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TOS forum

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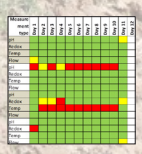

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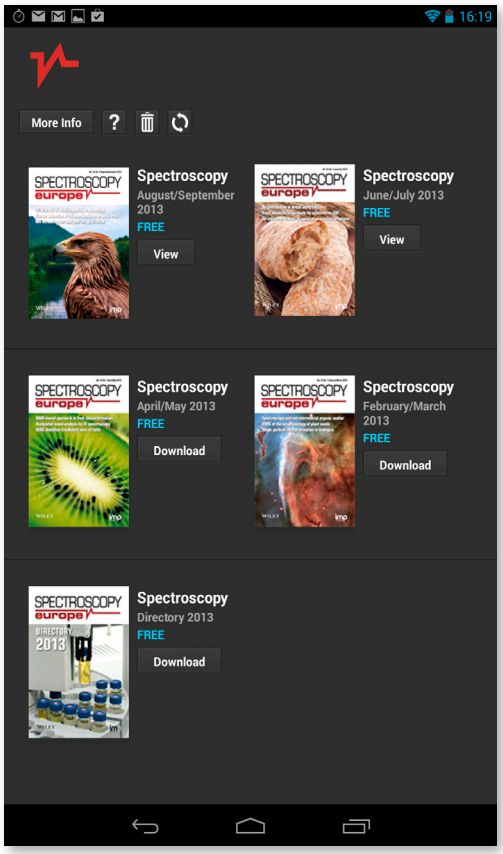

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From sorrow to moving on… with renewed spirit

Greetings to all members of the TOS community—and indeed *beyond,* to the degree that our efforts to reach wider audiences are successful. This issue of *TOS Forum* follows the publication of the memorial issue dedicated to Pierre Gy, *TOS Forum* no. 6. We have all experienced sorrow and reflection on the monumental life and *oeuvre* of Pierre Gy—but we have all also hopefully started to move on. This theme is appropriately reflected in the present issue.

This issue contains a veritable *pot-pourri* of features. You will find a major feature designed as a didactic overview of Gy's seminal work on *chronostatistics* and *variographics*, putting in perspective his entire 66-year scientific career. There are reports and descriptions of ongoing projects and activities of the most varied kind, spanning geographically from northern Sweden and southern Norway to Nicaragua, and content-wise ranging from a literature survey of the last 15 years on the quite specific topic "BH vs RC—which is optimal?" via one of the more spectacular comprehensive composite sampling processes ever encountered (2000 increments, no less) and carried out entirely by hand, to particularly revealing experiences with implementing Six Sigma in the industrial complex, as complemented by variographic screening. On top of all this, the present issue starts with a real eye-opener, a draft proposal for a "Constitution of the International Pierre Gy Sampling Association (IPGSA)". Lastly, but in no way least, there is also a timely presentation of the registration brochure for the upcoming 8th World Conference on Sampling and Blending, WCSB8. Indeed something for everybody…

Since the first organised global event (WCSB1, August 2003), our sampling community has endeavoured to move from one WCSB to the next seemingly with little trouble, organising and conducting a remarkable series of successful conferences. This is in the main due to the willingness of a succession of chairmen and their invaluable committees. However, how long can this continue? From an outside point-of-view this evolution may have looked smooth and easy, but appearances, as we know, can be deceptive. The sacrifices, toils and sweat behind *any* of our conferences are truly staggering. There is in reality no natural guarantee that a new willing chairman will always show up at just the right moment. And quite apart from this critical personal aspect, how do we secure a reasonable *spread* of locations around the world for "the next WCSB"?

Truth be told, from *behind* the lines, there is a danger that the world conference after next (WCSB9, 2019) may not necessarily have a place to go, and will not necessarily have a willing *chairperson*… (we need a female chairperson soon).

There is also another issue that has been tended to by an *ad hoc* and quite unofficial committee: the task of selecting the next recipient of the Pierre Gy Sampling Gold Medal (PGSGM). This has been in the hands of a non-elected committee consisting of all former award recipients, *only*. However, it is felt in some circles, present Editor included, that this is not sustainable in the long run, plainly because of the *principal* risk of scientific nepotism. I am *not* saying there has been *any* of that, or that there necessarily will be any of that in the foreseeable future, considering the upstanding gentlemen in this select group at present. But from a strict organisational point of view, it would be best to bring this very important committee under some form of elected system. At the very least to open it up to other members than only those who already carry a PGSGM around the neck.

Both the above issues have impacted on the draft proposal you will find as the opening feature in *TOS Forum* no. 7: a *proposal* for a constitution for **The International Pierre Gy Sampling Association**. The

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Barefoot sampling in Nicaragua. See page 30 for the full story.

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International Pierre Gy Sampling Association (IPGSA)

Kim H. Esbensen and Ralph Holmes

There has been discussion for several years whether the informal community attending the series of WCSB conferences would benefit from the establishment of a formal organisational body representing the interests of the international sampling community. An *ad hoc*, pro tem committee, Kim H. Esbensen and Ralph Holmes, has taken it upon itself to test this proposal. In order to kick off the possible establishment of an International Pierre Gy Sampling Association (IPGSA) in conjunction with WCSB8 in Perth in June 2017, the following procedure is proposed. An attempt has been made to keep the organisation of IPGSA as simple as possible.

A proposal for the Constitution of the International Pierre Gy Sampling Association is published here. The Editor will make every effort to reach the largest possible percentage of the international sampling community as well as relevant institutions and organisations. In order to facilitate an appropriate discussion at an inaugural assembly proposed to take place at WCSB8, responses, comments and suggestions on the proposed Constitution are invited. These must be received by the pro tem committee before 5 May 2017. The following e-mail address should be used: khe.consulting@gmail.com. All comments and suggestions will be made available to all WCSB8 delegates at the opening of the conference (or will be forwarded before the conference via e-mail, if deemed necessary).

The outcome of the inaugural assembly may be either the establishment of an International Pierre Gy Sampling Association (decided by a simple majority of assembled attendees) or failure of the proposal.

Proposal

Constitution of the International Pierre Gy Sampling Association (IPGSA)

[To be established at the inaugural Association meeting at WCSB8, Perth, 11 May 2017]

1 Preamble

The purpose of the International Pierre Gy Sampling Association (hereinafter referred to as IPGSA) is to oversee various formal activities of the international sampling community and to represent the views of this community as it sees appropriate from time to time. The Association is not a legally constituted body, but is a common-interest, unincorporated association of persons solely bound together for the common purpose of promoting the interests of the international sampling community and its various activities. The Constitution of the Association incorporates the rules identifying in whom control of the Association and its funds are vested. The Association is headed by a Council, the membership of which is specified in Clause 2 below.

The Council of the Association primarily has the authority to award the right to a country to host the World Conference on Sampling and Blending (WCSB), which draws together the world's leading sampling practitioners, manufacturers and experts to promote, network, discuss and reveal the latest advances in the theory and practice of sampling and blending in scientific research, technology development and industry, including large, medium and small government, community and private organisations. A special focus of the biennial WCSB conferences is on advancing the Theory of Sampling (TOS), and attendance at the eight conferences held so far has been very active since the first conference in 2003. The conference Proceedings, published by various institutional or learned journal publishers, play an important role in representing and documenting the current state of the art in sampling and blending.

The IPGSA Council informs the international sampling community of all relevant scientific news and the latest organisational matters through the scientific magazine

TOS Forum, which is a free-of-charge publication available from IM Publications (http://www.impublications.com/tos-forum) and elects its Editor for a two-year period. The Council also has the responsibility for securing adequate funding for the continued publication of *TOS Forum* (nominally three issues per year), more specifically for the difference between the production costs and the sponsorship/advertising funding successfully procured by the Editor.

With respect to conference Proceedings, the Council is responsible for establishing and maintaining an IPGSA internet portal from which all WCSB Proceedings can be downloaded or can be located if specific publications need to be acquired commercially. The *TOS Forum* and this portal shall work closely together.

Finally, the IPGSA Council is responsible for recognising outstanding contributions and excellence in the teaching and application of the Theory of Sampling via the Pierre Gy Sampling Gold Medal, as well as recognition of distinguished service in the sampling community and encouragement of young authors at WCSB conferences. If necessary, the Council is responsible for securing any additional funding needed for production of the Pierre Gy Sampling Gold Medal.

2 Roles

Chair of the IPGSA Council: elected for a four-year term by the sampling community at a general assembly which takes place at WCSB conferences held approximately every two years.

Vice-Chair: elected for a four-year term in the same way as the Chair of the IPGSA Council and represents the Chairman when required. The Vice-Chair also acts as Secretary of the IPGSA Council.

Council Member: elected for a four-year term in the same way as the Chair and Vice-Chair of the IPGSA Council in accordance with the composition of the IPGSC.

Editor *TOS Forum*: elected for a two-year term by the IPGSA Council.

Pierre Gy Sampling Gold Medal Committee: consists of all past Pierre Gy Sampling Gold Medal recipients plus two additional members elected by Council at each WCSB conference.

Life Member: all past WCSB Chairs and IPGSA Chairs.

Conference Host Country: responsible for appointing a WCSB Chair and establishing an Organising Committee responsible for organising the next conference and its Proceedings.

WCSB Delegate: a registrant at a conference.

The role of WCSB Chair and Chair of the IPGSA Council may, or may not, be filled by the same person.

3 International Pierre Gy Sampling Association Council

The IPGSA Council shall consist of the current Chair, Vice-Chair (Secretary), Editor *TOS Forum* and three other Members elected by WCSB delegates at the general assembly held at WCSB conferences. Council membership shall be decided upon by ballot conducted by the IPGSA Council at the general assembly based on nominations received from the Council and the sampling community. Outgoing elected members are eligible for re-election. Nominations shall reach the Secretary at least one month before the beginning of the relevant WCSB conference at which elections will be held. The Chair and all other Council members will be elected by a simple majority of votes cast by WCSB delegates. In the event of a tie, the successful candidate shall be determined by a draw from a hat. The term of office for elected Council members is the period between alternate WCSB conferences, i.e. approximately four years.

Council will meet as required but, as a minimum, at each WCSB conference and once between conferences. Council meetings shall be conducted either in person or via video conference, Skype or similar electronic communication means.

The Council is responsible for sampling community policy decisions, selection of the Host Country for the next WCSB conference and all dealings with the Host Country in this context. The Council also develops strategic plans for *TOS Forum* based on proposals from the Editor and takes the necessary actions to secure the funding needed to publish *TOS Forum*. Finally, the Council deliberates and formulates other IPGSA initiatives as it sees fit.

When choosing a Host Country or voting on any other contested matter, voting shall be by a simple majority of those attending Council meetings either in person or via electronic communication means. The Chair shall have a second or casting vote when needed.

In the absence of the Chair, the Vice-Chair will manage Council proceedings and will hold the proxy vote of the Chair, his own vote as Vice-Chair, and the Chair's casting vote if required.

The procedure for selecting Host Countries is detailed in section 7 below.

Unless otherwise determined, Council members are personally responsible for the costs of attending meetings of Council.

4 IPGSA Taskforces

The IPGSA Council may from time to time establish Taskforces on specific matters which in the view of the Council require review. The Council determines the Taskforce terms of reference and deadline, and constitutes the Taskforce members directly from willing members of the sampling community with the appropriate knowledge and competencies. Taskforces will be required to report formally on their activities at Council meetings. The Chair of a Taskforce should preferably, but not necessarily, come from the Council membership at the time of its establishment. The Council will regularly review the continuation of Taskforces, which generally have a limited lifetime.

5 IPGSA Awards

5.1 Pierre Gy Sampling Gold Medal

The Pierre Gy Sampling Gold Medal (PGSGM) in memory of the father of the Theory of Sampling (TOS), recognises a lifetime of distinguished service and advancement in the teaching and practice of the Theory of Sampling. The PGSGM Committee appointed by the IPGSA Council will select the recipient to be presented with the medal at each WCSB conference, which normally will take place at the conference dinner. Members of the IPGSA Council and the international sampling community may nominate candidates for the PGSGM, and submit nominations to the Chair of the PGSGM Committee. Nominating members must provide a CV of the candidate, the names and contact details of two supporters, and a summary of the achievements and contributions of the candidate. Voting will be by simple majority of the PGSGM Committee. The Chair will have a casting vote, but only when there is no apparent conflict of interest in exercise of the casting vote. In unusual circumstances, the Committee may decide to make the award to a maximum of two candidates.

The Pierre Gy Sampling Gold Medal may be awarded posthumously if decreed by sad necessity.

5.2 Distinguished Service Award

The IPGSA Council may from time to time announce an award, to be known as the "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 such an award may be nominated by any member of Council or individual members of the Association. The Award may only be awarded posthumously when the recipient dies in the period between the decision to make the Award and the presentation ceremony.

5.3 Young Author Awards

The Organising Committees of WCSB conferences may arrange Young Author Awards for the two most outstanding papers presented to encourage the participation of young authors in WCSB conferences. To qualify, the principal author must be less than 33 years of age at the date of the conference. The Award will be a scroll signed by the Chair and all other members of the Council which may, at the discretion of the Organising Committee, be accompanied by a monetary prize the cost of which will be met by the Organising Committee. The Organising Committee shall ensure that it is communicated clearly to all authors submitting papers for a conference that Young Author Awards are proposed. In this case, authors who consider themselves as a potential recipient shall submit a document, signed by a senior colleague, confirming that the author is eligible in terms of the age criterion and is the principal author of the paper submitted. The Organising Committee may choose not to make a Young Author Award. The Award can be made only once to any person and no Award can be made posthumously.

6 IPGSA Financial Arrangements

The IPGSA has no permanent source of income. However, in order to support the role of the Chair and the administrative costs of the informal IPGSA secretariat, a levy of EUR 35 per WCSB delegate shall be paid to the IPGSA by the Host Country within six months of the end of a WCSB conference. This levy may also be used to cover part of the costs of the Pierre Gy Sampling Gold Medal. The IPGSA Council will review the amount of this levy from time to time.

7 Host Country Selection Procedures

The IPGSA Council will call for proposals to hold the next WCSB conference at least one year in advance of each awarded conference. Issues to be addressed in bids include:

- The name of the professional/scholarly/scientific organisation under which the Congress will be organised.
- The person most likely to head the Organising Committee as Chair.
- The probable structure of the Organising Committee.
- The ability to finance and underwrite the cost of the Congress.
- Previous experience in organising similar international conferences.
- Availability of a suitable conference venue and appropriate accommodation.
- Availability of international and domestic flights to the host city.
- Potential to obtain sponsorship from industry and government.
- Any specific topics/themes for the conference.
- Visa regulations of the host country.
- Estimated cost of delegate registration.
- The likely person to act as the Editor of the WCSB conference Proceedings.
- Processes to be used to referee abstracts and final papers submitted for the conference.
- The proposed timing for submission of abstracts and full papers.
- Proposed Keynote speakers.
- Any other matters which the bidding country considers relevant.

Bids are to be received by the Chair of the IPGSA Council by the date indicated in the Call for Proposals. The call should, as a minimum, be made public in an issue of the *TOS Forum* at an appropriate time determined by Council. The Council will then meet, either in person or via video conference, to review and discuss all bids and whether they meet the selection criteria.

The final decision on the successful bid will be by a ballot of Council members. In the event of a tie, the Chair, having no conflict of interest, shall have the deciding vote and choose between the tied bids. If the Chair has a conflict of interest, the Vice-Chair shall have the casting vote.

8 WCSB Conference Organising Committee

The Conference Organising Committee as broadly outlined in the successful conference bid is responsible to the IPGSA Council for implementing an enabling structure to ensure effective planning and carriage of the conference. While the structure of the Organising Committee is not predetermined, it shall be established so that its composition and operation is appropriate to carry out all the necessary duties and meet its responsibilities. The Organising Committee will regulate its own affairs in accordance with internal rules and procedures. In the interests of continuity, the Organising Committee is encouraged to co-opt the Chairs of previous WCSB conferences. These persons do not need to physically attend meetings, but should be kept informed of deliberations to enable provision of advice and other assistance as required.

The Organising Committee is required to fund production of the Pierre Gy Sampling Gold Medal as well as the Young Author Awards if a monetary prize is included by including these costs in the WCSB conference budget.

9 WCSB Conference Proceedings

Copyright of WCSB conference papers will be held by the authors and not the organising body. Consequently, authors will be free to submit their work after the conference to a journal of their choice. The journal will apply its own reviewing rules to such submissions. Notwithstanding the above, the organisers of a conference may publish a book of Proceedings for the conference. The Organising Committee must as a minimum provide delegates with copies of all abstracts accepted for the conference in an appropriate electronic form with a hard copy version for use at the conference. Furthermore, the Organising Committee must undertake to make the Proceedings of WCSB conferences publicly available after the conference in an appropriate way, e.g. via the IPGSA internet portal.

10 Amendments to the Constitution

Proposals for amendments to this Constitution can be submitted by any member of the IPGSA Council with at least ten supporting signatures from the international sampling community. Such submissions must reach the Chair of the IPGSA Council at least one month prior to the next meeting of Council. A two-thirds majority of those present at the Council meeting either in person or via video conference is required to approve an amendment.

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Pierre Gy's development of the Theory of Sampling: a retrospective summary with a didactic tutorial on quantitative sampling of one-dimensional lots

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This paper presents an overview of the pioneering work of Pierre Gy on the Theory of Sampling (TOS) over a period of 66 years—a monumental legacy of dedicated work to the science of sampling. It starts with the early years, beginning in 1949, when Gy worked tirelessly, often in isolation, and not without resistance from other scientists, to create a systematic, mathematically based framework within which every error arising from sampling of a heterogeneous material can be identified (named), analysed and explained. Gy is widely remembered for "The Formula", which expresses the variance of the fundamental sampling error (*FSE*) in terms of the mass, fragment top-size and various other compositional attributes of the minerals of interest. But as early as 1947 the seeds of two related questions were planted: "How... to sample" and "How much... material should one take", which eventually evolved to become the Theory of Sampling, essentially completed in 1975. Presentation of this famous equation to the scientific community led to a period of intense practical experimentation on stationary lots that continues to this day. Gy also addressed the challenge of sampling from fluxes and here identified the variability due to autocorrelation between samples taken from flowing streams that led ultimately to the discipline of *chronostatistics*, a study of variability in one-dimensional streams. The scope and depth of Gy's research endeavours grew during the 1960s and led to his encounter with metallurgical balance and reconciliation. As a result, Gy developed the idea of *proportional sampling* that allows several material streams to be sampled according to the same selection probability with very efficient reconciliation benefits. The TOS was also further extended to include the problems that producers faced in regard to *bed blending*. Gy was a prolific writer and published over 250 different articles and books, with his last four papers published in the *Proceedings of the First World Conference on Sampling and Blending*, WCSB1 (2003), Esbjerg, Denmark. In these papers, Gy left us a personal history of the development of TOS as well as three fundamental tutorials, with practical examples, summarising how to manage both the qualitative and quantitative aspects of sampling of discrete materials. This tribute focuses especially on Pierre Gy's pioneering applications of *variography* to understanding the large-scale variability in process plants and process control from as early as the 1950s, and he devoted a major part of the development period of TOS to this critically important subject. The *variogram* allows one to identify sources of variability and provides valuable insight into correlations between successive samples. Poor understanding of the analytical capabilities of the variogram mean that it has not been widely applied in process control. Failure to address the concept of stream heterogeneity means that conventional statistics and statistical process control (SPC) fail to identify and distinguish the *sources of variability* in a process stream. For each type of heterogeneity, there is a matching variety of process variability. Although the method is powerful in terms of the insights one is able to gain in regard to plant performance and management, there are surprisingly few examples of its application in the literature, although there has been greater acceptance of the method. The authors believe the concluding didactic presentation of Pierre Gy's approach to process sampling may be a useful starting point for newcomers to TOS.

Introduction

The fascinating story of Pierre Gy's interest in sampling and development of the Theory of Sampling is presented in the Proceedings of WCSB1, a collection of papers as a tribute to his work and personal history.[1]

Pierre Gy began his career in French Equatorial Africa (Congo) working on the small M'Fouati lead mine as the Mineral Process Engineer in 1946, where he was in charge of the processing plant and associated laboratories. In 1947 the Paris-based head office asked Pierre to estimate the grade of a 200,000 t, apparently low-grade stockpile that had been dormant since 1940. He soon recognised i) that fragments on the stockpile varied from several tonnes to fine dust, ii) he knew *nothing* about sampling, iii) there was no meaningful literature available and iv) that he would have to improvise. This request planted the seed of life-long interest in his mind.

On his return to Paris in 1949, his work in a mineral-processing laboratory also constantly brought issues of "sampling" to his attention, in particular the question of "the minimum sample weight necessary to achieve a certain degree of reliability".[1] In his search through the available literature, such as there was, Gy found that Brunton[2] claimed that the minimum sample weight was proportional to the cube of the top particle size, while Richards[3] suggested that the square of the particle size was important. Brunton[2] based his ideas on the "constant proportionality factor", meaning that for samples with different fragment top sizes, the same number of fragments was required, but Gy[1] was concerned that variations in grade or density had not been properly incorporated.

It was the magnitude of financial transactions in the coal trade based on assays for ash and sulphur in "coal samples" that promoted much of the early research into sampling. Gy tells about UK- and USA-based researchers that "realised that sampling actually generated errors that could have a financial impact", and so began the interest in investigating coal properties in regard to particle top size, sample mass and sample variance. He mentions a Professor Hassialis from Columbia University, New York who

wrote a chapter on sampling based on a statistical multinomial model in the *Mineral Processing Engineer's Bible*, first published in 1927. The number of influencing parameters that were never known meant that this approach could not be practically implemented. A French Mining Engineer, R. Duval proposed a binomial model (in which the world is made up of white and black balls only) representing pure gangue and pure mineral, in which *all* fragments were considered to have the *same* physical mass. While Gy understandably found aspects of this model "dangerously misleading" (*sic*), it germinated the seed of interest sown by his earlier experiences, leading to his 1949 decision to study the theoretical issues around sampling in earnest.[1]

Gy expressed his intention to develop a mathematical model relating the variance of the sampling error to the mass of the lot, the sample mass and the *knowable* physical properties of the material being sampled. Such a relationship would allow the minimum sample mass needed to achieve an acceptable sampling variance to be determined. Gy's hopes of addressing the question "how much" had to be pursued in his own off-work time, as his employer provided neither time nor resources for this research. Surprisingly, even with these obstacles, he devised and wrote up the formula and the basic tenets of the TOS in two internal, unpublished notes for his company *Ste Minerais et Metaux*, entitled: "A formula for the minimum sample mass" and "Minimum sample mass required to represent a batch of ore" as early as 1950. Historically the TOS was born in 1950.

This endeavour led to a first theoretical model specifically for *particulate solids*, but generalised models for solids of animal and vegetable origin, types of domestic and industrial waste, liquids and gasses were also developed. By this stage Gy recognised that the models had *universal* validity and that it was *scale* rather than physical state that differentiated between the range of applications.

