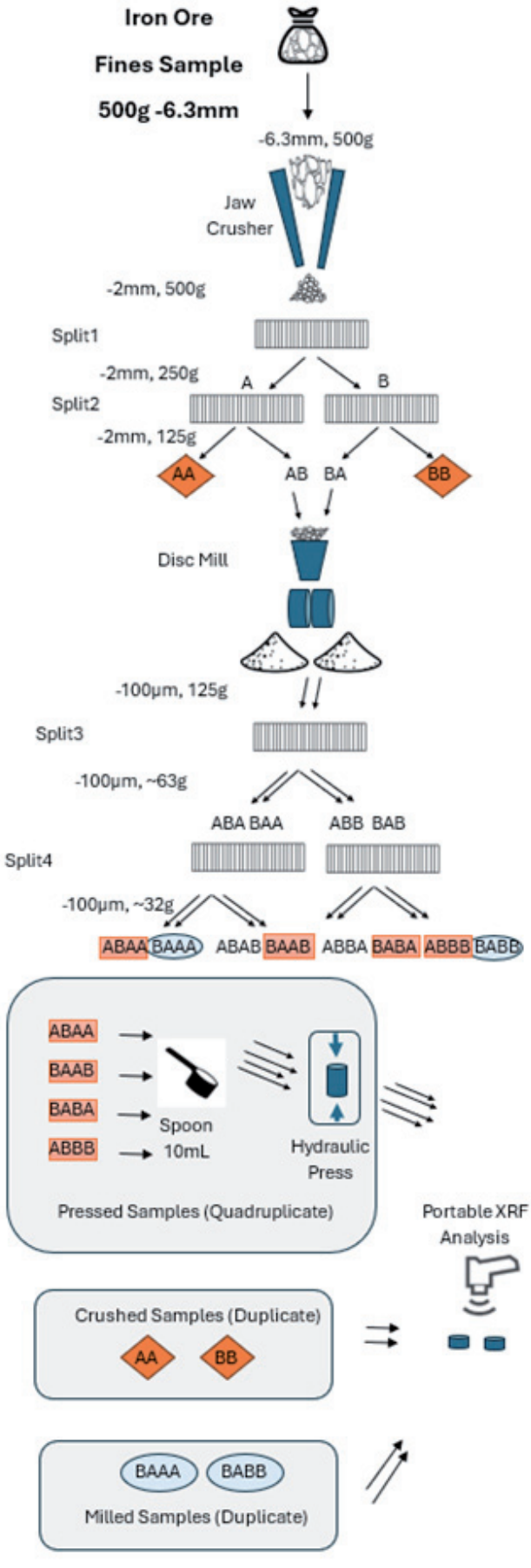
8. Example: Iron Ore Fines Sample

As an illustration of the effects of crushing, milling and pressing, an iron ore fines sample (<6.3mm top-size) was processed and analysed with an Evident Vanta™ Handheld XRF Analyzer, following the methodology outlined above to obtain quadruplicate pressed pucks. Separate splits were also taken at different stages of the full field sample preparation pathway, (see Fig. 6):

- Three "grab" samples with 10mL scoops were taken directly from the sample bag and analysed without further preparation.
- Duplicate (riffle split) samples analysed after first crushing to 2mm top size.
- Duplicate (riffle split) samples were analysed after milling to 100µm top size.

For analysis of loose powders, a cup with polypropylene or mylar film bottom is typically used to present a uniform 'flat' sample surface which is then analysed from below with an upwards projecting pXRF instrument. Loose powder samples do not have the same sample packing density and surface smoothness as a pressed puck but can be simpler to prepare (notwithstanding the assembly of the consumable cup and film can be fiddly, time consuming and costly).

The relative sampling + analysis variability (RSV), or Relative Standard Deviation (RSD), also known as the Coefficient of Variation (CV), is defined as the ratio between the standard deviation (σ) and the mean (µ), RSV = σ / µ of a replicated sampling or sub-sampling operation + analysis. As a dimensionless measure (i.e. expressed as a percentage) it allows for effective comparison of the precision between assays as a function of different sub-sampling operations.



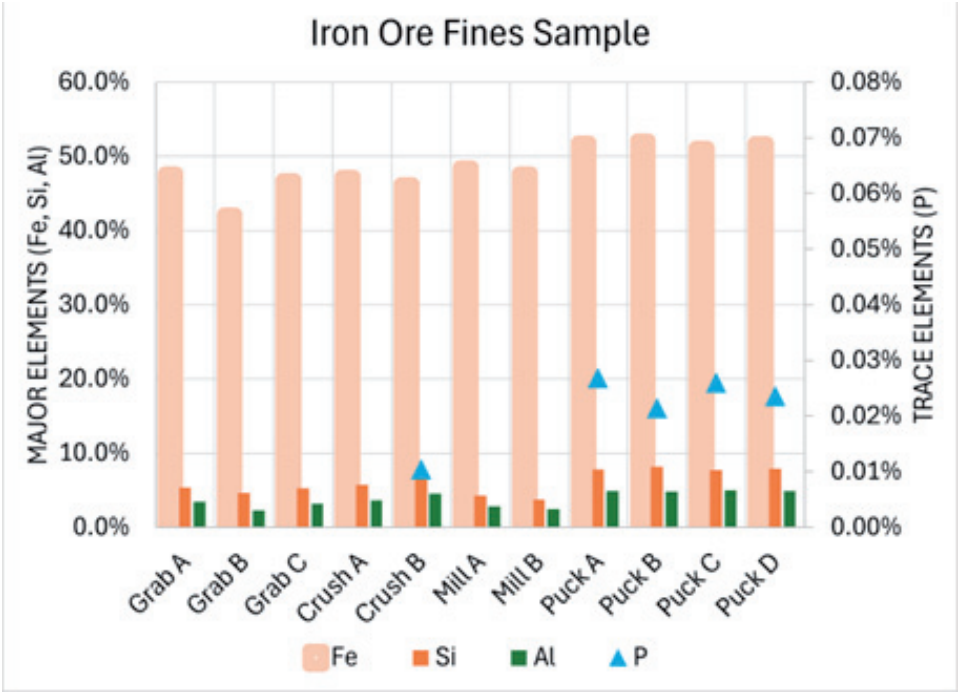
Credit: Block10; used with permission.

Figure 6: Process flow for iron ore sample preparation evaluation

Although in this limited first evaluation the data sets are small (more expansive testing will be performed in future), it is nonetheless illustrative in highlighting the benefits of well-executed sample preparation in the field. The RSV% is calculated across four main elements of interest in the iron ore sample, the valuable element being iron (Fe), and the deleterious aluminium (Al), silicon (Si) (as silica) and phosphorus (P), as presented in Fig. 7 (concentration) and Tab. 1 (RSV%).

The assay precision for Fe, a heavier element existing in high grades (~50%), improves with additional sample preparation (crushing, milling, pressing), but only incrementally; if the only concern was for an Fe result, it may be perfectly acceptable to stop at crushing, or analyse the fines sample at its raw size (<6.3mm).

Deleterious elements are also of significant interest, since they can have a negative impact on the produced ore value and must therefore also be quantified. For Al and Si, both much lighter / lower energy elements and hence more susceptible to sample surface roughness, there is a marked improvement (reduction) in RSV% when the sample is pressed into a puck, Table 1. The most striking example of the benefits of pressed pucks concerns measuring phosphorus, P. As a light and low (trace) concentration element it fails to be detected at all for most of the preceding sample preparation steps; it is only after being pressed into a pellet that it can be measured reliably.



Credit: Block10; used with permission.

Figure 7: Iron ore sample prep + analysis comparisons.

Table 1: RSV% for replicated prep + analysis.

Sample Prep	n	RSV (Fe)	RSV (Al)	RSV (Si)	RSV (P)
Grab	3	6.5%	17.7%	6.3%	~
Crushed	2	1.6%	11.1%	8.2%	~
Milled	2	1.2%	8.2%	6.3%	~
Pressed	4	0.81%	1.4%	2.2%	8.7%

9. Pressed Puck Analysis

For sample analysis, pressed pucks provide superior results. The Block10 BX-P press can be used with two different dies:

- the standard die produces ~26mm diameter pucks from the sample material only, and typically have sufficient integrity to allow analysis, but may chip around the edges or crack during excessive transportation, handling, storage, etc.
- For longer lasting pucks the premium die may be used, where a plastic retaining ring (and optional caps) are used.

Credit: Block10; used with permission.

Figure 8: Block10 sample press dies.

'Infinite Thickness' is the minimum thickness a sample must have in order to absorb all the x-rays of the primary X-ray beam emitted from an XRF instrument. If the infinite thickness is not met, then some of the x-rays pass through and are lost from the sample and result in underreporting of some elements. (Portable Spectral Services, 2024) The eventual thickness of the pressed pucks varies with the compressibility of the sample material, but typically a 10mL volume of freshly milled sample is suitable for creating a well-formed puck, with sufficient thickness.

10. Binders

Many sample types will bond into a puck easily, under hydraulic pressure (Block10 uses 19 tonnes of force in producing 26mm pucks), however some will fail and further additives are required:

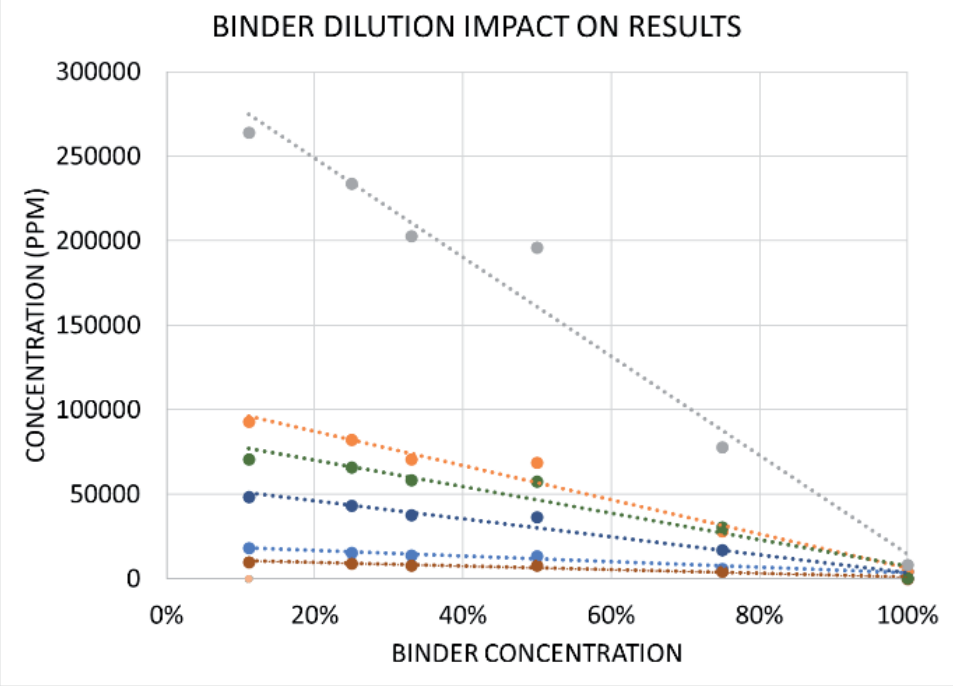
- For hard materials like high quartz samples, the puck may not bond properly without the addition of a wax / cellulose binder. Block10 produces a binder that is typically used in dilutions of between 3% and 12%; the binder is placed in a mixing vessel with 10mL of milled sample prior to puck pressing.
- Less commonly, samples that may fluidise under pressure (e.g. higher clay content) may also fail to form a puck; rather than bind into a solid, the sample behaves like a liquid and is simply forced out of the (small) clearances in the die components. In such cases some "roughage" may need to be added to the sample to ameliorate this fludising propensity; a milled bluemetal (basalt) or feldspar material added in ~25% dilution will usually suffice. Alternatively, the sample may be pressed at a lower pressure.

Whenever a sample is diluted with binder material, it is important that both the volume and composition of the binder are known, so that a correct compensation may be applied to the results.

The composition of the Block10 supplied wax binder is shown in Tab. 2. With a typical general composition of CnH2n+2, the bulk of the binder (~98%) consists of light elements that are outside the focus of pXRF mineralogical analysis (typically starting at Mg on the periodic table).

Table 2: Block10 binder chemical composition

Element	Concentration
Mg	0.42%
Al	0.45%
Si	0.84%
P	0.0070% (70ppm)
K	0.030%
Ca	0.050%
Ti	0.010%
Mn	0.0015% (15ppm)
Fe	0.030%
Cu	0.0003% (3ppm)
Zn	0.0004% (4ppm)
Th	0.0039% (39ppm)
U	0.0014% (14ppm)



Credit: Block10; used with permission.

Figure 9: Binder dilution impact.

Diluting the sample with a binder will be apparent in the results; however, for many samples in the grade ranges of interest, these low-level concentrations can be considered to have negligible impact on the results. The binder material also makes for a good reference blank, but it is challenging to prepare a puck at 100% concentration. Block10 can supply such reference blanks, although not yet as a certified reference material (certification is likely in the near future).

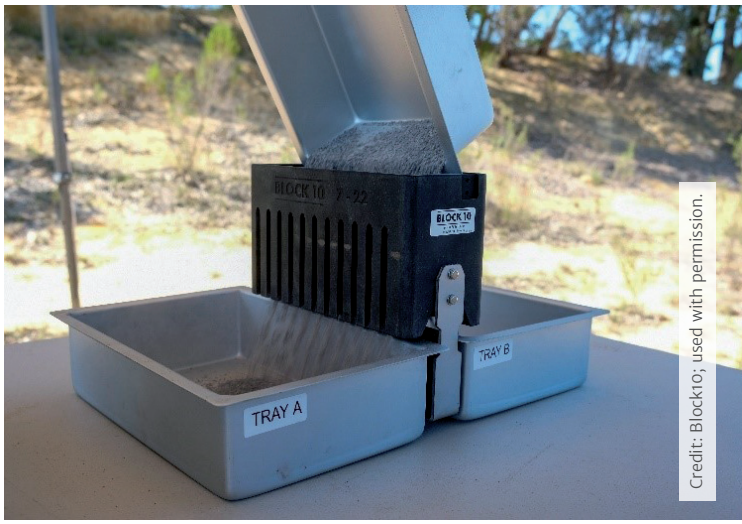
Even if assumed blank, the dilution impact of the added binder should be considered. At low level dilutions (e.g. 6%, as used for many of the samples in this testwork) the impacts are minimal, and where the focus is comparative rather than absolute results, may be disregarded, but at elevated levels (only used if necessary, i.e. previous attempts to form a puck with less binder have failed) the grades can be corrected (raised) proportionate to the dilution ratio.

Fig. 9 illustrates the impact on measured concentrations (highest to lowest Si, Al, Fe, Ca, Mg, Ti) for pressed pucks prepared from IMS-393 certified reference material and diluted with between 12% and 100% binder.

11. The Block10 BX-R Riffle Splitter

As the latest addition to the sample preparation range, the BX-R riffle splitter was developed to address the several sample division stages typically used when preparing samples with the rest of the Block 10 product range.

The BX-R riffle splitter features 13 chutes on each side for a 50:50 split. The chutes are 7mm wide to give >3x the maximum particle size after crushing (2mm) to prevent clogging. Although enclosed riffle splitters designs have been recommended as good practice (Esbensen & Wagner), the Block10 design is open, to allow constant visual inspection of the flowing particles. This is a more reliable configuration that limits the risk of internal sample hangup and carryover between samples. However, if an enclosed specification is required then a hood option is also available.



Credit: Block10; used with permission.

Figure 10: Block10 field deployable BX-R riffle splitter.

The splitter is machined from a single piece of high lubricity plastic. By machining rather than adopting the fabrication processes (bending, cutting, welding) used in most other rifflers, the geometry of the chute spacing can be much more tightly controlled, and a perfectly uniform chute width is key to the performance. The plastic construction means that it is very lightweight and can pack into its carry case along with size-matched lightweight aluminium trays for a highly portable splitting solution.

Splitting equipment is judged on two key criteria: performance precision and bias.

A. Khan's much-cited thesis (Khan, 1968) compared sample division methods, and concluded that rotary sample division was optimal, followed by riffle splitters. Khan considered a single sample mixture (60% fine / 40% coarse sand), calculating a standard deviation of 0.125% for rotary and 1.01% for chute riffling. A similar methodology using a 1:2 mixture of iron chips to sand, was conducted more recently (Nenuwa, Oke, & Sanya, 2018), with much less favourable rotary division performance; with standard deviation of ~2% and RSV of ~4%.

The Block 10 Portable Field Sample Preparation Equipment for pXRF is destined for extensive further assessment, planned to cover a wide range of rock types and relevant operating conditions. Systematic DOE (Design of Experiments) will be used.

Specifically, to evaluate the riffle splitting equipment at this point in time a test was devised utilising a binary mixture of white rice and chia seeds. Both are free flowing materials, but with a significant size disparity (see Fig. 11) that will cause segregation, and which also allow for easy separation by sieving. Both rice and chia seeds have good integrity and do not crumble easily, an important attribute for replication testing involving re-mixing of the original material batches.

This test was conducted at chia seed concentrations of 1%, 5%, 10%, 20%, 50%. Sets of replicated 4-fold sample divisions were performed at each concentration, with the component parts re-mixed completely after weighing the rice and chia fractions of the LH and RH splits (simulating 'analysis' of component concentrations).

Conceptually, a perfect single particle split should have a mean of 0.5 and an RSV of 100%, whereas when the number of particles approaches infinity, the RSV% should decrease asymptotically to zero.

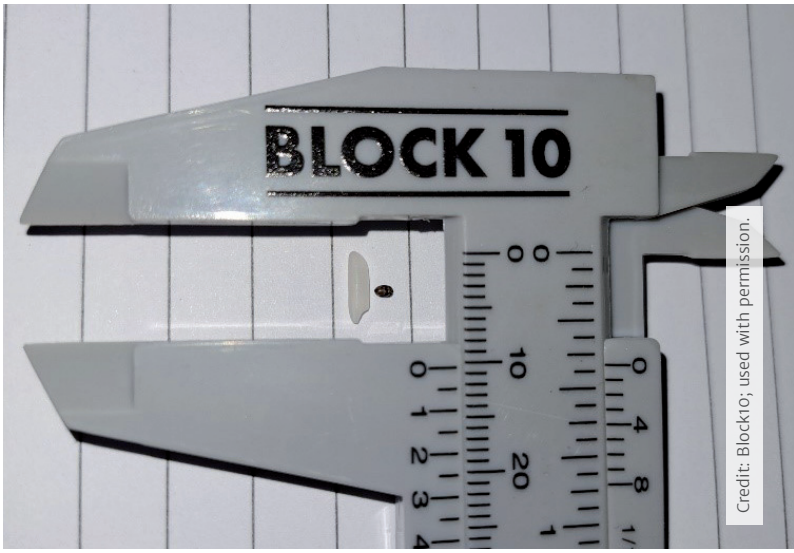


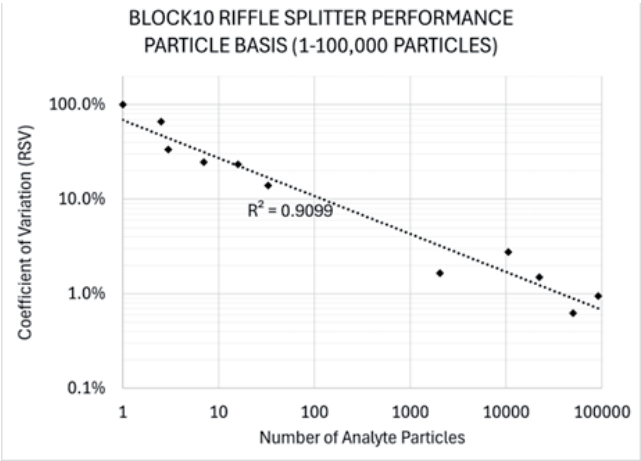
Figure 11: Size comparison for rice and chia seeds.

However, in practice, sub-sampling errors and measurement uncertainties, will cause variability in the RSV% results.

At extremely low particle counts, all the way down to the trivial case of sample division of a single particle, a number of rice grains (1, 2, 5, 8, 16, 32) were counted and put into 90 grams of chia, then divided with the 50/50 BX-R riffle splitter (again replicated four-fold). Why should consideration be given to such low particle counts? For many commodities at major and minor grades it is not relevant, but in a case such as gold analysis, where typical fire assay aliquots of 30g are prepared at 75µm top-size for grades that can be below 1ppm, there really aren't many analyte particles to split! Samples with coarse gold where most of the particles are at or near the milling specification have few Au particles in the aliquot. In this very challenging range, performance results indicate a likely RSV of 15-25% for the BX-R splitter.

The precision results are plotted in Fig. 12 and Fig. 13, depicting the predicted trend in practice – a 100% RSV at a particle count of 1, improving to a sub 1% RSV for analyte grades >20%.

The splitter performance is likely to differ for different materials, and will be impacted by particle shapes, flow properties, etc. However, the following of the theoretical trend, and the RSVs lower than 3% across all tested major grades suggests the BX-R riffle splitter produces high quality, precise results.



Credit: Block10; used with permission.

Figure 12: Riffle splitter evaluation.

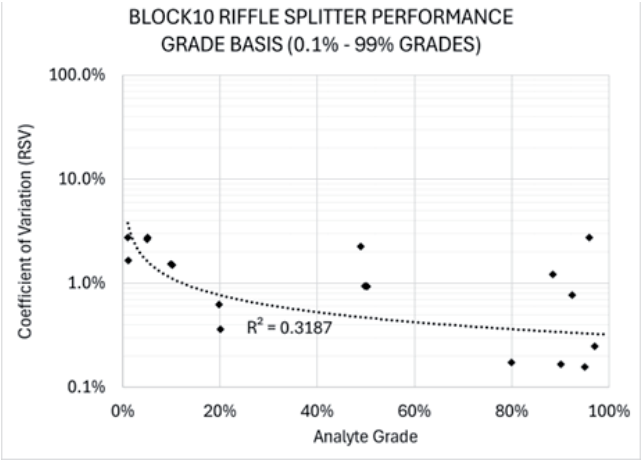


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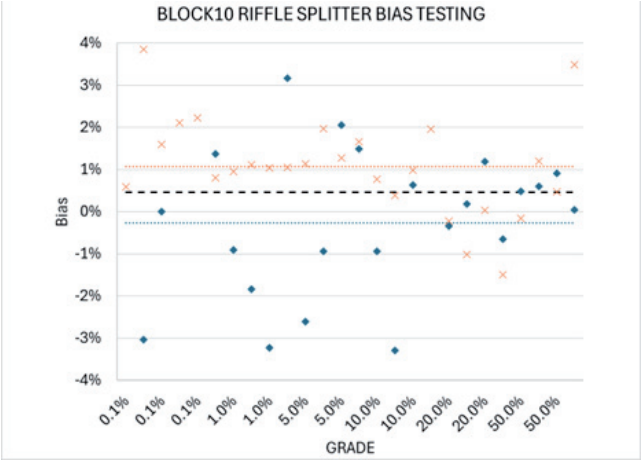
Figure 13: Riffle splitter evaluation.



Credit: Block10; used with permission.

Figure 14: Riffle splitter bias test.

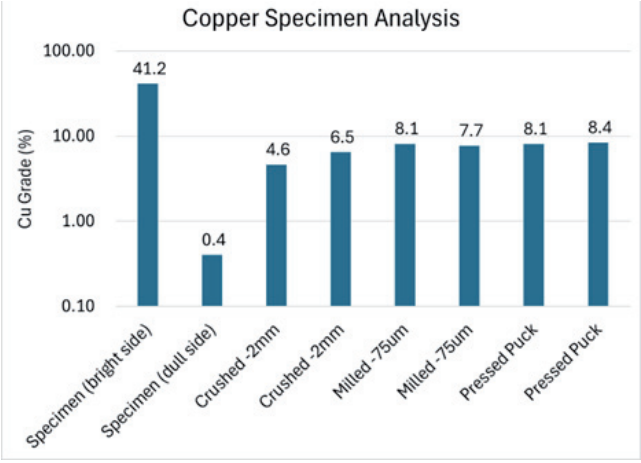
LH vs RH split bias is plotted in Fig. 14, and depicts an average rice (orange crosses) bias of +1.07%, an average chia seed (blue dots) bias of -0.26%, with a combined bias across both analytes of 0.4%. The bias is mainly attributable to the geometry of the splitter, and any slight variations in the flow, particularly of the last-most chute on either side, will impact this balance. This is where a rotary divider whould outperform a stationary splitter, with the potential for much greater than 13 sub-divisions making up each split. However, for the trade-off with size, weight, cost and complexity that is required for a portable and field-deployable solution, this is a welcome quality result.

Further test-work could include similar evaluation of a range of different splitter designs and configurations. It would be most useful across a range of equipment manufacturers to produce similar precision performance curves, allowing evaluation that the splitting device (and the corresponding KPIs) are fit for purpose at a given grade.

12. Example: Cu Specimen Analysis

Similar to the iron ore fines sample, a single azurite (copper) specimen rock, weighing approximately 100g, was also considered.

When in the field, coming upon an interesting sample, the immediate temptation when armed with a pXRF instrument is to analyse. This is fine for identification purposes, as long as no inference is made on grade based on these data.



Credit: Block10; used with permission.

Figure 15: Copper specimen analysis example.

Credit: Block10; used with permission.

Figure 16: Copper specimen, crushed chips, puck.

Table 3: RSV% comparison of crushed, milled and pressed sample

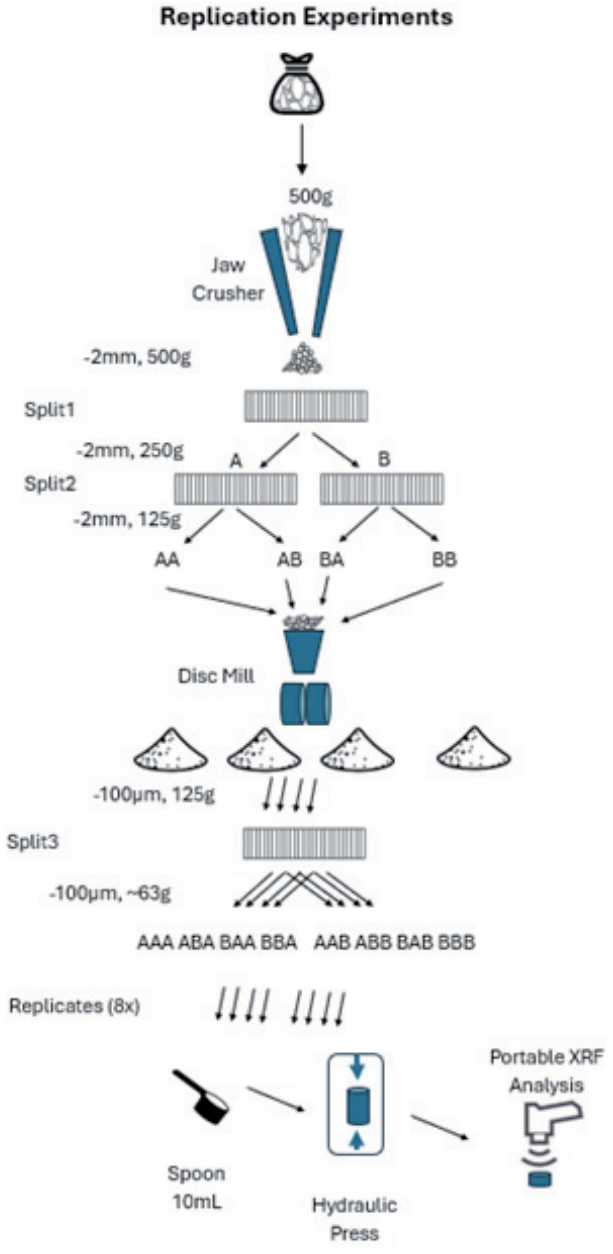
Sample Prep	n	RSV
Cu Specimen Only	2	98.1%
Crushed (-2mm)	2	16.5%
Milled (-75um)	2	2.6%
Pressed Puck	2	2.0%

As shown in Fig. 15, the results will unsurprisingly vary wildly depending on whether the interesting (blue) side of the rock or the barren back is facing towards the pXRF, while duplicate measurements of crushed, milled and pressed stages again highlight an improved RSV% with further sample preparation. There is still considerable variability at crushed (2mm) size chips (clearly visibly evident in Fig. 16), and interestingly, at these grades (~8% Cu) there is only a marginal improvement between the milled and pressed samples.