The Formula established

The progression in Gy's logic in regard to formulating the variance model, as early as 1950, is fascinating. He first identified all the unknown, but physically well-defined parameters, including the number of N_L fragments making up the lot, the corresponding N_S fragments making up the sample, a_S the grade of the sample, F_i the number of fragments and M_i the individual fragment mass. From these he devised strict, algebraically simple mathematical relationships into which he introduced *simplifications* and *approximations* to produce easily implementable and practical formulae. He first devised formulae for the mean and the variance of a population of "equally probable samples of N_S fragments". At some point in this work, he realised that he needed to educate himself more properly in statistics—and only a few years later he was awarded his second PhD—et voilà! This was necessary in order to able to work more stringently with the crucial approximation simplifications of the full mathematical descriptions.

In this context, among his most germane ideas was the concept of a quantitative measure of *heterogeneity*, which "lies at the root of all sampling errors", which in Equation 1 can be introduced as:

$$h_i \frac{(a_i - a_L)}{a_L} \times \frac{M_i}{M_i^*} \qquad (1)$$

where h_i represents the constitutional heterogeneity carried by one fragment F_i in the lot L, M_i and M_i^* are the individual and the average mass of all fragments. The importance of h_i is that it expresses the *heterogeneity contribution* carried by each fragment in the lot, which crucially can be summed up and, when divided by the number of fragments in the lot and given an appropriate statistical weight, leads to the desired approximate measure of the variance of the total sampling error (*TSE*), as shown in Equation 2.

$$\sigma^2_{TSE} = \left(\frac{1}{N_S} - \frac{1}{N_L}\right) \bullet \frac{\sum_{i=1}^{N_L} h_i^2}{N_L} \qquad (2)$$

After a very long process of trial and error (many years) in which he tested out a plethora of simplifications and approximations for correlating the sampling error to the physical properties of the lot material (this is where Pierre Gy decided his second PhD in statistics was essential), he arrived at the by now well-known general equation which is publically referred to as "Gy's formula" (but which he strongly preferred to call the "Formula" only),[1] shown in Equation 3.

$$\begin{aligned} \sigma^2_{FSE} &=\sim \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \bullet cfg\ell d^3 \\ &=\sim \frac{cfg\ell d^3}{M_S} \quad \text{when } M_S \ll M_L \end{aligned} \qquad (3)$$

Here c is the *mineralogical composition factor* having units of (but not the meaning of) specific gravity (g cm^{-3}), taking the average grade and densities of all components into account. Factor c is to be understood as the density of the "mineral of interest" (i.e. the phase carrying the analyte) divided by the grade, so c becomes larger as the average grade of the material decreases—the sampling variance increases the lower the grade of the increasingly more heterogeneously distributed phase. ℓ is a dimension-less *liberation factor*, defined by Francois-Bongarcon[4] as $\ell = (d_\ell / d_N)^{0.5}$ that varies between 0 for completely liberated components and 1 for non-liberated ores. f is a dimension-less *particle shape* factor having a general value of 0.5 (a kind of early "mission impossible" trying to input quantitative information in the formula representing the principal *form* of the mineral phase of interest with a simple number in the interval [0,1]) and g is a dimension-less *size range factor* with a general value of 0.25; this latter is a measure of the *sorting* of the material being sampled. d is the top particle size in centimetres through which 95% of the material passes, aka d_{95} (the *cube* of the top particle diameter won out—not the square, see re. Brunton vs Richards above).

This early explanation was followed by a more elegant proof in Gy[5] in which *TSE* is generated by each element of the lot being submitted to the selection process (extraction), the *sampling*, with a certain selection probability P_m. In this case *TSE* is now to be understood as the sum of i) the correct sampling error (*CSE*), only related to material composition and heterogeneity, and ii) the incorrect sampling error (*ISE*), specifically related to sample extraction and materials handling (i.e. the errors produced by the sampling process itself, if not effectively mitigated), such that $TSE = CSE + ISE$ from which two cases arose.

If sampling is *correct*, $P_m = P = \text{constant}$ and $ISE = 0$, meaning that $TSE = CSE$, and if in addition elements are selected individually and independently, then $TSE = CSE = FSE$. However, in practice the condition that samples be collected individually and independently is never satisfied, the best we can do is extract *groups* of neighbouring fragments (groups = increments) with uniform selection probability P. The likelihood of a spatial correlation between selected fragments in the spatial

volume of the lot generates a new error referred to as the grouping and segregation error (*GSE*) where:

$$TSE = CSE = FSE + GSE$$

Historically all manner of adverse problems have since manifested themselves whenever people applying Gy's formula do not take proper account of *GSE*—scores of frustrated *rapporteurs* lament that the level of total sampling errors estimated are usually larger (not seldom much larger) than what is indicated by var(*FSE*). This is all due to missing out on understanding and managing (reducing, eliminating as much as possible) *GSE*.

Further Gy[1] also makes the key point that using the formula to determine the sampling variance without appreciation of the possible crucial sampling bias (i.e. the gamut of all incorrect sampling errors) would render straightforward application of the formula *meaningless*. This is a point very well remembered. This is where all serious sampling starts, lest all possibilities of representativity are lost.

Practical experimentation with the Formula

Gy[1] tells the story of how he originally attempted to validate the formula by calculating the variance of a lead ore using 16 "equally split" samples of pulverised material, splitting here taking the role of sampling. His experimental *TSE* was several times *larger* than the theoretical value, something he interpreted as indicating that the *FSE* was only *one* of several components in the game. The other components of sampling error he suggested were the *GSE* as well as the sampling bias, see above, introduced through incorrect use of the riffle splitter. His research in the mid-1950s then led to the development of a circular cardboard sampling nomogram and later a sampling slide rule. The formula was first presented in English to the Society of Mining Engineers of the American Institute of Mining Engineers (SME of AIME) in 1957. However, it was only in 1965 that his research was presented in London at a meeting of the Institution of Mining and Metallurgy (IMM).

Sampling of flowing streams

Pierre Gy's 1960–1962 research into flowing streams of materials on conveyor belts and liquid launders brought to his attention the importance of sampling the "whole stream" for a fraction of the time, i.e. any increment must be a physical full slice of the stream. He identified the key issues in regard to cross-stream sampler operations, namely that the cutter velocity through the stream, the width of the cutter opening and the shape of the cutter are all-important, but it was first in 1977 that these issues were scientifically resolved.

He also recognised that increments extracted at constant intervals from a flowing stream are not independent of one another, but that some level of *auto-correlation* exists between most time series sample data. As early as 1962 Gy started published his work on *chronostatistics*, as it later became known, by borrowing the idea of spatial correlation between samples using concepts and data from the semi-variogram feature proposed by Matheron,[6] and later by David,[7] within geostatistics and transferring it to linear auto-correlation of time series data.

At this stage of his life, Gy made the choice to dedicate himself to writing and further research around the theory and practice of sampling, rather than to continue in his rather comfortable managerial position at Minerais et Metaux in Paris. This led him to what history now recognises as a grand 40-year period of theoretical research, consulting, trouble-shooting, lecturing and teaching regular courses at schools and Universities, and writing articles and books gradually being disseminated all over the world. The reader is encouraged to peruse Pierre Gy's complete bibliography.[1]

Theory of Sampling introduced—and challenged

This time of progressive successes was not without serious challenges, however, as some parties and individuals strongly opposed Gy's ideas and objected to his 1967 publication in French, "Sampling of Particulate Materials".[8] No story is only about success—it is a sad historical fact that the response from ISO standards committees has been less than unanimously accepting of the work and insights of Pierre Gy (although this situation has begun to be significantly turned around since 2003 by a dedicated effort by the sampling community). The world now has at its disposition a first standard dedicated to the universal principles of representative sampling, DS 3077.[9] This last part of the history of TOS can be followed in detail in the proceedings from the WCSB conferences and in *TOS Forum*.

Interestingly, the notion of correct sampling and its linkages to probabilistic sampling were only first proposed by Gy in 1972. In modern parlance, the fundamental tenet is that a sample is correct if and only if each lot fragment has the same statistical probability of being selected for the sample as every other fragment in the entire lot. Under any other circumstance, the sampling procedure is said to be incorrect and will therefore result in unrepresentative lot "samples" (better designated "specimens" for optimal distinction).

About this time Gy found that some members of the scientific community resisted his ideas about sampling as a scientific endeavour. His 1971 book entitled *Sampling of Particulate Materials, Volume 2* was soon followed by another book *The Theory and Practice of the Sampling of Particulate Materials* in 1975, but only a few hundred copies were ever sold. In this particular book, Gy made a very significant step in that he built "the mathematical bridge between selecting conditions and sampling errors". He identified for the first time, the distinction between *a priori* conditions of sample selection (conditions we can do something about before taking the sample), and *a posteriori* conditions (conditions we can observe, but about which we can do very little after the fact). The selection process itself can further be either probabilistic or non-probabilistic—and even if probabilistic, it can be correct or incorrect. Sampling errors are random errors, characterised by their statistical distribution and moments. Sampling can be accurate or biased (property of the *mean*), reproducible or not (property of the *variance*), and representative or not (property of the mean-squared error).

Pierre Gy also tells of the difficulties he faced in 1978–1979 writing his first book in English, a translation of this seminal 1975 text. The book, published in 1979, was followed by a second edition in 1982.

Between the release of the Second Edition of the 1979 text and his latest book in French,[10] Gy developed a number of new applications of his theory including the computation of auxiliary functions of the variogram, the ideas underlying proportional sampling and a theory of bed-blending.

Proportional sampling

Gy's first encounter with metallurgical balance reconciliation was in some North

African lead-zinc flotation plants where he summarised the idea of balance saying that "whatever comes in must ultimately come out, one way or another". He noted that if this principle of balance is not observed, then there must be "measurement biases or unsuspected losses", and that with a single exception in his 45 years of consulting, what came out was always *less* than what went in.[1] Eventually, after checking every sampling and measurement device, he reached the conclusion that the principal culprit for the 2–3% deficit was the calibration of the conveyor belt scales. After observing numerous conveyor belts over the years, Gy concluded that they suffer from a structural lack of reliability, the main problem being the conversion of an electrical current into an accurate measurement of tonnes of ore. Rather than the 0.5% accuracy *claimed* by manufacturers, plant personnel found a more realistic figure to be about 10% deviation from accuracy.

In view of the importance of proportional sampling to metallurgical balances and based on the excellent exposé of this subject provided by Wavrer,[11] the explanation that follows here is somewhat detailed. During his development of the theory of sampling Gy concluded that if the probability of selection P is a uniform distribution in time, then sampling is correct, and the mean of the sample mass M_S is a random variable equal to P times the mass of the lot M_L (Equation 4).

$$m(M_S) = \mathrm{P} \times M_L \qquad (4)$$

The corollary is that accurate estimation of P means that M_S/P is an unbiased estimator of the mass of the lot, M_L. If the number of increments is large, the minimum and maximum values that the sample mass could take with given sampling equipment is very accurately known, and confidence limits for these sample masses, M_S, are very small. Proportional sampling must not only be correct, but the mass and volume of the sample must be proportional to the mass and volume of the lot. Thus all concentrates, tailings and feed streams, sampled according to the same proportional ratio (selection probability), make the proportionality factor constant. According to Gy,[1] M_S/P is a much more reliable *unbiased* estimator of the mass of the lot M_L, than any that can be obtained by weightometers, and this became the basis of his revolutionary idea of *proportional sampling*. Wavrer's simple and elegant explanations[11] are presented in Table 1. Gy defined a time sampling ratio and a mass sampling ratio, for which equations are presented in Table 1. All feed streams, concentrates and tailings are to be sampled according to the *same* proportional ratio (selection probability).

In this way sampling from all material streams are now completely *comparable*, making the calculation of the material balance a simple and very accurate task. The critical success factor is that the selection probability is kept constant under all circumstances.

Bed blending

Perhaps the most important aspect of feeding a metallurgical furnace is to blend the raw materials in such a way that the average composition of the feed will be more-or-less uniform and homogenous in the one dimension of the ingoing material stream. Gy's work on *bed blending* began with a study of material processed in cement kilns. The lack of flexibility and sensitivity of cement kilns is such that feed materials must be as uniform as absolutely possible to avoid costly damage. For this reason, a large cement company introduced a bed blending system in order to homogenise, as best as possible, the ingoing raw materials. Good sampling equipment aided by on-line analysers allowed major components in the cement to be determined every few minutes. Computerised assistance to calculate the average composition of the stockpiled kiln feed allowed the composition of the blending pile to be known with accuracy, providing an almost ideal feed to the kiln.[1]

On one occasion the failure of the blending system to comply with Gerstel's theory,[12] Gy was asked to advice on the process and found that the bed blending theory was easily derived from existing sampling theory. In this case the manufacturer received an excellent explanation of how his equipment actually worked. Other cement producers approached Gy sometime later when it was proved that his new theory on bed blending was in perfect agreement with practice. The theory and practical aspects were published in 1981,[13,14] with a presentation on the subject to the Canadian Institute of Mining and Metallurgy (CIM) in Montreal.[15,16] Theories that Gy published over the years have consistently proved to be correct, and were easily adapted to the science of bed blending.

Gy's publications

It is not possible to tell Gy's story of discovery without at the same time telling what and where he published over 250 scientific books and papers on the TOS. His last textbook publication: *Heterogeneite, Echantillonnage, Homogeneisation* (Heterogeneity, Sampling, Homogenising), published in 1988 in French, was immediately translated into English and was published in 1992. It was the French version of this book that Dr Francis Pitard digested and shortened to produce his volume entitled: *Pierre Gy's Sampling Theory and Sampling Practice, Heterogeneity, Sampling Correctness and Statistical Process Control*. It is the Second Edition of this latter volume that has become a world famous publication used by many practitioners and now also taught in leading universities.[17]

In 1999, Allen Royle performed an English translation of Gy's 1996 text originally entitled *Echantillonnage pour Analyse Granulometrique* (Sampling for a size analysis). Royle's translated 150-page, 1998 English

Table 1. Wavrer's derivation for proportional sampling.[11]

Explanation	Equation	Parameters
"Time sampling ratio" of a lot, τ'	$\tau' = \frac{QT_i}{T_L}$	T_L is the flow time of the lot L Q is the number of increments between $t=0$ and $t=T_L$ T_i is the time to take one increment
"Mass sampling ratio" of a lot, τ	$\tau = \frac{M_S}{M_L}$	M_S is the mass of the sample S M_L is the mass of the lot L
If sampling is to be correct	$\tau' = m(\tau)$	$m(\tau)$ is the mean of τ
Rearranging	$\tau' = \frac{m(M_S)}{M_L}$	$M_L = \frac{m(M_S)}{\tau'}$

version was entitled *Sampling for Analytical Purposes* and was published by John Wiley, with a second edition in June 1999.[18] This has become a cherished avenue for newcomers into the sampling world. Pierre Gy was forever grateful to Royle for his help at a critical time, so much so that Gy *insisted* that the first Pierre Gy Sampling Gold medal was to be awarded to him. Allen Graham ("Bon") Royle was honoured by the TOS community in an obituary in *TOS Forum* Issue 1.

The WCSB1 in 2003 and the Proceedings of that meeting[19] were in honour of and a tribute to Pierre Gy's whole life and his work on the TOS. This volume contains 24 articles covering a very broad swath of the breadth and depth of the TOS as of 2003, five of which Pierre Gy wrote himself. (The 137 attendees to this first ever sampling world conference represented pretty much 90% of the then active individuals in the whole world.) This volume was to be the first in a series of Proceedings, the eighth will be from the Eighth World Conference on Sampling and Blending (WCSB8) being held in Perth in May 2017. The series of WCSB Proceedings is indispensable for anyone wanting to get into the theory and practice of sampling.

Summary of Pierre Gy's last contributions

Pierre Gy's last technical contributions to the sampling fraternity came in the form of a three-part theory and practice tutorial entitled "Sampling of discrete materials I, II, III" in the WCSB1 Proceedings.[19] While it was not possible for him to cover all aspects of sampling, these three papers nevertheless represent a formidable distillation of more than 50 years' work. Together with the accompanying feature article,[1] "Theory of Sampling—a personal history", these papers rank amongst some of the most important contributions from Gy, and deserve our full attention a.o. because he took the pains to rectify the admittedly bewildering, often changing three-letter-abbreviations (TLA) that have always characterised TOS, while laying down his final resumé of logical development history of the *theory* of sampling. These three tutorials specifically address the two fundamental questions that inspired Gy's original dedication in 1949, questions which cannot be dissociated from one another: "*How* should one select a representative sample? and *How much* material should be selected for this purpose?". Part I covers the foundation for the qualitative approach, and Parts II and III the quantitative approach to answering these two questions.

Part I: A new introduction to the TOS—qualitative approach

Part I is an up-to-date introduction to the TOS in which Gy explains the seriousness of good sampling science and technique and how without it, not only money, but also lives could be at stake. He set forth the basic definitions and notations, drawing a distinction between samples (representative) and specimens (worthless), showing how the sampling conditions and the proper definitions are strongly related. Explanations of the concepts of constitutional and spatially distributional heterogeneity (*CH*, *DH*) are followed by outlining the processes and methods of mass reduction, such as grab sampling, splitting and incremental sampling that again distinguish samples from specimens. Gy[8] summarised Part I by saying that sampling is a science, and while the TOS, which explains the generation of errors and proposes practical solutions, *may* be contested by those who wish, it can never be ignored. The inadequacy of most standards dedicated to or containing sections pertaining to sampling even today is because they fail to recognise TOS, while bias in other standards is deliberate (*sic*). TOS is the only scientifically consistent and exact means of extracting correct, unbiased, accurate and reliable samples. Non-probabilistic or incorrect probabilistic sampling will always produce biased and unreliable specimens, making meaningful decisions impossible.

The taxonomy and nomenclature of sampling errors is admittedly somewhat complex, but Gy[8] presents ultimate definitions of errors arising from sampling in Part I of his tutorials. Sampling consists of progressive stages of comminution and representative mass reduction of the original lot L to produce sub-samples. The primary sampling from any lot is very often accomplished by problem-specific composite sampling (often comminution is not possible at the primary stage). Errors at this stage are the primary sampling errors (*PSE*). Secondary sampling of the primary sample typically occurs in the laboratory, and here typically consists of progressive comminution and representative mass reduction of sub-samples, ending up with an *aliquot* a_L for analysis. Errors at this stage are secondary sampling errors (*SSE*). The combination of *PSE* and *SSE* errors give rise to the total sampling error ($TSE = PSE + SSE$). Combining *TSE* with the total analytical error (*TAE*), produces what Gy refers to as the global estimation error (*GEE*).

Part II: Quantitative approach—sampling of zero-dimensional objects

Parts II and III cover the quantitative approach and the sampling of zero-dimensional and one-dimensional objects. Part II introduces the concept of the sampling dimensions of the lot, stockpiles being typical of 3D lots, open-pit operations being typical of 2D lots, material on a conveyor belt being typical of a 1D lot, while discrete, independent, easily accessible objects lending themselves to easy *manipulation* (mixing, sub-sampling) are described as zero-dimensional lots. Gy[20,21] only deals with sampling of zero and one-dimensional lots.

Gy theoretically defines a lot made up of discrete material, L, as consisting of a set of units (potential sampling increments), the sets being either a population of non-ordered, zero-dimensional units (as in a stockpile), or as ordered, chronological cross-sections of one-dimensional units in a flowing stream of material on a conveyor belt (one-dimensional lot). In general, the one-dimensional units are characterised by a correlation between the position of the unit in the series and their composition. Such autocorrelation requires a very different mathematical approach to identifying and quantifying sampling errors compared to zero-dimensional lots, and hence the sub-division into Part II and Part III of Gy's papers.[20,21] While Part I covered the general concepts of heterogeneity and homogeneity in detail, Gy devotes Part II[21] to explaining the mathematical relationships between sampling errors as a result of the salient aspects of heterogeneity.

Gy provides a progressive mathematical basis for a model describing the contribution of heterogeneity to the lot by an unspecified unit, U_m, and uses this to define constitutional heterogeneity, CH_L in the case when U_m is a single fragment F_i. He then expands

this by alternatively viewing the unit, U_m, now as a *group*, G_n, of neighbouring fragments F_i to define the distributional heterogeneity DH_L of lot L, and further describes the relationship between CH_L and DH_L. In these steps Gy mathematically quantifies the various forms of heterogeneity and then expresses the sampling errors in terms of their mean, variance and mean squared errors. Part II[22] also describes the heterogeneity invariant HI_L before examining the notion and nomenclature around zero-dimensional probabilistic sampling and the related topics of correct and incorrect sampling.

Gy then describes a method of experimental estimation of what is now commonly referred to as the *heterogeneity test*, providing three practical examples of implementation of this technique from industry using lateritic iron ores, fragments of precious metal ore and sphalerite flotation concentrate pellets. Gy[21] finally makes a number of important summary statements about sampling in zero-dimensions iterating the most fundamental understanding that "sampling errors are the consequence of one form or another of heterogeneity". He notes that sampling of homogenous materials would be an exact operation according to the definition of homogeneity, but that it is *never* observed in the real world. The simplest way to convey the progression in Gy's layout of the quantitative approach to sampling zero-dimensional objects is to use the summary that he provided (p. 37), describing the relevant equations in support of these concepts, shown in Table 2.

Gy defines the *TSE* as the sum of only two components, i) the *CSE* and ii) the *ISE*, for which the probability of selection can be uniform or non-uniform.