In reality, copper deposits have much lower sub-percentage cut-off grades, where the difference between milled powder and pressed pucks will likely be much more pronounced. Pressed puck preparation is highly recommended for more precise, less variable analysis.

13. Replication Experiments

In the domain of sample preparation (rather than primary sample collection, which should be considered separately) a Replication Experiment (TOS) was devised to provide insight into the variability (precision) of the final analytical results, when using the present Field Sample Preparation Methodology and analysing all 8 split pathways (see below). The complete subsampling pathway is shown diagrammatically in Fig. 17.



Credit: Block10; used with permission.

Figure 17: Replication Experiments process flow.

The Replication Experiment was conducted on two very different sample types: a Certified Reference Material (CRM) vs. a field collected mineralized sample:

- a 500g CrushedCRM™ produced by IM Standards (IMS-393).
- a 500g Arsenopyritic / Quartz sample from the WA Goldfields region.

The CrushedCRM™ is designed with inherent heterogeneity, to allow sample preparation processes to be assessed with a reference material. Reference materials introduced at the pulp (milled) stage only validate the analysis rather than the preparation.

A good replication result (low RSV% across 8 splits, across each element analyte) on its own is not necessarily conclusive – what if the sample was very homogeneous to begin with?

To provide contrast with sub-optimal sampling practice, a comparable Replication Experiment was also conducted on the 'as is' field sample IMS-393 (also 500g), but this time 8 pucks were produced by spooning 8 x ~60g grab samples from the bag (with visible "Brazil nut effect", surely biasing the later scoops to have higher fines content). This scenario (scooping sample from a bag) is not uncommon in the rough-and-tumble field setting where time is money.

For the WA Goldfields sample an alternate "poor subsampling" method was also used for comparison, in which four specimen rocks were selected at random from a second 500g sample.

These required initial fragmentation with a hammer, before crushing, milling, and pressing duplicate pucks for each. One of the pucks failed to press (due to the high quartz content and lack of binder), so only 7 results were recorded, though enough to still allow a comparison Replication Experiment.

Credit: Block10; used with permission.

Figure 18: WA Goldfields sample specimens – pre fragmentation

Credit: Block10; used with permission.

Figure 19: WA Goldfields specimens – post primary fragmentation

The results were compelling across the multielement suite of 23 analytes (other elements, at or near the limits of detection, were not included). For the well- prepared IMS-393 (plotted in Fig. 20 as 8-way bar clusters for each element analysed) the only elevated variability occurs in several minor or trace elements, and even then it is relatively consistent.

The spooned sample (grab sampling) (Fig. 21) shows visibly higher variation across most elements, and a calculation of the improvement ratios (see Tab. 4) show a significant improvement in RSV across all major (Mg Al Si P K Ca Ti Fe) and most minor/trace elements (S Mn Sr Zr Ni Sn W As Rb), with V, Cr, Co, Zn, Cu and Y showing no improvement.

Consider that although the CrushedCRM™ is intended to feature some heterogeneity, it is nonetheless a standardised product, so it is significant that there was such a discernable difference between poorer practice, and Block10 sub-sampling methodology.

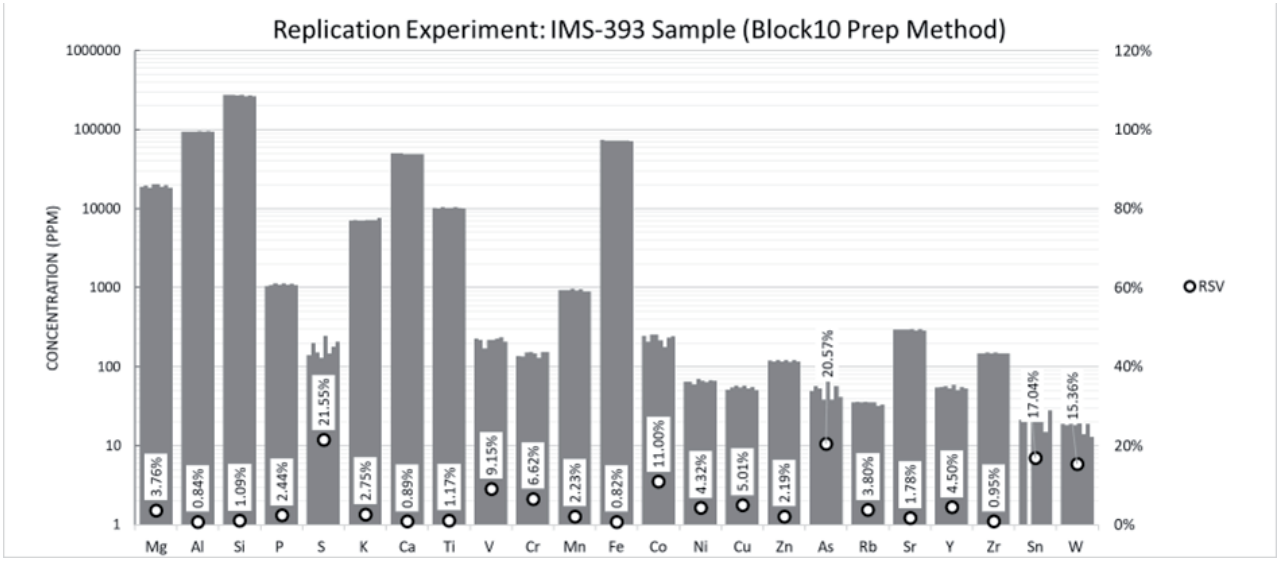


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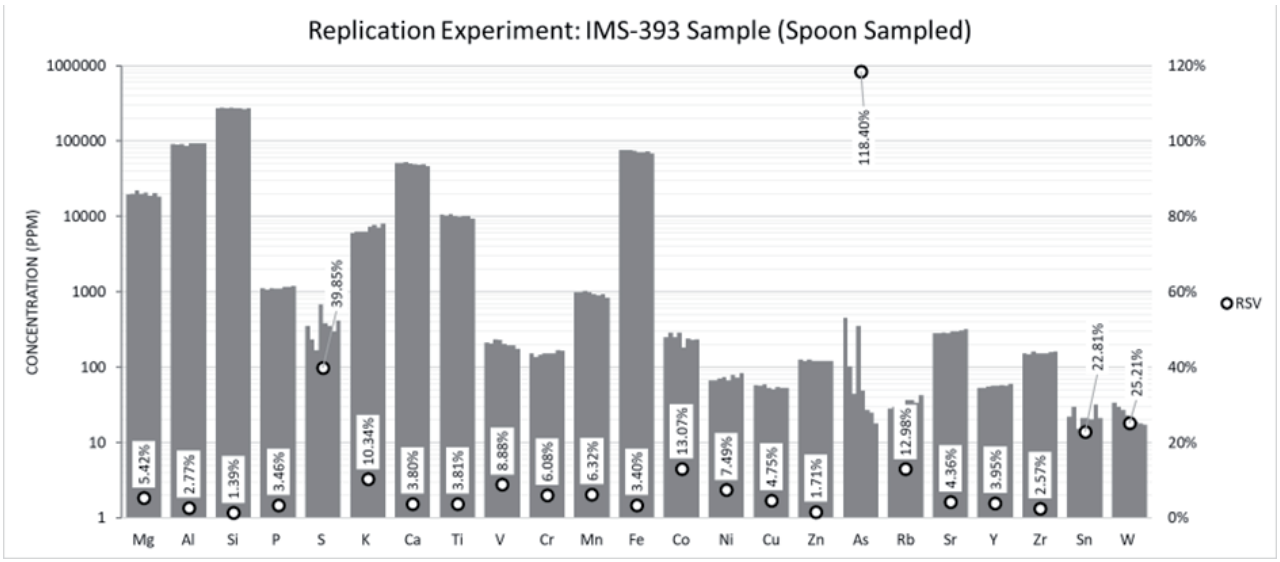
Figure 20: Replication Experiment results: well prepared IMS-393 sample (Heavy circles: RSV%).

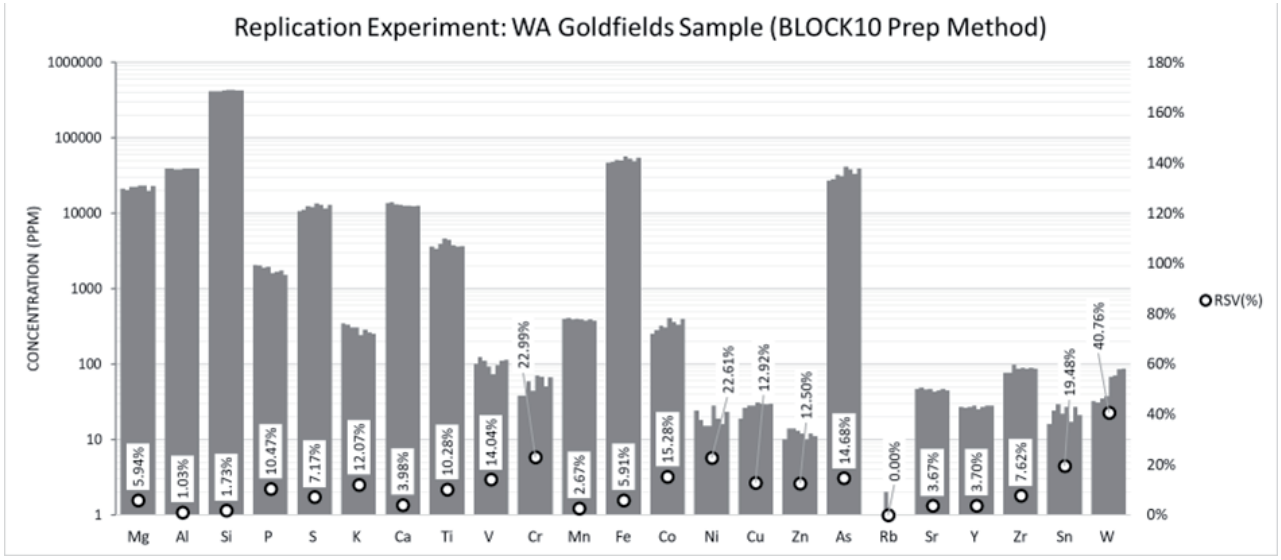


Credit: Block10; used with permission.

Figure 21: Replication Experiment results: spooned IMS-393 sample (Heavy circles: RSV%).

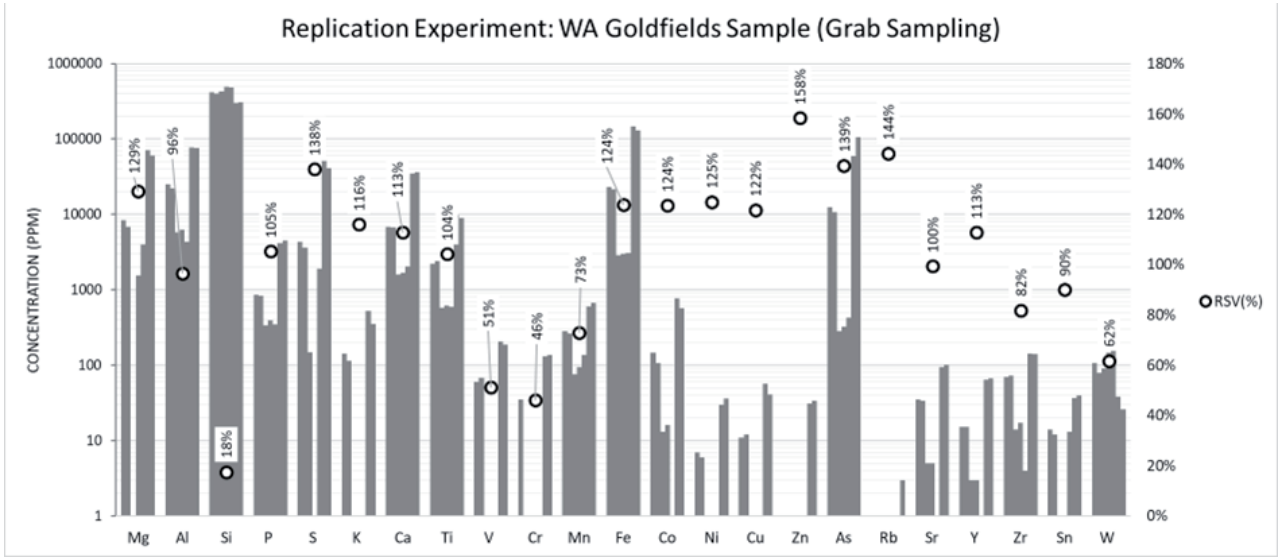
Table 4: IMS-393 Replication Experiment – analyte improvement ratios between well prepared and spooned samples.

Impact	Impact Ratio	Major Elements (>1%)	Minor Elements (0.1% - 1%)	Trace Elements (<100ppm)
No Impact	0.8x – 1.2x		V Cr Co Zn	Cu Y
Improvement	1.2x – 3x	Mg Si P	S Mn Sr Zr	Ni Sn W
Major Improvement	3x – 6x	Al K Ca Ti Fe		As Rb



Credit: Block10; used with permission.

Figure 22: Replication Experiment results: well prepared WA Goldfields sample (Heavy circles: RSV%).



Credit: Block10; used with permission.

Figure 23: Replication Experiment results: grab sampled WA Goldfields sample (Heavy circles: RSV%).

The WA Goldfields sample is visibly a much more heterogeneous sample (with distinct differences between the quartz and pyritic matrices), and there are clearly visible differences between the 4 specimen rocks. The well-prepared sample (Fig. 22) showed similar traits to the CrushedCRM™ when prepared with the same Block10 methodology – many results between 1% - 5% RSV, several in the 5-15% range, and only a handful of trace elements (Cr, Ni, Sn, W) with elevated variability.

By comparison, the grabbed specimens (Fig. 23) can best be described as wildly fluctuating – with 100% RSV across many elements, and a distinct lack of concentration across many elements for the grab samples with visibly higher quartz (lines 3,4,5 in each 7 results cluster). This sample provides an even clearer contrast and strengthens the conclusion that the Block10 equipment, following the correct field sample preparation methodology, produces highly consistent results even for a very challenging rock type.

14. Conclusions

The Block10 Sample Preparation Methodology is presented for the first time in this document, with Replication Experiments and other comparative examples across gold, copper and iron ore samples. This inaugural performance evaluation, although based on a limited experimental layout, demonstrates encouragingly accurate, high-precision results for field sample preparation for multielement pXRF analysis. Concerns over the latest sub-sampling stages using a grab sampling scoop as the final aliquoting tool have been addressed. As a quality assurance evaluation, Replicate Experiments specifically covering the procedural steps after the coarse comminution process (the jaw crusher) were performed. This means that measurement uncertainty (MU) contributions from preceding steps in the full 'lot-to-aliquot-to-analysis' pathway are not included in the present results, Tab. 4 (also see Further Work).

A coefficient of variation in the low single digits (<3% RSV) is achieved when using the BX-C jaw crusher, BX-M disc mill, BX-P hydraulic puck press, BX-R riffle splitter and BX-D dust extraction equipment to produce high quality pucks.
The BX-R riffle splitter was subjected to an augmented test regimen across a wide range of sample concentrations using a proxy rice & chia seeds mixture, which indicates a bias of less than 1% and a precision within 3% RSV at concentrations above 1%, and within 1% RSV at most higher concentrations above 10%.

15. Further Work

The full sub-sampling pathway, and further riffle splitter tests, shall be conducted to include comparison with other techniques and equipment, across a broad range of concentrations, repeated with different rock types and varying particle types, - size and density. A comprehensive DOE (Design of Experiments) approach shall be invoked.

Further test-work is also needed to quantify contamination levels for all wear part materials (tungsten, hardened steel, etc.), to guide wear part materials selection for specific sample types. With regard to carry-over and cross contamination, staggered blank / high grade tests will help quantify the impact, and validate the necessary cleaning regimes for optimal performance.

ACKNOWLEDGEMENTS

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Special Section:"Heterogeneity Testing & Optimal Sample Mass – HOW?"

By Kim H. Esbensen (Editor)

There can hardly be a more important issue in the Theory and Practice of Sampling than that of arriving at a reliable estimate of the necessary sample mass to be representative for a given target material. There are two principal avenues into this challenge: i) empirical heterogeneity characterization of the target material to be sampled (duplicate sample approach, variograohics); and ii) calculation of the variance of the Fundamental Sampling Error, s^2(FSE) according to several variants of Gy's famous formula, based on a set of characterizing material parameters. However, there are rather sharply divided opinions of what constitutes the correct way to do all this. This issue has been debated for several decades within the sampling community, at times with pointed arguments pro and con.

Yet, for many who are not initiated to the higher levels of TOS, this debate appears somewhat high-brow, a bit like "Pi in the sky".

Therefore, the editor has decided to open up the pages in SST for a thorough initiation to this important topic and has invited three key players to present opening shots. SST is happy to be able to bring the first three contributions to this debate in SST#2, with the clear anticipation that the discussion may very well spill over into SST#3 (at least).

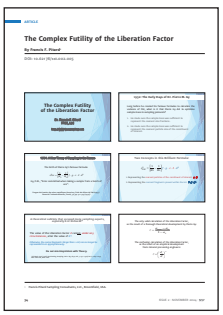
Readers who feel obliged to weigh in are most welcome to submit further contributions to the Journal.

A Brief Review of Heterogeneity Tests for Estimating the Variance of the Fundamental Sampling Error

By Ana Carolina Cheregati

Theory of Sampling (TOS) – Up for Debate?

By Dominique François-Bongarçon



The Complex Futility of the Liberation Factor

By Francis F. Pitard

A Brief Review of Heterogeneity Tests for Estimating the Variance of the Fundamental Sampling Error

By Ana Carolina Chieregati[1]

DOI: 10.62178/sst.002.003

ABSTRACT

The fundamental sampling error (FSE) is generated whenever a sample with mass M_S is randomly selected, fragment by fragment, each with the same probability, from a particulate material lot with mass M_L. FSE represents the sampling error between the actual (but unknown) grade of a lot and the grade estimated from a selected sample. This is the smallest possible error for a sample selected under ideal conditions, hence the term "Fundamental Sampling Error". FSE is characterized by its variance, calculated relative to the measured grade of the lot, using the well-known "Gy's formula". The variance of FSE can either be calculated theoretically by applying a set of material factors or can be estimated experimentally by conducting empirical heterogeneity tests to estimate the constant factor of constitution heterogeneity, aka the "Intrinsic Heterogeneity of the Lot," IH_L. Several 'competing' ways to conduct heterogeneity tests and to calculate IH_L have been proposed historically, but a perennial question in the sampling community is: "Which procedure and formulation reveal the *actual* variance of the fundamental sampling error?" To this day, this question has not been answered to the satisfaction of everybody, because (to paraphrase Edward Deming) "without data, you're just another person with an opinion," and no study has so far proven superior validity of one method over another. This paper surveys and explains, in a simplified way, the main experimental methodologies and formulations for estimating the variance of the fundamental sampling error, highlighting the many remaining challenges of heterogeneity testing, which can be seen as a most fascinating topic, however, because of its complexity. It is possible that no singular best approach should be sought in view of the highly complex realm of economic geology and its many types of ore and mineralisation.

1. Introduction

The fundamental sampling error (FSE) is the only error defined in Pierre Gy's Theory of Sampling (Gy, 1967; 1979; 1992) that can never be eliminated and is related to the constitution or intrinsic heterogeneity (*IH*) of the material in question. To calculate the relative variance of the fundamental sampling error, s^2_{FSE} (Equation 1), for a certain sample taken from a certain fragmented lot, crushed to a certain size, the intrinsic heterogeneity of the lot (IH_L) must be estimated, which can be done theoretically applying the Gy's material-characterising factors, or experimentally performing heterogeneity tests.

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) cfgld^3 = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) IH_L \quad [1]$$

where s^2_{FSE} is the relative variance of the fundamental sampling error, M_S is the mass of the sample (given in g), M_L is the mass of the lot (given in g), *c*, *f*, *g*, and *l* are the four Gy's factors that characterise a specific material (dimensionless, except *c*, given in g/cm³), and *d* is the nominal top-size of the fragments or d_{95} (given in cm).

The AMIRA Metal Accounting Code of Practice (2007) states that there are three basic methods that can be used for determining the value of IH_L: (1) individual particle analysis, also known as the '50 (or more) piece analysis' method, (2) use of scanning electron microscope data from particle sections, and (3) multiple sample analysis. The Code warns that these methods all have their limitations.

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According to Chieregati (2024), there are also three main methods for experimentally determining the value of IH_L: the original heterogeneity test (Gy, 1988; Pitard, 1993) with its several variations, the sampling tree experiment (Minnitt et al., 2007), and the segregation free analysis (Minnitt et al., 2011), from whose results it is possible to calculate the variance of the fundamental sampling error.

The question that looms high is: Which test reveals the *true* s^2_{FSE} and which one reflects what happens in the daily *reality* of sampling processes?

The aim of this article is to present to the reader the existing methodologies as well as their simplified mathematical approaches, but deliberately not to answer the cardinal question above. Chieregati et al. (2023) proved that the tests yield different results, however, to this day, it has not yet been proven which of them allows for a more accurate estimation of IH_L and, consequently, of s^2_{FSE}. Despite considerable theoretical and practical efforts, the issue remains open (see sections Discussion and Conclusions for some reflections on why this may be the case).

2. Metodologies: to select individual fragments or to split the lot?

This section presents the experimental procedures of the main heterogeneity tests proposed over the years, starting with Pierre Gy, almost five decades ago... Note that different notations can be assigned to the same variable. This paper follows the notation from the original authors' work.

2.1 Pierre Gy's 50-fragment method

The "50-fragment method" was proposed by Pierre Gy in his 1988 book, "*Hétérogénéité, Échantillonage, Homogénéisation*" (Gy, 1988), on which the first and second editions of Francis Pitard's books (1989a; 1989b; 1993) were based. Item 4.11 (p. 102) of Gy's book is titled "Experimental estimation of the intrinsic heterogeneity IH_L – the so-called 50/100 fragment method" (*Estimation expérimentale de l'invariant d'hétérogénéité* IH_L *– Méthode dite "des 50/100 fragments*", in French) and describes a method of experimentally estimating the intrinsic ('invariant') heterogeneity of the lot, IH_L.

The operational procedure proposed by Gy (1988) is similar to that presented in his previous works (Gy, 1975; 1982).

While the experimental procedure is practically the same, the interpretation of the results differs significantly.

The newest approach described by Gy (1988) is simpler than the one described earlier because it does not involve measurement of the volume of fragments, an operation little appreciated by practitioners and highly imprecise:

1. Collect randomly, one by one, at least 50, preferably 100 fragments F_i (i = 1, 2, ..., NF) belonging to the coarsest size class of the material lot under study. This can be done by operating with the material retained on the $d/2$ sieve (Figure 1) – if such sieving can be performed – or simply by visually selecting the coarsest fragments 'manually'. The set of all fragments F_i collected constitutes the lot E_1.
2. Wash the fragments (unless otherwise indicated) and dry them.
3. Weigh them dry, obtaining their individual masses M_i.
4. Analyse each fragment for all critical components (analytes): contents a_i, b_i, etc.

Figure 1: Coarsest size class of the material lot under study retained on the d/2 sieve.

According to François-Bongarçon (2024), it is advisable to collect fist-sized fragments so that the mass is adequate for preparation and chemical analysis. Otherwise, analysing a single fragment becomes unfeasible, leading to the method proposed as follows.

2.2 AusIMM's modified 50-piece test

The "Modified 50-piece test" suggests that it is the practitioner's decision to either select 50 individual fragments or 50 groups of individual fragments, each group composed of an equal number of fragments, selected one by one, randomly. The modified test presented by the AusIMM (2023) does not specify how many fragments each group should contain, so the professional has some leeway and may consider a number of fragments representing the mass of the subsample required for physical preparation in the laboratory.

The modified 50-piece test protocol, described below, essentially follows Gy's "50-fragment method" approach:

1. Select at least 50 individual (or subsamples consisting of groups of) fragments from the coarsest size class of a bulk sample with mass M.
   a. Note that the coarsest size class ranges from $d/2$ to d, with d being the nominal top size or d_{95}.
   b. Individual particles may be selected from the coarse size fraction after screening, or by visual estimate, which is often adequate in the case of ores with a large top size.
2. Dry the selected fragment/subsample separately.
3. Measure the dry mass M_j of each fragment/subsample.
4. Crush and pulverise each fragment/subsample separately to produce a pulp that is sufficiently fine (<150 µm) to serve as an analytical test portion.
5. Determine the concentration a_j of each fragment/subsample.

2.3 Simplified 4-size-class heterogeneity test

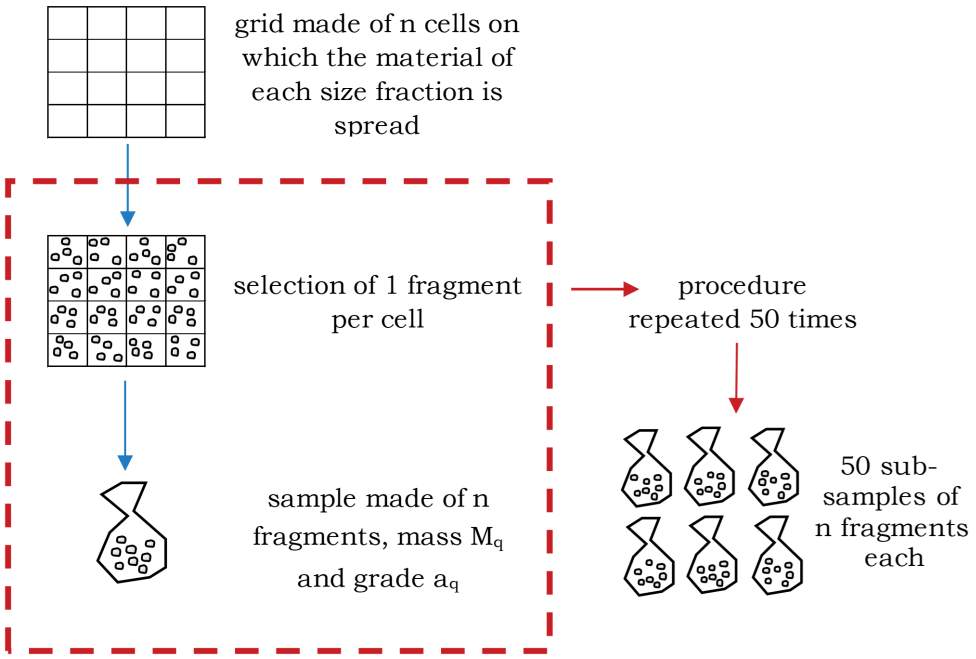
Also based on the "Modified 50-piece test", there are two ways to perform the heterogeneity test when aiming to obtain IH_L for more than one size fraction: (1) dividing the initial lot (ideally 250–500 kg) into four equal parts, crushing each part to a *different* top size, d, and then screening each part down to $d/2$; or (2) screening the entire lot (250–500 kg) into four different size fractions and performing the test for each size fraction separately. Because in the second method the lot is screened at the beginning, rather than being crushed into four different size classes and then screened, it was called the "simplified 4-size-class heterogeneity test" (SHT).