Selection with uniform probability will only incur the correct sampling error (*CSE*) arising from the constitutional and distributional heterogeneity of the material, which give rise to the fundamental sampling error (*FSE*) and the grouping and segregation errors (*GSE*), respectively. Selection with non-uniform probability will incur additional, bias-generated incorrect sampling errors (*ISE*), which are a combination of the incorrect delimitation error (*IDE*), the incorrect extraction error (*IEE*) and the incorrect preparation error

Table 2. Concepts and supporting equations for the quantitative approach to sampling of zero-dimensional objects according to Gy.[21]

Relevant concepts	Supporting equations
The contribution h_m of a given unit U_m to the heterogeneity of the set L of units. Unit U_m can be either a single constituent F_i or a group G_n of constituents such as an increment I. The heterogeneity contribution h_m is a function of the mass and composition of unit U_m and lot L.	$h_m = \frac{(a_i - a_L)}{a_L} \times \frac{M_m}{M_m^*}$
The constitutional heterogeneity, CH_L, of lot L considered as a population of single elements. CH_L is the variance of the corresponding population of h_i.	$CH_L = \sigma^2(h_i) = \frac{1}{N_F}\sum_i h_i^2$ and $\sum_i h_i = 0$
The heterogeneity invariant, HI_L, derived from CH_L for practical purposes and usage.	$HI_L = CH_L \frac{M_L}{N_F}$
The distributional heterogeneity, DH_L, of lot L considered as a population of groups of neighbouring elements. DH_L is the variance of the corresponding population of h.	$DH_L = \sigma^2(h_n) = \frac{1}{N_G}\sum_n h_n^2$ and $\sum_n h_n = 0$
The *TSE* generated when selecting constituents in a probabilistic way (non-probabilistic sampling cannot be analysed theoretically).	$TSE = \frac{a_S - a_L}{a_L}$
TSE is disaggregated into the sum of two components, *CSE* and *ISE*. Additional *ISE*s are observed when the sampling is incorrect.	$TSE = CSE + ISE$
The *CSE*s, observed when the sampling is correct; there is a first and second approximation.	$m(CSE)_1 = 0$ $m(CSE)_2 = -\frac{1-P}{P}\sum_m \frac{a_m - a_L}{a_L} \times \frac{M_m^2}{M_L^2}$
The *FSE* is the *CSE*, observed in ideal conditions, when the constituents are selected correctly, one by one and independently.	$\sigma^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \bullet cfg\ell d^3$
The variance of *FSE* is proportional to the CH_L, and, in practical applications, to the HI_L.	$\sigma^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \bullet HI_L$
A practical, experimental method to estimate HI_L and hence the variance of *FSE*.	$HI_S = \sum_i \frac{(a_i - a_S)^2}{a_S^2} \times \frac{M_i^2}{M_S}$
The *GSE* is the additional error generated when selecting constituents with a uniform probability P, by groups (increments) of non-independent constituents. The variance of *GSE* is proportional to the DH_L.	$\sigma^2_{GSE} \propto DH_L$

(*IPE*) (sometimes also including an incorrect weighing error, *IWE*). *IDE* results from incorrect geometrical delimitation of the physical three-dimensional increments, *IEE* result from incorrect extraction of material increments from the delineated increments and *IPE* results from incorrect preparation and handling of material increments and samples after having been extracted (correctly or incorrectly). All these latter errors arise from ill-informed and/or poor mechanical design, or improper use, or poorly maintained sampling equipment, or improper handling of the samples after extraction. In this case the *TSE* is given by:

$$TSE = CSE + ISE$$
$$= FSE + GSE + ISE + IEE + IPE.$$

Quantitative approach—sampling of one-dimensional objects

Pierre Gy pioneered applications of *variography* to understanding the large-scale variability in process plants and process control from as early as the 1950s and devoted a major part of the remainder of his TOS development period to this subject. The variogram allows one to identify *sources of variability* and provides valuable insight into correlations between successive samples. Poor understanding of the data analytical capabilities of the variogram means that it has not been widely applied in process control up until this day, except in the industry sectors which have embraced TOS (mining, cement and certain parts of process industries). Failure to address the concept of stream heterogeneity means that conventional statistics and statistical process control (SPC) fail to identify and distinguish the *sources of variability* in a process stream.[22] For each type of heterogeneity, there is a matching variety of process variability. Although the method is powerful in terms of the insights one is able to gain in regard to plant performance and management, examples of the application of this particular method is surprisingly absent in the literature.

Process variability

Large-scale variability, usually in the form of product composition cycles and chemical cyclic variations may be manifest throughout a sampling system or a process plant, but this type of variability is most easily discernible in one-dimensional process streams. In terms of the nomenclature of errors arising from the sampling of one-dimensional objects, Gy[21] considers a flowing stream of material or lot *L* as a sequence of zero-dimensional increments with adjacent potential increments centred on point increments. One-dimensional sampling is a two-step process, which may be correct or incorrect. It consists, first, of sample selection at a correct or incorrect *position* on a time axis of immaterial, point-increments, giving rise to the point selection error (*PSE*). Second, the *extraction* of material increments at the point increments of materialisation (the sampling event by cross-stream or cross-belt sampling), giving rise to the materialisation selection error (*MSE*). *MSE* is in turn the sum of only two components, the correct sampling error (*CSE*) and the incorrect sampling error (*ISE*), whereas the *TSE* is:

$$TSE = PSE + CSE + ISE = PSE + (FSE + GSE)$$
$$+ (IDE + IEE + IPE).$$

The overall contribution of the three main sources of variability, random error *V*[0], process error *V*[1] and cyclical error *V*[cyclic] can conveniently be represented, for example, in a pie diagram.

Examples of one-dimensional lots include moving aggregate mixtures on conveyor belts, liquids, suspensions and slurries in pipes or chutes. Pitard[23] explained that the components of variability around a targeted average are the integrated accumulation of heterogeneity arising from three sources, and expressed as: $h_T = h_1 + h_2 + h_3$, where:

Heterogeneity h_T = total heterogeneity,
Heterogeneity h_1 = random, discontinuous heterogeneity that is a property of the materials,
Heterogeneity h_2 = non-random, continuous heterogeneity that is a function of time,
Heterogeneity h_3 = cyclic, continuous heterogeneity that is a mechanical function of the system.

The variability, and by analogy the heterogeneity, in a moving stream of material can best be represented by a variogram, a model for which is shown in Figure 2.

Different sample intervals in the stream provide the data for calculating the variability between samples. In the first step, the variance between the closest consecutive samples is calculated and averaged according to Equation 5.

$$\gamma_1^2 = \frac{1}{2N}\sum\left(a_i - a_j\right)^2 \qquad (5)$$

This provides the first data point on the variogram. The second step is to calculate and average the variance between every second sample, and then every third sample and so on, giving rise to a typical variogram in Figure 2. The relative variogram simply divides the average variance for each point in the variogram by the mean

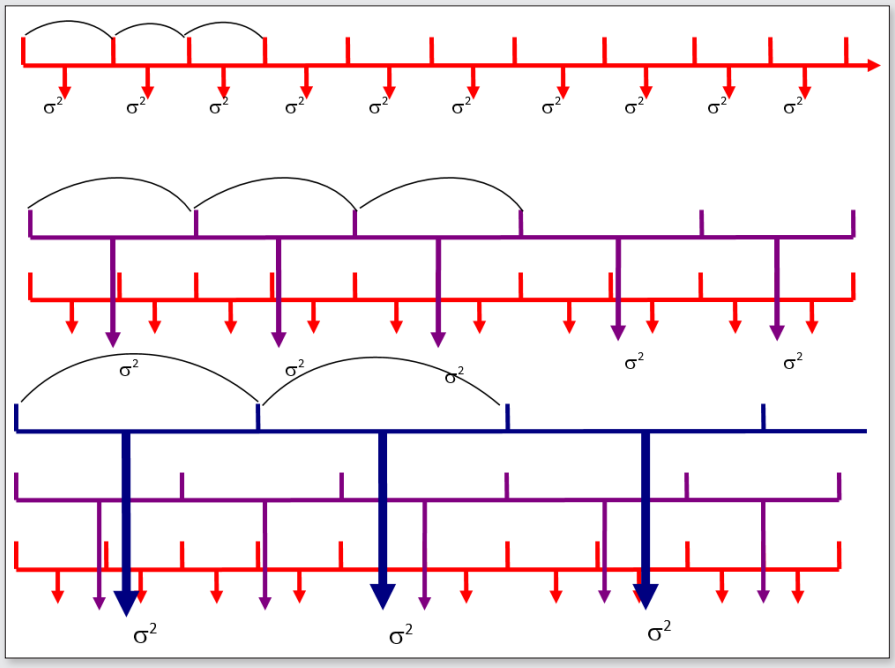

Figure 1. Top row: A series of sample points separated by distance *j* (the lag). Second row: a series of points separated by distance 2*j*. Third row: a series of points separated by distance 3*j* etc.

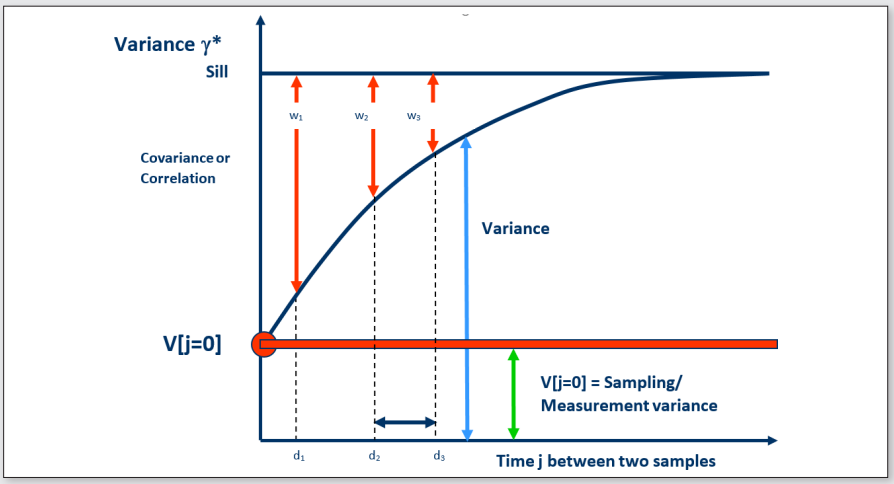

Figure 2. The components of the variogram indicating $V[j=0]$, commonly referred to as the nugget effect.

grade squared. The same effect, but also mass-corrected, is achieved by basing the entire variogram calculation on heterogeneity contributions. The difference between the absolute and relative variograms is only in the way they are calculated.

Absolute and relative variogram based on concentration values:

$$V(j)=\frac{1}{2(N_u-j)a_L^2}\sum_m\left[a_{m+j}-a_m\right]^2$$

Relative variogram based exclusively on heterogeneity contributions:

$$V(j)=\frac{1}{2(N_u-j)}\sum_m\left[h_{m+j}-h_m\right]^2$$

A modified set of 445 normally distributed data from a process plant is used below as an example of the principles and application of variography in process control.

The *moving average* allows one to identify small- and large-scale variability as well as cyclical behaviour in the process streams, while the variogram is a custom-built tool that allows components of variability to be identified and resolved. The components of variance are read off the variogram at specific line intersections. Variances are converted to standard deviations and plotted on the control charts, allowing an operator to manage the upper and lower control and specification limits.

Hydrometallurgical process plant example

Chemical variations in plant systems shown on the variability plot in Figure 3 provide perfect understanding in hindsight, but this information is by itself of only little use to a superintendent trying to stabilise plant variability.

At settings of 8.7% X and 9.28% X, several samples lie outside the upper and lower specification limits (Figure 4), indicating the difficulty of maintaining plant stability. Widening specification limits may help, but the erratic variation suggests the system is easily subject to overcorrection.

Large-scale variability: the moving average

Large-scale, cyclical variability is identified in the sampling data using a wide moving average window. Such cycles may have a regular period, but irregular amplitude, as in the case of metal% X (Figure 5). A 30-point moving average shown in Figure 5 emphasises the strong 118-hour cycle with numerous smaller superimposed cycles.

A 5-point moving average over the same 445-data (Figure 6) illustrates the much smaller scale cyclical behaviour in the data. These cycles have an average period of 14 h, but periods could vary from 10 h to 16 h.

Absolute variogram

The absolute variogram in Figure 7 was calculated for a 445-hour period although only 357 lags (hours) are shown on the x-axis. Three distinct cycles, each having cycles of 118-hour periods are evident in the variogram.

Although the periods are strongly regular, the amplitude of the cycles in the variogram is irregular.

Sources of variability

The following points are evident in the variogram (Figure 7) and are summarised in Table 3.

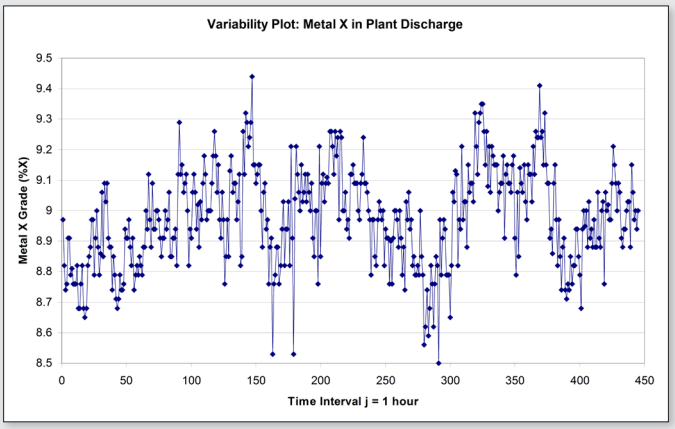

Figure 3. Variability plot of metal% X with highly variable sample data.

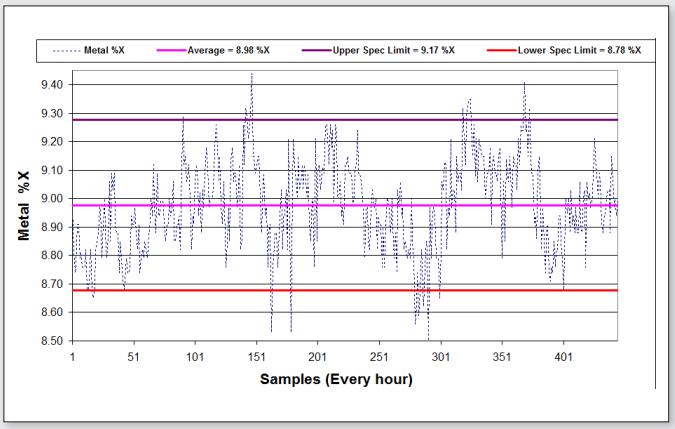

Figure 4. Variability plot of metal% X showing the average, as well as upper and lower specification limits.

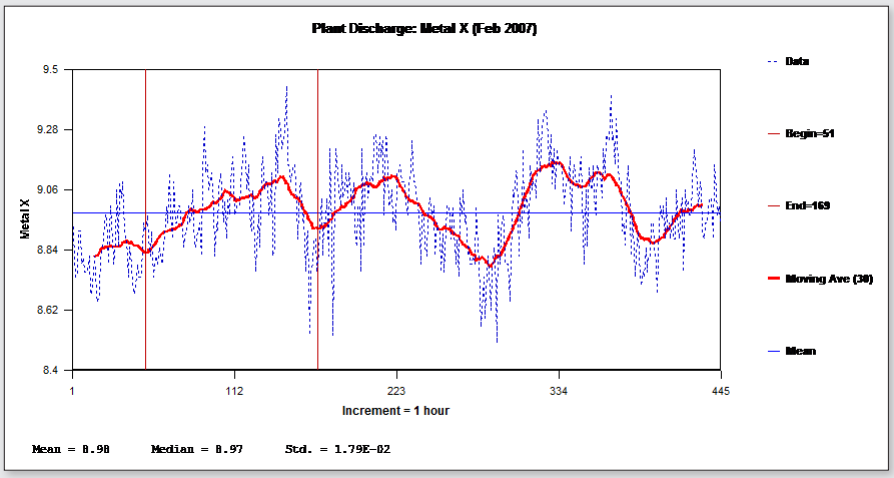

Figure 5. 30-point moving average showing four major cycles in the 445-data, with a 118-hour period (5-day period) superimposed on the data.

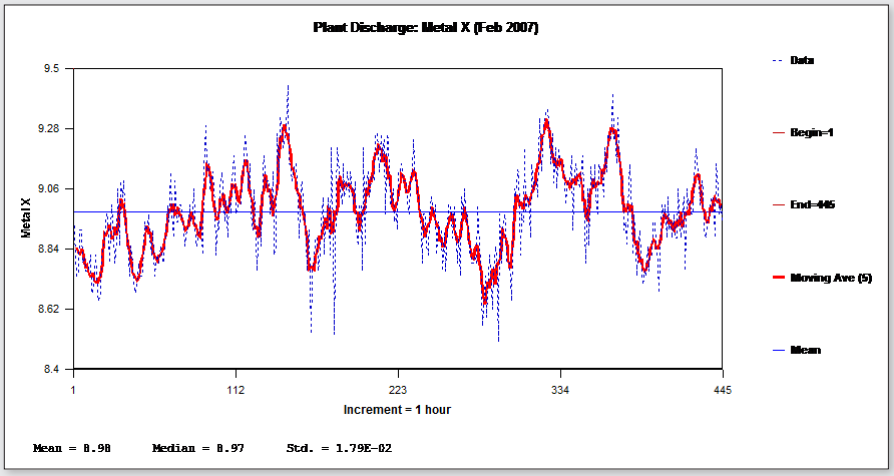

Figure 6. 5-point moving average showing the fine detail in the large scale 118-hour cycles.

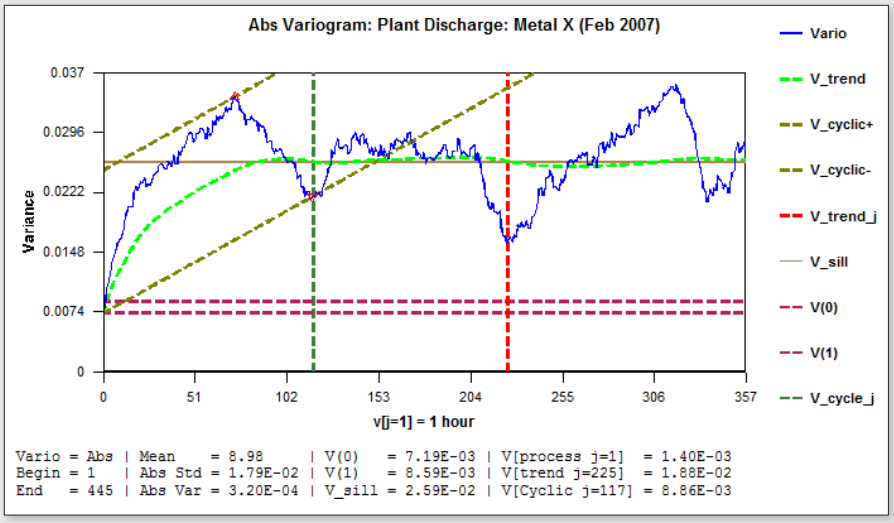

Figure 7. Absolute variogram for metal% X showing three distinct cycles.

The information derived from the variogram is transferred to the control chart and a variogram-based hierarchy of control limits is defined in Table 4, which shows how the various control Limits are defined and calculated.

The data listed in Table 4 can now be plotted on the control chart for metal X as a guide to what the sample variability is likely to do within the given constraints shown in Figure 8.

Analysing the sources of variability

Dr W. Edwards Deming clearly demonstrated in several experiments that one should not react to variations within $\pm 3\sqrt{V}[0]$. Indeed, there is no logical reason why anyone should react to some variability that does not exist in the process (i.e., *UCL* and *LCL*).

This concept allows us to optimise the sampling/measurement protocol. An active on-going reaction philosophy should be established to correct for all true process movements, when they are large enough to show through the random noise. For these movements, we must be ready to apply corrective methods.[22]

The variations characterised by $V2[j=1]$ are true process movements, predictable over a certain time frame, and potentially correctable. Therefore, we do not want to let variations become larger than $\pm\{(3\sqrt{V}[0]) + \sqrt{V}2[j=1]\}$ without reacting (i.e., *UCL′* and *LCL′*).

However, adding $\sqrt{V}2[j=1]$ to the limits *UCL* and *LCL*, to obtain *UCL′* and *LCL′*, makes a calculated allowance for process movements that are outside our control. Indeed, we are unable to react at time intervals shorter then $j=1$. This concept allows us to optimise the sampling/measurement interval.

Adding $\sqrt{V}3$ to the limits *UCL′* and *LCL′*, to obtain *UCL″* and *LCL″*, makes a calculated allowance for a process cycle beyond our control. It is very difficult to effectively correct a cycle. If this addition becomes too large, it is necessary to investigate the causes of the cycle. Sometimes, engineering modifications of the process are necessary to eliminate a cycle. Sometimes the way we organise our work results in a cycle. Understanding the cyclic nature of variability provides an opportunity to minimise a visible or invisible cost.

Table 3. The symbol, source, and amount of variability in the absolute variogram.

	Mean	**8.98% X**
	Absolute relative standard deviation	**0.0179% X**
	Absolute relative variance	**0.00032% X**
V[0]	**Short-range random variability** *V*[0]: this short-range random or irrelevant variability is shown by the lowest horizontal, dash/dotted line and corresponds to the total sampling, sub-sampling and measurement variability; it is not related to the plant process. It is a combination of inherent heterogeneity (random variability), fundamental error (*FE*) and grouping and segregation errors (*GSE*) and all uncontrolled sampling errors arising from a poor sampling procedure, and is a function of the total sampling and measurement variability. In the rest of this text *V*[0] is simply referred to as the random variability or "sampling variability". If *V*[0] is large, other errors are also likely to be large. The nugget effect accounts for a certain proportion of the total sill which is a relative measure of the overall variation. This component of error could be reduced if appropriate attention is paid to the sampling protocol. If the *V*[0] to sill ratio is high relative to the overall variation, anything above 65%, it suggests that the sampling errors associated with short-range random variability such as the grouping and segregation error (*GE*) and the fundamental error (*FE*) of the sampling protocol, are a significant problem. Upper and lower control limits (*UCL* and *LCL*) are applied to the control charts by adding three times the standard deviation of *V*[0] to the mean or the target average, in order to cover the 99.7% confidence interval.	$V[0] = 0.00719$ $\sqrt{V}[0] = 0.0848$ $S = \pm 0.0848$ $UCL = \text{Mean} + 3S = 9.23\%$ X $LCL = \text{Mean} - 3S = 8.73\%$ X
V[1]	**Total process variability** *V*[1]: is the typical value of *V*[1] at the first lag point in the variogram. It is the total non-random variation that occurs in the plant between any two consecutive analyses. This is a combination of the total sampling and measuring variability of the process, as well as the daily sampling interval (or process) capability. This variability cannot be controlled unless the routine sampling interval is reduced. The non-random component of variability is due to bias in the sampling process related to the delimitation error (*DE*), the extraction error (*EE*), the preparation error (*PE*) or the analytical error (*AE*) and can be eliminated through implementing an optimised sampling protocol. When *V*[1] is placed around the target mean (*TA*) it provides an indication of the upper and lower sampling capacity.	$V[1] = 0.00859$ $\sqrt{V}[1] = 0.0927$ $UCL' = 8.98 + 3S + \sqrt{V}[1] = 9.32\%$ X $LCL' = 8.98 - 3S - \sqrt{V}[1] = 8.64\%$ X
V[process]	**Process variance** *V*[process]: is the continuous, non-random variability in the plant between any two consecutive analyses; it is simply the difference between *V*[1] and *V*[0] at the first lag point. It is variability due to the sampling process related to the delimitation error (*DE*), the extraction error (*EE*), the preparation error (*PE*) or the analytical error (*AE*) and can be eliminated through establishing a sound sampling protocol. Process variability cannot be controlled unless the sampling interval is reduced. If it is relatively high in relation to *V*[0], almost three times, and it is likely that process variations are responsible for this. *UCL′* and *LCL′* include both the random variability due to the random, and the non-random process variability that takes place between two consecutive samples. These upper and lower control limits combine three standard deviations of the random variability *V*[0] and the process variability *V*[process], to give a 99.7% level of confidence in the control charts. The upper and lower control limits (*UCL′* and *LCL′*) are set in the control chart by multiplying the standard deviation of *V*[0] by 3 in order to cover the 99.7% confidence interval, and then adding the contribution from $\sqrt{V}$[process]. The position of the sampling, measuring and process variability relative to the product stream is shown in the control chart as *UCL′* and *LCL′*.	$V[\text{process}] = V[1] - V[0]$ $= 0.00859 - 0.00719$ $= 0.0014$ $\sqrt{V}[\text{process}] = 0.0374$

V[cyclic]	**Cyclical variability** *V*[cyclic]: the value for *V*[cyclic] is half the total amplitude of the process cycle, between the highest and lowest points on the variogram, usually associated with the first cycle. It is a non-random variable related to specific activity in the process. This variability is introduced as a direct consequence of interventions on, or interactions with, the process stream. It may be related to mechanical or human interventions, but is usually due to periodic changes in the diurnal performance of or maintenance interference with equipment, or due to changes in manpower behaviour or material inputs on the plant. The regularity of both the period and the amplitude of the short-range cycles in the variogram suggest that this effect is introduced by mechanical equipment. The reason for the cycles should be identified and adjustments made to the sampling equipment.	*V*[cyclic] = 0.00886 √*V*[cyclic] = 0.0941 *UCL*″ = +3*S* = 9.41% X *LCL*″ = –3*S* = 8.55% X
V[sill]	**Average variability of the process measured across the total data set** *V*[sill]: *V*[sill] is measured across the entire variogram and is a measure of the total variance in the data set, whereas *V*[trend] is the value of the sill at a given lag. This should be the same as *V*[trend] since they measure the same thing.	*V*[sill] = *V*[trend] = 0.0188% X at *j* = 225 lags
V[trend]	**First order integral of the variogram at any given lag** *V*[trend]: this indicates what the average variability of any individual sample might be at any given lag. Is the difference between *V*[0] and the first integral of the variogram at any lag and is specifically for the purpose of extrapolating the variogram back to the y-axis to obtain a value for *V*[0]. *V*[trend] can also be measured at any lag distance, but usually at the specific lag point where the variogram reaches a maximum. The trend, being the first order integral of the variogram, rises sharply in the early lags of the variogram and flattens out as it approaches the range of influence. Beyond the range of influence, *V*[trend] is more or less constant. It is in fact the geostatistical dispersion variance for a sample of a given support. The trend provides an explanation of the variogram behaviour during the period of the large-scale cycle. This component of variability is due to some mechanical or human intervention that takes place approximately every two to three days and introduces variability into the system. Generally, the trend of the variogram is upwards until a point (the range of influence) is reached, beyond which the variogram is level or declines.	*V*[trend] = 0.0188% X at *j* = 225 lags

Components of variability

The relative variogram allows the components of variability to be standardised and compared against one another as shown in the pie diagram of Figure 9.