For both methods, the selection of subsamples must be done separately for each size class, as described below:

1. If the material is wet, dry the entire lot before starting the test.
2. Screen – or crush and screen – the lot into four size fractions, starting with the top size class ($-d_{95}+d_{95}/2$).
3. Spread the material of each size class evenly on a grid previously drawn with masking tape, ensuring that no fragment overlap with other fragments.
4. Select at least 50 subsamples, made of groups of n-fragments, from each size class. To give all fragments the same probability of selection, the subsamples are composed of one fragment randomly collected *from each cell* of the grid, making up 50 n-fragment subsamples, as n is the number of cells. Note that the cell sizes vary according to the particle size fraction.
5. Measure the dry mass M_q of each subsample.
6. Crush, pulverise, and split each subsample separately to serve as an analytical sample.
7. Determine the grade a_q of each subsample.

Figure 2 shows an example of n-fragment subsamples being produced from the −½"+¼" size class. In this example, n = 90 and each subsample will be made of 90 fragments (6 × 15 grid cells).

Figure 2 Fragments being collected during the simplified 4-size-class heterogeneity test (−½"+ ¼" size class).



Credit: A. C. Chieregati; used with permission.

Figure 3: Simplified heterogeneity test procedure for each size fraction (Chieregati et al., 2023).

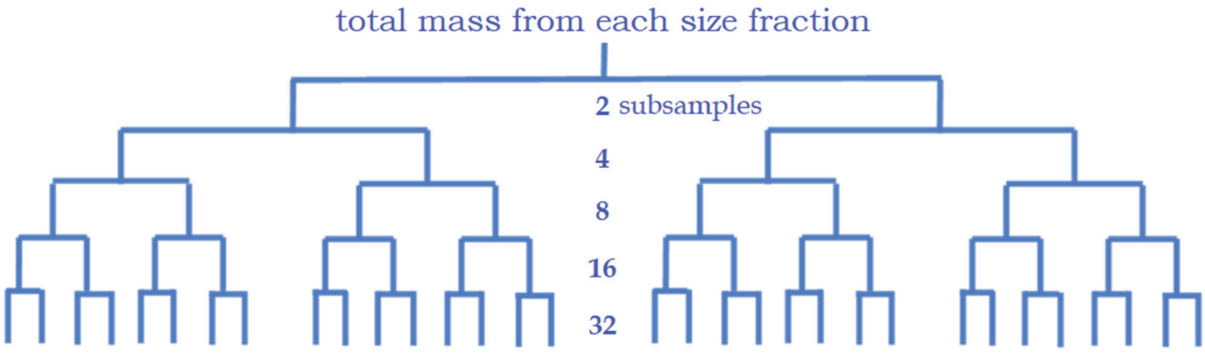
The simplified heterogeneity test procedure is schematised in Figure 3, where 50 *n*-fragment subsamples are generated. Consequently, the total number of subsamples will be 200 (4 size fractions × 50 subsamples). It is important to emphasize that the test can also be performed using three size fractions instead of four, indeed also with an even higher number of size fractions – where and when deemed necessary (of course at a greatly increased workload). The idea is to have 3, or 4 points (or even more) in a graph to calibrate the sampling constants, which will be detailed in the next section.

2.4 Sampling tree experiment and segregation free analysis

The sampling tree experiment (STE) was proposed by François-Bongarçon (1993; 1998; 2008) and is well described with a practical example by Minnitt et al. (2007). The segregation free analysis (SFA) was proposed by Minnitt, François-Bongarçon and Pitard (2011).

The experimental procedure of both methods is similar and is based on the binary sampling tree. The difference is the way each size class is prepared and the number of size classes to be tested: (1) in the STE, the initial lot (approx. 60 kg) is divided into four equal parts, after which each part is crushed to a different top size to be tested; (2) in the SFA, the initial lot (approx. 200 kg) is screened in fourteen different size fractions to be tested. The following steps are common for both tests:

1. After preparing the four or fourteen size classes, riffle split each size class material into a series of 32 subsamples, resulting from five splitting stages and forming the binary sampling tree shown in Figure 4.
2. For the STE, two subsamples can be chosen at random from each size fraction for granulometric analysis to check the d_{95}, leaving 30 subsamples per size fraction for chemical analysis.
3. Measure the dry mass M_s of each subsample.
4. Crush, pulverise, and split each subsample separately to serve as an analytical sample.
5. Determine the grade of each subsample.



Credit: A. C. Chieregati; used with permission.

Figure 4: STE and SFA sampling tree procedure for each size class (adapted from Minnitt et al., 2007).

Figure 5 shows the STE/SFA riffle splitting procedure, where 32 subsamples from each size fraction are generated, two for granulometric analysis (STE) and the remaining 30 (STE)/ 32 (SFA) for chemical analysis. Consequently, the number of subsamples will be 120 (4 size fractions × 30 subsamples) for the STE and 448 (14 size fractions × 32 subsamples) for the SFA. It is important to emphasize that the SFA can be performed using a different number of size fractions. Chieregati et al. (2023) used only four size fractions and called the modified test "simplified segregation free analysis" (SSFA).

Credit: A. C. Chieregati; used with permission.

Figure 5: Material of the −½"+ ¼" size class being riffle split during the STE/SFA procedure.

3. Formulations: to calculate or to calibrate the sampling constants?

This section presents the simplified mathematical approach on which each heterogeneity test described in the previous section is based. Note that different notations can be assigned to the same variable. Out of respect, this paper follows the notation in the original authors' work.

3.1 Pierre Gy's 50-fragment method

With the results of the mass and grade determined for each of the 50 selected fragments, and using Pierre Gy's original formula (Gy, 1988, p. 360):

$$s^2_{EF} = \frac{1-P}{P\,M_L}\,IH_L = \left(\frac{1}{M_E} - \frac{1}{M_L}\right) IH_L \quad [2]$$

where s^2_{EF} is the relative variance of the fundamental error, P is the selection probability, M_E is the mass of the sample (given in g), M_L is the mass of the lot (given in g), and IH_L is the intrinsic ('invariant') heterogeneity of the lot (given in g), it is possible to calculate s^2_{EF} after the experimental estimation of IH_L described as follows:

1. Calculate the mass M_{E1} of the lot E_1:

$$M_{E1} = \sum_i M_i \quad [3]$$

2. Calculate the grade a_{E1} of the lot E_1:

$$a_{E1} = \frac{\sum_i a_i M_i}{M_{E1}} \quad [4]$$

3. Calculate the unbiased random estimator $EST\,[IH_{E1}]$ of the intrinsic heterogeneity of the lot E_1:

$$EST[IH_{E1}] = \sum_i \frac{(a_i - a_{E1})^2}{a^2_{E1}} \cdot \frac{M_i^2}{M_{E1}} \quad [5]$$

4. Evaluate the proportion M_{L1}/M_L of the class. If the lot E_1 is obtained by sieving a lot E with a mass M_E, the estimator $M_{L1}/M_L = M_{E1}/M_E$ can be used. In the absence of objective information, the average value of 0.30 can be adopted.

5. Calculate the estimator of $[IH_L]_1$, which is the standard estimator of IH_L when $[IH_{L1}]$ approaches $[IH_{E1}]$:

$$EST[IH_L] \approx EST[IH_L]_1 \approx EST[IH_{L1}]\frac{M_{L1}}{M_L} \approx EST[IH_{E1}]\frac{M_{E1}}{M_E} \quad [6]$$

According to Gy, the validity of this method depends mainly on the 'invariance' of the intrinsic heterogeneity IH_{L1}, which is a random function of the mass M_{L1}.

3.2 AusIMM's modified 50-piece test

With the results of mass and grade of each of the 50 fragments/subsamples, the following procedure should be carried out for the estimation of IH_S and s^2_r:

1. Calculate the combined dry mass M_S of all fragments/subsamples as:

$$M_S = \sum M_j \quad [7]$$

2. Calculate the combined concentration a_S of all fragments/subsamples as:

$$a_S = \frac{\sum a_j M_j}{M_S} \quad [8]$$

3. Calculate the parameter IH_S as:

$$IH_S = \sum \frac{(a_j - a_S)^2}{a^2_S} \cdot \frac{M_j^2}{M_S} \quad [9]$$

4. Evaluate the mass proportion M_A/M, where M_A is an estimate of the ore weight retained in the size class $d/2$ to d. For example, if the +12.5 mm size fraction in a <25 mm mill feed stream constitutes 25% of the total material flow, the ratio M_A/M would be expressed as 0.25.

5. Calculate the constitution heterogeneity IH of the ore as:

$$IH = IH_S \cdot \frac{M_A}{M} \quad [10]$$

6. The relative variance V_r or s^2_r is calculated as:

$$V_r = s^2_r = \frac{IH}{M_{sample}} \quad [11]$$

3.3 Simplified 4-size-class heterogeneity test

With the results of mass and grade of each of the 50 subsamples per size fraction:

1. Calculate the combined mass M_Q of all subsamples:

$$M_Q = \sum_q M_q \quad [12]$$

2. Calculate the weighted average grade a_Q of all subsamples:

$$a_Q = \frac{\sum_q a_q M_q}{M_Q} \quad [13]$$

3. Calculate the unbiased random estimator *EST IH*$_L$ of the intrinsic heterogeneity of the lot. Note that Pierre Gy's Equations 5 and 6 can be rewritten as Equation 14 (refer to Pitard, 1993, p. 176):

$$\text{EST IH}_\text{L} = g \sum_i \frac{(a_q - a_Q)^2}{a_Q^2} \cdot \frac{M_q^2}{M_Q} \quad [14]$$

4. Use Gy's granulometric factor *g* (0.25 for uncalibrated material and 0.55 for calibrated material) as the mass proportion of the top size fragments for each size class.

5. Calculate the nominal diameter of the fragments, where d_{MAX} and d_{MIN} are the openings (in cm) of the upper and lower screens of each particle size fraction, respectively:

$$d_N = \sqrt[3]{\frac{d_{MAX}^3 + d_{MIN}^3}{2}} \quad [15]$$

6. Plot $IH_L \times d_N$ on a log-log graph and the power regression line (example in Figure 6). In the regression line equation $y = ax^b$, y represents IH_L, a represents K, x represents d_N, and b represents α of the IH_L calibrated formula, K and α being the sampling constants:

$$\text{EST IH}_\text{L} = K d_N^{\alpha} \quad [16]$$

7. The relative variance of the fundamental sampling error, s^2_{FSE}, is then calculated as:

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \text{EST IH}_\text{L} \quad [17]$$

It is important to emphasize that calibrating the sampling constants *K* and *α* (François-Bongarçon, 1998; Minnitt et al., 2007; Minnitt et al., 2011; Ganguli et al., 2017; Bortoleto et al., 2019; Chieregati et al., 2023) is manifestly not unanimously accepted as the best alternative among sampling experts, between which rather adverse attitudes have been prevalent at times. However, a preliminary study on aluminium ores (Marques and Chieregati, 2023) shows a significant correlation between the theoretical IH_L calculated using Gy's material factors and the experimental IH_L calculated using the calibration of *K* and *α* through the simplified 4-size-class heterogeneity test. This is encouraging as it shows the way forward for more studies in an incredibly complex mineral realm (see sections Discussion and Conclusions).

The calibration proposition based on the simplified 4-size-class heterogeneity test suggests that a log-log plot be constructed with the nominal fragment size d_N on the x-axis and the corresponding values of *EST* IH_L on the y-axis. By plotting the power regression line of the four points on the graph, estimates of the parameters *K* and *α* from Equation 16 are obtained, where *K* is a constant factor representing the product of all Gy's material factors, and *α* is the exponent of the nominal fragment size, equal to 3 in Gy's original formula and determined by the slope of the regression line on the heterogeneity graph.

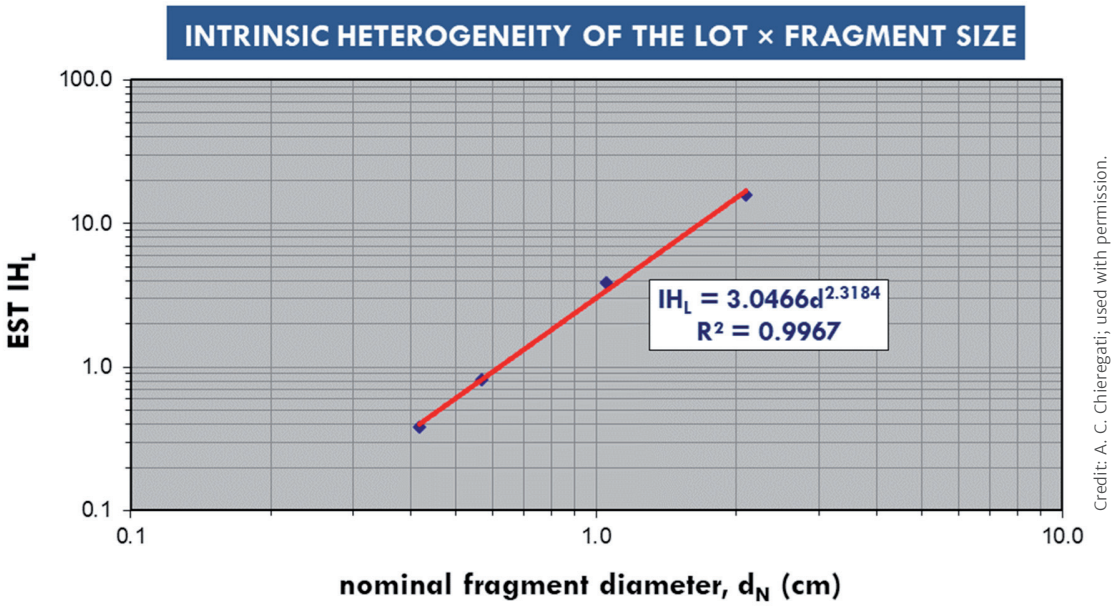


Figure 6: Example of sampling constant calibration using the simplified 4-size-class heterogeneity test.

3.4 Sampling tree experiment and segregation free analysis

Although the STE and SFA experiments involve more complex data processing, including removal of outliers, reduction of the analytical data, calculation of the standardised variance, calculation of the liberation size, etc., the author chose to present a simplified formulation, focusing solely on the calibration of the sampling constants *K* and *α*. For a detailed description of all data processing steps, please refer to François-Bongarçon (1993; 1998; 2008) and Minnitt et al. (2007; 2011).

With the results of mass and grade of each of the 30-32 subsamples per size fraction:

1. Calculate the total relative variance σ^2 of the data for each size fraction.

2. Calculate the residual relative variance σ_R^2 for each size fraction, subtracting the analytical variance σ_A^2 from the total variance:

$$\sigma_R^2 = \sigma^2 - \sigma_A^2 \qquad [18]$$

Note: According to Minnitt et al. (2011) and François-Bongarçon (2024), this adjustment to the variances is necessary, because it has influence on the values for the slope *α* and the intercept *K*, and probably affects the series with the smaller d_N. The variance derived from the 30-32 chemical analyses of each size fraction is a multi-stage variance that includes both the pulp variance and the analytical variance.

The authors state that it is important that the variances from the analytical (pulverised) stage are subtracted from each of the respective multi-stage variances (sets of 30-32 analyses) to provide an unencumbered single-stage variance.

3. Rearrange the simplified Gy's formula (Equation 19) to give a linear equation in logarithmic graph (Equation 20):

$$\sigma_R^2 = \frac{Kd_N^\alpha}{M_S} \qquad [19]$$

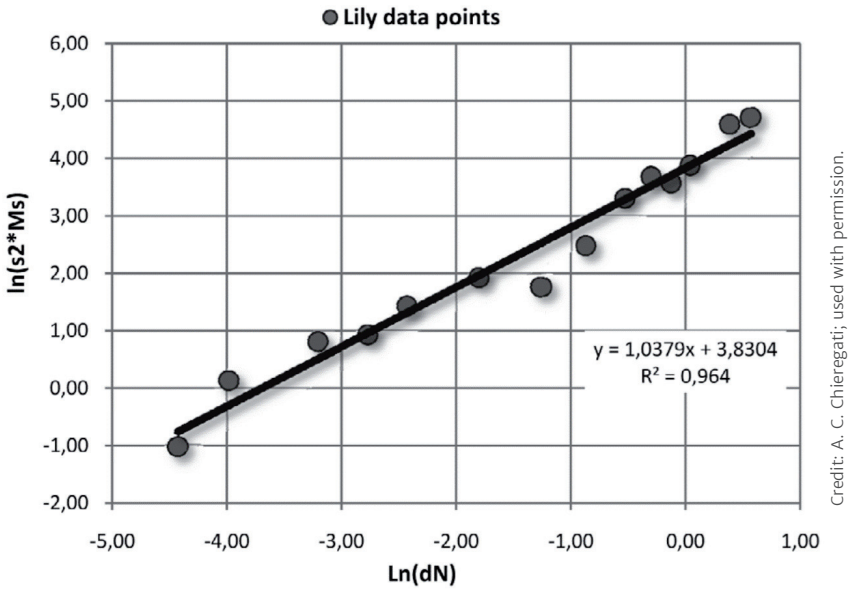
$$\ln(\sigma_R^2 * M_S) = \alpha \ln(d_N) + \ln(K) \qquad [20]$$

4. Plot $ln(\sigma_R^2 * M_S) \times ln(d_{N/MAX})$ on a graph and the linear regression line (example in Figure 7). For the STE, plot each point for its nominal top size d_N; for the SFA, in turn, plot each point for its d_{MAX} (upper screen opening of the size class), not the average diameter.

5.The slope of the line provides a value for *α*, while the constant is the intercept on the y-axis and provides an estimation of a value for *K*. The regression line in Figure 7 shows $\alpha = 1.0379$ and $K = e^{3.8304} = 46.08$ (as for Equation 20).

6. The relative variance of the fundamental sampling error, σ^2_{FSE}, is finally calculated as:

$$\sigma_{FSE}^2 = \frac{Kd_N^\alpha}{M_S} \qquad [21]$$



Credit: A. C. Chieregati; used with permission.

Figure 7: Example of sampling constant calibration using the SFA (Minnitt et al., 2011).

4. Discussion

After the brief review presented in this paper, the question "which procedure and formulation reveal the *actual* variance of the fundamental sampling error?" remains unanswered, despite ongoing studies. Chieregati et al. (2023; 2024) studied different types of ore and concluded that the simplified segregation free analysis, compared to the simplified 4-size-class heterogeneity test, tends to underestimate the sampling constant α and to overestimate both the sampling constant K and the total s_{FSE} of the sampling protocol. Two thirds of the 16 chemical elements analysed in these studies presented lower values of α and higher values of K and s_{FSE}. These trends are partially explained by Pitard and François-Bongarçon (2011), who state that there are two main types of heterogeneity tests: (1) to estimate exclusively the variance of the fundamental sampling error (FSE), or (2) to estimate the variance of the quality fluctuation error, component 1 (QFE_1), which includes both the fundamental sampling error and the grouping and segregation error (GSE). The first type of test estimates exclusively the intrinsic constitution heterogeneity of the lot because the samples are composed by collecting individual fragments one by one at random, the only condition under which GSE will cancel; the second type includes the distribution heterogeneity between extracted replicate splits or groups of fragments. According to these authors, the variance of QFE_1 better reflects what is happening in daily reality in sampling protocols.

Based on all available results from empirical studies that can be found in the open literature, there does not seem to be conclusive systematic patterns for a 'best' heterogeneity test behaviour representing specific types of ore or mineralisation across the mining and exploration industry. Rather there is a strong analogy to the findings of Engström (2017) and Engström and Esbensen (2017) in the study of blast hole sampling versus reverse circulation drilling, which found a similar lack of correlation with respect to specific ore types. Each case is best served with being evaluated individually.

One might be tempted to speculate that a two parameter (K, α) mathematical relationship may be *too simple* a formalism for covering the extremely complex realm of Geology. With so many different types of mineralisation and ores, there is perhaps no reason to expect a singular universal best practice.

Pierre Gy himself once wrote (Gy, 1982, p. 279): "*[...] the method which was developed 25 years ago, breaking up as it does the fundamental variance into a product of simple factors, precises remarkably well the influence of the various characteristics of the material to be sampled.*" According to Gy, then, applying the set of four material-characterising factors (Equation 1) for each type of ore may still be the best option for calculating the relative variance of the fundamental sampling error.

5. Conclusions

Even though a much greater discussion and detailing about heterogeneity studies could be made – and in fact has already been done by François-Bongarçon (2008; 2024) –, the aim of this paper is only to present the general outlines of different experimental procedures and data processing. There will always be a need for carefully planned and meticulously executed empirical characterisation of the material for which a quantitative heterogeneity characterisation is needed, either to estimate s^2_{FSE} or to calculate realistic optimal sample masses.

The most important issue is to keep in mind that depending on how the heterogeneity test is conducted and how the data is processed, different results can be obtained and, consequently, different conclusions will be drawn. According to François-Bongarçon (2024), *designing* the heterogeneity experiment may be the most important step which, when poorly done, can trigger irreversible damage to the conclusions of the study. The author addresses the main problems of heterogeneity studies in detail and affirms that "*on-going recipes and publications are unclear and often false, [...] articles are never supposed to be recipes to follow blindly, instead they should be viewed at most as enlightened suggestions*" (François-Bongarçon, 2024, p. 21).

This is exactly the didactic purpose of this paper: to bring the complex heterogeneity tests to the readers' attention, so they can reflect on them, study the different methods in greater depth, and draw their own conclusions to conduct their own studies, rather than claiming that one approach has superior validity over another. And perhaps one day the cardinal question will have a definitive answer.

The seed has been sown... who would like to take on this challenge?

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Theory of Sampling (TOS) – Up for Debate?

By Dominique François-Bongarçon[1]

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1. Introduction

Scientific debates are useful. The World Conferences on Sampling and Blending (WCSB), besides being the biannual highpoints of social interaction for the International Pierre Gy Sampling Association (IPGSA) community, are very much also about presenting, debating and discussing the latest research results. And here there are healthy debates between ‚experts' about a number of Theory of Sampling (TOS) issues. But these have by and large rendered the understanding of the general theory and its direction of development somewhat cloudy in the minds of the general audience beyond the IPGSA boundaries.

Scientific research does not progress based on certainties, but instead on systematic skepticism. But undisciplined casting doubt about the next steps of development are not a good way to disseminate the Theory of Sampling (TOS), which is otherwise very well established, and very useful in practice - nor is this a good way to gain new adepts, nor to convince students they should be interested in pursuing it.

Here a partial selection of these issues will be reviewed, if only in a few sentences or paragraphs each, and in loose ordering, hoping to clarify the real concepts behind them, irrespective of the amount of debate they are currently triggering. No precise references are given, the reader is referred to the abundant literature on each subject, particularly in the series of WCSB conference proceedings.

2. The Legacy from Pierre Gy

The first comments will be about clarifying the WCSB conferences and their raison d'être. The first WCSB conferences were not initially conceptualized and designed to be a debating forum. The inaugural conference was specifically designed to honor Pierre Gy and his legacy. But from there, the conference concept developed itself along the way, very much without specific guidance.

But our biannual conferences have been very useful over the last twenty years for disseminating Gy's ideas and the details about his admirable work. Gy was not a promoter, he worked alone, with no associates, his circle of followers was scarce, he never read other people's works on TOS issues – so over the last 30 years much work was needed by his followers, to promote TOS, and to motivate the industrial world to use it, and universities to teach it.

Gy's work was dual: he discovered and designed the first principles of sampling, relating to how samples should be taken physically, creating the concepts of sampling correctness and segregation - and subsequently he worked out the mathematical modeling of the sampling variance for randomly taken samples, resulting in an elegant equation, famously now known as "Gy's Formula". It is important to observe that Pierre Gy was often distinctly dissatisfied with the way 'his formula' was misused, often grossly, based on a far too superficial understanding of the basic assumptions behind its derivation.

He also addressed how a new tool, the variogram, developed by G. Matheron, could be used to characterize one-dimensional estimation problems that were in fact improperly likened to sampling by users at large. Indeed, the distinction between sampling s.s. on the one hand, i.e., extracting a small mass intending to represent the whole lot, and on the other hand, measuring a concentration of interest at specific points with coordinates in some 1D (e.g. time), 2D or 3D space, over a measurement support (not a ‚sample' per se) with the aim of performing a geostatistical estimation has been very indistinct and blurred, even up to this day in many users' minds, courtesy of our relaxed day-to-day vocabulary, alas often misleading. This important distinction should hopefully clarify matters, especially in the minds of new students of TOS.

1 Agoratek International Consultants Inc., Canada.

3. Some Particular Applications of TOS First Principles

3.1 Sampling Methods

A debate exists, for instance in surface mining, about comparing blast hole (BH) and reverse circulation (RC) rig sampling. Articles have been published on a false debate that never really existed. The economics, the practicalities/applicability and the performance of each of these two sampling methods are for the users to compare in each particular case. Making it a matter of religious preference is contrary to science and detrimental to TOS dissemination. The rest is a matter of proper implementation, avoiding extraction errors, and addressing the major issues as best as possible with the right priorities. A particularly illuminating contribution to this 'debate' can be found in a paper published from a recent Ph.D. by Karin Engström (2017).

3.2 Sampling Equipment Design

There are issues about sampling equipment that are also confusing, due to the sometimes misleading terminology used by some OEM manufacturers. The only quantitative samplers that we know how to use with good results to sample a flow are i) linear and ii) circular (Vezin) cross-stream devices, provided they are used within certain limit conditions. Apart from these, we are in the unknown, with no guarantees of unbiasedness. In the case of two families of 'samplers', which have a certainty for size distribution biases: a) ‚rotary distribution' samplers (sometimes called "rotary dividers', or sometimes improperly called Vezin subsamplers), and b) classical cross-belt samplers (which are in fact mere ‚material pushers', with no reasonable chance of ever being unbiased). These are harsh judgements on some OEMs, but somebody has to state them.

In particular, all 'process control samplers' used in metallurgical plants are non-quantitative, without exception, and almost always biased. The base metal industry would be well advised to imitate the precious metals industry by adding TOS-compliant quantitative samplers to plants in order to achieve objective metal balancing.

3.3 Segregation

The propensity for segregation to be omnipresent in aggregate mixtures of minerals and similar mixtures of unit elements with different density, surface roughness, etc., cannot be effectively combated using mechanical mixing.