The main contributor to the variability is the overall trend (or sill) that accounts for 51% of the variability. This is the height of the sill at the end of the third cycle shown in Figure 7. The reason for the high contribution of *V*[trend] may be a possible strong autocorrelation between one sample and the next because of the mixing of liquids.

The next major component of variability is due to the cyclical nature of the process, which accounts for about 24%. The source of this variability lies in the incorrect delimitation error (*IDE*), the incorrect extraction error (*IEE*), the incorrect preparation error (*IPE*) or the total analytical error (*TAE*), and should be identified and eliminated.

The variability due to the differences between one sample and the next (*V*[1]–*V*[0]) only accounts for about 4% of the variability. This is a relatively small value and it tends to suggest that there is not much in the way of correlation between one sample and the next.

The value of 20% for *V*[0] suggests that there may be issues in regard to the sampling protocol that could be improved to

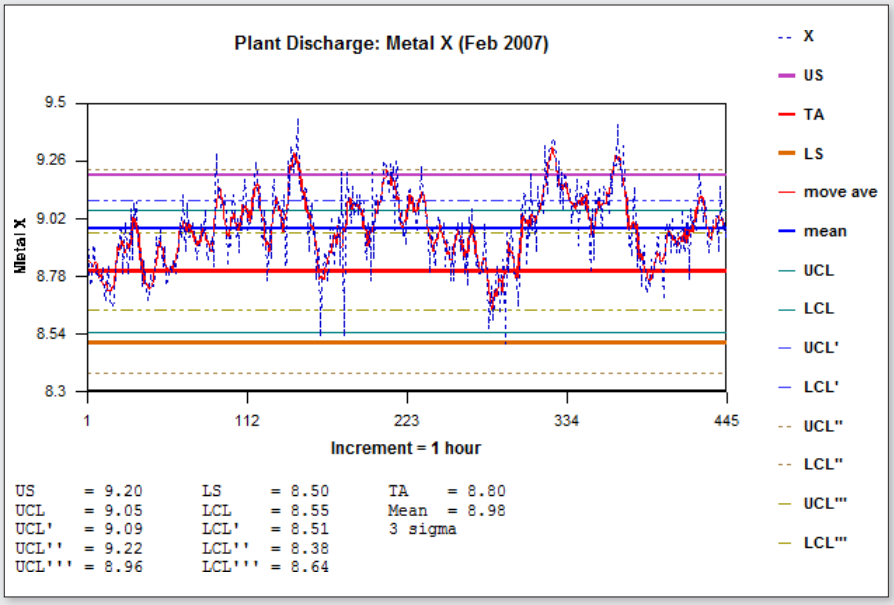

Figure 8. Control chart for metal %X showing lower control limits and lower specification limits.

Table 4. Hierarchy of control limits derived from the variogram.

Component of variability	Variances	Control limits
$V[0]$, the combined *NE*, *FE* and *GE*: UCL and LCL = Mean ± $3\sqrt{V[0]}$	$V[0] = 0.00719$ $S = \sqrt{V[0]} = 0.0848$ $3S = 0.2544\%$ X	$UCL = 8.98 + 3S = 9.23\%$ X $LCL = 8.98 - 3S = 8.73\%$ X
+Plus process allowance: UCL' and LCL' $= UCL \pm \sqrt{V[1]}$ and $LCL \pm \sqrt{V[1]}$ $= \pm\{(3\sqrt{V[0]}) + \sqrt{V[1]}\}$	$3 \times \sqrt{V[0]} = 0.2544\%$ X + $\sqrt{V[1]} = 0.0927$ $= 0.3471$	$UCL' = +3S + V[1] = 8.98 + 0.3471 = 9.33\%$ X $LCL' = -(3S + V[1]) = 8.98 - 0.3471 = 8.63\%$ X
+Plus cyclicity allowance: UCL'' and $LCL'' = \pm\{(3\sqrt{V[0]}) + \sqrt{V[1]} + \sqrt{V[\text{cyclic}]}\}$	$3 \times \sqrt{V[0]} = 0.2544$ + $\sqrt{V[1]} = 0.0927$ + $\sqrt{V[\text{cyclic}]} = 0.0941$ $= 0.4412$	$UCL'' = 3 \times \sqrt{V[0]} + \sqrt{V[1]} + \sqrt{V[\text{cyclic}]} = 8.98 + 0.441 = 9.42\%$ X $LCL'' = 3 \times \sqrt{V[0]} + \sqrt{V[1]} + \sqrt{V[\text{cyclic}]} = 8.98 - 0.441 = 8.54\%$ X

reduce $V[0]$. The main contributors to this sampling error are the nugget effect (NE), the fundamental sampling error (*FSE*), and the grouping and segregation errors (*GSE*), each of which could be investigated to identify the main contributor to this error.

Conclusions from the control chart

A control chart that compares the variability of metal% X against the sources of sampling error and control limits is shown in Figure 8. The main conclusion from the chart is that the lower specification limit is set appropriately. The sampling interval is more than adequate and management may even think of reducing the sampling frequency. Even though the sampling procedures and protocol may require some more attention, the plant operators should be able to control the process well. It is essential that the dynamic characteristics of the process be acknowledged, and that the reason for the cyclical behaviour be identified and eliminated. Under present conditions, it is unlikely that large invisible financial losses are occurring.

The legacy of Pierre Gy

While history turned out so that Pierre Gy only attended the first of what was to become the biannual WCSB Conferences (but happily in the company of his wife Sylvia, see *TOS Forum* Issue 5), these meetings of sampling practitioners, engineers, scientists and enthusiasts have acted to consolidate the research and application of sampling theory and practice at a global level and have presented a regular stream of high quality research since 2003. The global sampling fraternity owes much to those who initiated, contributed and have continued to organise and support these outstanding conferences.

Pierre Gy passed away on 5 November 2015, but his legacy lives on with force. His tireless work, depth of insight and completeness of scope of sampling issues from 1949 to 2015, a period of 66 years, established a formidable foundation that ensures that sampling of materials will remain a fertile field of research—and of a superior informed practise—for ever. The Theory of Sampling (TOS) provides the only complete scientific basis for sampling, including a basis for relevant standards and guidelines, and will prove itself to all who will take the time to investigate the solutions that this logical, modular approach to sampling problems offers. The ideas and approach taken by Gy in adopting Matheron's variogram concept in its application to problems associated with process control is a special feature of TOS and led to the field of study referred to as chronostatistics and, as an extension, to the logical, coherent theory of bed blending. A seminal paper in which Matheron himself assessed

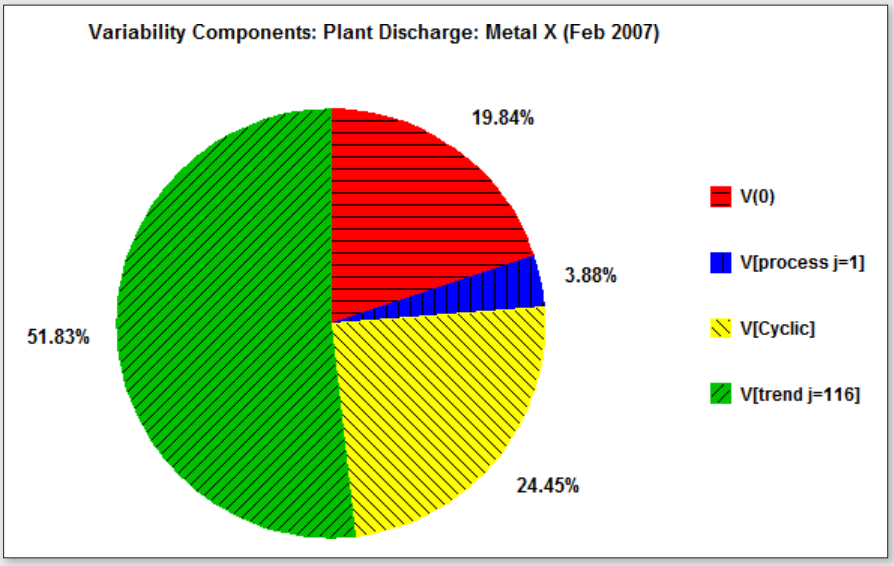

Figure 9. Components of variability in the sampling process for metal% X.

the validity of Gy's application of the variogram approach to linear sampling was a highlight of WCSB7, Bordeaux, 2015 and can be found in the Proceedings of the meeting.

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References

1. P. Gy, Pierre, "Part IV: 50 years of sampling theory—a personal history", *Chemometr. Intell. Lab. Syst.* **74,** 49–60 (2004). doi: https://doi.org/10.1016/j.chemolab.2004.05.014
2. D.W. Brunton, "The theory and practice of ore-sampling", *Trans. AIME* **XXV,** 826–844 (Atlanta Meeting of October 1895) (1896).
3. R. Richards, *Ore Dressing*, Volume 2. McGraw-Hill (the sampling table is on page 850) (1908). https://www.brainshark.com/malvern/vu?pi=192277776&text=M021507
4. D. Francois-Bongarçon, *Extensions to the Demonstration of Gy's Formula* (January 1998).
5. P. Gy, "L'Echantillonnage des Minerais en Vrac" (Sampling of Particulate Materials), *Revue de Industrie Minérale* (St Etienne, France) Numéro Special (Special issue) (15 Janvier 1967).
6. G. Matheron, *Les Variables regionalisees et leur estimation* (Regionalized Variables and their Estimation). PhD thesis, Masson, Paris (1965).
7. M. David, *Handbook of Applied Advanced Geostatistical Ore Reserve Estimation*. Elsevier, Amsterdam (1988).
8. P. Gy, "Sampling of discrete materials-a new introduction to the theory of sampling. I. Qualitative approach", *Chemometr. Intell. Lab. Syst.* **74,** 7–24 (2004). doi: https://doi.org/10.1016/s0169-7439(04)00167-4
9. DS 3077, *DS 3077. Representative Sampling—Horizontal Standard*". Danish Standards (2013), www.ds.dk
10. P. Gy, *Heterogeneité, Echantillonnage, Homogeneisation* (Heterogeneity, Sampling, Homogenising). Masson, Paris (1988). Complete textbook in French
11. P. Wavrer, "An automatic linear proportional sampler based on the principles of the Theory of Sampling", *TOS Forum* **Issue 6,** 25–27 (2016). doi: https://doi.org/10.1255/tosf.85
12. R. Wohlbier, "Stacking, Blending, Reclaiming", in *Bed Blending*, Ed by A.W. Gerstel. Trans-Tech Publications, Clausthal, Germany, Ch. 24 (1977).
13. P. Gy, *Theorie de la Prehomogeneisation derivee de la Theorie de l'Echantillonnage* (Theory of Bed-Blending derived from the Theory of Sampling). Industrie Minerale-Les Techniques (1981).
14. P. Gy, "A new theory of bed-blending derived from the Theory of Sampling—development and full-scale experimental check", *Int. J. Miner. Process.* **8,** 201–238 (1981). doi: https://doi.org/10.1016/0301-7516(81)90013-2
15. P. Gy, *Optimisation de la Strategie d'Exploitation d'un ensemble Mine-Metallurgie ou Carriere(s)-Cimenterie* (Optimising the Operational Strategy of a Mine-Metallurgy or Quarry-Cement Works Complex). Mines et Carrieres-Les Techniques, pp. 118–124 (1998).
16. P. Gy, "Optimizing the operational strategy of a mine metallurgy or quarry-cement works complex", *Explor. Min. Geol.* **7(1&2),** 175–180 (1998).
17. F.F. Pitard, *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness and Statistical Process Control*, 2nd Edn. CRC Press (1993). ISBN: 0-8493-8917-8
18. P. Gy, *Sampling for Analytical Purposes*, Translated by A. Royle. John Wiley, Chichester (1998); 2nd Edn (1999).
19. K.H. Esbensen and P. Minkkinen (Eds), WCSB1 Proceedings, Special Issue of *Chemometr. Intell. Lab. Syst.* **74(1),** (2004).
20. P. Gy, "Sampling of discrete materials II. Quantitative approach—sampling of zero-dimensional objects", *Chemometr. Intell. Lab. Syst.* **74,** 25–38 (2004).
21. P. Gy, "Sampling of discrete materials III. Quantitative approach—sampling of one-dimensional objects", *Chemometr. Intell. Lab. Syst.* **74,** 39–47 (2004).
22. F.F. Pitard, *The Theory and Practice of Sampling*. A short course presented through the Geostatistical Society of South Africa (GSSA) in the School of Mining Engineering, University of the Witwatersrand (2006). See also Reference 17.

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continued from page 3

introduction further outlines the motivations of the *ad hoc, pro temp* committee behind the proposal. It will be vital for the future of the sampling community that all have read, contemplated and made up their minds as to this proposal, which will form the background for an inaugural assembly at WCSB8. It is important that the proposal keeps the by-laws to an absolute minimum. Check it out…

In the beginning of a year of a WCSB there will be an unavoidable hesitation to submit manuscripts to *TOS Forum*—there will be a quite natural wish to present contributions at our biannual conference, and to publish in the Proceedings. In spite of this the influx of features for the Forum is quite satisfactory, commensurate with two to three issues per year. Still, as always, the Editor would be remiss in his duties if he did not issue the obligatory "Call to Arms". There is a place for our biannual gatherings, the highlight of our dispersed scientific and technological community, and there is a time for similar interaction and communication between all its members in the intervening two years. *TOS Forum* will be there expressly for this purpose. All communication does not necessarily have to be in the form of fully fledged scientific papers—there is a place for other contributions too. Please view the present issue of *TOS Forum* in this light. The Editor sincerely hopes that the efforts of the authors of the present features will be inspirational for many others. The next issue of *TOS Forum* is planned for September/October 2017. Will it contain a contribution from... you?

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Improvement practices in process industry—the link between process control, variography and measurement system analysis

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In modern process industry it is necessary to find "smart" ways to continuously satisfy customers' demands, strive for more efficient production and prioritise the focus on safety, health and environment, allowing dedicated companies to obtain a competitive advantage. To achieve individual goals in these areas, companies increasingly implement a so-called business system approach, which ensures systematic, continuous improvement initiatives in all sectors. Most business systems address improvement in process control, since in many cases there is a significant positive economic potential which often only requires minor capital investments. This is also the case for Glencore Nikkelverk, Norway, where it has been decided to transform a former project-based improvement framework into a holistic business system, the Nikkelverk Business System (NBS). One pillar consists of different tools to improve process performance, with the objective to identify critical processes and process stages a.o. and also to ensure that the measurement systems in use can be geared, and validated, to mainly capture process variations, allowing to document valid process improvements. At Nikkelverk there are between 6000 and 7000 measurement points, of which one-third are used for process regulation purposes. It is therefore critically important to be able to monitor and verify that measurement system uncertainty is suitably low—ideally at a level of 10% relative or below compared to the variation of the process being monitored. This will become a key priority. An optimal measurement system should therefore not contribute significantly to the total apparent process variation as revealed by raw process data that are used to track when a process is deviating from steady state or dangerously close to control limits, or gets outside relevant safety limits. Critical processes must be kept under constant monitoring and control to ensure that all improvement attempts work from a solid database. As but two examples, energy and material consumption, can be fine-tuned to a lowest possible level *only* when both processes themselves and the measurement systems are monitored and controlled properly. This not only contributes to the cost efficiency of the company, but enforces process monitoring and control to take a leading role. We here focus specifically on the relative merits of variographic analysis (Theory of Sampling) and measurement system analysis (Six Sigma) and show how the former can function as a very effective screening for the much more costly latter.

Historical background

Since the 1930s, processes have mainly been monitored by control charting, starting out with the well-known Shewhart chart, a concept of quality control in manufacturing which was first advanced by Walter Shewhart.[1–4] Many graphical methods have been developed under the umbrella of "statistical process control" (SPC), for example the Quesenberry chart, Moving Range chart, EMWA chart etc., which all have the purpose to make room for the voice of the process. But with such a wide range of possible methods, one can at times be bewildered instead of informed. It is quite a task to find suitable (and simple) tools that give you good enough results to work on for continuous improvement of key processes.

It should not, however, be necessary to be an expert in statistics to be able to improve an industrial process. Tools and methods used must be relatively easy to grasp for process technicians, enabling involvement of as many stakeholders as possible to create a common understanding of the current process state. Hence the best analysis is the one that provides the greatest insight with the simplest approach. But this organisational challenge will not be further discussed in this paper, which instead focuses on two salient technical issues, measurement system analysis (MSA) and variographic process characterisation.

Introduction

Glencore Nikkelverk is situated on the seaside of the city of Kristiansand at the south tip of Norway. Since 1910, nickel and other metals have been refined, produced and exported from the production plant in Kristiansand. Nikkelverk's history ties in with the development of industrialisation in Norway at the beginning of the 1900s. Today, Nikkelverk produces 92,000 t of nickel, 39,000 t of copper, 4700 t of cobalt and 115,000 t of sulphuric acid per year, and is one of the most efficient nickel refineries in the world. In the process industry, stabilisation and control of the process is a key factor to continue production as a sustainable company.

Proper monitoring and control of the measurement system also play important roles for all industrial process performance. Therefore, the magnitude of the contribution to the total observable process variation stemming from the measurement system itself has to be known in order for it to be compensated for when trying to perform the most efficient process control. This can be determined by conducting a measurement system analysis (MSA) in which one splits the measurement system into suitable compartments and determines their individual specific variation by involving several operators to repeat the same procedure several times over. The variation stemming from the measurement system should be kept to a minimum in order to be able to achieve production under stable conditions.

Measurement system analysis is a vital part of the Six Sigma philosophy, which was introduced at Nikkelverk in the first years of the current millennium. Experiences from

this period were that the MSA process was rather cumbersome and burdened the operators with significant additional workloads. Application of the knowledge obtained by a MSA study was also sometimes questionable. It was estimated that the time spent for one MSA could range between one-half and three days, excluding reporting. Approximately 2000 measurement points/systems are currently used in process regulation at Nikkelverk; application to all would therefore lead to a resource expenditure of several years purely for MSA fieldwork. This does not seem a feasible work load as seen from a management point of view and it is certainly even harder to sell this level of many MSA studies to the personnel involved. Even if we only focus on the critical processes identified, the number of MSA studies necessary would be overwhelming. Something had to give—enter variographic characterisation. Notably also, apart from being a very time-consuming task, the result from such studies only reveal the variation of the measurement system at the moment of the test. Concluding that the measurement system is not a significant contributor to the process variation based on single MSA studies therefore runs risks with respect to generalisation over long production periods.

What is the objective of a MSA study? The goal is to record process facts in a database with which to evaluate the adequacy of a particular working measurement system in relation to getting access to the reliable information regarding the process variability proper.

What if one can in fact do this quality check for each measurement system only, or mainly, by using online data and/or the extensive historical process database?

What if one could initially classify measurement systems as capable, conditionally capable or capable, compared to the process variation only involving stored *online data*?

What if this type of control on the measurement systems could be performed automatically, on *routine* on-line process data?

Such an approach has in fact existed for quite a while, as used within *geostatistics* and within the *Theory of Sampling* (TOS) regimen; an approach called *variographics*. At Nikkelverk we see variography as a skilled detective for both processes measurement and process characterisation. The following sections will outline and explain *why*.

Variography vs MSA

The first issue on the agenda is to clarify the difference between variographics and a MSA study in order to describe how they may complement each other in relation to improving industrial processes.

The variogram—absolute and relative

Figure 1 shows a simple example of an increasing variogram and how the lag distance affects the resolution of observable process variability. To develop a variogram one needs a time series with equidistant sampling. By calculating the average of the variances characterising *pairs of observations* spaced by different *lags* (distance between samples) a variogram is obtained with relative ease. Several literature resources exist which explain how to do this and how to interpret variograms,[5–8] making it unnecessary to repeat this here.

The master formula for an *absolute variogram* based on absolute concentration values is:

$$V_a(j) = \frac{1}{2(N_u - j) \bullet a_L^2} \sum_m \left(a_{m+j} - a_m\right)^2 \quad (1)$$

where $(N_u - j)$ is the number of sample pairs and N_u the total number of datapoints; j is the contemporary *lag* distance; a_m is the concentration value at position m in the time series and a_{m+j} is the value of the datapoint at position $(m+j)$. The squared estimated average a_L from all data is part of the denominator in order to scale the variance measure for lag j, so that $V(j)$ becomes a unitless variance estimate.

The formula for the relative variogram based on *heterogeneity contributions* at each sampling location in the measurement series, h_j, is shown in equation 2:

$$V_r(j) = \frac{1}{2(N_u - j)} \sum_m \left(h_{m+j} - h_m\right)^2 \quad (2)$$

The absolute variogram, $V_a(j)$, is convenient when comparing the information with actual measurements and specification

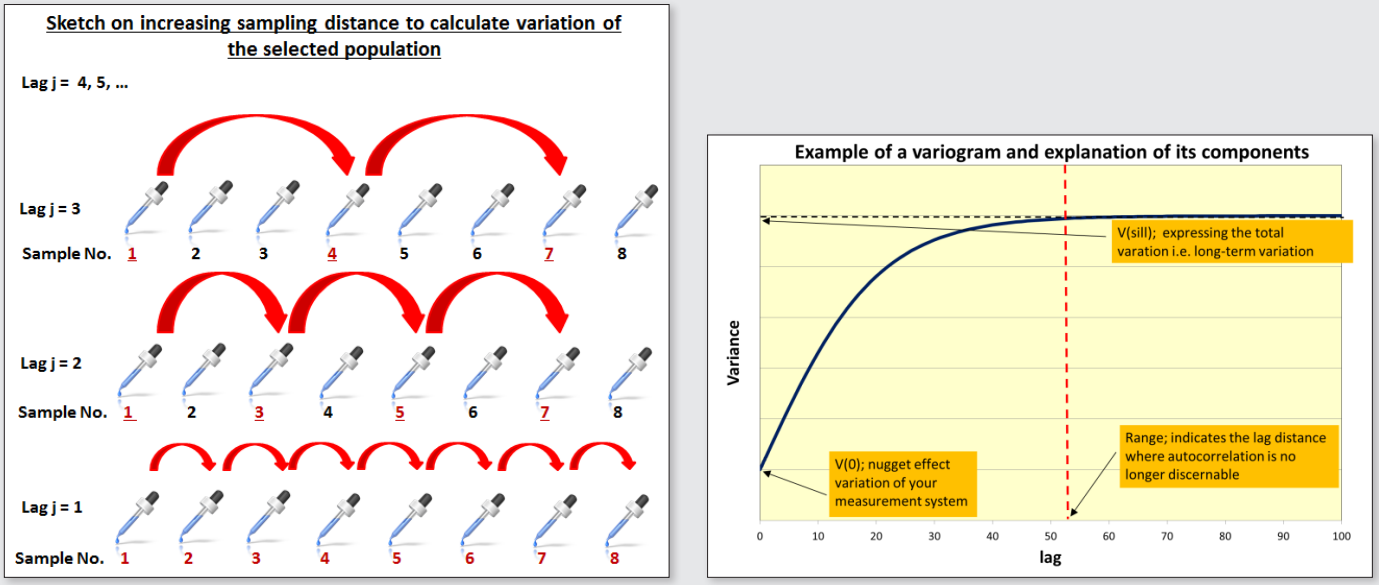

Figure 1. Example of an increasing variogram (right), illustrating the nugget effect, $V(0)$, the range and the $V(\text{sill})$ as well as how the contemporary lag distance reflect the sampling resolution employed.[5–8]

limits, while the relative variogram, $V_r(j)$, is more suitable when comparing process information from one variogram with another; for example, from two (or more) measurement locations/measurement systems or from two variograms at different process times.

The variogram example in Figure 1 shows $V(0)$, the *nugget effect*, which expresses the total error variance of the measurement system (containing contributions from all incorrect sampling errors, all correct sampling errors as well as the total analytical error).[5–8] After a certain lag distance (about lag 50 in the example presented), termed the *range*, the variogram approaches the total variation of the process, $V(\text{sill})$, also called the long-term variation. This level is called the variogram *sill*.

There are further sources of variation, e.g. process cycles and/or linear trends, which can be deduced (as well as their proportion of the total variances) and quantitatively estimated from a full variographic analysis. These variance source effects are easily incorporated in the subject matter described here. However, these variations are not the focus of this article and will not be addressed further here; see Reference 8 for details.

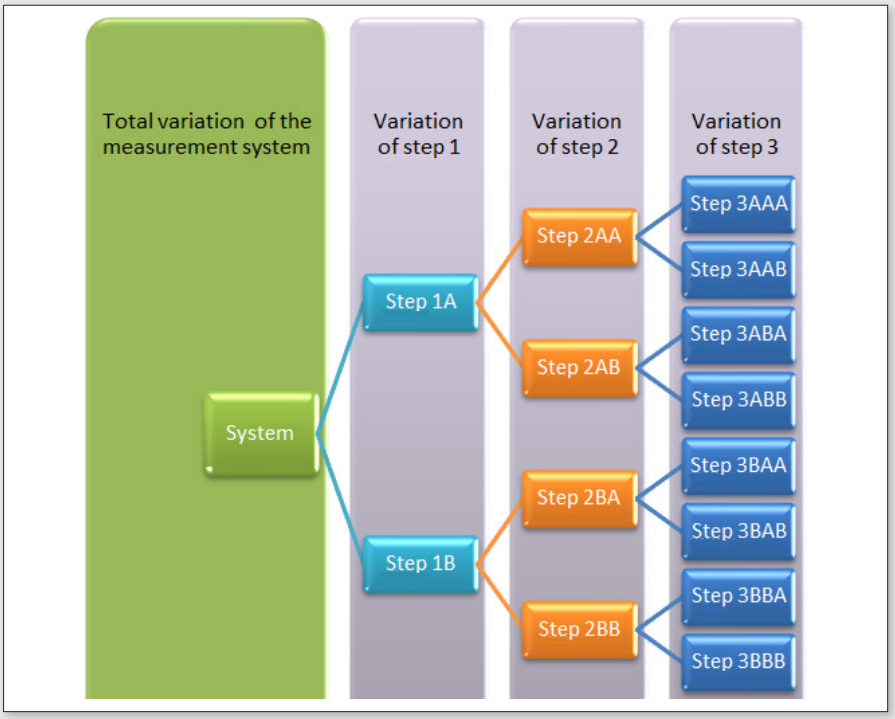

Figure 2. Generic illustration of a MSA study where *parallel samples* for each stage allow determination of the effective variations ascribable to each step. The illustration amounts to a duplicate split design, but more intense sampling replication is an option as well.

Measurement system analysis (MSA)

The purpose of a measurement system analysis (MSA) is to evaluate and verify the measurement system by quantifying its accuracy, precision and stability.[9] Thereby one is able to check if the variation of the measurement system is "small" compared to the process variations themselves. Depending on the industry sector and the specific process involved, an often stated rule-of-thumb is that the ratio between measurement system variation and process variation will be lower, or between 1/10 and 3/10.

If one wants to control process variation using a particular measurement system as a basis for decision making, process data should of course not be overshadowed by variations stemming from the measurement system. A MSA study is a vital part of a Six Sigma project. There are several approaches on how to conduct a MSA. For a study on variations regarding a sampling system which delivers samples to a laboratory for analysis, the steps sketched in Figure 2 can, for example, be the sampling procedure (step 1), the sampling reduction (step 2) and the sample preparation (step 3). Additionally, a step 4 would be the analytical instrument on which the samples are analysed.

In a MSA the measurement system is divided into sequential steps and parallel samples (replicates) are obtained to identify the resulting variations in each step. Care must be observed that all variance contributing factors are taken into account in the respective steps, for example operator errors, sampling errors, crushing and weighing errors etc. all of which contribute to the total observable variation.

This master design can be done in different optional ways, for example by using more parallels at each stage. The designer of the MSA in collaboration with the operative personnel has to decide why and when what level of replication is necessary.

Observe that this general MSA setup is equally applicable if the "sampling" consists of a direct process measurement instead of extricating a sample to be analysed in the laboratory. Esbensen and Paasch-Mortensen describe the duality of such process sampling with the physical sampling extraction, and show that there are no essential differences, both approaches give rise to the potential full complement of sampling errors (incorrect sampling as well as correct sampling errors).[6]

Another way to set up a MSA would be to focus on the reproducibility and repeatability of the final analytical results when different operators, instruments or other devices are used. The possible sources of the total observed process variation can then be decomposed as:

$$\sigma^2_{\text{Observed Process}} = \sigma^2_{\text{Actual Process}} + \sigma^2_{\text{Measurement System}}$$

$$\sigma^2_{\text{Measurement System}} = \sigma^2_{\text{Repeatability}} + \sigma^2_{\text{Reproducibility}}$$

where the variation due to *repeatability* and *reproducibility* are considered as the main contributors to the variation of the measurement system. The results of these kinds of MSA are often reported as "Gauge R&R" numbers. The stability and accuracy of the analytical measurement system can be determined by the use of *control samples* and/or *certified reference materials* where available. It is here necessary to distinguish between the analytical part of the measurement system in the strict sense and the total, effective measurement system, which specifically also includes the sampling system(s).[†]

[†]Observe that these attributes are only defined for *analytical* methods/analytical systems, but that there is no carrying-over option to the *sampling* process, see Esbensen & Wagner (2014),[10] who describe these crucial differences in full detail.

For a MSA there are several established statistical tools *(linearity, stability, gauge repeatability and reproducibility, GRR)* that can be deployed to ensure that measurement setups are within acceptable conditions to meet manufacturing capabilities and customer requirements. Observe, however, that this approach does not recognise issues arising from sampling process variances. The central issue is that TOS shows how the influence of so-called "incorrect sampling errors" lead to a *sampling bias* which is **inconstant**, and which therefore cannot be straightjacketed into the standard analytical variance decomposition scheme above, see References 6 and 10.

Variographics and MSA complement one another

At Nikkelverk we would like to use the variogram as a *screening and grading tool* of the measurement system performance in order to decide if application of the tedious MSA is necessary. Figure 3 shows where the variogram and the MSA work well individually and where their scope coincides. Figure 3 shows that the total observed process variance, either delineated by a variogram or via a MSA can be decomposed.

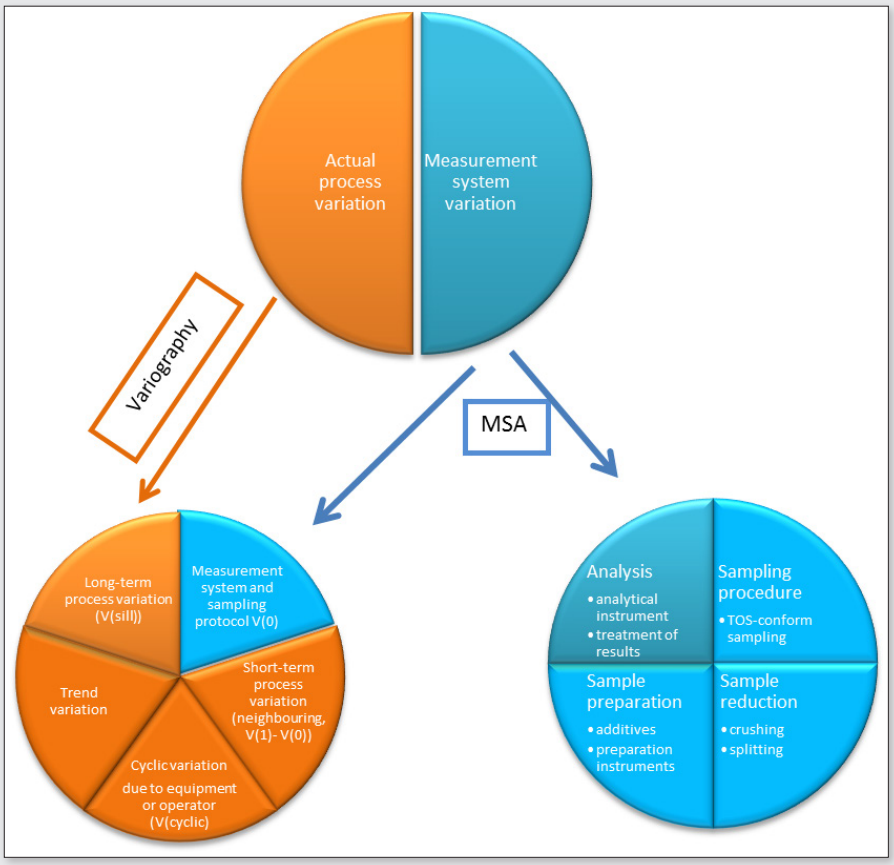

Figure 3. Different variance components as revealed by the variographic and MSA, respectively. Variographics gives an overview on all variance components of the process system, while MSA specifically allows detailed information on all steps involving the sampling and analysis procedure, with the proviso that *all* incorrect sampling errors (sampling bias) can be assumed to be *absent*. However, this is far from always a realistic assumption.[6,10]

Examples of effectively categorising measurement system conditions

The first step in a MSA is to understand how the results appear under normal operating conditions (NOC). In the software used for this feature this can be done either by looking at the time series and checking the distribution pattern of the data series by means of a histogram or by comparing a Q–Q plot against a fitted normal distribution.

It is recommended to test the measurement system with a slightly oversampled unit lag distance when performing a variographic characterisation; in this way an optimal correct lag distance can be derived.[6] This optimised lag distance should then be used when calculations are performed to determine the status on the measurement system in question (each day, each week or whatever is appropriate to the process). Below we present three examples of how variography can be applied to check particular measurement systems at Nikkelverk.

pH measurements

Measurement of pH is one of the most important measurements at Nikkelverk (where nickel refining is performed by a complex hydro metallurgical process) and therefore serves as a major indicator. Pilot studies of the use of variograms have been started regarding monitoring of the variation stemming from the measurement system itself. The software used for detailed study to calculate variograms is EMPV (Effective Management of Process Variability), from Francis Pitard Sampling Consultants.

There are many process steps at Nikkelverk which are controlled by pH measurements. A typical sampling station is presented in Figure 4a. The electrodes are immersed in the process reactor solutions, and they are cleaned every hour by lifting the electrodes into an acidic solution (Figure 4b).

Figure 5 shows a time series plot of online pH measurements at reactor 1, and a conventional statistical distribution of the measurement data from the selected time series is shown in Figure 6. The lag "distance" is 4 minutes and 19 seconds, corresponding to a complete series of 72 hours; the 1000 measurements used here were obtained from the historical process system database.

The resulting variogram (absolute variogram) is shown in Figure 7. The sill, $V(\text{sill})$ is defined by the horizontal dashed line in black. The variance for the measurement system, $V(0)$, is defined the intersection of the back-projected curve with the y-axis corresponding to lag = 0.

pH measurements obtained for six different weeks with the same time periode of 72 hours are extracted from the process database for reactors 1 and 2. The variograms of all weeks for each reactor are normalised with respect to the largest sill variance allowing all variograms to be presented and easily compared in one graph, as shown in Figure 8 for reactor 1 and Figure 10 for reactor 2. The coloured areas in the graphs indicate if the variance of the measurement system is well below company threshold limits to ensure that the process is the main contributor to the observed variations. These characterisations follow the Six Sigma approach.

Figure 4. a: Sampling station for pH measurement; b: lifted pH electrodes during the cleaning cycle.

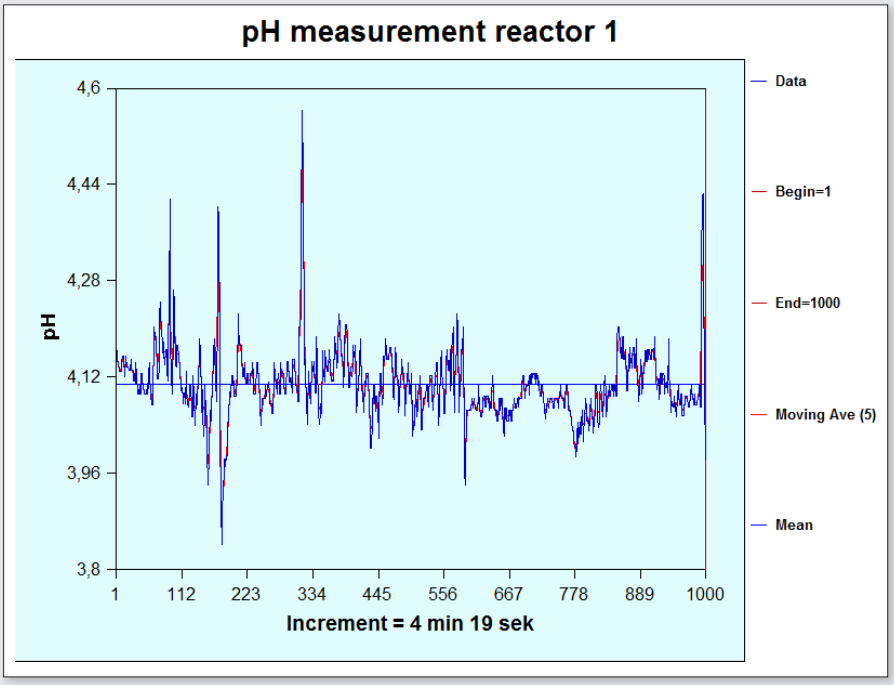

Figure 5. Time series plot of pH measurement of reactor 1 for three consecutive days in week 49, 2015.

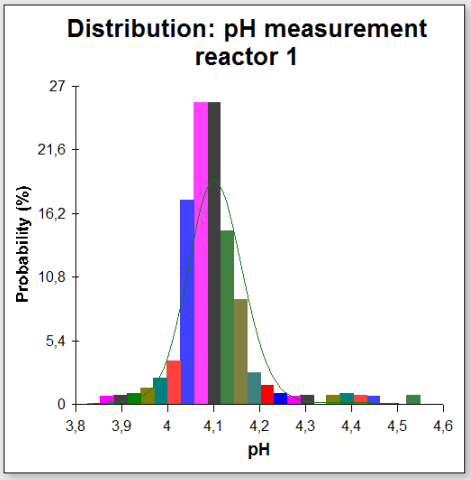

Figure 6. Distribution of the pH measurements presented in Figure 10.

In the study at Nikkelverk, classification of the different capability levels of a measurement system follows from terms used in MSA studies, Figure 9. The measurement systems for pH are therefore classified either as "capable" (green area) where the variation of the measurement system, $V(0)$, is 10% or below compared to the process variation, "conditionally capable" (yellow area) where the variation of the measurement system is between 10% and 30% relative to the process variation and "not capable" (red area) where the measurement system variation is above 30% compared to the process variation.

The picture of the sampling station for the pH electrodes, Figure 4a, shows several measurement points for the different reactors. Spillage of solution from one to another measurement system is unfortunately not

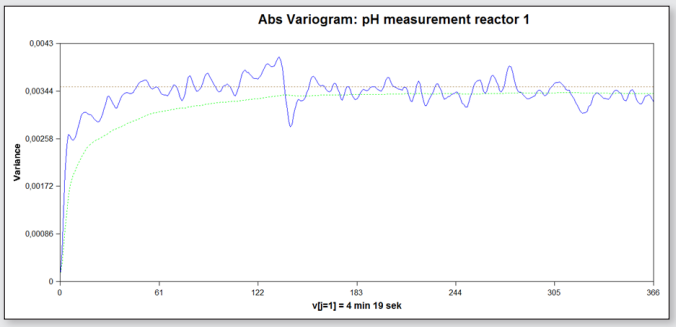

Figure 7. Absolute variogram of pH measurements presented in Figure 5. The green dashed line represents the first order integral W that is used to extrapolate the variogram back to *V*[0] and the red dashed line represents the total variance of your process, *V*(sill).

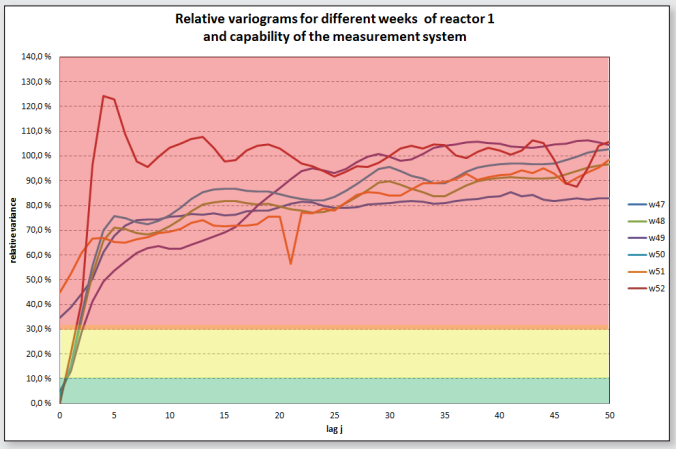

Figure 8. Six consecutive one-week variograms of pH measurements in reactor 1. The variance (y-axis) is normalised with regards to the largest *V*(sill).

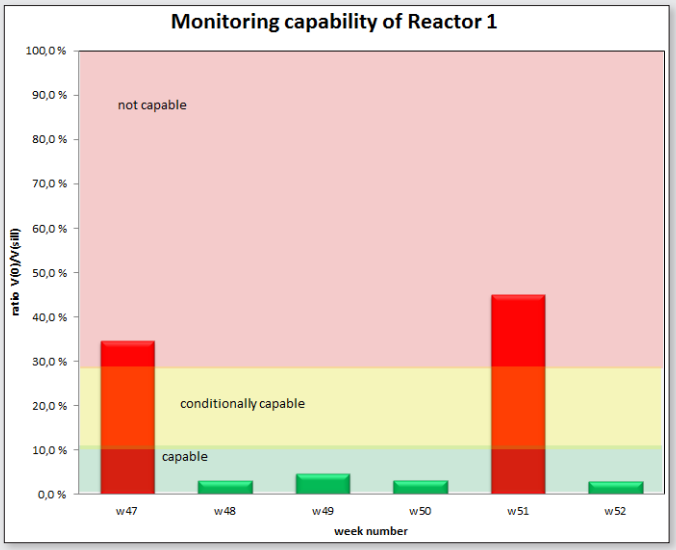

Figure 9. Measurement system *conditions* of pH in reactor 1 presented as ratio of *V*(0)/*V*(sill) per week showing capable, conditionally capable and not capable conditions following the Six Sigma approach.

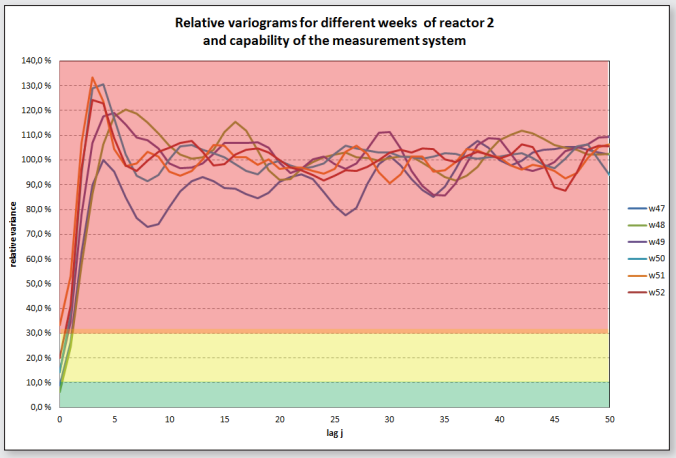

Figure 10. Variograms of pH measurements in reactor 2 per week presented over 6 weeks—one variogram for each week. This presentation uses the relative variograms.

completely ruled out, by an inconvenient design (it is, however, minimised). If something is wrong in the sampling station and the monitoring of the capability shows that the capability is in the red zone for parallel positions, this might be an indication of cross-contamination by spillage and can be checked by visual observation as well as corrected for.