Within bulk materials, a variety of bed-blending methods turning the segregation to the advantage of better sampling, on the one hand, and multi-incremental sampling on the other, are, conversely, fully effective. A well-used riffle splitter is effective in removing most of the effects of segregation, but the method with the highest score is rotary splitting over a rotating carrousel fed by a vibrated feeder of the proper length, i.e., long enough for the migrating layer of material to fall over the carrousel as a fragment-fine layer.

Segregation is likely to be the next large field of research in TOS. Gy's demonstrations have included the segregation term in the theoretical variance formula, but it was deemed to be non-quantifiable and was therefore not pursued further, while Visman, using the same formalism, actually proposed some very useful quantification experiments in some particular cases.

4. Numerical Control and Mathematical Modeling

4.1 Is TOS Mathematically Complex?

The statistical model on which Gy based his demonstration of a variance formula is heavy and cryptic. The only fully rigorous demonstration, that vindicated Gy's own, was purely mathematical, though even much more cryptic and complex and it was published in French by Matheron (2015). That demonstration, more recently translated into English, augmented and commented by François-Bongarçon and Pitard is extremely complex, with heavy use of integral and differential calculus and at the end it reaches a formula that is only a first order approximation.

It is always possible to establish more didactic, simplified demonstrations, that better show the underlying theoretical structures, but the price to pay for these useful derivations, is a lack of rigor. There is not such a thing as a demonstration both simple and rigorous of the variance of a sample of particulate material when the particles have different physical properties. Matheron's demonstration shows this beyond the shadow of a doubt.

In essence, yes, TOS mathematics is complex, because corners cannot be cut while rigorously establishing a theory, but, after it has been established, there are various options for clearer derivations.

4.2 Liberation Factor

An additional difficulty that cannot be overstressed, is that the first order approximation reached by both Gy and Matheron independently of each other, can only be calculated explicitly in the case of fully liberated units (mineral grains, fragments). There is no such a thing as a general variance formula for sampling of non-liberated materials, unless the formula is added a diminishing factor between 0 and 1, called the liberation factor, l, for which no practical and generally valid model was initially proposed by Gy.

Gy knew that factor to be correlated with the degree of liberation, i.e. the proportion of liberated component of interest, and to depend on the liberation and comminution sizes dL and dN. The model $l = \sqrt{dL/dN}$ had once been proposed (but later rescinded) by Gy, because it invariably resulted in erroneous variances. In precious metals dealings, the erroneous nature of that model was directly obvious (for monetary reasons). An example from Gy published in Pitard (1993) was published, which, pushed to the limit, could be proved to be absurd by simply eliciting the liberation size.

To date only one model has been offered, which is a generalization of the above formula for l, with a variable exponent, b, between 0 and 3 (instead of the fixed approximation 0.5 used by Gy). That model has been used successfully for 30 years now. One should note though, that a model of liberation factor is strictly required only if the variance formula is to be used for predictive purposes, i.e. for predictions in which the concentration, or the comminution size, will vary. As an example, for an existing sample preparation protocol, with fixed comminution sizes, characterizing the variance of each sampling stage and optimizing only the required sample masses, can be done from duplicate samples generated at each stage.

4.3 Heterogeneity, heterogeneity testing

This brings up the issue of heterogeneity characterization and heterogeneity testing. Full heterogeneity characterization makes use of the liberation factor - explicitly, when the developed version of the variance formula is used, or implicitly, when it is replaced using a ‚Heterogeneity Factor' divided by the sample mass. Indeed, if the material is not liberated, the liberation factor is always present but embedded in said heterogeneity factor, and changes in concentration (which may trigger changes in liberation size) or in comminution size, require an explicit model of the liberation factor, lest calculations become completely illusory. The experimental calibration of that model is often called heterogeneity testing.

Even though only one model so far has been proposed for the liberation factor, many experimental methods have been proposed for heterogeneity testing. If they are well performed, their choice largely is a matter of preference. However, some common errors are often seen in heterogeneity testing studies that unfortunately invalidates them. The most common error consists of equating the one-stage formula to be calibrated to multi-stage sampling variances that have parasitic, non-primary components. The other common mistake is to ignore the variations of the liberation factor with dL an dN, by simply not using a model for it, also resulting in illusory results.

4.4 Bad Sampling Consequences

While the first principles of TOS dictate how samples or measurements should be extracted, even when they are properly applied, difficulties can still arise. In particular when sampling variances grow too large (e.g. if sample masses are too small). What happens then is the distribution of possible sample values, or its translated distribution of sampling errors, may then become overly skewed. As a result, the empirical median is much lower than the mean of the real-world distribution (which, for an unbiased sample, is the true, unknown concentration value). Consequently, more than 50% of the samples will return a value lower than the true value. Note that this is not a mathematical bias, as occasional very high values will also be returned so that, on average, all converges towards the true value. When taking a single sample, however, this is not a real consolation, especially as, on top of this, the occasionally compensating high-flier will often be capped or suppressed (the fallacy of considering all outliers faulty). Additionally, the most probable sample value generally is the mode, which is then even lower than the median.

This phenomenon is sometimes illustrated as the infamous Poisson effect (due to the Poissonian nature of the distribution of the grains of the component of interest). A graph is then built that shows the most probable sample value (MPSV) as a function of the sample size. This very didactic graph, however, needs to be carefully interpreted. It does not represent a bias (again there is no real bias), and the most probable sample value is not the one that generally will be obtained - it is only the value with the highest frequency of occurrence, if the sampling would be repeated an infinite number of times. On this graph, the average sample result is simply the horizontal line on which the graph is centered.

The difference between the MPSV curve and this average simply illustrates the skewness of distribution mentioned above - and nothing more.

If the relative sampling variability standard deviation (RSD) reaches or exceeds 32% or above, then in the binomial/Poissonian case of fully liberated material, the distribution will become noticeably asymmetrical. In practice, it commonly accepted that that 32% limit is a good and safe criterion to apply, for liberated or non-liberated materials alike, not only to each sampling stage, but also to the overall, combined RSD in the case of a multi-stage protocol.

It is fair to say however, that there are "healthy debates" as to what constitutes a reasonable upper threshold for acceptable sampling variance (both 16% and 20% have been suggested). This evergreen debate is perhaps a good example of ..... "considering the issue case-by-case."

5. Conclusion

Based on these brief reflections, the author welcomes one or more "healthy debates", preferentially in this journal.

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The Complex Futility of the Liberation Factor

By Francis F. Pitard[1]

DOI: 10.62178/sst.002.005

The Complex Futility of the Liberation Factor

Dr. Francis F. Pitard
FPSC, LLC

http://www.fpscsampling.com

1952: The Early Days of Dr. Pierre M. Gy

Long before he created his famous formulas to calculate the variance of FSE, what is it that Pierre Gy did to optimize sample mass in sampling protocols?

1. He made sure the sample mass was sufficient to represent the coarsest size fractions.
2. He made sure the sample mass was sufficient to represent the coarsest particle size of the constituent of interest.

1954: A New Theory of Sampling to the Rescue

The birth of Pierre Gy's famous formula:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot g \cdot c \cdot \ell \cdot d^3$$

Gy, P.M., "Error committed when taking a sample from a batch of ore".

Congres des laveries des mines metalliques françaises, Ecole des Mines de Paris(1953). Revue de l'Industrie Minerale, France, 36, pp. 311-345 (1954).

Two Concepts in this Brilliant Formula:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot g \cdot c \cdot \ell \cdot d^3$$

1. Representing the coarsest particles of the constituent of interest: $c \cdot \ell$
2. Representing the coarsest fragments present within the lot: $f \cdot g \cdot d^3$

A theoretical subtlety that escaped many sampling experts, especially R.H. Richards*

The value of the Liberation Factor ℓ cannot, under any circumstances, alter the value of d^3.

Otherwise, the coarse fragments (larger than 1 cm) can no longer be represented in an appropriate way.

Do not mix Empiricism with Theory.

* Richards. R.H. (1908) Ore dressing. Sampling: Vol.2: 843-852; Vol. 3: 1571-1578; Vol. 4: 2031-2033. Mac-Graw Hill, New-York

The only valid calculation of the Liberation Factor, as the result of a thorough theoretical development by Pierre Gy:

$$\ell = \frac{a_{\max} - a_L}{1 - a_L}$$

The confusing calculation of the Liberation Factor, as the result of an empirical development from mineral processing engineers:

$$\ell = \left(\frac{d_\ell}{d}\right)^x$$

1 Francis Pitard Sampling Consultants, LLC., Broomfield, USA.

The damage was done, leading to:

Massive confusion,
Unjustified arguments,
Misleading modifications in TOS,
Unnecessarily complex theoretical developments,
Sampling practitioners struggling to find the best approach,
A state of TOS unattractive for International Standards,
Weak testing programs,
Showing obvious lack of maturity.

CONCLUSION:

It would be wise to return to the old strategy making the calculations of the appropriate sample mass twice, to find out what is the most stringent requirement.

CARDINAL RULE #1:

The selected sample mass must be such that all size fractions are represented in line with appropriate Data Quality Objective (DQO).

A sample that is too small to represent the coarsest fragments in the lot cannot, and will not, be representative of anything.

Pierre Gy provided a wonderful formula to satisfy Cardinal Rule #1:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot \rho \left[\left(\frac{1}{a_{Lc}} - 2\right) d^3_{FLc} + \sum_x d^3_{FLx} \cdot a_{Lx}\right]$$

$$s^2_{FSE} = \frac{f \cdot \rho}{M_S}\left[\frac{1}{a_{Lc}} - 2\right] d^3_{FLc}$$

A wise habit to prevent the misuse of Gy's formulas in domains where they do not apply.

- The wrong strategy:

$$s^2_{FSE} = \frac{f \cdot \rho}{M_S}\left[\frac{1}{a_{Lc}} - 2\right] d^3_{FLc}$$

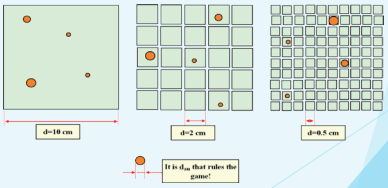
- The right strategy for a preselected DQO:

$$M_S = \frac{f \cdot \rho}{s^2_{FSE}}\left[\frac{1}{a_{Lc}} - 2\right] d^3_{FLc}$$

CARDINAL RULE #2:

The selected sample mass must be such that the maximum size d_M of the grains of the constituent of interest, liberated or not, be fairly represented in the collected sample, in line with appropriate DQO.

A sample that is too small to represent the coarsest particles of the constituent of interest in the lot cannot, and will not, be representative of anything else.



FUNDAMENTAL SAMPLING ERROR (FSE)
Gy's Many Applications

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot g \cdot c \cdot \ell \cdot d^3 \quad \Rightarrow \quad \ell = \frac{a_{max} - a_L}{1 - a_L}$$

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] \frac{f_{INT} \cdot g_{INT} \cdot \rho_{INT} \cdot d^3_{INT}}{a_L} \quad \Rightarrow \quad \ell = 1$$

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot \rho \left[\left(\frac{1}{a_{Lc}} - 2\right) d^3_{FLc} + \sum_x d^3_{FLx} \cdot a_{Lx}\right]$$

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K d^{3-x} \quad \Rightarrow \quad \ell = \left(\frac{d_\ell}{d}\right)^x$$

EMPIRICISM vs THEORY

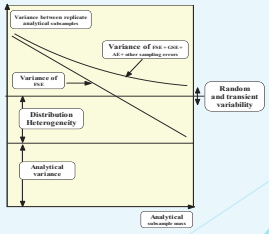
Do not confuse
Prediction & Comprehension.

Prediction from experiments are valuable, however causes of effects are multiple and the final analysis is not easy.

If observation from experiments can lead to prediction, only comprehension allows access to laws expressed in TOS.

Replicate samples variance and its components

This is where R.H. Richards in 1908 was totally confused.



From the rigorous TOS (economically impractical) to necessary approximations (economically practical)

Approximations diminish the precision of predictions; however,

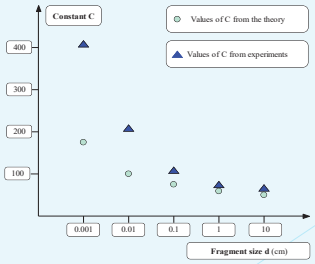
when **well understood**, should not alter the comprehensive rigor of TOS.

The reconciliation of theoretical prediction with empirical observation

Example:

More often than not, the theoretical estimate of the **variance of FSE < the variance observed in reality** from experiments using replicate samples.

Theoretical prediction vs empirical observation



Comparing oranges and apples

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K \cdot d^{3-x}$$

smaller than

$$s^2_{QFE1+AE+X} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K \cdot d^{3-x}$$

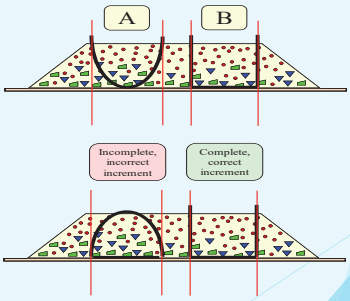
x ?

The many hurdles of empirical experiments

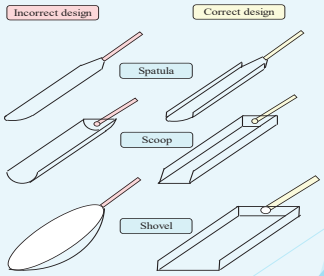
- Fragments not collected one by one at random
- Unrecognized delayed comminution of minerals of interest
- GSE
- AE
- Correctness:
  - IDE
  - IEE
  - IPE
  - IWE

Basically, empirical experiments do not have access to FSE.

IDE and IEE



Correctness is in the details



Only the Theory of Sampling has access to FSE

Which leaves us with:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot g \cdot c \cdot \ell \cdot d^3 \Rightarrow \ell = \frac{a_{max} - aL}{1 - a_L}$$

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] \frac{f_{INT} \cdot g_{INT} \cdot \rho_{INT} \cdot d^3_{INT}}{a_L} \Rightarrow \ell = 1$$

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] f \cdot \rho \left[\left(\frac{1}{a_{Lc}} - 2\right) d^3_{FLc} + \sum_i d^3_{FLs} \cdot a_{Ls}\right]$$

With all their **well-known and well-addressed limitations** of course.

Step 1. A simple and pragmatic strategy to address FSE

Allow a **Total Allotted Uncertainty** considered as an upper maximum limit.

DQO or SQC?

Examples:

Exploration for gold:	**± 32%**
Exploration for copper:	**± 20%**
Material Balance for gold:	**± 10%**
Material Balance for copper:	**± 5%**
Sales of concentrates for gold:	**±3%**
Sales of concentrates for copper:	**±1%**
Environmental assessments:	**± 32%**

DQO: Data Quality Objectives / **SQC:** Sample Quality Criteria

An important step to address the validity of simplifying assumptions

If the desired precision for FSE is ±16% or ±10%, approximations for IH may be acceptable.

However, if a precision of ±1% is required for FSE, then a careful size/density analysis may be required.

Further Reading: See WCSB10 presentation by Stephane Brochot et al.

Step 2. A good understanding of Geology and Mineralogy is important

Information from logging diamond core samples is extremely valuable to obtain information to get started with FSE, such as:

a_{max}

d_M

Mineral associations

Beware: the potential for delayed comminution
Beware: the potential for Poisson Processes

Step 3. Create a reliable model for the liberation factor

The following model must be based on reliable geological and mineral information:

$$\ell = \left(\frac{d_\ell}{d}\right)^x$$

This gives access to **D. François-Bongarçon's** favorite approach:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K . d^{3-x}$$

The necessary reconciliation & the myth

$$f . g . c . \ell . d^3 = \frac{K}{d^x} d^3$$

Both sides **must be the same, although in a different language.**
However:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K . d^{3-x}$$

and

$$s^2_{QFE1+AE+X} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K . d^{3-x}$$

are incompatible!

CONCLUSIONS

Empirical experiments are useful to detect problems:
They are whistleblowers.

However, they cannot provide solutions.

Only TOS can provide solutions through a thorough understanding of all sampling errors.

RECOMMENDATIONS

If TOS, as presently structured, seems incapable to provide solutions, it is because we don't understand TOS well enough.

All necessary approximations made in the daily applications of TOS have been well addressed a long time ago by Pierre Gy.

Reinventing the wheel does not help and most of the time leads to confusion, chaos and unnecessary expensive tests.

Anyone who wants to improve TOS first *needs to be familiar* with the subtleties of Pierre Gy's work.

Giants of Sampling 2: David W. Brunton

By Alan F. Rawle[1]

DOI: 10.62178/sst.002.006

1. Introduction

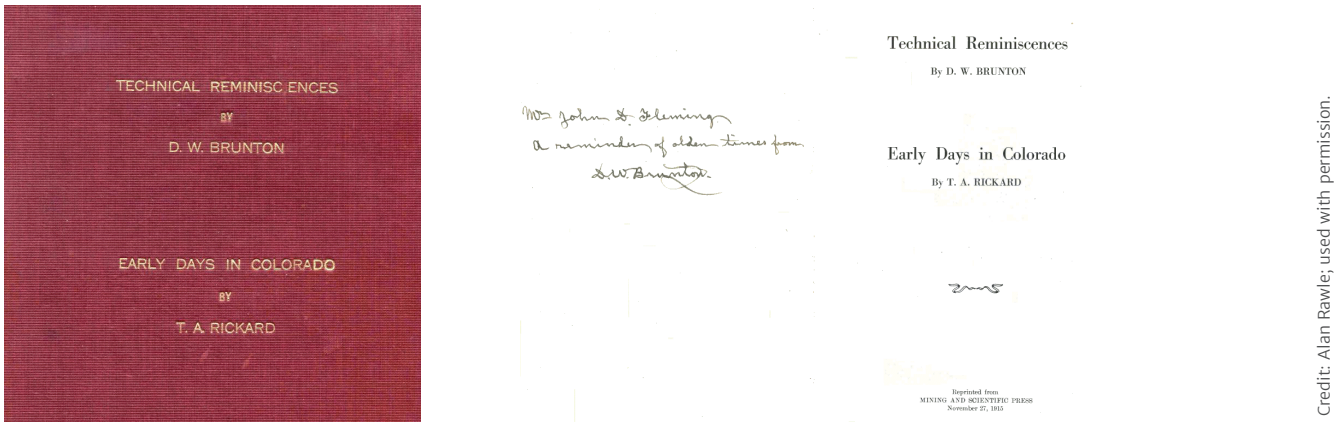
In contrast with Henry Vezin, of whom we wrote about in the last Sample Science and Technology, we have an enormous amount of information related to David (William) Brunton to draw upon. We have an extensive contribution from him in virtually every area of engineering and not simply in sampling science. He published and patented extensively and held positions of authority in the mining field. Brunton was President of the American Institute of Mining Engineers from 1909 - 1910 (and Vice President before that in 1897 – 1898). He was inducted (#181) into the National Mining Hall of Fane and Museum as late as 2004. We note, though, that Brunton was preceded by Georgius Agricola (Inductee # 180), author of De Re Metallica!

Brunton was interviewed by T A Rickard in 1921 in the Mining and Scientific Press in which many details of his early and scientific life were highlighted in the 13 pages of that article in a Question-and-Answer format. Another article written by Brunton entitled 'Technical Reminiscences', published in both the Mining and Scientific Press in 1915 and then reprinted in a small book of the same name, contains extensive details of the early pioneering days in the Colorado and Nevada mining fields (21 pages in prose format).

My copy of the latter with a dedication and signature from him is shown attached.

From these articles, we learn that Brunton was born in Ayr, Canada of Scottish parents in 1849 (June 11th) and came to the US in 1873 subsequently studying geology and chemistry at the University of Michigan in 1874 and 1875. There is a detailed autobiography written by Ginny Kilander in 'Enterprise & Innovation in the Pikes Peak Region' (Editor: Tim Blevins Pikes Peak Library District, 2011).

This excess of detailed career information relating to David Brunton means that we can only be selective in highlighting some of his achievements and inventions. Furthermore, Brunton made a fortune from an area unrelated to mining and sampling devices and that was his invention (and patent protection/defence of) of the pocket transit. He was also an expert witness in mining matters and an authority of the 'Law of the Apex', such legal dealings probably brought him significant income. His engineering skills lent themselves to car couplings, wooden mine beams, tunnelling, safety, and explosives in mining as well as several devices related to sampling of ores from a redesigned shovel and basic riffling devices to oscillating sampling devices dividing based on time.



Credit: Alan Rawle; used with permission.

Figure 1: Technical Reminiscences (AFR personal collection).

1 Retired. Hardwick, Massachusetts, USA.

Even a new design of circular slide rule was part of his skills. We'll deal with the ancillary parts first and then move onto Brunton's extensive contributions in the sampling field.

2. Car adventures

The extent of David Brunton's diverse engineering skills is displayed with relation to his purchase of the first automobile licensed in Denver, Colorado. This was no simple matter like buying a car today. In 1898, David Brunton travelled to Boston and went to an automobile show at Mechanics Institute and tested several motor cars. His selection was shipped to Denver in parts. A few months later in May 1899, Brunton noted in his diary: "May 7. Left Butte, reaching Denver on the 9th. Found Columbia electric automobile awaiting me. Spent day setting it up. May 10. Ran electric carriage on the streets in Denver." We note the early use of an electric vehicle! A picture is shown below of the 4 Brunton children going for a ride in a later car. I assume that this car was not assembled from parts.

Figure 2: The Brunton children going for a ride in a later vehicle.

It was not just fun and games for David Brunton and automobiles. From the Monthly Bulletins American Mining Congress Volume 13 Number 4 April 18th, 1910, we learn of the "Injury of D W Brunton".

"A wide circle of friends were grieved to learn of the injuries sustained by Mr. David W. Brunton of Denver in an automobile accident on Monday morning, March 25, 1910. Mr. Brunton, in company with Mr. Wellington Hibbard and Mr. Aiken, officials of the Laramie-Poudre Reservoir & Irrigation Company, was on a tour of inspection of the company's works in the vicinity of Fort Collins, Colo., when the automobile in which they were riding got beyond the control of the driver on a steep grade. The machine, after a wild career down the road, overturned, throwing the Occupants out. All were injured, Mr. Hibbard dying shortly after. Mr. Brunton sustained severe injuries on the right side of his body, his right leg being badly lacerated. He was rushed to a hospital in Denver, where he is recuperating nicely.

The accident was most unfortunate, and while the death of Mr. Hibbard and the injury of Messrs. Brunton and Aiken is deplored, friends of the latter were very glad to learn that they were not more seriously injured".

Another newspaper article claims that the accident was as a result of the driver not slowing down when implored to do so by Brunton.

He had an earlier patent (1908) for a 'Safety lock for autos' and it was stated that his wife, Katharine Kemble, was the true car enthusiast in the family.

Figure 3: The car after the "smash-up".

3. The pocket transit

This invention was the one that ensured a place in mining geology and military history for David Brunton. Prior to the invention of the 'pocket transit' mining engineers had to carry a large amount of unwieldy equipment to get basic information from a mine.

After experimentation and development, Brunton devised a portable (carried in the pocket) compass that would allow this basic information to be quickly obtained.

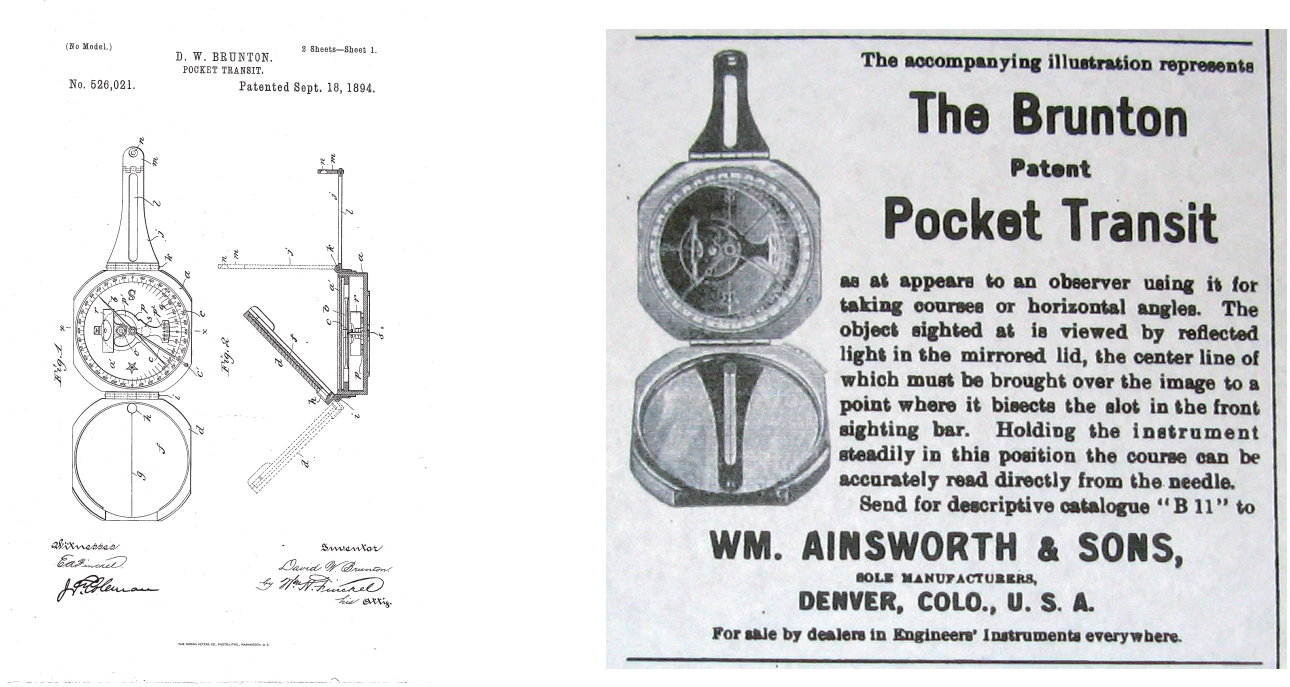


Figure 4: The Pocket transit first patent (1894) and early advertisement.

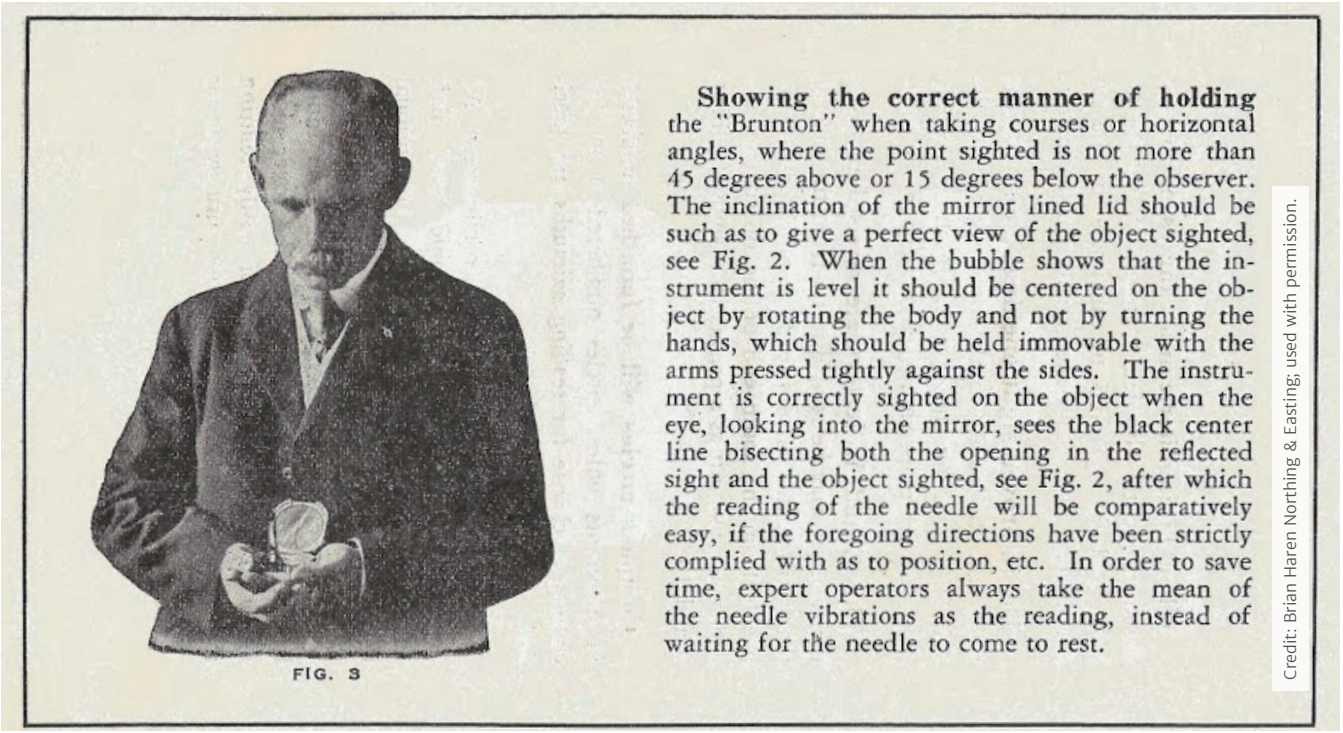


Figure 5: David Brunton using the transit (from the 1929 manual courtesy Brian Haren Northing & Easting | Making sure things are where they really are (oldtopographer.net)).

The advantage of the device was that it replaced bulky instruments. It has an accurate mirror compass, level and clinometer that reads in both degrees and percent of slope. The pocket transit could record the direction of a horizontal or vertical feature while also sighting the feature itself. With an enclosed spirit level, it could be used to measure the angles of dip and strike. He patented the initial design in 1894 (see below) but the device would have many iterations and upgrades over the years (e.g. degrees to 'mils' – dividing the circle into 6400 parts not 360 - for the US military version, the M2. This means that one mil is approximately 1000 of the radius of a circle and thus 1 mil at 1000 meters distance would mean a deflection of 1 meter, 2 mils – 2 meters etc. Gun pointing could then be carried out more quickly and effectively). Unusually, it was constructed from aluminium which was an expensive metal in those days. Many hundreds of thousands have since been sold since the early days.

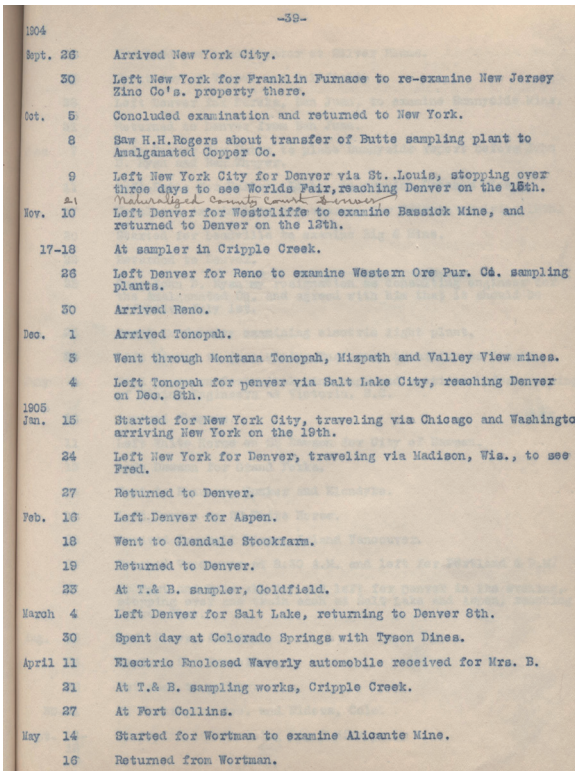
Interestingly, the opening of the patent states 'Be it known, that I, David W. Brunton, a subject of the Queen of Great Britain, but having declared my intention of being a citizen of the United States'. This was when he was still Canadian and finally naturalized on October 21st, 1904, in between trips to the World's Fair in St Louis and the Bassick mine in Westcliffe, Colorado. See the hand-written addition in (page 39 of) his diary entry below.

The first 2 or 3 transits were made by Negretti and Zamba in London in 1900 and 1901 but soon manufacture was switched to William Ainsworth and Son(s) in Denver. In 1972, following the closure of Ainsworth, manufacture was resumed in Riverton, Wyoming by the Brunton company formed specifically for that purpose.

4. Mine Tunnelling

In 1914 Brunton published a government tome entitled 'Safety and Efficiency in Mine Tunneling', (United States Department of the Interior, Bureau of Mines, USBM Bulletin, B 57). In 1916, another document entitled Safety in Tunneling (Department of the interior, Bureau of Mines Miners' Circular 13) published with John A Davis contained the important maxim 'Don't shoot into explosives with a rifle or pistol, either in or out of a magazine'.

One of Brunton's most publicised successes was in the Cowenhoven tunnel (1893) which was: double-track, 2¼ miles long under Smuggler Mountain for Aspen Mines. It was drilled through water-saturated dolomite sand up to 421.5 feet/month (!). It led to the tunnel miners for presenting him with a "large gold medal" in "recognition of his arrangements for their comfort and safety".



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1904		
Sept.	26	Arrived New York City.
	30	Left New York for Franklin Furnace to re-examine New Jersey Zinc Co's. property there.
Oct.	5	Concluded examination and returned to New York.
	8	Saw H.H.Rogers about transfer of Butte sampling plant to Amalgamated Copper Co.
	9	Left New York City for Denver via St. Louis, stopping over three days to see Worlds Fair, reaching Denver on the 18th.
	21	Naturalized County Court Denver
Nov.	10	Left Denver for Westcliffe to examine Bassick Mine, and returned to Denver on the 13th.
	17-18	At sampler in Cripple Creek.
	26	Left Denver for Reno to examine Western Ore Pur. Co. sampling plants.
	30	Arrived Reno.
Dec.	1	Arrived Tonopah.
	3	Went through Montana Tonopah, Mizpath and Valley View mines.
	4	Left Tonopah for Denver via Salt Lake City, reaching Denver on Dec. 8th.
1905		
Jan.	15	Started for New York City, traveling via Chicago and Washington arriving New York on the 19th.
	24	Left New York for Denver, traveling via Madison, Wis., to see Fred.
	27	Returned to Denver.
Feb.	16	Left Denver for Aspen.
	18	Went to Glendale Stockfarm.
	19	Returned to Denver.
	23	At T.& B. sampler, Goldfield.
March	4	Left Denver for Salt Lake, returning to Denver 8th.
	30	Spent day at Colorado Springs with Tyson Dines.
April	11	Electric Enclosed Waverly automobile received for Mrs. B.
	21	At T.& B. sampling works, Cripple Creek.
	27	At Fort Collins.
May	14	Started for Wortman to examine Alicante Mine.
	16	Returned from Wortman.

119796

[FORM FOR NATURALIZED CITIZEN.]

DEPARTMENT OF STATE
FEB 20 1922
ISSUED
WASHINGTON.

United States of America,
State of Colorado
County of Arapahoe

I, David William Brunton, naturalized and loyal citizen of the United States, hereby apply to the Department of State, at Washington, for a passport.

I solemnly swear that I was born at Ayr Ontario Canada on the eleventh day of June 1869; that my father, James Brunton, was born in Shetland and is now deceased; that I emigrated to the United States, coming from Mt. Pleasant Ontario Canada about Nov 5 1892; that I resided 30 years, uninterruptedly, in the United States, from 1892 to 1922 at Buffalo, Leadville, Denver; that I was naturalized as a citizen of the United States before the County Court at Denver City and County of Denver Colo on Oct 21 1904 as shown by the Certificate of Naturalization presented herewith; that I am the identical person described in said certificate; that I have resided outside the United States since my naturalization at the following places for the following periods:

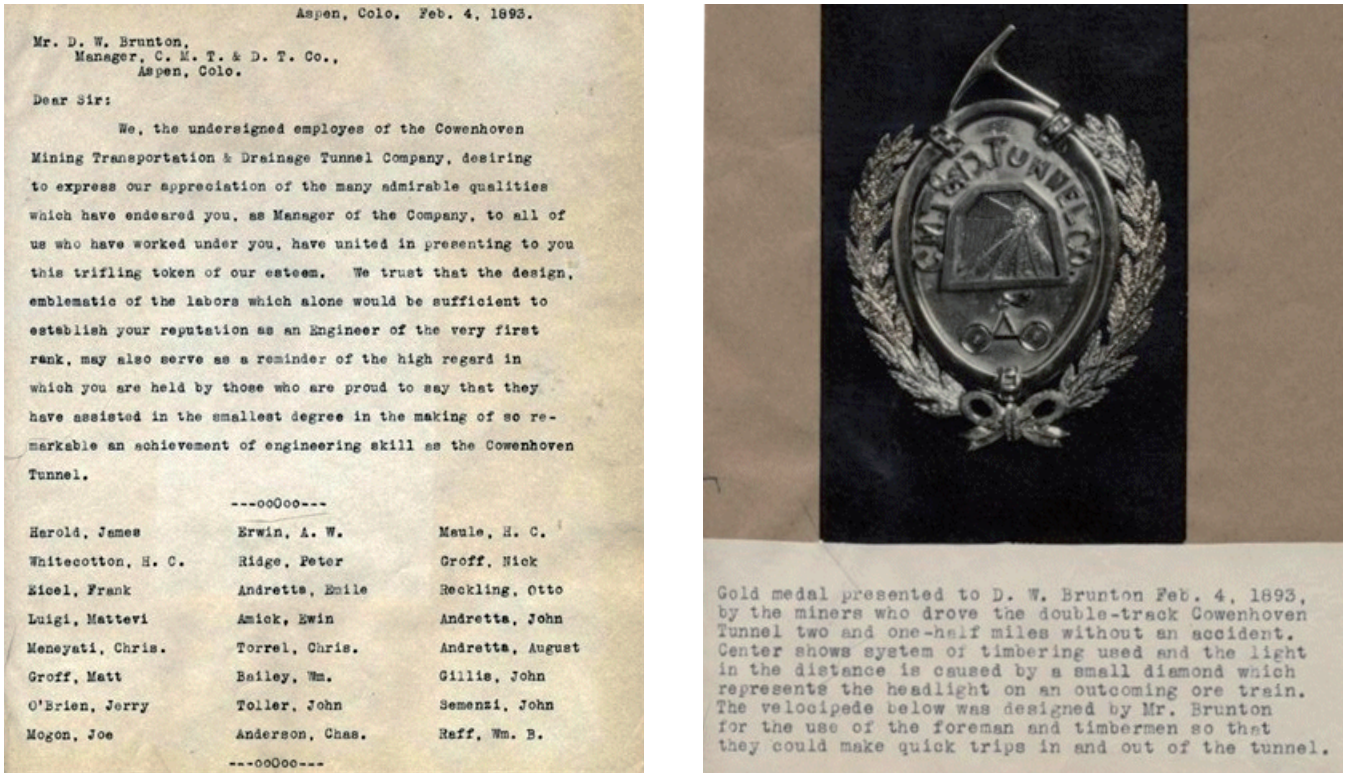
From _____ to _____
From _____ to _____

and that I am domiciled in the United States, my permanent residence being at Denver in the State of Colorado, where I follow the occupation of Mining Engineer.

My last passport was obtained from Washington on _____

Credit: Penny Larsson; used with permission.

Figure 6: Naturalization entry and 1922 passport application.



Aspen, Colo. Feb. 4, 1893.

Mr. D. W. Brunton,
Manager, C. M. T. & D. T. Co.,
Aspen, Colo.

Dear Sir:

We, the undersigned employes of the Cowenhoven Mining Transportation & Drainage Tunnel Company, desiring to express our appreciation of the many admirable qualities which have endeared you, as Manager of the Company, to all of us who have worked under you, have united in presenting to you this trifling token of our esteem. We trust that the design, emblematic of the labors which alone would be sufficient to establish your reputation as an Engineer of the very first rank, may also serve as a reminder of the high regard in which you are held by those who are proud to say that they have assisted in the smallest degree in the making of so remarkable an achievement of engineering skill as the Cowenhoven Tunnel.

---ooOoo---

Harold, James	Erwin, A. W.	Maule, H. C.
Whitecotton, H. C.	Ridge, Peter	Groff, Nick
Eicel, Frank	Andretta, Emile	Reckling, Otto
Luigi, Mattevi	Amick, Ewin	Andretta, John
Meneyati, Chris.	Torrel, Chris.	Andretta, August
Groff, Matt	Bailey, Wm.	Gillis, John
O'Brien, Jerry	Toller, John	Semenzi, John
Mogon, Joe	Anderson, Chas.	Raff, Wm. B.

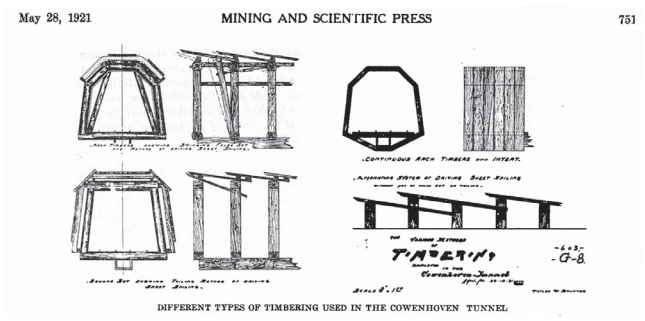
---ooOoo---

Gold medal presented to D. W. Brunton Feb. 4, 1893, by the miners who drove the double-track Cowenhoven Tunnel two and one-half miles without an accident. Center shows system of timbering used and the light in the distance is caused by a small diamond which represents the headlight on an outcoming ore train. The velocipede below was designed by Mr. Brunton for the use of the foreman and timbermen so that they could make quick trips in and out of the tunnel.

Credit: Penny Larsson; used with permission.

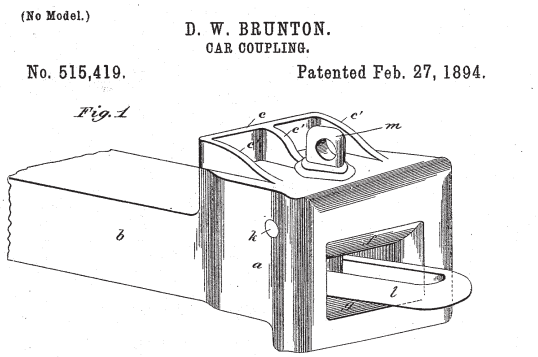
Figure 7: Letter and medal presented to David Brunton by miners of the Cowenhoven Tunnel.

There were a number of other innovations pertaining to the tunnel. One was in a bonus system instigated by Brunton where the miners received extra renumeration for all work in excess of 150 feet/month. Some machine drillers could then reach the lofty salary of 24 Pounds (strange as the $ was the currency of the US) per month. Because of the nature of the terrain new ways of supporting the tunnel with mine timbers were found. This led to patents US 692111 Mine-Timber January 28, 1902 and US759418 Mine Timber May 10, 1904 plus car couplings (US 515419 Car Couplings February 27, 1894).



May 28, 1921 MINING AND SCIENTIFIC PRESS 751

DIFFERENT TYPES OF TIMBERING USED IN THE COWENHOVEN TUNNEL



(No Model.)

D. W. BRUNTON.
CAR COUPLING.

No. 515,419. Patented Feb. 27, 1894.

Fig. 1.

Figure 8: Mine Timbers and car coupling.

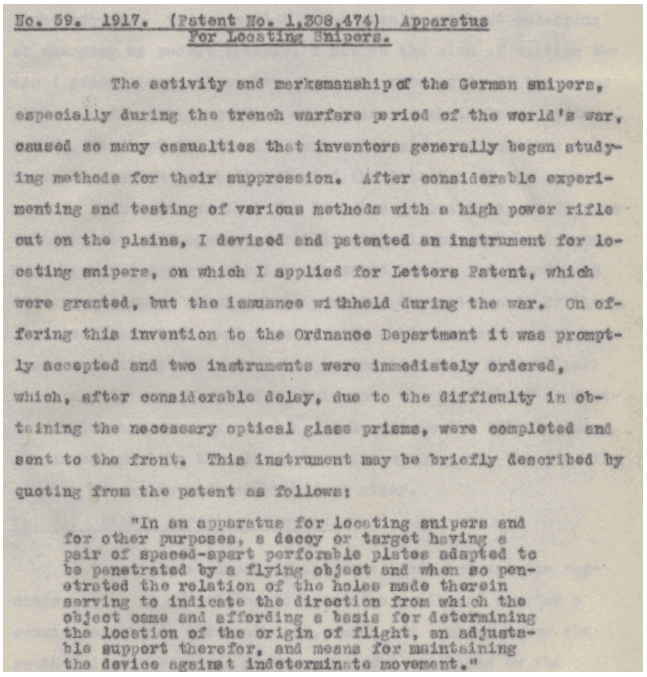
5. Mine mapping and the Law of the Apex

In the early days of mining, it was relatively easy to follow a find or vein until it potentially could (or did) venture under another property or claim. These issues led to the 'Law of the Apex' (or The General Mining Law of 1872 (as amended)). In broad terms, the law refers to the principle that title to a given tract of mineral land, with defined mining rights, goes to the individual who locates the surface covering the outcrop or apex. They had the right to mine the vein even if its subsurface extension continued beneath other mining claims. To prevent conflicts and resulting litigation, accurate mapping of mines was essential.

Brunton devised a method where various sections through the mine were displayed on (Vellum) tracing paper and placed one on top of another in a book. In this manner, the workings, faults, and ore bodies could be seen in relation to one another. Brunton used the approach successfully in court. He states in his 1921 interview with Rickard 'The best method of placing actual mine conditions before a judge or jury is by some graphic method of visualization. Verbal descriptions of mine workings convey little or nothing to a man who has never been underground'.

6. Work during World War 1

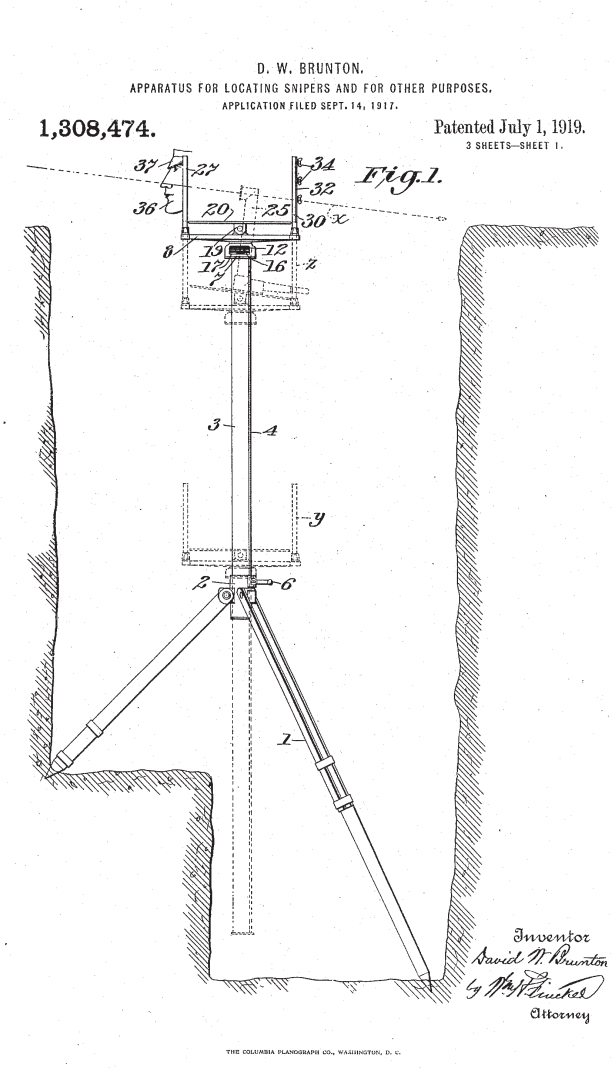
David Brunton represented AIME on the War Committee of Technical Societies first as a member and then as Chairman of that committee. Something like 135000 suggestions as to inventions that could help the war effort were made and these needed evaluation (most of them being useless). Brunton himself patented an idea for locating snipers. This remained classified until after the war.



No. 59. 1917. (Patent No. 1,308,474) Apparatus For Locating Snipers.

The activity and marksmanship of the German snipers, especially during the trench warfare period of the world's war, caused so many casualties that inventors generally began studying methods for their suppression. After considerable experimenting and testing of various methods with a high power rifle out on the plains, I devised and patented an instrument for locating snipers, on which I applied for Letters Patent, which were granted, but the issuance withheld during the war. On offering this invention to the Ordnance Department it was promptly accepted and two instruments were immediately ordered, which, after considerable delay, due to the difficulty in obtaining the necessary optical glass prisms, were completed and sent to the front. This instrument may be briefly described by quoting from the patent as follows:

"In an apparatus for locating snipers and for other purposes, a decoy or target having a pair of spaced-apart perforable plates adapted to be penetrated by a flying object and when so penetrated the relation of the holes made therein serving to indicate the direction from which the object came and affording a basis for determining the location of the origin of flight, an adjustable support therefor, and means for maintaining the device against indeterminate movement."



Credit: Penny Larsson; used with permission.

Figure 9: Brunton's notes on locating snipers and the published patent.

7. Sampling and samplers – theory and practice

Key (almost obligatory!) reference documents in this journey are:

- Ore-Sampling Conditions in the West (T R Woodbridge) Technical Paper 86 Department of the Interior Bureau of Mines (1916). Woodbridge worked at the Taylor and Brunton sampling works (of which we'll read more later) and this work contains a lot of really useful information, photographs, and pictures
- Mechanical Ore Sampling in Montana (H B Pulsifer) University of Montana Bulletin No 3 State School of Mines, Butte, Montana (March 1920). Lots of excellent diagrams and pictures of the early sampling days making much reference to the document immediately above. It does, however, have many excellent photographs of sampling devices and methods
- The 3 (classic) sampling papers in AIME published by Brunton:
    - A new system of ore sampling Trans AIME Volume 13 (actually XIII) 639 – 645 (1884)
    - The Theory and Practice of Ore-Sampling Trans. AIME Volume 25 (actually XXV) 826 – 844 (1895)
    - Modern Practice of Ore-Sampling Trans. AIME Volume 40 (actually XL) 567 – 596 (published 1910)

Also, we note than Geoff Lyman in his article (A brief history of sampling Aus IMM Bulletin, 39 – 45, June 2014) discussed Brunton at length so we'll try not to duplicate his (recommended) commentary which can be found as a free download on ResearchGate.

Brunton looked at all aspects of sampling from cone-and-quarter, through shovels, rifflers, and mechanical systems. Invariably he patented something related to each of those inventions. Further, this was not just theoretical work but employed in large scale sampling works in several very large mines in the west.

From a theory perspective, let's talk about Brunton's last 2 papers. The basic synopsis is that one particle added or subtracted would not make a difference to the "allowed" error and Brunton considered the addition of 1 particle of gold in 1/16 assay ton causing a 1% error in an ore running at $5200/ton. Other points:

- Used the cube as the reference shape, not the sphere. No description of the effect of shape or a shape factor
- The smaller the sample taken, then the finer the crushing needed
- Considered the maximum concentration of valuable ore in relation to the average grade
- The s.g. of the richest mineral (taken from Dana)
- 'The number of particles of richest mineral in excess or deficit'
- Dealt in tons and pounds.....and "screen cloth". No SI units or ISO/ASTM screen dimensions

In the 1895, paper Brunton displays a number of tables and graphical plots that are not easy to digest and there is not a single diagram or picture shown. It attempts to differentiate sampling for rich and lean ores based on the metallic content – something that still causes controversy today (the liberation factor in the Gy equations, for example). The 1909 paper contains some pictures (e.g. of cone and quartering and shovelling) and diagrams (e.g. sections taken by the Snyder, Vezin, and Brunton samplers) as well as detailed tables justifying the Brunton techniques of sampling. There are diagrams of the huge Taylor and Brunton samplers (more on this later) and flow charts of how huge ore deliveries are divided up: "The results of the investigations recorded in this paper show how absolutely necessary it is that ore-samples should be re-crushed after each successive "cutting-down," so that as the sample diminishes in weight, there may be a nearly constant ratio between the weight of the sample and that of the largest particle of ore contained therein"

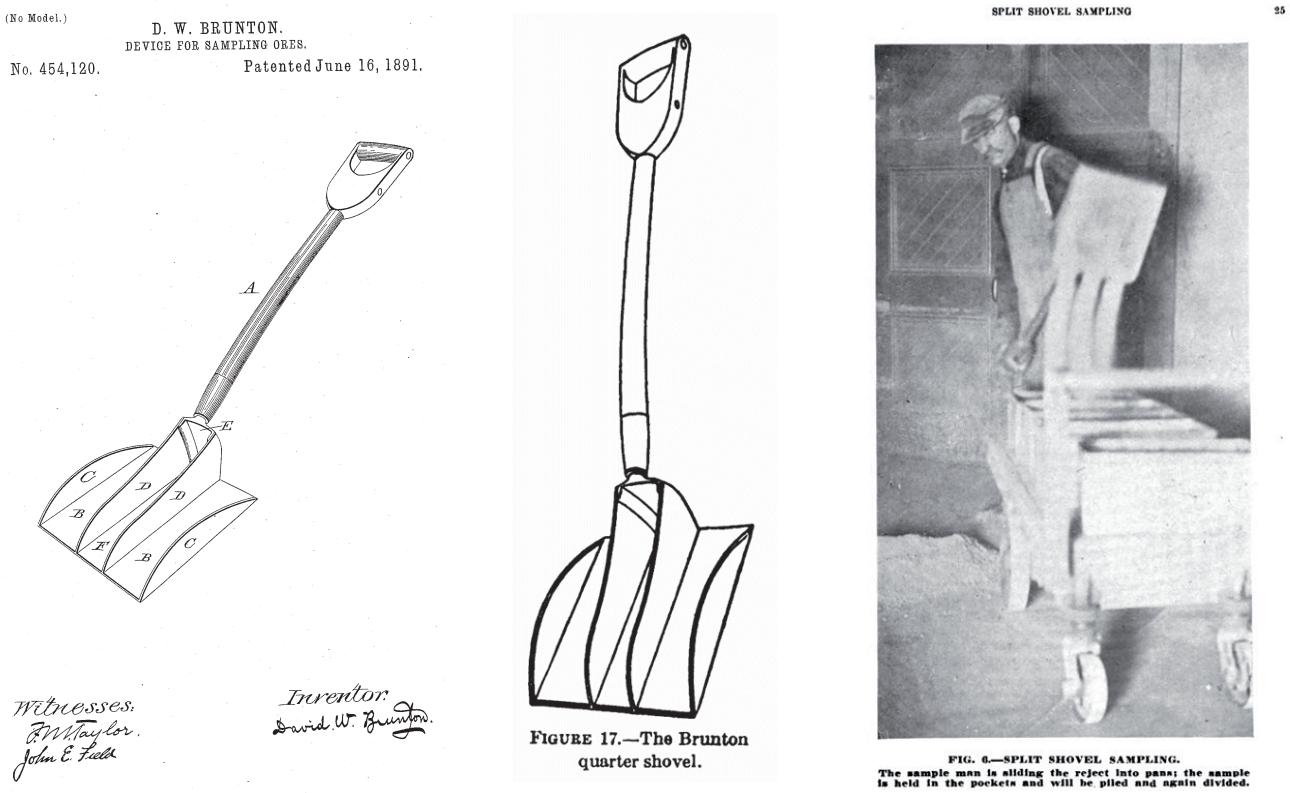


Figure 10: The 'simple' Brunton Shovel and its use (delimitation errors?).