Alternative variographic characterisation of the measurement system

DS 3077 (2013),[5] Esbensen and Romanach[11] describe how the fraction of *V*(0) of the sill variance level, expressed as a percentage, can be used as a grading facility for the measurement system delivering the on-line time series involved. In this approach a single threshold demarcation is recommended; measurements systems must not give rise to a *V*(0)/sill fraction larger than 30% in order for the measurement system to be reliable for valid insight into the process variances proper.

This furthers an alternative on-line measurement system characterisation that does not involve an experimental design and active intervention, as is the case for MSA. The six-week time series for pH measurements from reactor 2 is shown in Figure 10, here based on the relative variograms. It is easy to augment the variographic threshold 30% with the three-fold variance fraction classification system employed in MSA, as is shown in Figure 11.

From Figure 9 it is observed that for both weeks 47 and week 51 the measurement system in reactor 1 contributed to well above the specified limits for "not capable". For reactor 2, Figure 11, this is only the case in week 51, while the variation of the measurement system seems to be at least "conditionally capable" in weeks 49, 50 and 52 or even "capable" in week 47 and 48.

Whether to use absolute or relative variograms can be debated, but in the present context both will lead to a measurement system classification that can be appreciated within the three-fold MSA brackets. The main issue is that it is possible to *grade* any process measurement system based directly on routine on-line process data. This is a huge efficiency improvement of the

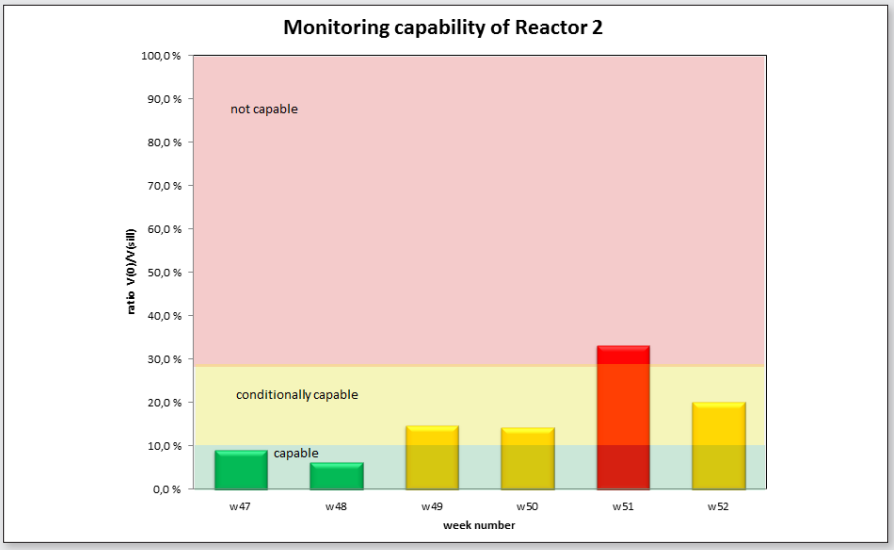

Figure 11. Measurement system condition of pH in reactor 2 presented as ratio of *V*(0)/*V*(sill) per week. The three-level grading classification system is adopted from Six Sigma MSA.

measurement system check over having to instigate the cumbersome full MSA procedure for all measurement locations.

Online process analysis of Pb

There are different chemical elements which must be monitored closely to ensure high quality products at the end of Nikkelverk's production line. One of these elements is lead, which has to comply with very strict, low concentration specifications in the final nickel product. Formerly, lead was monitored by using polarography with mercury electrodes. However, recent restrictions by the authorities on using mercury in workplaces started a project in which a new method for lead analysis should be found.

One of the tentative new test systems is show in Figure 12, which is installed close to the process in the so-called at-line configuration. There are several systems installed through the full process line and two examples of process data for reactor 1 and 2 are shown in Figure 12.

The sample is withdrawn in batch mode from the process, with a time interval of several minutes. The measurements are recorded continuously in the company database.

For reactor 1, a time series of 7 days is presented in Figure 13. Due to known external disturbances in the system, the first 120 measurements have been excludeded from the calculations producing the variogram.

Figure 12. Newly tested alternative at-line voltametric cell for measurement of lead.

Since the measurement period is several minutes, a lag distance of 20 minutes was chosen to be used for the variogram shown in Figure 14 in concordance with the intimate general process knowledge accumulated over several decades.

For reactor 2 the same time frame and starting date are used to draw the time series shown in Figure 15. As for reactor 1 the time series for reactor 2 was also reduced to 380 measurement points, here to eliminate the highly irregular outlier shown in Figure 15. The resulting variogram is shown in Figure 16.

From these variographic analyses it easily appreciated that i) both measurement systems operate with a low *V*(0) relative to the sill level (very clearly below 10%), which is highly satisfactory; and ii) the largest contributor to the overall process variance is a cyclic phenomenon. So the challenge

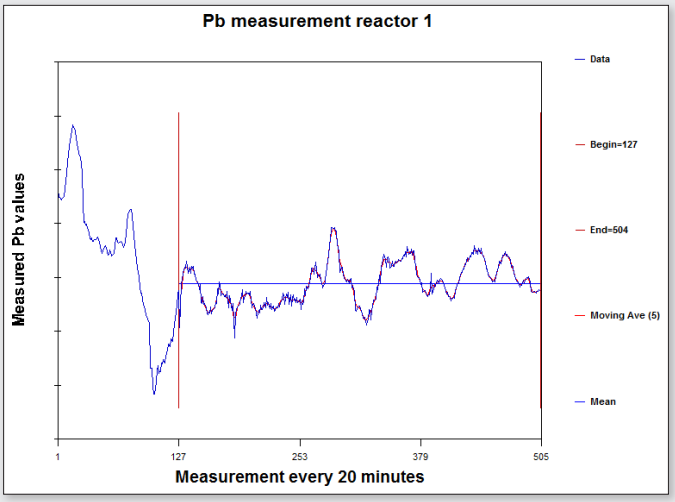

Figure 13. Time series of Pb measurement in reactor 1 (the first 120 minutes were excluded due to know irregularities).

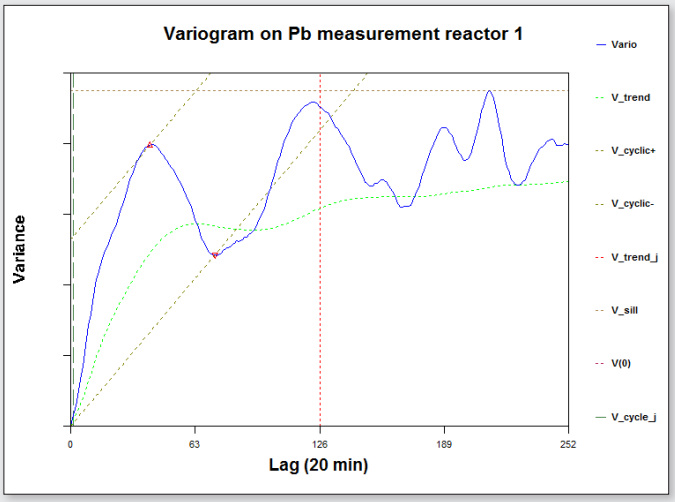

Figure 14. Relative variogram on Pb measurement in reactor 1 with lag distance 20 minutes.

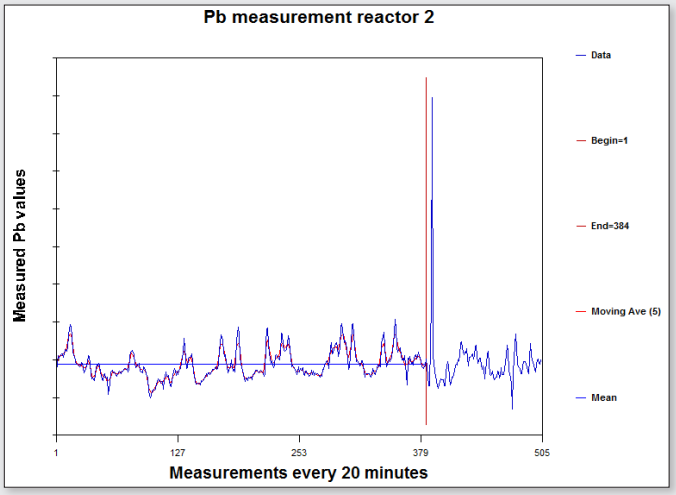

Figure 15. Time series of Pb measurement in reactor 2.

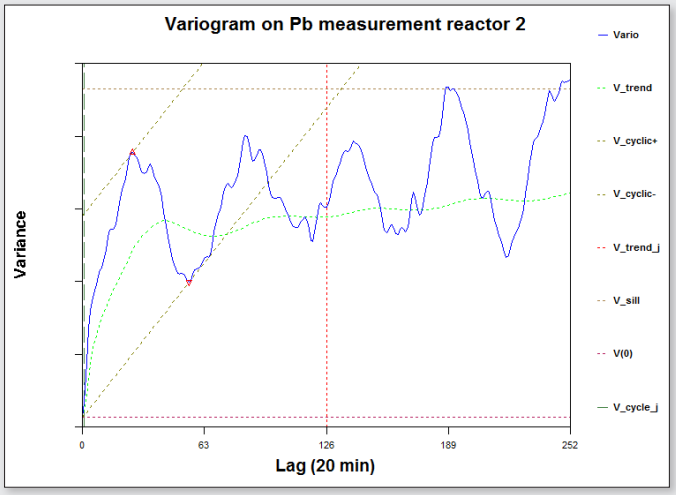

Figure 16. Relative variogram on Pb measurement in reactor 2 with lag distance of 20 minutes.

for this new alternative lead analysis is not the measurement system performance but the observed process cyclicity with a frequency of 20–22 hours. It is critical to identify the root cause of this behaviour since it contributes with ~50% to the total process variation. Nevertheless, the intention to use the *V*(0)/sill ratio to express the condition of the measurement system can still be upheld as it is not influenced by the large cyclicity. For the two reactors, the lead measurement systems are both capable due to both *V*(0)/*V*(sill) being considerably smaller than 10%.

This verification of course has to be repeated after the observed cyclicity is reduced or has been completely removed from the process.

Redox measurement

As for pH measurement, redox measurements are used extensively to determine the status of the process. The measured mV signal gives an indication of the degree of process reaction equilibrium. The redox electrodes are installed at the same sampling station as the pH electrodes (Figure 4a). For reactor 1 a time series of a mV signal from a redox electrode is shown in Figure 17. As for the other examples it is important to check whether the data series includes significant shifts or if pervasive trends are present during the measurement interval.

The variogram of the redox measurements in Figure 18 show a cyclic behaviour with a distinct periodicity of one hour which is clearly a representation of the washing cycle of the bank of electrodes. The intersection with the y-axis determining *V*(0) is again low compared to the total process variation; the ratio is ~8% which signifies a "capable" status of the redox probe at the time.

Synoptic overview of measurement system status

To illustrate the overall benefits of using the rapid on-line variographic measurement system, data series for time frames of three day's duration were extracted from the reactor 2 process database, resulting in seven separate time series equal to the example presented for the pH measurements. The resulting capability classification is shown in Figure 19.

In Figure 19, as well as Figures 9 and 11 for the pH measurements, the ratio between *V*(0) representing the variation of the measurement system and *V*(sill) representing the variation of the process are shown and company-developed MSA terms applied to the three ratio levels: <10%, 10–30% and >30% following the Six Sigma approach.

This is the same measurement system *quality index* approach described in DS 3077,[5] with the mandate that this index

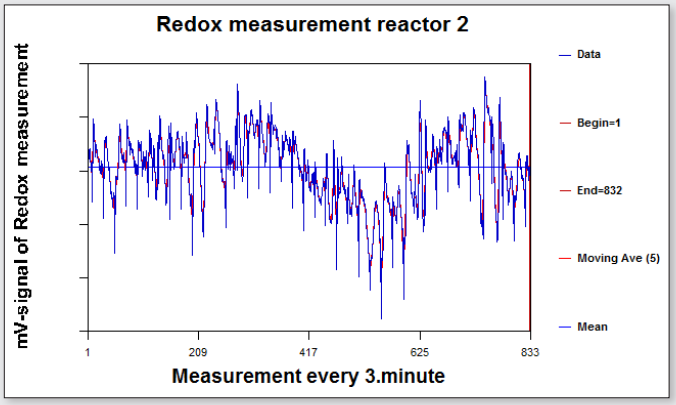

Figure 17. Time series of redox measurement in reactor 2.

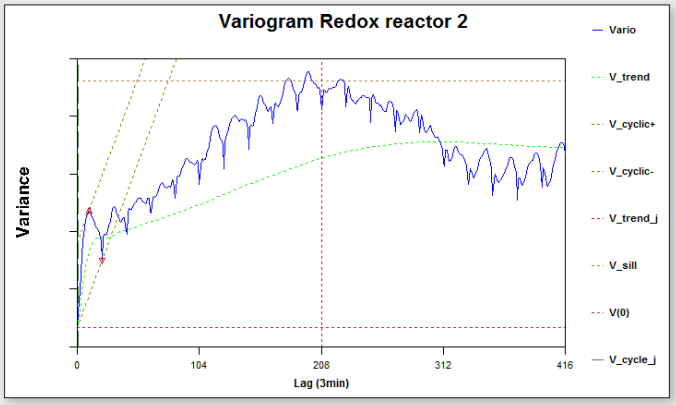

Figure 18. Variogram of redox measurement in reactor 2 shown in Figure 17.

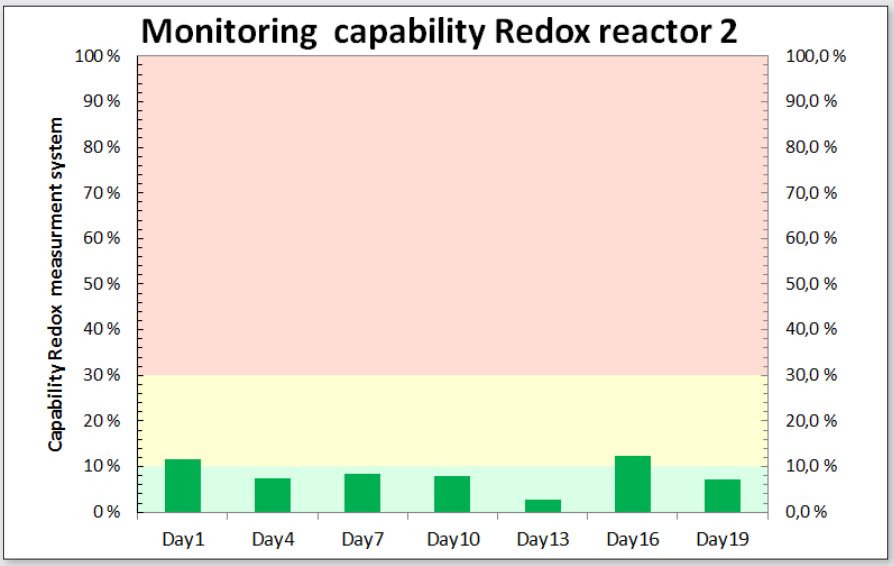

Figure 19. Monitoring overview graph to classify the capability of the redox measurement for reactor 2 with a time interval of 3 days for each bar.

must be made public in order for the quality department (or any other user of the process data or by product end-users for that matter) to get the necessary *insight* into the measurement system performance in relation to the total magnitude of the observable process variations. A parallel example, although applied to a very different industrial process (mixing in pharmaceutical productions) was presented by Esbensen and Wagner using the same approach as the one adopted here.[11] This parallels how complete process-and-measurement system evaluation can be achieved by simply "piggy-backing" on the existing process data acquisition, i.e. no specific MSA experimentation necessary.

Discussion and conclusion

Figures 9, 11 and 19 show how the condition of a measurement system can be derived from on-line data and compared to the magnitude of the contemporary process variation.

The specific time period selected for variographic characterisation is important. Too long lag distances may lead to the impression that the measurement system is a major source of variation. Too short distances between the measurements can give several data points with the same value following each other, since the process system is programmed to not change the stored data if the measured data does not deviate from previous data. The choice of the time period to monitor the variation of a measurement system must be competently and carefully defined for each system separately, always based on the most comprehensive process experience, and validated and revised at regular intervals.

Measurement system changes (electrode replacements, setups etc.), for example due to maintenance, should show up and will be documented with the conventional monitoring graphs and will thus give an easy understanding on why systems might "suddenly" show a higher variation. When skilled at working with these monitoring graphs (e.g. Figures 9 and 11), specific measurement system patterns can be detected, for example as for the pH measurements in week 51 for reactors 1 and 2 which were defined "not capable". Operators and process engineers familiar with the design of the process know that both pH electrodes used in this study are situated at the same measurement station. At the measurement station several reactor liquids are collected in defined compartments running with a steady flow and pH for all reactors are measured with separate pH electrodes. However, cleaning routines are common for all pH electrodes. Assuming that these routines are **not** followed (for a variety of *possible* reasons), the pH electrode measurement system variances at the measurement station, *V*(0), might increase and this could be easily observed with monitoring graphs of the types shown in Figures 9, 11 and 19.

It will also be possible to review all preventive maintenance intervals in which exchange of parts in measurement systems have taken place to inspect the resulting performance quality index changes—and to adjust the maintenance intervals, for example, if changes are unnecessarily made too often. Since the redox electrodes are located at the same sampling station as the pH electrodes, combining the results from both measurement types might even give an indication on what might be the root cause if direct faults are discovered.

It would appear that evaluation (grading) of measurement systems by on-line variographic analysis can be very helpful in selecting which measurement systems are in need of a more thorough evaluation (MSA). Variographic process variability characterisation will easily show whether a critical process measurement system has a *V*(0) of 10% or below, relative to the contemporary process variation *V*(sill), fully qualifying the existing system for duty. Including the *V*(0) and *V*(sill) values in the monitoring chart should be considered in order to reveal sudden shifts in time series data which would be camouflaged in a high *V*(sill)-value and resulting in a low *V*(0)/*V*(sill) ratio.

Further work for Nikkelverk with this monitoring opportunity will include development of a standard operating procedure (SOP) on how to estimate the correct time range for the process where the measurement system variation is best investigated. Today operators/process engineers acquire knowledge of system performance by manually checking *individual* data series with standard SPC charts and/or (recently) by using variographic analysis. One has to be aware of the consequences of too-small time intervals when extracting data from the process monitoring system. The responsible operator/process engineer has to have been properly trained, have proper experience and has to know the historical circumstances of the measurement system—as well as its interaction with the process. It is necessary to work as a *team* with the operators in the field. This type of team approach has been well described, for example, in chapter two of the standard work *Process Analytical Technology*.[6]

At Nikkelverk, weekly monitoring has to be setup automatically in a thoroughly user-friendly context to ensure that system characterisations, and therefore also system benefits, are easy to acquire and understand, and appropriate graphs must be actively used by all operators and process engineers, as well as by maintenance personal. An ideal example is shown in Figure 20.

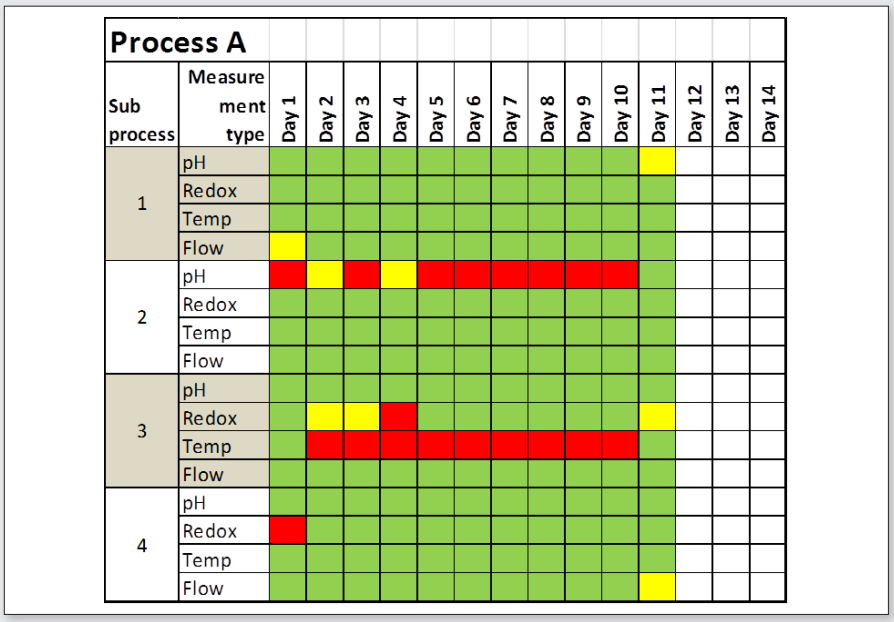

Figure 20. Schematic example of measurements system capability overview.

An example of an overview sheet for different measurement systems is shown in Figure 20. This synopsis allows an easy overview of all measurement systems performance and conditions and will help to find the root cause of the compound process variation.

As in earlier figures, colour codes show if the graded measurement system is capable (green), conditionally capable (yellow) or not capable (red) in the time period selected.

Furthermore, standard operation procedures (SOP) giving out-of-control-action plans also have to be developed to specify in all necessary detail what to check and what to do when a measurement system shows up as "not capable".

It seems obvious that this type of variographic monitoring will help to stabilise processes at Nikkelverk further and increase our understanding of what to address when tending to run out of control. Measurement system analysis is still viable and available, but can now optimally be conducted **only** where it proved necessary—either due to high variations of the measurement system or at process locations which are not monitored frequently by on-line or laboratory measurements. The use of the recently introduced variographic characterisation is clearly a further step forward as part of Nikkelverk's policy for controlling all critical processes with the ICCM (identify, control, capable and maintain) approach.

The application of variography as a method in process industry is endless. It is simple to *dare* just to start using it—and consider every irregularity (and many will always be observed) as a golden opportunity to learn more about the process(es), the measurement system(s) and their critical role in the broader company landscape.

Consider the case in which measurements systems problems are **not** discovered by the suggested frequent variographic scanning of the full array of systems. The consequences would be that the total variation cannot be understood with certainty, but would very often likely be attributed to the process alone since in this case there would be no fact-based evidence that the measurement systems are not working properly. This will invariably lead to *unnecessary* correction of the process parameters, or lead to faulty and quite unnecessary "corrections" of the measurement system. As an example, an undiscovered error in a pH measurement by a 0.2 pH unit over several days will in such a case lead to an excess use of correcting chemicals which is in reality completely unnecessary. It is critically important to control both process and your measurement system with help of variography.