8. Manual Division - The Brunton shovel

Shovelling (e.g. alternate shovels) has been a widely used method of sample division over the years and Brunton obviously thought carefully about the method and how to improve it.

There are actually 2 types of Brunton shovel. The simple 3 compartment (the centre one being half the volume of each of the outer 2 providing a quarter of the total in the centre) one patented in 1891.
The device works by shovelling in the normal manner and tilting the shovel backward in order to remove ¾'s of the total taken. The ¼ remaining in the centre compartment is then recovered by tilting the shovel forward into the appropriate lot. It appears to be a recipe for repetitive strain injuries! We can also see with segregated piles (always the case) that there's a tendency for the larger material to be retained in the outer compartments and the finer ore (probably richer) confined to the centre sampled compartment.

The 7-compartment shovel, with 3 sample and 4 reject divisions, may be used with higher-grade and smaller samples, and for original and duplicate samples.

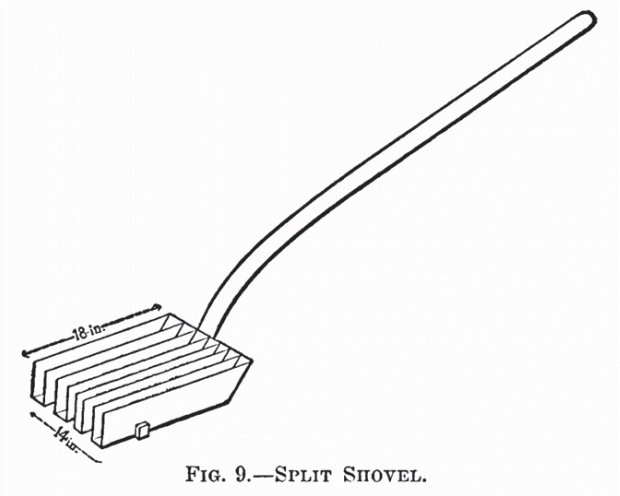
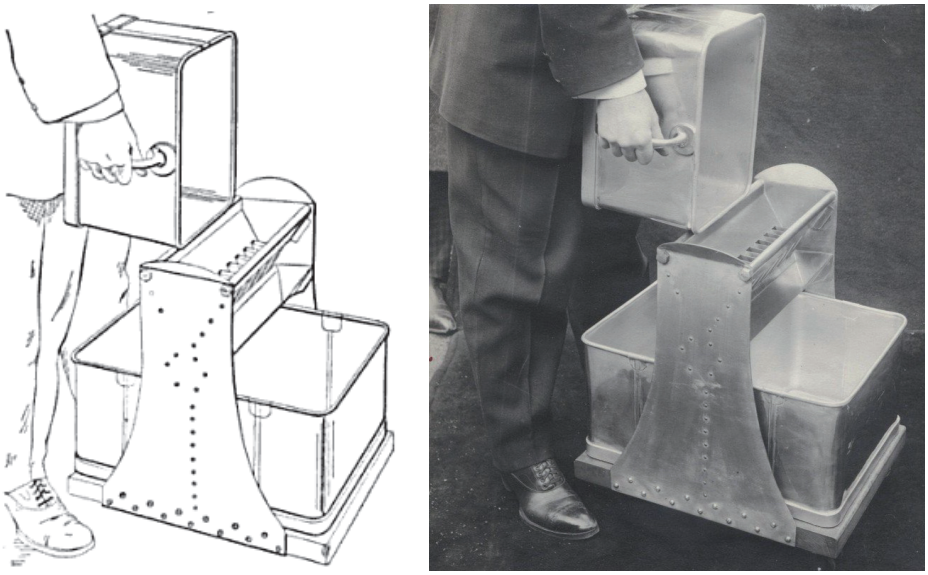


Figure 11: 7-compartment split shovel (from Hofman Metallurgy of Lead 1899).

9. Riffle divider

This is a slight improvement to the shovelling method and the cone and quarter method (for which Brunton had an attempt at mechanization – patent in 1896.

There are a number of similar photographs including one in the 1921 Interview article. This type of (non-rotary) sample divider still sees regular use.



Credit: Penny Larsson; used with permission.

Figure 12: Diagram from Richards' Ore Dressing. Picture shows David Brunton's lower half including trousers/pants.

It is important to note that the methodologies behind these huge constructions that we will describe have, in many instances, been forgotten by other industries. It seems ridiculous to talk to those in the pharmaceutical industry about needing shovelfuls of sample, but the problems related to the largest particles in the system hold true there as well as the mining industry.

The basic concepts of sampling were well known and thought out by players such as Brunton and Vezin and were summarized in Warwick's Notes on Sampling (see Figure 13).

The fundamental constraint is 'The entire stream for a fractional portion of the time' and is the philosophy of Brunton's oscillating sample dividing on the basis of time. Several such dividers would be employed in the large sampling towers present in mines.

Brunton's oscillating sampler was shown in diagrams and pictures in his 1909 paper and those pictures are reproduced in Geoff Lyman's text. It was regarded as an improvement on his earlier vibrating method.

10. Mechanized routes of sample division

This probably is the zenith (or the apex?) of sample division and the practical outcome of years of thought and many (rejected) designs. I am remined of the comment by Warwick in 'Notes on Sampling': "But it is quite delusive to attempt to check a machine by a notoriously inaccurate method".

The oscillation is up to 72 times per minute and, typically, would sample in a representative 1/625 of the whole stream. This is from taking 4 portions each at 20% of the whole stream – $(1/5)^4$. Like most sampling devices it does not cope well with slurries or damp/sticky materials.

NOTES ON SAMPLING. 17

scoop across the stream of ore, yet in order for it to do its work perfectly the stream of ore should be kept as solid as possible. A falling stream of ore is difficult to control; it scatters and strikes the edge of the sampler with great force, and at all angles. But, by confining the ore so that it is delivered in a steady stream and at a minimum velocity, the correctly designed sampling machine will do its work quietly and according to the plans of the designer.

In addition to the principles already laid down as to the method of workng automatic samplers, in order for such machines to be really efficient, they should fulfil certain other requirements.

1. The machine should be simple, readily cleaned, stand much wear and tear, and not require to be stopped at frequent intervals for adjustment or repair.

2. As the quantity of the ore is reduced in bulk, the ore should be recrushed so that the ratio of the largest piece to the weight of the whole sample shall be within limits already laid down in a previous article.

3. The cut should be taken as frequently as possible.

And to recapitulate what was said in the beginning of this article:

4. The sample should be taken across the entire stream of ore.

5. It should be taken evenly from all parts of the stream, i. e., as much from one part as from the other.

6. That the stream should be delivered steadily to the sampler, and in as solid a condition as possible.

Any automatic sampling machine or plant built according to these principles will certainly give a most accurate sample in the quickest possible time, and at a very small expense.

Figure 13: Basic concepts for mechanized sampling (from Warwick).

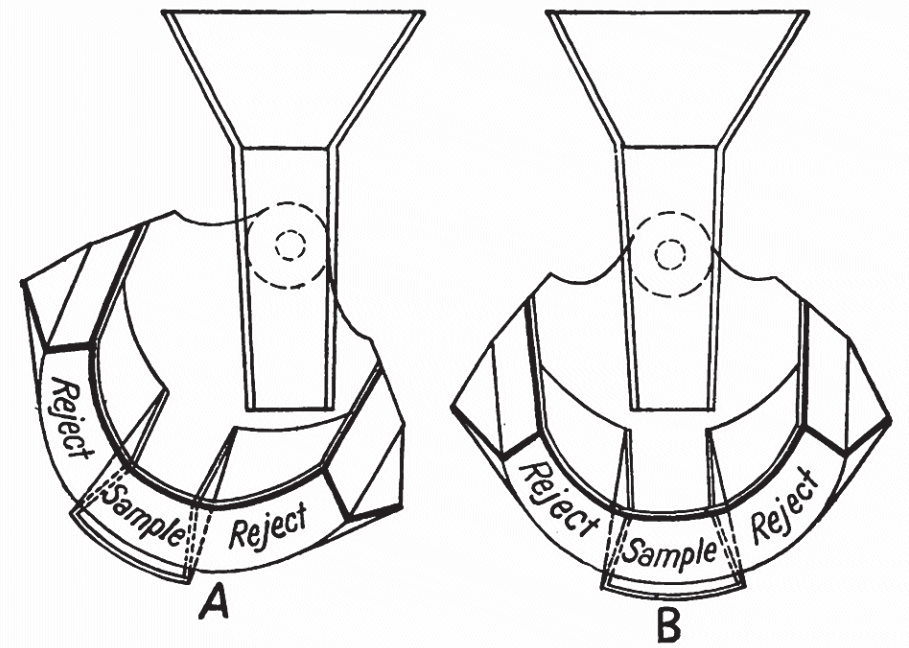


FIGURE 16.—Brunton oscillating time sampler. A, position of sample spout when ore is going to reject; B, position when ore is going to sample.

Figure 14: Brunton's oscillating time sampler (from Ore-Sampling Conditions in the West).

Brunton formed a partnership with Frank M Taylor and constructed 'Taylor and Brunton' sampling works all over the mines in the west of the US. Many close relatives of both Brunton and Taylor were employed within these works. W S Copeland was DWB's brother-in-law and was married to DWB's sister, Aggie E Brunton. WS managed the Aspen works and WS's brother, Lewis A managed the Utah works. Another brother of WS, George E, managed the Cripple Creek works. Tyler Woodbridge, to whom we have made extensive reference to his Ore-Sampling tome was "Tyler R. Woodbridge, Civil Engineer, care Taylor & Brunton Sampling Co., Victor, Colo".

This 4-storey building, making use of gravity, enabled a 1-ton sample from a rail cart to be automatically reduced down, via a number of comminution stages, to 8 ounces (a 99.84% reduction) and the remainder ('reject') loaded into delivery cars for further processing. This 8 ounces (~ 227 grams) lot sample is still almost 8 times larger than the standard 30-gram assay routinely employed in gold mines nowadays.

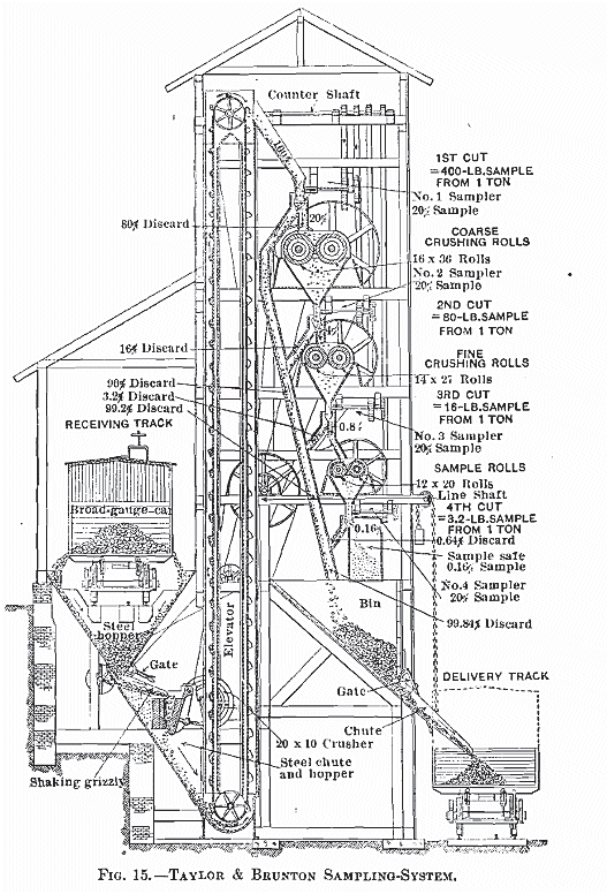


FIG. 15.—TAYLOR & BRUNTON SAMPLING-SYSTEM.

Figure 15: Diagram and the T & B sampling system (from Brunton's 1909 AIME Paper).

Figure 16: Taylor and Brunton Sampling Company – Cripple Creek district History Colorado. Accession # 90.156.384.

Customers submitted ore for analysis and were issues a simple report detailing the valuable metals (usually Au or Ag), moisture content (the buyer does not want to pay for water). Sometimes an analysis would simply state 'No gold'.

It's interesting to note that the importance of sampling was well-known in the late 1800's and early 1900's.

One wonders if so much care and attention is employed now or whether "The work of sampling is often looked upon as within the realm of boys and pensioners only' (William Glenn AIME Volume XX "Sampling Ores Without Use of Machinery" page 155 (1892)) and 'It is perfectly evident, as Mr. Glenn says, that a vast amount of skill and precision is daily wasted by our chemists in the delicate analysis of samples that mean nothing' (Dr R W Raymond in the Discussion following the above article (quote spans pages 164 – 165)).

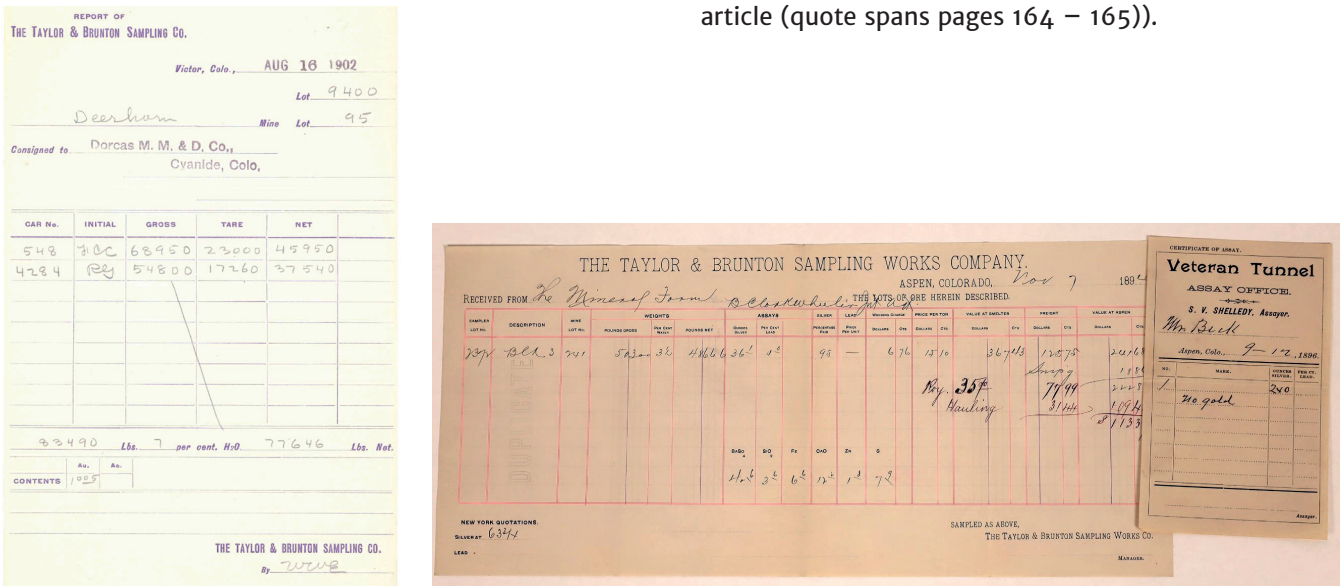


Figure 17: Sampling Reports from the Taylor and Brunton Sampling Works.

11. Brunton's genealogy and heritage

Father: James Brunton (1820 - 1867)

- Born in Galashiels, Scotland (David Brunton states "Selkirk" in 1921 Mining and Scientific Press interview)
- 1820 to William Brunton and Ann Elizabeth Button
- Died: Aug 5th, 1867, in Ontario, Canada

Mother: Agnes Dickie (1824? - 1902)

- Born in Scotland (Kilmarnock) April 24 (?), 1824 to Thomas Dickie and Janet Halbert
- Died: Sep 4th, 1902, in Brantford, Ontario, Canada
- Year of birth probably incorrectly stated as 1833 in a number of texts

Spouse: Katharine Kemble Brunton (1865 - 1928)

- Mr. Brunton married, at Kingston, New York, February 11, 1885, "Miss Katharine Kemble, of that city. Mrs. Brunton is a lady of graceful accomplishments, and is descended from a distinguished colonial ancestry, one of whom was Colonel Johannis Snyder, one of the patriots of the American Revolution. Through his service, she is a member of the Daughters of the American Revolution"

Children:

- Fredric Kemble Brunton (1886 - 1929)
- John Teller Brunton (1892 - 1956)
- Harold James Brunton (1893 - 1941)
- Marion B. Brunton (April 26th, 1898)
  - Married Nelson Earle Barker (1892 - 1980)
  - Died: San Diego Mar 22nd, 1944)

In his 1909 AIME paper, Brunton stated 'The art of sampling has now reached a stage where a standardization of methods is both desirable and possible'. I'm not sure that this stage has been reached over 100 years later despite the pioneering work of David Brunton and others. As his legacy, we should simply quote a compatriot, W L Saunders of New York: "No one is more competent to discuss the modern conditions in mining and metallurgy than Mr. Brunton, for he not only speaks as one in authority, but his experience and his ability entitle him to a hearing as one of the first rank among mining engineers. The moral code set forth in the concluding paragraph of his paper is worthy to be placed as a classic in the annals of the Institute, and it should form the basis of instruction to mining engineers at the colleges". (In the Discussion following the reading of David Brunton's paper "Modern Progress in Mining and Metallurgy in the Western United States" Trans. AIME, Volume XL, 543-561, (1910). Same volume as the "Modern practice of ore-sampling" 567-596).

ACKNOWLEDGEMENT

David Brunton was a keeper of detailed notes and much of this has been retained in various locations. When I first embarked on this journey for a webinar given in December 2012 (sadly no longer available), I had the considerably fortune to become acquainted with Penny Larsson. Penny was the great-granddaughter of DWB, on her mother's side). She provided access to a huge amount of information that she made available from her personal collection of DWB's photo albums, scrapbooks, diaries, and books and some of which she documented on ancestry.com (she allowed me access to this site too). I have been unable to reconnect to her (I guess she'd be in her 80's now), but her generosity and kindness enabled a superb presentation to be constructed with those personal touches unavailable elsewhere. I have used some of the material within this paper and I gratefully and humbly acknowledge her contribution.

DS3077 - Revised 3rd ed. Launched October 2024

By Kim H. Esbensen[1]

DOI: 10.62178/sst.002.007

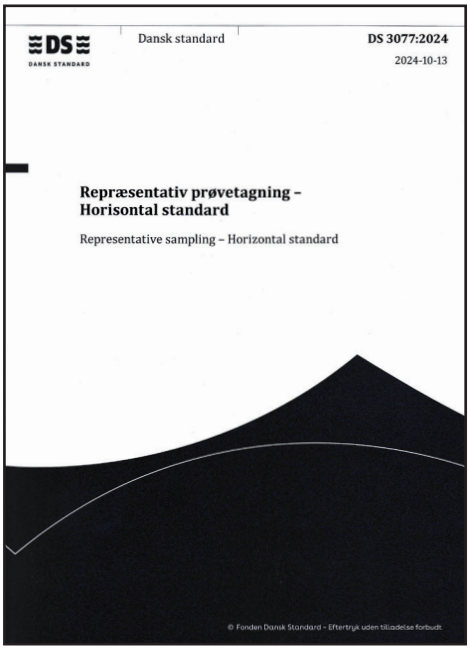
Danish Standard Foundation (DS) announces publication of the generic sampling standard DS 3077:2024, released in its 3rd revision October 2024. This represents the culmination of a 15+ year project, initiated in 2008. The 3rd edition supersedes the second edition, which have been available since 2013.

DS 3077:2024 "Representative sampling – Horisontal standard" has the ambition to be the de facto universal standard for sampling of all particulate, aggregate and mixture materials (including slurries):

DS 3077:2024 presents the authoritative foundation for all practical sampling activities 'from-lot-to-aliquot', including sampling in the laboratory, covering the gamut of science, technology, industry, commerce, trading and society (e.g. for supervisory and regulative authorities) a.o.

SCOPE:

"The Theory of Sampling (TOS) is a generic, matrix-independent framework for representative sampling of all types of aggregate and mixture materials (solid, slurries) in all grain-size brackets (from broken ores to powders). TOS' universal sampling principles can be applied uniformly to all types of materials, and lots composed by aggregate particular matter and slurries. This document describes a generic sampling process in sufficient detail and covers all elements necessary for the stated objective, enabling documentation of sampling representativity under the specified conditions for the sampling process employed. DS 3077 constitutes a complete competence basis for representative sampling, ensuring appropriate levels of accuracy and precision for both primary sampling as well as for all sub-sampling procedures and mass-reduction operations subsequent stages before delivering a guaranteed representative aliquot for analysis. This document outlines a systematic scientific basis for designing new and assessing, and if necessary improving, the performance of existing sampling procedures. The approach described in this document will contribute toward increased reliability in decision-making based on analytical measurement results. This document establishes a basis enabling professional sampling quality control (QC) by mandating disclosure of results from relevant sampling quality objectives (QO): For sampling of stationary lots: Replication Experiment (RE); for sampling of dynamic lots: Variographic Analysis (VA). This document contains an independent macro with variographic software (freeware) making variographic characterisation available for a set of samples restricted to 100 (Annex C)."



DS 3077:2024 can be downloaded from DS webpage: https://webshop.ds.dk/en/standard/M374267/ds-3077-2024

DS has offered to project manage a process with the aim of proposing DS 3077:2024 (3rd ed.) to become an ISO standard. The International Pierre Gy Sampling Association (IPGSA) wishes actively to support this project with expertise and competent personal resources.

1 Chairman standard committee S-890 (Danish Standard).

Sampling Science & Technology: Inaugural Editorial Board

DOI: 10.62178/sst.002.008

SST is proud to announce the SST inaugural editorial board (2024), which will resume its duties and responsibilities effective after publication of SST#2.

The editorial board is comprised by seven distinguished academics and scholars, industrial experts and consultants, all with profound expertise extensive experience regarding sampling in science, technology, industry, commerce and society.

The editor extends a warm welcome to all!

Richard Minnitt

Professor Emeritus, Witwatersrand University, consultant

Richard Minnitt completed a M.Sc. in geology in the Murchison Range and a Ph.D. in the Richtersveld regions of southern Namibia. He joined Anglo American and later JCI, after which he spent 14 years doing contract and consulting work. He completed a second MSc in mining and joined the School of Mining Engineering at WITS in 1995, where he taught courses in Mineral Economics and Geostatistics. His interest in sampling of particulate materials arose from the numerous visiting lecturers he invited to Wits University including Dominique Francois Bongarçon, Francis Pitard, Geoff Lyman and Kim Esbensen. Dick retired from Wits in 2017 but continues to consult for international mining companies and research in his fields of interest. He now holds a position as a Visiting Emeritus professor where he continues to teach postgraduate classes and supervises M.Sc. and doctoral students.

Stéphane Brochot

Managing director CASPEO

Stéphane Brochot obtained his Ph.D. in physics from the University of Orleans-Tours and completed his education with a degree in mathematical engineering and computer processing. He joined BRGM (the French Geologic Survey) in 1991 as a researcher and as head of software development. Since 2004, Dr. Brochot has been co-managing director of Caspeo, where he continues research on mineral processing modelling and simulation, sampling, data reconciliation and metal accounting. Brochot is one of the inventors of INVENTEO. His extensive expertise in sampling has been beneficial for several mining companies and other industries dealing with sampling of solids.

Claudia Paoletti

Program Manager, ENABLE Department, European Food Safety Authority (EFSA)

Claudia Paoletti did her Master in Biological Science at the University of Rome (Italy) and her Ph.D. in Plant Genetics at the University of Connecticut, USA. For three years she was at Dalhousie University (Canada) studying plant population genetics and biometry. She continued her activity at the Research Institute for Industrial Crops in Bologna (Italy) where she focused on the evaluation of the risks of transgenic crops. In January 2006 she joined the GMO Unit of the European Food Safety Authority (EFSA) first as Team Leader and then as Deputy Head of the Unit. In 2019 she was appointed manager of the programme designed to reorganise the EFSA in preparation for the new European Law on food safety. She has been the Italian expert for the definition of the European Commission sampling plans for GMO detection in conventional seeds. She coordinated the European sampling research project KeLDA and she has been the biometric officer of the EU Community Reference Laboratory for GMOs. She is expert consultant for ISO/IWA committees, OECD, CEN, the European Commission and FAO. She organised international training courses on food/feed safety for the European Commission, UNIDO, PHARE project and universities within and outside Europe. She has over 90 contributions either as book chapters, or as peer-reviewed papers.

Simon Dominy

Associate Professor, Camborne School of Mines, University of Exeter; consultant

Simon Dominy is a mining geologist-engineer with over 25 years based in operations, consulting and academia. He has experience across mine production, corporate business development, and multi-disciplinary studies. Simon has a background in underground operations management and technical/leadership roles, with multi-commodity and continent experience. He has worked across the mine value chain from project studies, through to mine reopening/development, operations and operational improvement. He is a Visiting Associate Professor at the Camborne School of Mines, University of Exeter, UK, and holds technical roles with Novo Resources Corporation, Artemis Resources Ltd and OCX Gold Group.

Rodolfo Romañach

Department of Chemistry, University of Puerto Rico at Mayaguez

Rodolfo Romañach is Professor of Chemistry at the University of Puerto Rico – Mayagüez Campus, and site leader for the Center for Structured Organic Particulate Systems. He worked in the pharmaceutical industry for over 12 years before joining the UPR Chemistry Department in 1999. He found his mission in training a new generation of pharmaceutical scientists capable of doing real time process measurements in the manufacturing area. He is presently continuing efforts to improve the teaching of chemometrics and the Theory of Sampling to further understanding the errors affecting real time process measurements—and what to do about it all.