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Feedback on this article will be highly appreciated, directed to Elke.Thisted@glencore.no.

References

1. R.J. Howarth, "Quality control charting for the analytical laboratory—Part 1 Univariate methods—A review", *Analyst* **120,** 1851–1873 (1995). doi: https://doi.org/10.1039/an9952001851
2. W.A. Shewhart, *Economic Control of Quality of Manufactured Product*. ASQC, Milwaukee (1931).
3. W.A. Shewhart and W.E. Deming, *Statistical Method from the Viewpoint of Quality Control*. Dover, New York (1939).
4. D.J. Wheeler, *Advanced Topics in Statistical Process Control: The Power of Shewhart's Charts*. SPC Press, Knoxville (2001).
5. DS 3077, *DS 3077. Representative Sampling—Horizontal Standard*. Danish Standards (2013), www.ds.dk
6. K.H. Esbensen and P. Paasch Mortensen, "Process sampling (Theory of Sampling, TOS)—the missing link in process analytical technology (PAT), in *Process Analytical Technology*, Ed by K. Bakeev. Wiley-Blackwell, Oxford, pp. 37–80 (2010). doi: https://doi.org/10.1002/9780470689592.ch3
7. R.C.A. Minnitt and F. Pitard, "Application of variography to the control of species in material process streams: %Fe in an iron ore product", *J. S. Afr. Inst. Min. Metall.* **108,** 109–122 (2008).
8. F. Pitard, *Pierre Gy's Sampling Theory and Sampling Practice*, 2nd Edn. CRC Press, Boca Raton, FL (1993).
9. B.C. McDaniel, *Measurement System Analysis (MSA)*. https://www.moresteam.com/toolbox/measurement-system-analysis.cfm [Accessed 26 December 2015]
10. K.H. Esbensen and C. Wagner, "Theory of sampling versus measurement uncertainty (MU)—A call for integration", *Trends Anal. Chem.* **57,** 93–106 (2014). doi: https://doi.org/10.1016/j.trac.2014.02.007
11. K.H. Esbensen and R.J. Romañach, "Proper sampling, total measurement uncertainty, variographic analysis & fit-for-purpose acceptance levels for pharmaceutical mixing monitoring", *TOS forum* **Issue 5,** 25–30 (2015). doi: https://doi.org/10.1255/tosf.68

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Barefoot sampling in San Juan de Limay, Nicaragua: remediation of mercury pollution from small scale gold mining tailings

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The increasing population on planet Earth has many impacts—one is a strong influence on the amount of mercury released to the environment. Burning of coal in power plants, particularly in China, has tripled several times during the last century and so has the use of coal for cooking for the ever-increasing population of India and elsewhere. These sources account for the second largest release of mercury to the environment. But the worst sinner is the rapidly increasing number of small-scale gold miners in Asia, Africa, Central and South America, who presently provide food on the table for 10s of millions of households. Small-scale gold miners use vast amounts of mercury for capturing the gold and much of this mercury is released directly to the environment. A large part evaporates to the atmosphere and the rest is transported downstream in rivers ending up in the oceans. The amount of mercury released is phenomenal, an estimated 3000 tons of mercury is released annually by small-scale gold miners alone. A vast proportion enters the food chain in fish and sea mammals, as well as in rice polluted by spillage waters which enters irrigation pathways. Human consumption of polluted fish and/or rice already today has a very severe impact on human health, and this will have even more severe consequences if the current situation is not changed radically. It is of particular concern if mercury-intoxicated women become pregnant because the foetus extracts mercury from the mother. The human foetus is much more sensitive to mercury intoxication and thus has a high risk of being born with brain damage as well as physical disabilities. Over time this will cause reduced intelligence for exposed children in the next generation. This grim outlook has prompted a group of concerned researchers to teach small-scale gold miners to work without the use of mercury and simultaneously to find ways to clean mercury-polluted gold mining tailings, which are one of the main polluting agents. Here we report on one specific part of this endeavour where the Theory of Sampling (TOS) was needed in order to secure reliable estimates of gold and mercury contents in dispersed mining tailings.

Background

Mercury pollution constitutes an environmental time bomb of potentially alarming proportions. The two main sources of global mercury pollution are small-scale gold mining (SSGM) and coal burning in power plants, as well as domestic cooking in developing countries.[1] The massive release of mercury to our environment will cause a serious global health issue for generations to come; the possibly worst scenario is that humanity will experience a dramatic decrease in intelligence in future generations. Small-scale gold mining is a low-technology, poverty-driven way for many tens of millions of people to provide for their daily needs.

Small scale miners crush and mill gold ore together with mercury. The mercury captures the gold by forming an *amalgam*. This is subsequently heated in open vessels whereby mercury evaporates and the gold is left behind for economic recovery. This is a technologically simple and very easy processing method that does not require any noticeable investment in equipment and in this way "nothing goes to waste" of the precious gold. Unfortunately, it is the key process element of milling gold ore together with mercury that creates the serious health problem described. During milling a large part of the mercury is ground to small drops called *mercury flour*.[2,3] Mercury flour cannot coalesce and can therefore not be recovered by the miners, but ends up in tailings (waste dumps from SSGM operations). This mercury loss is doubly unfortunate—both for the miners and for society. Not only is mercury flour harmful to the environment, but it also constitutes a financial problem since it still contains appreciable amounts of gold that cannot be recovered with the simple methods employed and thus reduces the economic viability of mercury-based SSGM. Over time mercury flour in SSGM tailings will *evaporate* or gradually be *washed* into the drainage system, ultimately ending up in the world's oceans from where the evaporated mercury will be distributed over the entire planet. The part of mercury that ends up in rivers, lakes and oceans will be transformed to the compound *methylated mercury*, or "organic mercury", which readily enters the food chain(s), where it will be bio-magnified, resulting in high concentrations of toxic mercury compounds in top-level fish and sea mammals. Polluted fish are unfortunately consumed by humans—this is the root cause of the very serious health problems that have been called the impending global mercury disaster.[4]

One principal way to *mitigate* this mercury disaster is to clean the hundreds of thousands of SSGM tailings containing mercury flour which are littering large parts of South-east Asia, Africa, Central and South America. If an efficient, inexpensive low-tech method can be found, it will benefit not only the global environment and health status of millions, but will at the same time also produce considerable amounts of gold in quantities that may well cover the costs of modified processing methods, and eventually result in more profitability for the SSGM communities.

Barefoot sampling in San Juan de Limay, Nicaragua

Experiments to extract mercury flour from polluted tailings by alternative, mercury-free

approaches have been carried out in the Philippines[2] and are presently being tested in Nicaragua. In Nicaragua, tailings from a number of different SSGM processing sites were subjected to the most advanced alternative recovery process currently available. The experimental tailing lots varied from 4 tons to 15 tons. The first critical step in these experiments was to obtain *reliable* estimates for the average mercury and gold contents in the tailing heaps available for this experimental campaign; these concentrations are known to be of the order of 2–15 ppm. This is no small challenge in a setting where tailings typically are of the order of ~10 tons, and everything has to be carried out *manually*.

It is critical to follow the tailing mercury and gold throughout the full multi-stage recovery process and to be able to carry out a complete metallurgical accounting. For this the original Au and Hg concentrations in the primary lots are the key information needed, as are the sampling processes employed for dealing with these very low abundances. We here report on *barefoot sampling* in which application of the principles of the Theory of Sampling (TOS) was *de rigeur,* but with only DS 3077 and willing, able hands available. For environmental and individual miner health reasons, it is even more important to keep track of the Hg concentrations at all sampling stages as well, which poses its own specific problems.

2015 Nicaragua field experiment—prospects

During spring and autumn 2015, feasibility tests were out carried in Nicaragua to estimate the efficiency of extracting mercury flour from SSGM tailings. To the degree this is feasible, and to the degree it can be successfully recovered at a sufficiently high recovery rate (~75%), this will be a significant driver to allow SSGM collectives to accept the alternative process.[†]

Investment capital for this type of local mining reclaiming is available a.o. from developed nations' development funds, and there is also direct commercial potential. The alternative process will be profitable, at *assumed* recovery rates larger than 75%, as long as tailing concentrations are above 3 ppm. The same process will remedy the otherwise continuing SSGM tailings pollution—a double whopper—and all essentially with barefoot technology!

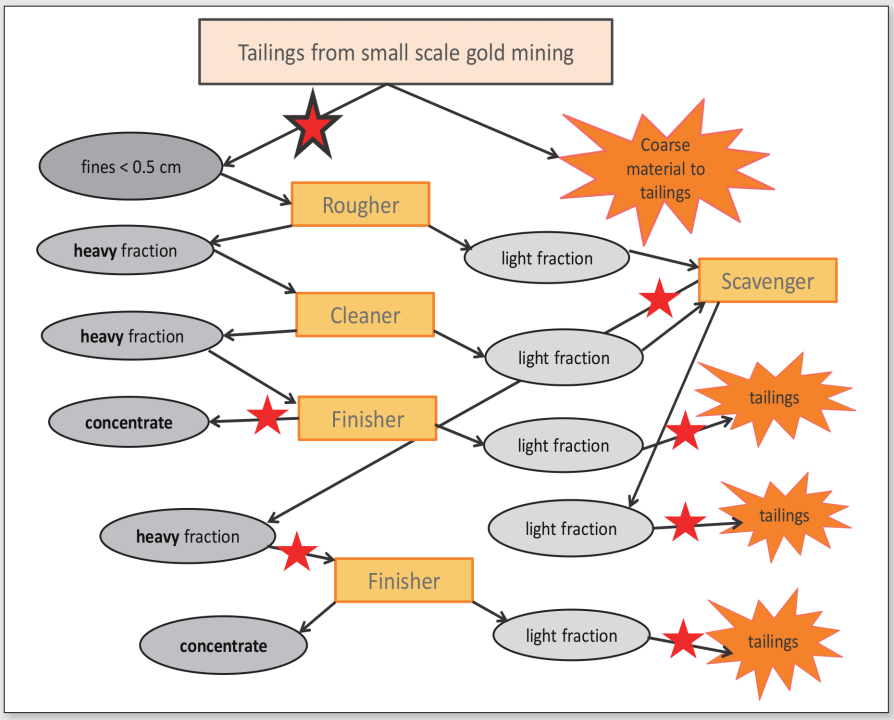

Figure 1. Schematic overview of the SSGM tailing reclaiming process with sampling points for the complete pilot study (small red stars) and the present experiment (large red star with black edge).

Field experiment design and sampling requirements (TOS)

Tailings for the experiments were gathered from five different SSGM processing sites. The tailings were selected to represent different types of gold mineralisations and thus different general compositions with presumably differing *processabilities*, and milling efficiency was indeed observed to vary widely locally. The overall mercury contents would therefore be expected to vary significantly between different tailings (corroborated by the analytical results, see Table 1). Tailing lot masses varied from 4 tons to 21 tons with an average of ~10 tons.

The crucial first step is to establish the *average* mercury content of each tailing lot with absolutely no primary sampling bias allowed because of the ultra-low grade levels present. For this reason, sampling expertise in the form of the TOS was called upon. It is equally important to be vigilant with respect to the representativity of all subsequent field and laboratory mass-reduction steps. From original tailing size to analytical mass, sampling rates are of the order of $1:10^7$. The principles of TOS have to be upheld scrupulously along the **entire** lot-to-analysis pathway. While this is trivial in most scientific, technological and industrial contexts in the developed world, the present project poses a highly challenging twist: almost everything must be carried out *manually*—which calls for *barefoot field sampling* (for the first two stages), but in the subsequent laboratory mass-reduction stages some innovative approaches were also called upon, as described below.

We welcome this challenge—how better to contribute to helping tens of millions of SSGM families with a life-threatening mercury danger?

SSGM tailing recovery process—a brief

Tailings were *scooped* into a drum, which selects and discard >0.5 mm material. The resulting fines are directed into a train of three spiral concentrators, which separate heavy from light minerals. These are termed "Rougher", "Cleaner" and "Finisher", respectively, in Figure 1. The heavy fraction

[†]There exists an alternative mercury-free gold extraction method, which is gradually gaining momentum in South-east Asia. This approach uses gravitational separation to produce a gold concentrate, which is subsequently smelted by using the environmentally benign chemical compound borax.[2,3,5,6] An introduction to this approach can be seen on an educational video here: https://www.youtube.com/watch?v=X6Sawj0HyF0.

Figure 2. First stage in the SSGM tailing reclaiming process feasibility project, initial particle size screening.

Figure 3. Halfway through the intensive task of moving a complete original lot one shovel at the time, taking great care to extract an increment from *each*, as detailed in Figure 4.

from each spiral is directed to the next spiral. The light fraction from "Rougher" and "Cleaner" is directed to a centrifuge, termed "Scavenger". The light material from here is directed to tailings while the heavy fraction is directed to the "Finisher". The heavy fraction from the "Finisher" is directed to a stack of copper amalgamated plates "Peter plates",[2] which finally capture the mercury flour and free gold particles to be reclaimed. Figure 2 shows the first stage of the full feasibility study (drum loading for initial particle size screening). Below we are exclusively interested the critical primary sampling from the original tailings: how to get a documentable representative analytical estimate of the average gold grade?

Primary lot sampling—the crucial stage

Manipulating lots of the size of 5–15 tons is usually not a problem when the appropriate industrial equipment is at hand, e.g. front-loaders, bobcats or the like—of which there most emphatically are **none** available to very poor artesian mining collectives. But able hands, picks and shovels are in abundance. It was decided to follow the principle: "move the original lot 10 m to the right" and perform process sampling along this 1-D transportation stage. Thus each tailing dump was transported manually, one shovel-full after another in order to facilitate sampling, Figure 3.

Figure 4 shows this primary composite sampling *in extremis*: the material in *each* shovel-blade (approx. 5 kg) is intercepted by a scoop of approximately weight 100 g. For an average 10-ton primary lot size, this translates into 2000 increments (each of ~100 g), by all standards an overwhelming coverage of each original lot with a solid guarantee for compliance with the Fundamental Sampling Principle (FSP). The resulting composite primary sample weighs ~200 kg. This material was subjected to forceful mixing before further sub-sampling, based on the abundant man-power available.

Figure 5 shows how the next sub-sampling stage was executed: *each* 200 kg primary sample was passed through a riffle splitter, in a series of 50/50 split sessions until the sub-sample mass had been reduced to ~1–2 kg, which was the sample size subsequently transported to GEUS, Denmark for further processing and preparation for analysis.

Slurry sub-sampling in the laboratory

After processing all primary tailings in the manner illustrated, quantitative analysis was carried out on a selected set of seven primary samples (project financing was at the time of the analysis also at a decidedly "barefoot" level). These samples were not easy to process, however, as they were all *slurries* and with very different Au and Hg contents. Slurry sampling is not easy under any circumstance, but especially not when

Figure 4. Incremental sampling from each shovel used to transport all original lots, see Figure 3.

Figure 5. Loading the project riffle splitter (kindly provided by GEUS). Sub-sampling is made effective by the fact that the sample to be split does not need to be split all in one, but can be subjected to riffle-splitting in an intermittent loading process.[7]

Figure 6. Two types of slurry sample containers as received from Nicaragua, plastic bottles and bags. The photo shows the frozen versions after 24 h in a freezer at –16°C, ready for sub-sampling, see Figures 7–8.

stringent counter-volatility demands are to be upheld. Also, sub-sampling, although here carried out in a well-equipped laboratory (GEUS, Copenhagen), had to be performed with procedures that potentially can be carried out under the relevant ambient field conditions in Nicaragua.

Due to the severe risk of segregation (free Au particles, mercury flour), handling the slurry samples became a critical issue, not easily tackled with the standard riffle splitters at hand. It is critical to *counteract* any-and-all segregation effects present in the sub-sampling procedure employed (while these effects *may* be small, intermediate or large, they are never absent and the only responsible approach is to assume such effects are always significantly present). For this reason, conventional riffle splitting could not be used. A better way was required, illustrated in Figure 6.

A novel twist had to devised: after vigorous and extensive mixing, the entire 1–2 kg slurry samples, which came in tightly sealed but otherwise conventional plastic bottles or bags, were stored in a freezer (–16°C) for 24 h, sufficient for the entire content to freeze solid. The "splitting" was then effectuated as a two- or three-step *longitudinal* sectioning of the solid bottle or bag content, see Figures 7 and 8. In this way **any** residual segregation affecting the *vertical* container contents in their slurry state was sampled in a fully representative fashion, while maintaining quite effective sampling rates of the order of 1 : 10 at this stage. This sub-sampling technique is only dependent on the cohesiveness of the frozen solid w.r.t. the thickness of the slice cut with the diamond saw (and subsequently with a hobby knife).

From the primary sampling stage all the way through the penultimate sample shipped off to the analytical lab, the critical success factor was counteracting segregation. Even the commercial, accredited ACTlab analytical laboratory was directed to document the in-house sub-sampling employed with reference to DS 3077.[8,‡]

All 34 final sub-samples were of analytical mass ~12 g when shipped off for multi-element analysis (standard economic geology element suite *plus* Au and Hg) at Actlabs Labs, Canada.

Sampling perspective

The original lots were on average of a size corresponding to 10 tons. The average assumed Au-grade in the mine tailings was of the order of 2–15 ppm, based on the best local mining experience available. The lot material, earlier mining tailings, is crushed to an average grain size diameter of 50 µm, but the tailings have never been subjected to mixing or blending to any extent. It follows that such very low-grade gold concentration carriers (flakes, particles) must be present in an exceedingly irregular spatially heterogeneous distribution pattern, i.e. for the present study the lot distributional heterogeneity is extreme. It would be close to a miracle if standard haphazard scooping of a small primary sample from 10 tons, archetype grab sampling, could ever be representative.[8] Only a very thorough composite sampling can be accepted. In order to meet these hard demands, the "field-to-analysis" pathway consisted of i) primary sampling (extremely effective composite sampling with a sampling rate of $1:10^{4+}$); ii) field mass-reduction (riffle splitting, sampling rate ~1 : 100); longitudinal diamond saw cutting of premixed vertical slices (1 : 10); in-house aliquoting sub-sampling (~1 : 10). The complete sampling pathway mass reduction thus spanned seven orders of magnitude (mass/mass). All stages were carefully designed and scrupulously performed in

‡Appendix 1 quotes our directions to ensure full disclosure of the in-house laboratory procedures w.r.t. sub-sampling. At the present extremely low average gold grade there is every possibility that the lot-to-aliquot integrity can be significantly affected by mindless grab sampling (using a spatula) even at this ultimate sampling stage. Indeed the size of gold particles and mercury flour drops may be fatally mis-sampled with a spatula.

Figure 7. Two-stage mass reduction of frozen sample bottles. First cutting is a 50/50 split, followed by a further slice of one of the randomly selected half cores produced, resulting in a 10–15% final sub-sample mass, which is guaranteed to be representative of the original container content in the vertical dimension irrespective of the degree of possible residual segregation present.

Figure 8. Three-stage mass reduction of frozen sample bag content. All cuts are vertical slices again resulting in a 10–15% final sub-sample mass, which is guaranteed to be representative of the original container content in the vertical dimension irrespective of the degree of residual segregation present.

accordance with TOS' every principle for representative sampling to a degree only rarely deployed within the geosciences, while at the same time **exclusively** only relying on manual processes.[7,9] The term "barefoot sampling" appears apt.

Project results—preliminary findings

Table 1 shows the analytical results from the primary samples representing the seven mining tailings addressed in the sampling feasibility pilot study.

Discussion

In industrial contexts, an average gold grade of above ~5 ppm is considered profitable; in the SSGM community, 3 ppm (and above) is eminently interesting. In this view the values revealed in Table 1 show the inefficiency of traditional gold extraction using mercury. There is therefore an important incentive to address these types of tailings. There are two options:

1) extraction of gold from original deposits using the alternative mercury-free procedure and/or
2) re-process SSGM tailings, also based on the alternative approach.

Re. 1) Teaching small-scale miners mercury-free extraction procedures constitutes an important objective—which will result in an increased profitability of at least 50%, while simultaneously contributing significantly to solving the global mercury poisoning threat.[2]

Re. 2) This will directly reduce the global mercury pollution—while at the same time being able to score economically from a source never mined optimally before, SSGM tailings. Some operators have run these tailings through a cyanide leaching process, which has its own, severe environmental problems of course.

Conclusions

This work originated as part of a global mercury pollution reduction endeavour to which SSGM is **the** major contributor. The mercury is hosted in tailings as tiny droplets, which are difficult/impossible to recover. The present feasibility project, financed by the Danish Ministry of Environment and Food, focuses on methods for mercury cleaning

Table 1

Analyte	Au	Ag	Hg	Cu	Pb	Zn	As	Se	Sb
Unit	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Detection limit	0.03	0.5	10	0.03	0.03	0.3	0.05	0.05	0.05
Analysis method	FA-GRA	ICP-MS	ICP-OES	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS
Limay1	13	7.1	10	45.1	54.2	50.2	10.4	4.89	4.78
Limay13	4.99	121	20	332	592	88.2	96.1	9.37	4.2
Limay23	4.41	28.2	20	317	241	55.7	8.03	7.12	2.62
Limay30	1.15	7	10	151	148	165	58	6.15	2.86
Limay36	9.87	8.9	10	46.9	65	61.6	6.74	6.14	4.13
Limay44	13.5	10.5	10	34.1	67.7	51.9	7.58	5.88	3.44
Limay51	3.95	222	40	176	709	197	224	7.71	7.36

with the aim of two potential bonuses for the SSGM community, and for the world, one environmental, the other economic.

In this context, a need for careful primary sampling was identified. Global SSGM tailings, destined for an improved, non-toxic reclaiming process, need to be characterised with extraordinary focus on reliable estimates of average grades for Au and Hg.

For this purpose, the TOS was invoked which had to be applied subject to stringent "barefoot" technology requirements. Amongst others, use was made of extraordinarily intensive composite primary sampling in full compliance with the FSP. The project also developed a "freeze-drying" technique for sampling "difficult" slurry samples with severe gold segregation and mercury integrity issues. As described and illustrated, these TOS tasks were satisfactorily resolved. There is likely a carrying-over potential for the freeze-dried sub-sampling procedure to other similar types of slurry material.