Pentti Minkkinen

Professor Emeritus, Lappeenranta University of Technology, Sirpeka Oy

Pentti Minkkinen received his MSc (eng.) from Helsinki University of Technology in 1969. He then worked as an Associate Expert in two UN Development Program mineral exploration projects in Turkey and in Egypt before completing his graduate studies at Helsinki. In 1976, he started as Associate Professor (Inorganic and Analytical Chemistry) at a newly founded University, Lappeenranta University of Technology, from which retired as full professor by the end of 2007, after a 40+ year tenure. Here he started teaching the theory and applications of sampling in 1978, soon also chemometrics, as an important part of process analytical chemistry. He has been lecturing sampling at undergraduate and graduate courses at several universities, at professional continuing education courses, and at numerous conferences and at industry courses. After retirement, he worked three periods as Visiting Professor at Aalborg University, Campus Esbjerg, Denmark in Prof. Esbensen's research group (2007, 2008 and 2009). In 2012, he founded Sirpeka Oy from which he offers consulting services on sampling, analytical quality control and in chemometrics. At his old university, now amalgamated and named Lappeenranta Lahti University of Technology (LUT), he continues his scientific career as Professor emeritus. Prof. Minkkinen was the founding chairman of the continuing biannual conference series, Scandinavian Symposium of Chemometrics. He was also co-chairman for the first World Conference on Sampling and Blending. He is the founding chairman of the Discussion Group of Chemometrics in the Finnish Chemical Society. He has published ~80 papers on chemometrics and sampling in refereed journals and conference proceedings; his invited and contributed lectures in various conferences and symposia contributions is close to 200. He has received three international awards, the Kowalski Prize in Chemometrics (2002), the Herman Wold Gold Medal in Chemometrics (2007) and the Pierre Gy Sampling Gold Medal (2005).

Claas Wagner

Professor, University of Applied Sciences, Lucerne, Switzerland

Claas Wagner originally trained as an economist, realized that his real interests were with environmental and energy related topics. Sustainable resource management, emission reduction procedures and energy efficiency issues share common ground: decisions need to be based on valid data. This led to Claas' Ph.D. on representative sampling and data analysis for quality monitoring in large-scale combustion plants. Since almost 10 years, Claas is professor in Environmental Engineering and Ecology at the University of Applied Sciences Lucerne, Switzerland, focusing on environmental impact analysis and evaluation tools. Claas also combines his fields of interest as a consultant providing quality assurance approaches for various industries.

Kim H. Esbensen

Editor

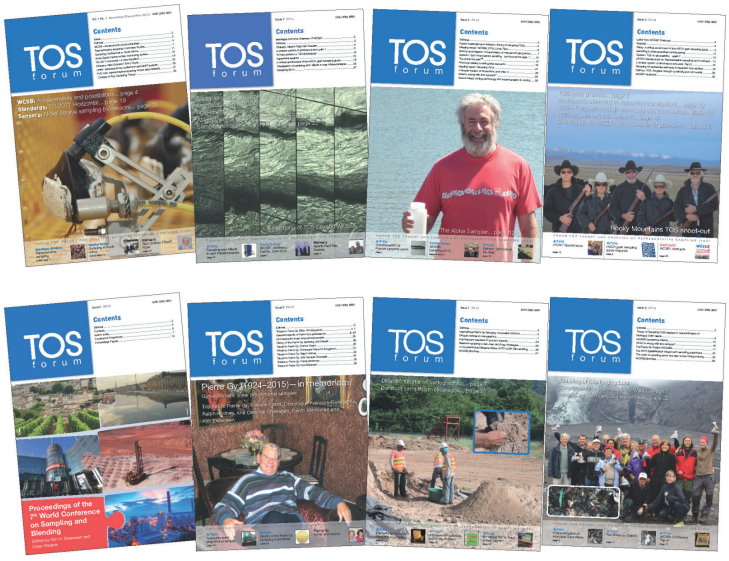
Kim H. Esbensen, M.Sc. (geology, Aarhus University), Ph.D. (Technical University of Denmark), Dr. (hon) (Technical University of Lappeenranta) has been research professor in Geoscience Data Analysis and Sampling at GEUS (National Geological Surveys of Denmark and Greenland (2010–2015), Chemometrics & Sampling professor at Aalborg University (2001–2015), professor (Process Analytical Technologies) at Telemark Institute of Technology, Norway (1990–2000 and 2010–2015) and professeur associé, Université du Québec à Chicoutimi (2013–2016). From 2015 he phased out a more than 30-year academic career for a new quest as an independent researcher and consultant. But as he could not terminate his love for teaching, he is still very active as an international visiting, guest and affiliate professor. A geologist/geochemist/metallurgist/data analyst of training, he has been working 20+ years in the forefront of chemometrics, but since 2000 has devoted most of his scientific R&D to the theme of representative sampling of heterogeneous materials and processes (Theory of Sampling (TOS), PAT (Process Analytical Technology) and chemometrics). He is a member of several scientific societies and has published over200 peer-reviewed papers and is the author of a widely used textbook in Multivariate Data Analysis (35,000 copies), (6.th edition 2018). He was chairman of the taskforce behind the world's first horizontal (matrix-independent) sampling standard DS 3077 (2013; 3rd rev. 2024). He founded the magazine TOS forum and the Sampling Column in Spectroscopy Europe/World, amalgamated into Sampling Science and Technology (2024), for all three steering matters as editor. In 2020 he published the textbook: Introduction to the Theory and Practice of Sampling. He received the Russian Chemometrics Society (RCS) Gold Medal (2012); the Pierre Gy Sampling Gold Medal (2013) and the IPGSA Distinguished Service Award (2024).

TOS forum

Starting 2024 *Sampling Science and Technology* (SST) is a direct continuation of *TOS forum*, which was published by IMPublications in the decade 2013-2023.

The complete archive can be found here:

sst-magazine.info/tos-forum



Advanced Continued Education (EDU)
The Complete TOS forum Archive

DOI: 10.62178/sst.002.009

The entire publication history of TOS Forum has been made public under the Sampling Science and Technology webpages. TOS Forum issues 1–11 contains a wealth of diverse opportunities for continued advanced education.

TOS Forum can be reached at sst-magazine.info/tos-forum. The following three articles are examples of the treasures to be found in the TOS forum archive:

"Critique of Gy's Sampling Theory": Misplaced expectations of Wikipedia's democratic intentions

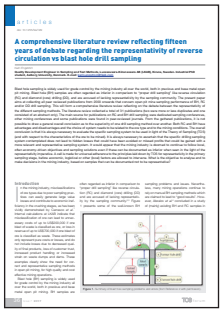
By Geoffrey J. Lyman and Kim H. Esbensen
doi.org/10.1255/tosf.11

In today's age of the internet and the cloud's many "blessings", Wikipedia is widely hailed as the pre-eminent internet source of readily available information. Wikipedia has especially been acclaimed for its apparent democratic attitude towards building a free, open encyclopaedia of the time. But there is also a darker side to all this enthusiasm—in that anybody can enter any new entry where none exists on a given topic, or edit any existing article. In fact, upon reflection, it dawns upon users that this democratic openness is not necessarily a blessing. Thus this institution has aptly been described by the following depressing characterisation: "Wikipedia is the medium in which your worst enemy can get to write your epitaph".

The Aloha Sampler™: concept, objective, design and implementation

By Charles Ramsey
doi.org/10.1255/tosf.25

The Aloha Sampler is an innovative new sampling tool to effectively collect and combine increments from dynamic, liquid, one-phase and two-phase systems. It is extremely inexpensive and very cost effective to implement and produces more representative samples than any other conventional techniques. TOS forum has asked EnviroStat to present the Aloha Sampler for its readers.



A comprehensive literature review reflecting fifteen years of debate regarding the representativity of reverse circulation vs blast hole drill sampling

By Karin Engström
doi.org/10.1255/tosf.99

Blast hole sampling is widely used for grade control by the mining industry all over the world, both in precious and base metal open pit mining. Blast hole (BH) samples are often regarded as inferior in comparison to "proper drill sampling" like reverse circulation (RC) and diamond (core) drilling (DD), and are accused of lacking representativity by the sampling community. The present paper aims at collecting all peer reviewed publications from 2000 onwards that concern open pit mine sampling performance of BH, RC and/or DD drill sampling.

Report to IPGSA on WCSB11

By Martin Lischka, Mariska Reyneke, Willem Slabbert, Letisha Smal, Sandra Ratsoma, Kim Esbensen, Sheryl Tittlemier, Chris Robben, Terance Nkosi and Richard Minnitt

DOI: 10.62178/sst.002.010

1. Overview of WCSB11: Diversity and Application of the Theory of Sampling

The Eleventh World Conference on Sampling and Blending (WCSB11) was held at Misty Hills Conference Centre, Muldersdrift in Johannesburg, South Africa from the 21-23 May 2024. This is the second time the conference took place in South Africa, the first being the WCSB4 in Cape Town, 2009. WCSB11 was attended by approximately 150 delegates from 18 different countries, each bringing insights and understandings of the way sampling in general and the Theory of Sampling (TOS) in particular affects our lives. In addition, there were approximately five on-line attendees who were unable to travel to South Africa, but who participated and made excellent on-line presentations of their research. The overall level of attendance was above average and may be considered a proxy for the measure of success of the Conference. In the three days of the Conference, fifty-four presentations were made by delegates, and five sponsors were given an opportunity to share their expertise with delegates. The theme of the conference "Diversity and Application of the Theory of Sampling" was to examine and explore the significant implications and inroads the Theory of Sampling has made into such diverse fields as economic, mineral, industrial, food and feed, agricultural, and pharmaceutical activity in which sampling is an important basis for making far reaching, important decisions.

Although many of the concepts affecting the accuracy and precision of particulate sampling were developed as early as in the period between the mid-1800s to the mid-1900s, it was Pierre Gy, a French chemist and engineer who began his work on sampling in 1949, who laid the foundation and formalised our understanding of sampling theory in full measure. What we now know and refer to as the Theory of Sampling was a work in progress between 1950 and 1975 and led to the 1967 publication in French entitled "L'Échantillonnage des minerais en vrac: Théorie générale" (Sampling of Particulate Materials: Theory and Practice) which was Gy's first comprehensive exposition of his theories. Gy's contributions were seminal in forming the theoretical framework for sampling of particulate materials in general, which includes the formulation of key principles and mathematical models to address the inherent variability and errors in sampling of heterogeneous materials and processes.

2. Theme and Scope of the Conference

The series of World Conferences on Sampling and Blending was initiated by WCSB1 in Esbjerg, Demark in 2003, and has subsequently been held in Brisbane, Australia (WCSB2 2005), Porto Alegre, Brazil (WCSB3 2007), Cape Town, South Africa (WCSB4 2009), Santiago, Chile (WCSB5 2011), Lima, Peru (WCSB6 2013), Bordeaux, France (WCSB7, 2015), Perth, Australia (WCSB8 2017), Beijing, China (WCSB9 2019), and Kristiansand, Norway (WCSB10 2022).

These World Conferences have become the cornerstone for fostering international collaboration amongst the sampling fraternity and interested professionals. The aim is to share knowledge about standardising practices within the scope of the Theory of Sampling, to minimize variability and uncertainty, and to enhance the reliability and accuracy of sampling methods. WCSB meetings provide a forum that bridges the gap between the Theory of Sampling and the Measurement Uncertainty, and thereby create a unifying foundation that will lead to the development of more universally accepted practices and standards.

Although TOS is a cornerstone for modern society's pursuit of sustainable processes and products, full acceptance and implementation across all the sectors of industry and society, it is still a work in progress. WCSB11 extended and amplified the efforts to reach the scientific and technological involvement of other industries besides mining and minerals, including applications in technology, industry, society, commerce, and trade. These areas include food, feed, agriculture, pharmaceutical production, with particular emphasis on Process Analytical Technologies (PAT), environment, and sustainability.

Additionally, the conference underscored the importance of representative sampling in quality management and with respect to the environment, as well as the optimization of natural and renewable resources while considering environmental impacts. WCSB11 specifically aimed to address the UN Sustainable Development Goals 9 and 12, focusing on sustainable industry, innovation, infrastructure, and responsible production and consumption.

3. Aims, Objectives, and Goals

The aims of the World Conferences on Sampling and Blending (WCSB) are principally to preserve, promote, and advance the Theory of Sampling by providing a platform for researchers and practitioners to collaborate, share knowledge, and develop standardized methodologies and best practices.

Continuance of the WCSB series will ensure that the principles of representative sampling, as laid out in TOS, are understood, regulate, and penetrate the worldwide practice of sampling. In general, the key aim is to improve the knowledge about heterogeneous materials, which arise in industrial and industrial contexts. The Conferences act as a knowledge exchange forum for the convergence of scientific inquiry and industrial practice, enabling the dissemination and exchange of insights and innovations in sampling and blending.

Interdisciplinary collaboration and exchange of knowledge is fostered in a collaborative environment where academia, manufacturers, engineering firms, and professional practitioners can interact and benefit from shared expertise and experience. The support and involvement of Original Equipment Manufacturers (OEM) responsible for promoting technological advancements and quality assurance in sampling practices highlight the need for this symbiotic relationship.

The technical program and high quality of papers and presentations form a strong, scientifically verified basis for the global sampling community to move towards analytical excellence, emphasizing the importance of both detecting and mitigating sampling errors. In this way a global appreciation and international recognition and application of TOS-based sampling practices is strengthened. Publication, availability, and accessibility of the proceedings in digital format promotes the wide dissemination and historical preservation of the knowledge shared at the conferences. Through these aims, the WCSB conferences strive to uphold and propagate the importance of sampling science and technology, ensuring its continued relevance and application across various industries and academic disciplines.

The WCSB11 was promoted and marketed by the SAIMM well in advance of the conference. Workshops and keynote speakers were promoted once they had been fixed in the program. An outstanding outreach was the "Crucible" podcast (https://iono.fm/e/1408876), which was hosted by the SAIMM, and is available on the SAIMM homepage and on Spotify. During the conference the SAIMM marketing team continuously posted content from the highlighting speakers and sponsors on LinkedIn. Even weeks after the conference, the posts still receive attention in this community. Access to the photos taken at the conference and at the evening events has been made available via the conference Dropbox (see below).

4. Organising Committee and Sponsors

Gratitude is extended to the dedicated members of the Organising Committee, including the Secretariat of SAIMM who acted as the host and organizing entities led by Camielah Jardine, Gugu Charlie, Patricia Takalimane, Nazli Mamdoo, and Sam Moolla. Members of the WCSB11 Committee included Mariska Reyneke, Willem Slabbert, Letisha Smal, Sandra Ratsoma, and Kim Esbensen, Sheryl Tittlemier, Chris Robben. The Chairpersons were Terance Nkosi and Richard Minnitt.

Sponsors and supporting organizations of WCSB11 included the following:

Headline Sponsor

- Rand Refinery – were the leading sponsor and sponsored all the awards and gifts and had a 10-minutes sponsor presentation slot at the conference. RR also had an exhibition stand.

Premium Exhibitors

- HERZOG Maschinenfabrik GmbH & Co.KG set up an exhibition stand and made a 10-minutes sponsor presentation slot (but they did not take up any of these benefits)
- Rocklabs set up an exhibition stand and made a 10-minutes sponsor presentation slot at the conference
- Multotec set up an exhibition stand and made a 10-minutes sponsor presentation slot at the conference

Corporate Sponsor

- Fl Smidth made a 10-minutes sponsor presentation slot at the conference

Exhibitors

- Block 10 set up an exhibition stand
- ITECA SOCADEI – set up an exhibition stand
- Qingdao Yosion Intelligent Technology set up an exhibition stand, and sponsored the delegate bags

Banner Sponsor

Measurement Process Solutions set up promotional banners displayed at the conference Qotho Minerals and displayed promotional banners at the conference

Figure 1: Thank you to all WCSB11 sponsors.

4.1 Scientific Committee and Reviewers

The conference's success is also attributed to the rigorous peer-review process conducted by a competent corps of reviewers:

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5. Conference Programme

Pre-conference workshops: Three workshops were offered:

- Introduction to the Theory and Practice of Sampling (TOS) in Science, Technology, Industry, Commerce and Society (Kim H. Esbensen);
- Sampling Theory, Sampling Practices and Their Economic Impact (Francis F. Pitard & Dominique François-Bongarçon);
- Sensor-Based Ore Sorting and Sampling (Christopher Robben)

The post-conference feedback points to broad satisfaction with this educational opportunity.

In a bold new step, the Organising Committee decided to select a range of younger and newer entrants to the sampling community as Keynote speakers and as Session Chairpersons, meant as an educational challenge for the younger cadres. The keynote speakers were: Ana-Carolina Chieregati, Rodolfo Romanach, Claudia Paoletti, Jean-Sebastien Dubé, Stephane Brochot. Oscar Dominguez Gonzales was also invited as a keynote speaker, but was unfortunately unable to present orally; however, his lecture is included in the conference Proceedings. See online proceedings for information on their presentations.

The scientific value and relevance of the accepted papers at the World Conference on Sampling and Blending (WCSB) are substantial, given the context and aims of the conference. The value and relevance of the papers contribute to the development and refinement of the Theory of Sampling (TOS), addressing both theoretical foundations and practical applications. New innovative methodologies and techniques that improve accuracy, efficiency, and reliability in various industrial and analytical contexts are presented. Interdisciplinary research findings and technological innovations with applications across multiple industries, from mining and pharmaceuticals to environmental studies and food safety, demonstrating the broad applicability of sampling techniques are now on record in the approved collection of papers. Industrial applications, enhanced decision-making, standardization and best practices, sustainability and environmental impact, the educational value, and the importance of global collaboration are emphasised. Overall, the accepted papers at the WCSB conference help setting high standards that benefit both academia and industry.

6. Papers and Proceedings

A call for the submission of papers for the WCSB11 was issued in July 2023 with a notice of acceptance being sent to authors on 6th October 2023. Papers accepted for the conference were subjected to peer review and the date for final paper submission was 29th January 2024. Reviewing criteria included scientific value and showcasing the current state of sampling and blending. The author deadline for revised papers was 12th February with final acceptance or rejection of the papers was issued on 19th February 2024. Papers were published as proceedings of WCSB11 in a convenient and readily available electronic format, which is distributed in a SAIMM Dropbox facility with a link provided to delegates 2-3 days before the conference.

The conference proceedings were professionally edited by Annette Thompson through SAIIMM and produced in a modern format, facilitating rapid submission, review, and publication. After each WCSB conference, the proceedings are the only tangible scientific evidence left for posterity.

SAIMM has given permission for the WCSB11 Proceedings to be available and downloadable as open access at both the SAIMM as well as the IPGSA webpages [https://www.saimm.co.za/Conferences/files/wcsb11-2024/WCSB11%20Proceedings%20Book.pdf; https://intsamp.org/], documenting the current state of sampling science and technology, furthering the opportunities for future developments and advancements. The IPGSA owes SAIMM a great debt of gratitude for this opportunity.

7. Awards and Recognitions

7.1 Pierre Gy Sampling Gold Medal:

Dr. Ana-Carolina Chieregati and Dr. Claudia Paoletti were awarded the Pierre Gy Sampling Gold Medal for: "Excellence in Teaching and Dissemination of the Theory of Sampling." (see separate "Award Justification" in this issue).

7.2 Distinguished Service Award

For the first time the award was presented by the IPGSA Council to Prof. Kim H. Esbensen (by kind courtesy of Rand Refinery donating a Gold Kruger Rand medal). Prof. Esbensen was awarded for his sustained and distinguished contributions to the organisational activities of the Association and its WCSBs over 20 years, including steadfast leadership of the committees involved behind "DS3077 Representative Sampling - Horizontal Standard", the world's only de facto international standard on this important topic (see separate "Award Justification" in this issue).

7.3 Young Authors Awards

Also, for the first time, four awards in the form of specialised commemorative coins by kind courtesy of Rand Refinery, were presented to Young Authors, to the organizing committee, and SAIMM secretariat team that facilitated for the success of the conference. The Young Author Awards are made for the most outstanding papers presented by young authors at WCSB conferences to encourage their participation. To qualify, the author must be less than 35 years of age at the date of the conference. These awards are made at the discretion of the Organising Committee.

- The Best Paper Award made to Killian Berelsmann of Herzog, Germany, for his outstanding paper entitled "Quality criteria in sample preparation – how to ensure full reproducibility and fully homogenization?" co-authored with Martin Lischka.
- The Best Presenter Award made to Hulisani Esra Madima of Multotec, for his presentation entitled "The case for using five-times particle nominal top-size cutter width for dry material primary increment sampling at 0.6 m/scutter speed" co-authored with Willem Slabbert.
- The Best Presenter Award made to Charles Tonongei, of Anglo-American Platinum, for his presentation entitled "Variographic analysis of a concentrator plant feed slurry stream data before and after replacement of the intermediate sampler hopper".
- The Best Presentation Award made to Debra Samuel of the Rand Refinery, for her paper entitled "Comparing sampling techniques for gold bullion to find the most effective sampling method".

Terance Nkosi and Richard Minnitt were awarded commemorative plaques by the IPGSA for their sustained efforts in the co-chairman ship of the WCSB11 Conference.

Figure 2: Terance Nkosi (left) and Richard Minnitt were awarded commemorative plaques by the IPGSA.

Figure 3: A 'random sample' from the Misty Hills venue auditorium – is it representative?

8. New Initiatives and Future Outlook

The next World Conference on Sampling and Blending WCSB12 will take place in Exeter, Cornwall UK in 2026, to be co-chaired by professors Hylke Glass and Simon Dominy. (NB. Bids for future conferences must be submitted to the IPGSA no later than one month before commencement of the next WCSB; bids must follow the guidelines published by the IPGSA Council).

The conference was given an extensive report on the current work aimed at concluding with the fully revised DS3077 (3rded.), to be published autumn 2024, and subsequently to be proposed as an ISO standard. This ISO process is likely to take a year or two, facilitated by participation by dedicated TOS-competent future members of the committee to be organised.

9. Special Panels and Discussions

A recurrent program element at WCSB conferences is a panel discussion, this time chaired by Dr. Ralph Holmes and Dr. Kim H. Esbensen. This was an open forum debate about teaching and training of TOS, inviting thoughts, ideas and experiences from conference delegates about training and education in the field of sampling. This point was initiated by a sponsoring OEM whose concern was the importance of the fundamental principles in the Theory of Sampling and the ways and means that such education can penetrate industry and society at large. The importance of establishing a single-source document on TOS, that can be made universally accessible, and on which the teaching and training of TOS can be stabilized and based, was emphasised. It was pointed out that this goal is actually in the midst of being realised through the sustained work behind the DS3077 national standard, which for 10 years has served as a de facto international standard supplying a first attempt at this objective.

10. Exhibition, Poster Sessions, and Networking

The exhibition featured 10 exhibitors, including five South African and five foreign exhibitors.

Participants engaged in fruitful discussions during coffee breaks, a wine tasting event, and poster sessions. Exhibitors from Rand Refinery, Iteca Socadei, Kingyosion, Block 10, Multotec, SAIMM, and Rocklabs showcased the latest in representative sampling, sample preparation and analysis, while poster sessions covered a wide range of topics in industry and academe. Networking opportunities: Coffee breaks and poster presentations facilitated networking among delegates, and many participants shared positive feedback, highlighting the conference as a prime opportunity to interact with the growing community of diverse-industry sampling professionals and the excellent organisation of both in-person and virtual components.

11. A sample of highlights

Figure 4: Ulrik Thisted presenting at WCSB11 from a very remote site in Norway, Elke and Ulrik Thisted's IT-equipped summer cabin – What's not to like with hybrid conferences?

Figure 5: Newest TOS R&D enthusiast from beyond traditional application fields: Jean-Sebastien Dubé (Canada) on the left and to the right steadfast industrial process expert from Pharma, Rodolfo Romanach (Puerto Rico).

12. Post-Conference Tours

Delegates visited Rand Refinery the headline sponsors of the Conference, and the OEM Multotec, a Premium Exhibitor at the Conference.

The technical visit to Rand Refinery, world-leading precious metals refinery "just around the corner", was commented on by participants who commended the company and hosting team, who, even though the Rand Refinery at the time of this visit was in the process of stock accounting, dedicated valuable time to ensure the visitors had an enriching experience. "From the moment of arrival, the team welcomed us warmly, providing a delightful breakfast, lunch, and thoughtful gifts, ensuring our comfort throughout the visit.

Figure 6: One of the pre-conference courses: "Sensor-Based Ore Sorting and Sampling" was conducted by Dr. Christoffer Robben (right) for a small, very enthusiastic group of participants.

The tour was informative and well- organized, covering key stages of the refining process:

- Receiving: How raw materials and deposits are received and logged.
- Sampling: Detailed explanation of the many stages of the Rand's unified sampling process.
- Preparation: Steps for sub-sampling and preparation of materials for analysis.
- Analysis and Research: Insight into analysis techniques and ongoing research. The Rand Refinery is a LBMA reference laboratory.

Our knowledgeable guides shared numerous interesting factoids, making the tour both educational and engaging. The visit to Rand Refinery was highly insightful, offering a rare look at its complex operations. The hosting team's dedication and hospitality made the experience memorable and valuable, deepening understanding and respect for the precious metals industry".

The theme for the technical visit to Multotec Process Equipment was: "Where the Theory of Sampling comes to life". The visit was hosted around an in-depth tour of Multotec's sampler workshop.

The in-person event allowed participants to evaluate, touch, see and interact with live sampling equipment and the detailed features refined into these machines to allow metal accounting precision sampling. The range of wet and dry sampling equipment on display included: linear launder samplers, radial vezin samplers, slurry handling hoppers, cross stream belt end cross-cut samplers, cross belt hammer samplers, control panels and ancillary sample storage equipment.

The attendees themselves were inspired by the theme and were enthusiastic, engaging and full of eager questions – even after attending a rewarding 3-day sampling conference packed with knowledge!

One of the attendees posted on LinkedIn: "A big thank you to the Multotec team for hosting us in a technical visit at the end of the World Conference on Sampling and Blending 11 at their Spartan facilities near the Johannesburg airport, and for sponsoring the conference. I enjoyed learning about Multotec's multiple products to support the mining industry, and the important role that the Theory of Sampling plays in day-to-day operations."

13. Conclusion and Future Conferences

In summary, the achievement of the World Conferences on Sampling and Blending (WCSB) has significantly contributed to the advancement and dissemination of the Theory of Sampling (TOS). TOS addresses the complexities of particulate sampling and its inherent errors, providing systematic methods to minimize sampling uncertainties.

The conferences serve as a vital platform for researchers, academics, and industry professionals to exchange knowledge and improve standards in sampling theory and practice. The conferences have facilitated truly global teaching and understanding of TOS, leading to its inclusion in postgraduate courses across various countries including the US, Denmark, Brazil, Mexico, South Africa, and Australia. The WCSB series have fostered dialogue between proponents of TOS and total Measurement Uncertainty (MUtotal), emphasizing their complementary nature.

TOS effectively identifies and minimizes the effect of sampling errors, while MU primarily identifies and reduces analytical variance. The conferences have invigorated research and development in TOS, ensuring continuous improvement and innovation in sampling methodologies.

This has been particularly crucial in maintaining the relevance and application of TOS in various industries. Forward-leaning Original Equipment Manufacturers (OEMs) have benefited from the theoretical and practical insights provided at WCSB, leading to improved sampling equipment design that adheres to TOS principles.

This collaboration has resulted in generous sponsorship from OEMs for the conferences. The conferences honour significant contributors to TOS with the Pierre Gy Sampling Gold Medal.

Since the first conference in 2003, held biennially (with a delay in 2021 due to the Covid-19 pandemic), WCSBs have played a pivotal role in advancing the theory and correct application of sampling, ensuring it remains a vital scientific discipline in both academic and industrial contexts.

There is every intention to continue on this global path, also laying the foundation for a current crop of complementary regional sampling conferences in South America, Australia and South Africa. This significantly increased activity is a tribute to the highly successful role and achievement of the eleven WCSB conferences to date.

ACKNOWLEDGEMENTS

Technical Support: A special thank you to the managers of the IT aspects, who impeccably ensured seamless high-quality integration for online participants.
Venue: Appreciation goes to the venue Misty Hills, which handled all logistical challenges efficiently, providing a conducive environment for the conference.

Post-conference Resources

- Master-link to Conference presentations, proceedings, and photos available at: https://www.dropbox.com/scl/fo/olv2w12xue84yzy8uy4c4/AONrHivlaEubxE_kz4QdSKE?rlkey=hojx73q356kotbuds9x6m45gf&e=2&st=mart7fyv&dl=0
- Link to a "Crucible" podcast: https://iono.fm/e/1408876

Pierre Gy Sampling Gold Medal 2024 Award Justification

DOI: 10.62178/sst.002.011

The "History of the Pierre Gy Sampling Gold Medal 2003–2015" is described in [1].

The committee has deliberated extensively on the merits for candidacy for the PGSGM 2024. In a situation in which all 'natural candidates' for being awarded the PGSGM for "Excellence in teaching and application of the Theory of Sampling" having all been so honored in the period 2003-2022, the opportunity had (finally) arrived to 'catch up' and reach across a significant age gap in the PGSGM candidate pool at WCSB10 – It is also pertinent to address other representation issues if-and-only-if in compliance with the main scientific merit. A stray comment was heard from a committee member (undisclosed): "These two women also deserve gratitude and admiration for their professional achievements in a world often dominated by aggressive men".

Considering this, the committee has decided on two worthy candidates as recipients of the PGSGM 2024 Medal to be awarded at WCSB11, May 21-23 2024, Gauteng, South Africa:

- Ana Carolina Chieregati
- Claudia Paoletti

The committee argues:

In solidum. Both Ana Carolina and Claudia have been ardent participants and supporters at nearly all World Conferences on Sampling and Blending. Additionally, they have extended considerable efforts to contribute to advancing the TOS and applications hereof - in two distinctly different TOS application arenas.

Table 1: The legacy of the PGSGM.

WSCB	Conference location	Recipient of the PGSGM
WCSB1 (2003)	Esbjerg	**A. G. 'le Bon' Royle** (1924-2013) - *awarded 2010*
WCSB2 (2005)	Brisbane	**Pentti O. Minkkinen**
WCSB3-4 (2009)	Cape Town	**Francis Pitard, Dominique François-Bongarçon**
WCSB5 (2011)	Santiago de Chile	**Pedro Carrasco** (1950-2011) - *awarded posthumously*
WCSB6 (2013)	Lima	**Kim H. Esbensen**
WCSB7 (2015)	Bordeaux	**Ralph Holmes**
WCSB8 (2017)	Perth	**Richard Minnitt**
WCSB9 (2019)	Beijing	**Geoffry Lyman**
WCSB10 (2022)	Kristiansand	**Simon Dominy**

Ana Carolina Chieregati

Ana Carolina is a Mining Engineer from University of São Paulo, has a master and a PhD degree in Mineral Engineering from University of São Paulo, and has a geoscience post-doctorate from the University of Aalborg (Denmark). Since 2002 she has been a lecturer and professor at the Department of Mining and Petroleum Engineering of University of São Paulo, teaching Mineral Exploration, Mine Reconciliation, Quality Assurance/Quality Control - and the Theory and Practice of Sampling. With 20 years of experience in sampling and reconciliation, she taught in South America and Australia, published several technical papers and book chapters, and participated in many mining projects in Brazil, Argentina, Chile, Honduras, New Caledonia, and Mongolia, most of them related to the optimization of sampling equipment and procedures in gold, zinc, copper, nickel, niobium, iron, phosphate, and bauxite mines.

Since her first presence at WCSB3, it has been a pleasure to witness Ana's enthusiasm in developing a deep understanding of the Theory of Sampling through WCSB conferences and interaction with eminent sampling experts around the world. As a professor, Ana is highly respected for her professional and personal care for her students, whom she regularly encourages to present contributions at scientific conferences etc. not the least at WCSB. She continues to prepare many students in the field of TOS for Masters and PhDs, supporting them with great vigor, while deliberately co-publishing with many of them. She is teaching TOS extensively, including outside academe and passes her critical knowledge and enthusiasm on to everyone she meets. This significantly reduces the age profile of those with a good knowledge and appreciation of correct sampling theory and practice.

In addition to her teaching, she has been a consistent contributor to research and development in the field of particulate material sampling, especially regarding empirical studies of heterogeneity, in which arena she has published extensively with a noticeable impact. She has taught TOS courses widely both at the University of São Paolo and elsewhere in South America. She has published ~15 focused sampling papers over the last 15 years or so, all of which provide great practical advice to the sampler.

In her mining rich home country Brazil, she has been instrumental in promoting TOS and has developed a strong profile at the mining scene all over South America. As a leader of practical projects in collaboration with Brazilian mining industries, she is much revered.

Ana Carolina is the right person to be awarded the PGSGM at this time of her career, representing the younger generations of scientists and technologists who have eagerly advanced the applications of TOS both in theory and in practice. Her efforts are massive and deserve this ultimate reward because she creates new „troops" for us - using Pierre Gy's own words. Ana Carolina made a deliberate point to visit Pierre Gy during his last days (when at WCSB7); He was well aware of her efforts and very happy for the visit. As it turned out, Ana Carolina was the last of our community to be with him at that occasion, as is beautifully recorded in TOS Forum, Issue 6.

Figure 1: Ana Carolina Chieregati was the last of the sampling community to meet with Pierre in Bordeaux, the day after WCSB7, 2015.

Claudia Paoletti

Claudia Paoletti did her Master in Biological Science at the University of Rome (Italy) and her PhD in Plant Genetics at the University of Connecticut, USA. She was for three years at Dalhousie University (Canada) studying plant population genetics and biometry. She continued her activity at the Research Institute for Industrial Crops in Bologna (Italy) where she focused on the evaluation of the risks of transgenic crops. In January 2006 she joined the GMO Unit of the European Food Safety Authority (EFSA) first as Team Leader and then as Deputy Head of the Unit. In 2019 she was appointed manager of the programme designed to reorganise the EFSA in preparation for the new European Law on food safety. She has been the Italian expert for the definition of the European Commission sampling plans for GMO detection in conventional seeds. She coordinated the European sampling research project KeLDA and she has been the biometric officer of the EU Community Reference Laboratory for GMOs. She is an expert consultant for ISO/IWA committees, OECD, CEN, the European Commission and FAO. She organised international training courses on food/feed safety for the European Commission, UNIDO, PHARE project and universities within and outside Europe. She has over 90 contributions either as book chapters, or as peer-reviewed papers.

Claudia Paoletti arrived on the sampling scene with a BANG at WCSB2, Brisbane.

The committee is impressed by the enthusiasm with which Claudia has since embraced the objectives of the International Pierre Gy Sampling Association (IPGSA) and the application of TOS in the sampling of food, feed, and industrial agriculture: plants, seeds, fruits, nuts, grain. For 20 years she has played a key role in moving the sampling community forward, enhancing its profile and scientific integrity, and has played a pivotal role in ensuring that significant external impact is achieved through various International Pierre Gy Sampling association (IPGSA) working groups.

She has promoted TOS to many other important organizations in Europe and globally, WHO, ISO, OECD, CEN, FAO and FAO. She has organised international training courses on food/ feed safety for the European Commission, UNIDO, PHARE project and at universities within and outside Europe.

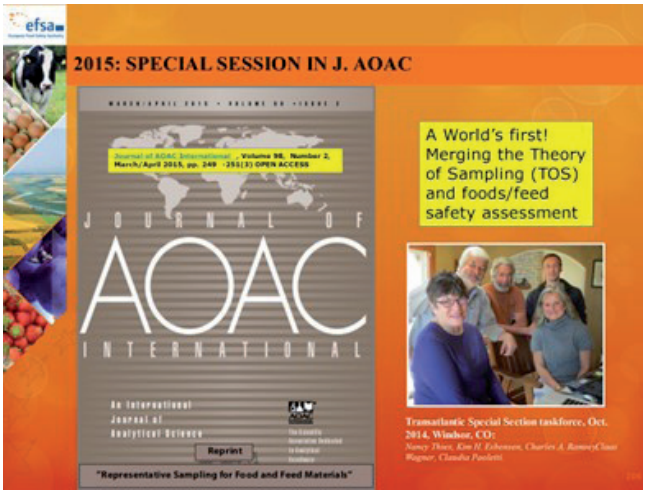
Claudia has been a most effective influencer within EU (EFSA) in showing the importance of TOS, an effort which is far from visible to the general public, but all the more important for "Food Safety". Her groundbreaking study on sampling of soybeans for GMO in cargo ships arriving at EU ports, masterly showed that the first adopted traditional sampling plans for grain in general were based on wrong assumptions, which lead to dramatically wrong conclusions, a.o. leading to official standards and guiding documents giving faulty confidence in the official quality control in current in use. This is known as the "KeLDA" study, which is highly respected in many contexts.

She has published over 90 contributions either as book chapters, or as peer-reviewed papers, two of which are brought to attention here.

Figure 2: Claudia Paoletti to Hans S. Møller (WCSB2, 2005): "It is so difficult to sample those 'sheeps' the right way" – sigh!

Figure 3: Pierre Gy Sampling Gold Medal Committee, augmented the two with freshly minted WCSB11 medallists (center).

1) Claudia played a prominent role in the fruitful trans-Atlantic collaboration behind the seminal publication "Representative Sampling for Food and Feed Materials" (Jour. AOAC International (2015), a world's first curated compendium of 15 contributions towards 'TOS for food/feed Materials'. This publication is in fact a mini TOS textbook.

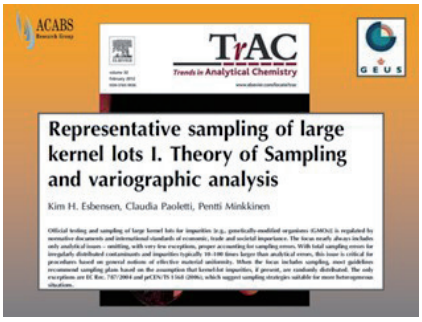


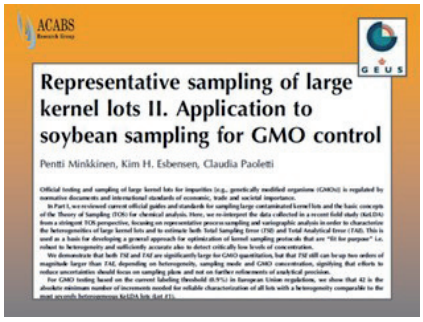
2) An important follow-up to the KeLDA study showed how TOS can be brought to bear on the issue of sampling for GMO components in ship cargoes of industrial feed soybeans. This study also introduced advanced variographics to elucidate several subtle sampling optimization issues.

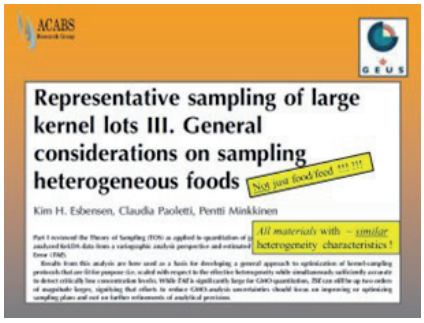
Claudia Paoletti has of late taken on a greater level of organizational responsibility within the IPGSA. She is a very powerful diplomat in the service of our cause. Her tireless work is a manifest reflection of the mandate for the PGSGM: "Excellence in teaching and application of the Theory of Sampling", for which she deserves this ultimate reward with all accolades.

Pierre Gy Sampling Gold Medal ab 2024

After the 2024 awardees were welcomed into the PGSGM committee, our community will appreciate a significant lowering of the average member age and a much-needed gender gap reduction – all undoubtedly for the greater good of IPGSA.







Presentation of IPGSA Distinguished Service Award to Prof. Kim H. Esbensen

By Ralph Holmes

DOI: 10.62178/sst.002.012

The Constitution of the International Pierre Gy Sampling Association (IPGSA) specifies that the IPGSA Council may present a "Distinguished Service Award" to persons who over a sustained period have made distinguished and noteworthy contributions to the organisational activities of the Association and/or its conferences. Recipients of this award may be nominated by any member of the IPGSA Council or International Advisory Committee prior to each World Conference on Sampling and Blending (WCSB).

For the first time since its establishment in 2017, the Council received a compelling nomination for this award on the occasion of the 11th World Conference on Sampling and Blending (WCSB11) held in Johannesburg, South Africa, in May 2024 - and after a very brief discussion the Council agreed that the inaugural Distinguished Service Award should be presented to none other than Dr. Kim H. Esbensen.

As the initiator of the WCSB conference series, the Council could not think of a more worthy recipient of the inaugural award. Kim was the creator of the WCSB concept and organised the very first conference (WCSB1) in Esbjerg, Denmark in 2003, the key aims being to bring together practitioners, experts and academics from all over the world involved in sampling, share their expertise, and promote the Theory of Sampling (TOS) developed by Dr Pierre Gy who was the guest of honour at WCSB1. This was a truly visionary development by Kim and initiated the entire WCSB conference series. Since then, WCSB conferences have been held around the world in Australia, Brazil, South Africa, Chile, Peru, France, China and Norway (next up is Cornwall, UK). Kim played a key role in all these conferences, including the establishment of the International Pierre Gy Sampling Association a.o. to assess bids and coordinate the allocation of WCSB conferences. He has also played a pivotal role in the publication of WCSB Proceedings, and since 2013 was editor of TOS forum and the Spectroscopy Europe/World "Sampling Column" that in 2024 evolved into the aggregated journal "Sampling Science and Technology" (SST).

Figure 1: Dr. Kim H. Esbensen with IPGSA's Distinguished Service Medal.

Kim H. Esbensen was well qualified to undertake these tasks: He has been Research Professor in Geoscience Data Analysis and Sampling at GEUS, the National Geological Surveys of Denmark and Greenland (2010-2015), Chemometrics and Sampling Professor at Aalborg University, Denmark (2001-2015), and a Professor (Process Analytical Technologies) at Telemark Institute of Technology, Norway (1990-2000 and 2010-2015). In 2015 he moved on from his 35-year academic career to undertake a new role as a consultant and independent researcher. But this did not terminate his love for teaching, as he regularly takes on international roles as a visiting, guest and affiliate professor. As a geologist/geochemist/metallurgist/data analyst by training, he first worked for more than 20 years at the forefront of chemometrics.

However, since 2000 he has devoted most of his R&D efforts to representative sampling of heterogeneous materials, processes and systems, in particular to the Theory of Sampling (TOS), PAT (Process Analytical Technology) and chemometrics. He is a member of several scientific societies, has published over 250 peer-reviewed papers, and is the author of a widely used textbook in Multivariate Data Analysis, the 6th edition of which was published in 2018. For 15+ years, he has been the driver behind the world's first horisontal (matrix-independent) sampling standard DS3077 (2014), the revised 3rd edition launched 2024, which is currently being progressed towards becoming an ISO Standard.

Congratulations Kim on receiving the inaugural IPGSA Distinguished Service Award.

This award complements the Pierre Gy Sampling Gold Medal that he received from the International Pierre Gy Sampling Association in 2013 at WCSB6, which is awarded to individuals who have made *significant contributions to teaching and dissemination of the theory and practice of sampling*.

WCSB12 – Sampling for a Sustainable World

By Simon Dominy and Hylke Glass

DOI: 10.62178/sst.002.013

The IPGSA is pleased to announce that the 12th World Conference on Sampling and Blending (WCSB12) will take place between Monday 29th June and Friday 3rd July 2026 in Cornwall UK, hosted by the Camborne School of Mines, University of Exeter.

WCSB12

29th June to 3rd July 2026
Camborne School of Mines, University of Exeter, Cornwall, UK.

WCSB12 aims to bring together the diverse international sampling community to present and debate concepts and ideas for a standardised approach to sampling embodied in the Theory of Sampling (TOS).

The opportunity to meet, exchange ideas, and share practical experiences will be a significant benefit for attendees. The Conference will provide understanding and insights for practitioners, academics, manufacturers and engineering firms aiming to achieve representative sampling through TOS. Topics around societal, industrial, and environmental aspects of particulate sampling in mining/minerals, metals, cement, food and feed, agriculture, aquaculture, and pharmaceuticals will be addressed. Sampling for environmental contamination studies and sustainability are also included.

We look forward to welcoming you to Cornwall in 2026.

Conference Chairs: Professor Hylke Glass and Dr Simon Dominy.

Contributors

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Ana Carolina is a Mining Engineer from University of São Paulo, has a master and a PhD degree in Mineral Engineering from University of São Paulo, and has a geoscience post-doctorate from the University of Aalborg (Denmark). Since 2002 she has been a lecturer and professor at the Department of Mining and Petroleum Engineering of University of São Paulo, teaching Mineral Exploration, Mine Reconciliation, Quality Assurance/Quality Control - and the Theory and Practice of Sampling. With 20 years of experience in sampling and reconciliation, she taught in South America and Australia, published several technical papers and book chapters, and participated in many mining projects in Brazil, Argentina, Chile, Honduras, New Caledonia, and Mongolia, most of them related to the optimization of sampling equipment and procedures in gold, zinc, copper, nickel, niobium, iron, phosphate, and bauxite mines.

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With an electrical and automation background, Ross has spent close to 20 years working with leading sample prep and laboratory OEM's across the globe, where his passion for innovation and technology has continued to grow. Ross also enjoys being a dad and husband in his spare time.

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Dr Simon Dominy is a mining geologist-engineer with over 25 years' experience based in operations, consulting and academia. He has a background in mine operations and technical/leadership roles, with multi-commodity and continent experience. He has worked across the mine value chain from project studies, through to mine reopening/development and operations. Simon is an acknowledged expert in the evaluation and exploitation of coarse gold-bearing high-nugget effect deposits. He has designed and managed numerous studies relating to geometallurgy; resource development; sampling protocol optimisation; bulk sampling programmes; resource/reserve estimation; and grade control. He has authored numerous technical reports (JORC 2012 and NI 43-101), and peer reviewed journal and conference papers. He is a Visiting Associate Professor at the Camborne School of Mines, University of Exeter, UK, and holds technical/advisory positions with Novo Resources Corp., Artemis Resources Ltd, Puma Exploration Inc., and OCX Gold Group. In 2022, Simon was awarded the Pierre Gy Sampling Gold Medal by the IPGSA.

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Esbensen, Kim H.

Dr Kim H. Esbensen has been research professor in Geoscience Data Analysis and Sampling at GEUS, the National Geological Surveys of Denmark and Greenland (2010–2015), chemometrics and sampling professor at Aalborg University, Denmark (2001–2015), professor (Process Analytical Technologies) at Telemark Institute of Technology, Norway (1990–2000 and 2010–2015). From 2015 he phased out a 35 year academic career for a new quest as consultant and independent researcher. But as he could not terminate his love for teaching, he is regularly active as an international visiting, guest and affiliate professor. A geologist/geochemist/metallurgist/data analyst of training, he has been working 20+ years in the forefront of chemometrics, but since 2000 has devoted most of his R&D to the theme of representative sampling of heterogeneous materials, processes and systems: Theory of Sampling (TOS), PAT (Process Analytical Technology) and chemometrics. He is a member of several scientific societies and has published over 250 peer-reviewed papers and is the author of a widely used textbook in Multivariate Data Analysis, which was published in its 6th edition in 2018. He was chairperson of the taskforce behind the world's first horizontal (matrix-independent) sampling standard DS3077 (2013), 3.rd.ed. soon to be inducted as an ISO standard. In 2020 he published the foundational „Introduction to the Theory and Practice of Sampling". Since 2013, he was editor of TOS forum and Spectroscopy Europe/World „Sampling Column", from 2024 amalgamated and metamorphosed into „Sampling Science and Technology (SST).

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François-Bongarçon, Dominique

Dominique François-Bongarçon graduated as a Mining Engineer and holds a Doctorate in Mining Sciences and Techniques at the Geostatistics Center from the Paris School of Mines (Paris Tech). He has more than 40 years of experience in the mining industry and works as a consultant in earth sciences for his own company, Agoratek International Consultants Inc., based in Canada. In 1992 he embarked on a career-long research in Gy's theory of sampling, and he worked with Pierre Gy as a consultant and on training courses. He contributed to the onset of the WCSB cycle of conferences (2003). In 2009, he was the recipient of the Pierre Gy Sampling Gold Medal. In recent times, he has been continuing his research in Sampling Theory, in the techniques and spirit of the QA-QC discipline and on mine-mill reconciliations. He is also making new advances in the handling of extreme grades in Geostatistics.

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Glass, Hylke

Hylke Glass is Professor at the Camborne School of Mines (CSM), a department of the University of Exeter, since 2001. He was originally introduced to sampling theory by Theo Zegers at the Delft University of Technology in 1994. Togetherthey investigated the quantification of the sampling variance and the effects of grade varying across particle sizes, degree of liberation, the particle size distribution itself, moisture content, and occurrence of very low grades. This led to aa number of publications, including a presentation at the Surface Mining 1996 conference. Following Theo's retirement in 1997, Hylke took over the teaching of sampling of particulate materials and, from 1999, worked with Bas Geelhoed on creating understanding about fundamental aspects of Pierre Gy's Theoty of Sampling (TOS). A series of papers were published in journals including Geostandards Newsletter and Statistica Neerlandica. He continues to take an active interest in the development of sampling theory, its application in resource estimation and control of mineral processing, as well as raising awareness of sampling in the mining, engineering and minerals processing courses taught at CSM.

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Holmes, Ralph

Ralph Holmes obtained a BSc degree in Physics from the University of Melbourne in 1967 and a PhD degree from the same university in 1972. Over the last 35 years Ralph has been involved largely in mineral processing research and managed CSIRO's iron ore processing research for more than 20 years. He is currently an Honorary Fellow with CSIRO Mineral Resources following his retirement from CSIRO and is recognised internationally as an expert in iron ore processing and sampling mineral commodities. He is an Honorary Fellow of the Australasian Institute of Mining and Metallurgy (Chartered Professional – Metallurgy), President of the International Mineral Processing Council (IMPC) and President of the International Pierre Gy Sampling Association. Ralph received a Pierre Gy Gold Medal in Bordeaux, France, in June 2015 for "Excellence in Teaching and Application of the Theory of Sampling" and in October 2015 received a CSIRO "Lifetime Achievement Award" for sustained and meritorious achievements over a CSIRO career spanning more than 43 years in the field of mineral processing and international standards development both as a research manager and practitioner benefitting both CSIRO and Australia.

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A manufacturing and mechatronics engineer with a passion for creativity and problem solving. Outside of work Harry enjoys music, basketball, and freestyle skiing.

Minnitt, Richard

Dick Minnitt completed a MSc in geology in the Murchison Range and a PhD in the Richtersveld regions of southern Namibia. He joined Anglo American and later JCI, after which he spent 14 years doing contract and consulting work. He completed a second MSc in mining, and joined the School of Mining Engineering at WITS in 1995, where he taught courses in Mineral Economics and Geostatistics. His interest in sampling of particulate materials arose from the numerous visiting lecturers he invited to Wits University including Dominique Francois Bongarçon, Francis Pitard, Geoff Lyman and Kim Esbensen. Dick retired from Wits in 2017, but continues to consult for international mining companies and research in his fields of interest. He now holds a position as a Visiting Emeritus professor where he continues to teach postgraduate classes and supervises masters and doctoral students.

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Dr Francis F. Pitard is a consulting expert in Sampling, Statistical Process Control (SPC) and Total Quality Management (TQM). He is President of Francis Pitard Sampling Consultants in Broomfield, Colorado, USA. Dr Pitard has six years of experience with the French Atomic Energy Commission and fifteen years with Amax Extractive R&D. He teaches Sampling Theory for the Continuing Education Offices of the Colorado School of Mines. He has a Doctorate in Technologies from Aalborg University in Denmark. He is the author of Theory of Sampling and Sampling Practice (Third Edition 2019). He is the recipient of the prestigious Pierre Gy's Gold Medal for excellence in promoting and teaching the Theory of Sampling.

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Rawle, Alan F.

Alan Rawle has had around almost 50 years' experience in various aspects of science and technology. Alan has a degree in industrial chemistry and a Ph.D in supported alloy catalysts both acquired at Brunel University, London, UK. Since 1990, Alan has been with Malvern Instruments as the Applications Manager based in Westborough, MA, USA since 2003. He is still is working part-time with Malvern Panalytical.
Dr. Rawle was (2005 – 2022) CoChair of E 56.02, the Characterization SubCommittee of the ASTM E56 Committee on Nanotechnology. He was the Technical Author (i.e. writer) for ASTM standards in particle size, zeta potential, size distribution calculation among others. Dr. Rawle is also a Fellow of the Royal Society of Chemistry (FRSC), a Distinguished Fellow of the International Engineering and Technology Institute (DFIETI), and a regular contributor to ResearchGate.

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Russell, Steven

Steven Russell has been designing, developing and implementing automation projects for the mining industry across a two-decade career. He has applied his background as a mechatronics engineer to innovative projects that deliver productivity, safety and quality outcomes to many of the world's top miners. Over the last decade, Steve has led teams and developed growth strategies in executive roles as Mining Director at Scott Technology / Rocklabs and Head of Sales at Southern Innovation. Since early 2022, Steve has been establishing and growing his own team at Block10, with a continuing focus on sensing, automation and mechanical applications in the mining industries and sample laboratories.

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How to contribute

Sampling Science and Technology (SST) serves as a collaborative platform fostering scientific and technological engagement within the global sampling community. Our primary objective is to have a significant educational impact, catering to various levels of interest.

SST embraces didactic studies, practical insights, illustrative case histories, and occasional theoretical articles tailored for the sampling community in both strict and broad senses. Your valuable contributions play a pivotal role in our mission to cultivate professional sampling competence across diverse societal sectors where sampling holds significance — spanning science, technology, industry, trade, food/feed, public health, and more.

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IPGSA
INTERNATIONAL
PIERRE GY
SAMPLING ASSOCIATION

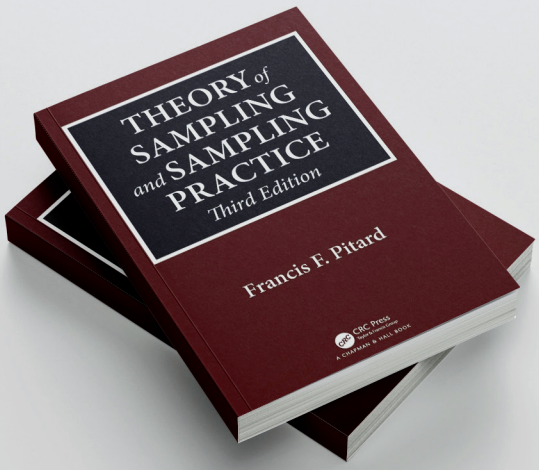
Introduction to the Theory and Practice of Sampling

by K.H. Esbensen

SIX Publishing (2024)

Price: € 85.00

six-publishing.com/tos-introduction



Theory of Sampling and Sampling Practice, 3rd Ed.

by F.F. Pitard

CRC Press, Taylor & Francis Group (2019)

ISBN: 978-1-138-47648-6
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Theory and Practice of Particulate Sampling: an Engineering Approach

by Geoffrey J. Lyman

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