Appendix 1

"These samples originate from a study of low (to very low) Au and Hg concentration in mine tailings and tailing dumps (estimated 2–15 ppm), implying a highly irregular distribution of elemental micro-Au flakes/fragments in the 15 ton original tailing dump. The project has invested a considerable effort in arriving at the seven sample flasks supplied (masses ~30g) with outmost care in using Theory of Sampling compliant primary and secondary sub-sampling throughout, as documented in DS 2077 (2013). It is critical that also the final mass-reduction needed for ACTLABS to extract the precise analytical aliquot mass/volume are fully representative, i.e. extracting the aliquot mass from the sample flasks supplied by spatula is unacceptable. We ask ACTLABS to follow one of the recommended procedures in Petersen et al. *(2004), Esbensen & Julius-Petersen (2009). Because this project is a method-development feasibility study in which sampling, handling and analysis are of equal importance, we ask ACTLABS to supply a complete documentation of the in-house sub-sampling employed."*

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References

1. AMAP/UNEP, *Technical Background Report for the Global Mercury Assessment 2013*. Arctic Monitoring and Assessment Programme, Oslo, Norway/UNEP Chemicals Branch, Geneva, Switzerland (2013).
2. P.W.U Appel, L.D. Na-Oy, Y. Hatsukawa, T. Osawa, J. Kystol and L.L. Sørensen, "Cleaning mercury polluted mine tailings in the Philippines", *Danmarks. Grønlands. Geol. Unders. Rap. 2011/127* (2011).
3. P.W.U. Appel and D.L. Na-Oy, "Mercury-free gold extraction using borax for small-scale gold miners", *J. Environ. Protect.* **5,** 493–499 (2014). doi: https://doi.org/10.4236/jep.2014.56052
4. S. Bose-O'Reilly, B. Lettmeier, R.M. Gohte, C. Beinhoff, U. Siebert and G. Drasch, "Mercury as a serious health hazard for children in gold mining", *Environ. Res.* **107,** 89–97 (2014). doi: https://doi.org/10.1016/j.envres.2008.01.009
5. P.W.U. Appel, A. Andersen, L.D. Na-Oy and R. Onos, "Introduction of mercury-free gold extraction methods to medium-scale miners and education of health care providers to reduce the use of mercury in Sorata, Bolivia", *J. Health Pollut.* **5(9),** 12–17 (2015). doi: https://doi.org/10.5696/2156-9614-5-9.12
6. A.R. Scialli, J.P. Bonde, I. Brüske-Hohlfeld, B.D. Culver, Y. Li and F.M. Sullivan, "An overview of male reproductive studies of boron with an emphasis on studies of highly exposed Chinese workers", *Reproduct. Toxicol.* **29,** 10–24 (2010). doi: https://doi.org/10.1016/j.reprotox.2009.10.006
7. K.H. Esbensen and L.P. Julius, "Representative sampling, data quality, validation – a necessary trinity in chemometrics", in *Comprehensive Chemometrics*, Ed by S. Brown, R. Tauler and R. Walczak. Wiley Major Reference Works, Vol. 4, pp. 1–20 (2009). doi: https://doi.org/10.1016/B978-044452701-1.00088-0
8. DS 3077, *DS 3077. Representative Sampling—Horizontal Standard*. Danish Standards (2013), www.ds.dk
9. K.H. Esbensen and C. Wagner, "Theory of sampling versus measurement uncertainty (MU)—A call for integration", *Trends Anal. Chem.* **57,** 93–106 (2014). doi: https://doi.org/10.1016/j.trac.2014.02.007

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

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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. This will form a comprehensive literature review reflecting on the debate between the representativity of the different sampling methods. The literature review collected a total of 31 publications (two were more or less duplicates and one consisted of an abstract only). The main source for publications on RC and BH drill sampling were dedicated sampling conferences, other mining conferences and some publications were found in peer-reviewed journals. From the gathered publications, it is not possible to draw a general overall conclusion as to the superiority of one drill sampling method over another. Both RC and BH have advantages and disadvantages and the choice of system needs to be related to the ore type and to the mining conditions. The overall conclusion is that it is always necessary to evaluate the specific sampling system to be used in light of the Theory of Sampling (TOS) (and with respect to the characteristics of the ore to be mined). It is always necessary to ascertain that the specific drilling sampling system contemplated does not lead to hidden losses that could have been avoided or missed profits that could be gained with a more relevant and representative sampling system. It would appear that the mining industry is doomed to continue to follow local, often economy-driven objectives and sampling solutions even if these can be documented as inferior when seen in the light of the representativity imperative. A call is made for universal adherence to the principles laid down by TOS for representativity in the primary sampling stage, before economic, logistical or other (local) factors are allowed to intervene. What is the objective to analyse and to make decisions in the mining industry, based on samples that can be documented not to be representative?

Introduction

In the mining industry, misclassifications of ore types due to poor sampling practices can easily generate large value losses and contribute to economic inefficiency in the crushing stages, as has been vividly demonstrated by Carrasco *et al.*[1] Internal calculations at LKAB indicate that misclassification of ore can lead to unnecessary costs of up to US$200,000 if one blast of waste is classified as ore, or loss in revenue of up to US$700,000 if one blast of ore is classified as waste. These estimates only represent pure costs or losses, and do not include losses due to decreased quality of final products, loss of customer trust, increased product handling or increased strain on waste dumps and dams. These examples clearly show the need for correct and representative sampling methods in open pit mining, for high quality and cost effective mining operations.

Blast hole (BH) sampling is widely used for grade control by the mining industry all over the world, both in precious and base metal open pit mining. 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.[2,3] Figure 1 presents some of the well-known BH sampling problems and issues. Nevertheless, many mining operations continue to rely on manual BH sampling methods which are claimed to lead to "good results". However, Abzalov *et al.*[4] concluded in a study of (mainly) existing BH and RC samples in

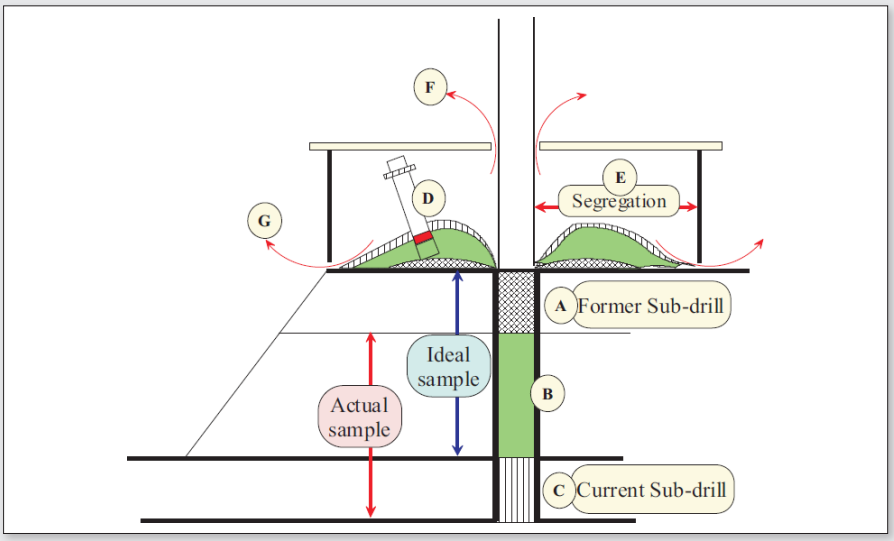

Figure 1. Summary of blast hole sampling problems and errors (from Reference 2 with permission).

Figure 2. Manual BH sampling at a LKAB open pit mine. a: Cutting a sectorial part of the BH cone. b: Collecting a vertical slice of uniform thickness from the bottom to the top in the centre of the blast hole cone.

an iron ore deposit, that both methods can be equally biased compared to full cone BH sampling. See Figure 2 for a contemporary example of manual sampling in iron ore open pit mining.

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. This will form a comprehensive literature review reflecting on the debate between the representativity of the different sampling methods. With a summary of published conclusions the authors will attempt to see if it is possible to find an overall consensus regarding the superiority of any of the sampling methods.

Method

This literature review is conducted with both a quantitative and qualitative focus. First, all identified papers covering the topic of open pit mine drill sampling performance were compiled in a complete reference list. The search for publications was done through the web-based databases SCOPUS and ScienceDirect with keywords: “blast hole (BH) sampling”, “open pit mine sampling”, “drill sampling” and “reverse circulation (RC) sampling”. The search also covered review of proceedings from the specific sampling conferences Sampling and World Conference in Sampling and Blending, as well as proceedings from various mining conferences and congresses. Last, all references in the hitherto gathered publications were reviewed for any further publications on the topic. Apart from internet based searches, some physical digging was also conducted, Figure 3.

The abstracts of all primary identified publications were reviewed to collect articles that specifically discuss the representativity or performance of at least one of the three drill sampling methods. Publications that concern a drill sampling method, but do not further discuss its representativity or precision of collected samples were excluded from the literature review during review of abstracts. The focus of the literature review is to assess performance, i.e. representativity and/or precision of open mine drill sampling methods; all publications that discussed this issue were included in the review. As the performance of actual drilling, *in situ* or bulk sampling or assay methods is not the main focus, publications that only discuss these matters were excluded from the review. Publications regarding underground drill sampling have also been excluded from the review as the present focus is on open pit mine sampling.

The ambition from the authors was to identify all publications from 2000 and onwards addressing the representativity of drill sampling methods. There might, however, still be some publications that could not be identified with the search methods used here. Any additional literature items that may surface in this context will be included in an updated survey which will be the base for discussions in the PhD thesis which includes the present feature. Defence is planned for 2019.

Results

The main sources for peer reviewed publications in the subject of open pit mine sampling and its representativity were specific sampling conferences, i.e. *Sampling* in Australia and the international *World Conference in Sampling and Blending* biannual series. A second source is other mining conferences and a few publications could be found in peer-reviewed journals. See Table 1 for the sources of all publications in the literature review. Comprehensive references for all publications can be found in the list of references. A brief summary of the most important conclusions from all collected publications can be found in Table 2.

The collected publications include seven theoretical discussions based on the Theory of Sampling (TOS) as well as previous publications and personal experience. In 22 of the publications, one or more case studies were conducted to evaluate the performance of one or both drill sampling methods. See Table 2 for a summary of all publications. Two publications were more or less identical with the same case study, results and conclusion; consequently, only one has been added to this summary.

Figure 3. A selection of some physical sources collected for the literature review.

Table 1. Sources for the collected publications.

Publication	Sampling specific conference	Other mining conference	Peer reviewed journal
Total: 31 publications	16	10	5
Abzalov *et al.*[4]		✓	
Abzalov *et al.*[5]			✓
Alfaro[6]	✓		
Caccioppoli *et al.*[7]		✓	
Carrasco *et al.*[1]			✓
Chieregati *et al.*[8]	✓		
Chieregati *et al.*[9]	✓		
Chieregati *et al.*[10]	✓		
Crawford *et al.*[11]		✓	
El Hajj *et al.*[12]	✓		
François-Bongarçon[13]	✓		
Goers *et al.*[14]	✓		
Gomes *et al.*[15]	✓		
Hapugoda *et al.*[16]	✓		
Hapugoda *et al.*[17]		✓	
Holmes[18]			✓
Holmes[19]			✓
Hoogvliet[20]		✓	
Kirk *et al.*[21]		✓	
Magri *et al.*[3]	✓		
Magri *et al.*[22]		✓	
McArthur[23]	✓		
Minkkinen *et al.*[24]	✓		
Niemeläinen *et al.*[25]	✓		
Ortiz *et al.*[26]		✓	
Pitard[27]		✓	
Pitard[2]	✓		
Séguret[28]	✓		
Spangenberg *et al.*[29]			✓
Young[30]		✓	
Ziegelaar *et al.*[31]	✓		

Last, one publication consisted only of an abstract, the study was presented orally at a conference in full but no article was prepared for the proceedings. In 12 of the case studies, existing grade control data was used while in 16 publications experiments were performed to generate new data, Table 3.

Table 4 shows a summary of the drill methods evaluated, the reference method and the most important conclusion of each publication. Table 4 also show which ore type is mined in the case studies presented. About half of the publications evaluate BH sampling and the other half compare BH to RC sampling. Four of the publications only evaluate RC sampling. The most common reference method used is DD sampling (nine publications), while full cone BH sampling, RC sampling and plant feed or reconciliation are used as reference in three to six publications. In as many as seven publications, RC drill sampling is assumed to be representative by the author(s), either from previous publications or "by experience". At the same time five (other) publications conclude that RC sampling can be non-representative as evaluations show several sampling problems and biases. Eleven publications conclude that RC sampling is more representative then BH sampling, while thirteen publications indicate that BH sampling can be representative or fit-for-purpose.

In summary, the results and conclusions show a very diverse picture of the debate between RC and BH sampling. One weak indication *could be* that base metal mining (iron ore) might show a slight tendency to accept BH sampling as representative. In contrast, the literature review shows that for sampling in gold mining, RC is generally concluded to be more representative than BH sampling. One exception is Chieregati[8,9] which both conclude that BH sampling can be fit for purpose if using correct equipment and sampling procedures, Figure 4.

Discussion

The different aspects of RC vs BH sampling are complex and in all cases clearly relate to the specific ore type and the prevailing mining conditions. The wide range of conclusions from all publications show that there is no universal answer to one sampling method always being superior. BH sampling is indeed accompanied by many problems like loss of fines, upward/downward contamination, influx of sub-drill material, pile segregation, pile shape irregularities, operator-dependent sampling, too small sample size, frozen BH cones and non-equiprobabilistic sampling equipment, see Figures 5 and 6 and References 2 and 6 among others.

Solutions do exist that handle some of the problems related to manual BH sampling and are able to reach a representative or fit-for-purpose status, however. Examples that counteract the most glaring sampling bias problems are channel sampling and sectorial sampling, Figures 4 and 7. Another solution that has proved to provide representative BH samples (in two publications) is automated BH sampling systems, Figure 8. Even though these can produce good quality samples, they have not made a breakthrough on the market for BH sampling, mainly due to the increase in drilling time when applying the automated sampling approach.

Figure 4. New modified sectorial sampler fitted to the PWH drill (right) and detail of the buckets/frame (left) (reproduced from Reference 9 with permission).

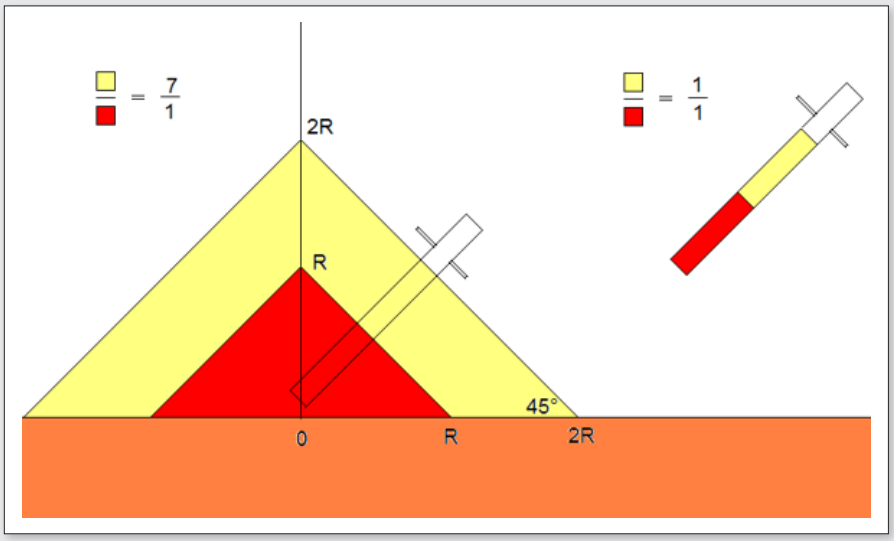

Figure 6. Non-equiprobabilistic sampling tube (reproduced from Reference 6 with permission).

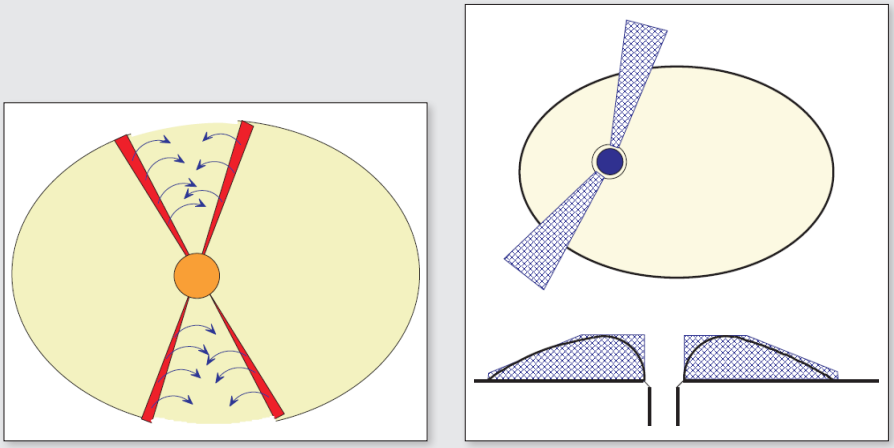

Figure 7. Left: digging two radial channels, from which to extricate four thin, radial increments to make a composite sample. Right: correct design and positioning of radial bucket/sectorial sampler. Reproduced from Reference 2 with permission.

Figure 5. Frozen BH piles are a big problem in some open pit mines, from Reference 6.

The scale of resolution of sampling grids is in many publications concluded to be more important than sampling performance. Typical RC sampling grids are *ca* 25×25 m compared to BH sampling grids that are in most cases around 5×5 m. This large difference in grid size often leads to a larger increase in the number of misclassified mining blocks than BH sampling imperfections. If great care is taken when developing sampling methods, adapted to the drill rig at hand and accommodating the need of each mining situation, BH sampling can come satisfactorily close to being fit-for-purpose in some mining situations. In other case studies, RC sampling is proved to be more representative and is proved

Figure 8. Top: Drillsampler™ from Harrison Cooper for automatic blast hole sampling installed underneath the drill deck (reproduced from Reference 2 with permission). Bottom: the Finnish "Autosampler" system with Softcore™ sample socks attached (reproduced from Reference 24 with permission).

Table 2. Main conclusion of collected publications.

Publication	Main conclusion from publication
Abzalov *et al.*[4]	Manual BH sampling with shovel from BH cone on 6 × 6 m grid, compared to RC drill sampling on 25 × 25 m grid. BH and RC sampling proved equally biased in comparison to full BH cone assays. BH and RC or DD results are consistent at distances of 1 m, but the variations in grade between twin holes increase when the distance increase and holes 10 m apart show excessively poor repeatability. Indicating that a sampling grid of 25 × 25 m will be sub-optimal at this mine. The quality of grade control procedures depends on both quality and quantity of grade control samples. In this case study, the amount of misclassified selective mining units would increase from approximately 5.8% to 12.3% when increasing sampling grid from 5 × 5 m to 25 × 25 m.
Abzalov *et al.*[5]	Comparison between BH and RC sampling in iron ore open pit mine. The study incorporates both sampling error and sampling grid to optimise sampling procedures. Currently, BH sampling is used for grade control, but RC sampling was considered as an alternative approach for grade control. Sample duplicates and twin holes with RC and BH sampling revealed that RC sampling does not guarantee improved sample quality. RC and BH exhibit similar precision errors and RC were biased, underestimating Al_2O_3 and SiO_2 grades, and overestimating Fe grades. Simulation showed that change to RC grade control with 25 × 25 m grid would not reduce grade control errors, but rather increase the number of misclassified ore and waste blocks.
Alfaro[6]	A case study of the Rio Blanco ore deposit is porphyry copper, located in the central zone of Chile. Many problems with manual BH sampling in winter due to moist and frozen material. Comparisons between BH and DD assay results is done by identifying holes with maximum inter distance of 5 m. The comparison indicates problems with the BH samples, especially for As and Mo.
Caccioppoli *et al.*[7]	Comparison between RC and manual BH sampling is done for flitch mining. The authors are using RC drilling as reference for the manual BH samples that are taken separately for top and bottom flitch. The result show differences between full cone BH and RC assay results in 20% of the blast holes. Improvement of the material recovery in the remaining blast holes could improve the accuracy of the BH assays.
Carrasco *et al.*[1]	A case study of BH sampling in a porphyry copper operation shows a nugget effect of 70% of the total variability. The sampling did not take the equiprobable rules into account and collected 250 g of material from a 2 ton lot with 2 cm top size. The variability was much greater than for diamond drilling even though DD had a much smaller support. By the use of statistical and geostatistical calculations, the authors calculate losses due to poor BH sampling to approximately 22 MUSD.
Chieregati *et al.*[8]	Summary of several aspects that make sampling gold challenging based, on two case studies. Even though RC drill sampling is regarded as a more appropriate sample method, a significant (up to 20%) loos of fines can occur through overflow of the cyclone. BH sampling also have problems with loss of fines due to wind, and manual sampling with a shovel is common and does not conform to TOS equiprobabilistic principles. The authors suggest that the use of a correctly designed sampler could eliminate problems with delimitation, extraction and weighting errors in BH sampling. RC sampling can also be improved, for example by adding a secondary cyclone to collect the fines.
Chieregati *et al.*[9]	Validation of a newly designed cupola stationary sectorial sampler for BH sampling. The sectorial sampler has a significantly higher recovery of fine material which minimised sampling error due to loss of fines. The sampler did not lower production compared to previous manual BH sampling. The two opposite sectorial sample collectors were unbiased to each other. The sampler did not show any bias to the reference used, which in this case was TOS correct sampling of the plant feed. The authors note that double-discharge drills are a completely different scenario and cannot be directly compared to this case study with single-discharge, narrow diameter drill.
Chieregati *et al.*[10]	Case study of RC drill sampling compared with manual BH sampling. Complete BH cone as well as complete RC material was used as reference. Result show that the BH drilling loses coarse material in the hole and the manual BH method oversamples the coarse particles. The RC sampling system is unbiased compared to the complete RC material. The new RC rig shows both representative sampling results as well as increased reconciliation reliability.
Crawford *et al.*[11]	Investigation of manual BH sampling proved it to inaccurate and have poor repeatability. Trials showed that RC sampling was able to produce better sample representativity and depth flexibility, but the cost of RC was too high for the operation. The solution for improved BH sampling was to implement an automatic sampling system for the BH rig. This sampler could collect four samples over the 8 m drill hole depth. Even though some loss of ultrafines resulted in a sampling bias, the flexibility and improvement of sampling representativity compared to manual sampling outweighed the concerns about ultrafines.

Publication	Main conclusion from publication
El Hajj *et al.*[12]	Manual sampling from a BH drill rig and RC sampling was compared to each other as well as to a DD reference. Both BH and RC sampling overestimate Au and Cu grades, but the bias for BH is about double to RC. The manual BH sampling is in turn underestimating the Au and Cu grade leading to satisfactory, but illusory, reconciliation results. Conclusion show that the manual BH sampling method was not suitable for reconciliation. There was also concluded that the estimate errors from the sampling method was not as significant as the errors from the type of drill rig used. Recommendations are to work with automated RC drill sampling in spite of extra cost and more traffic in the mine.
François-Bongarçon[13]	A comprehensive discussion of advantages and disadvantages of both BH and RC drill sampling without prerequisite assumption on one or the other's superiority. Conclusion states that the advantages and disadvantages are not clear-cut between the two methods, and certainly not as much as previously presented. Resolution is concluded to be a more critical factor than the performance of each sampling method as long as greatest of care is given to obtaining unbiased samples.
Goers *et al.*[14]	Three different RC drill sampling systems on two different drill rigs have been tested. The systems tested were: conventional cyclone and three tiered splitter sampling system, the Rotaport cone splitting system and the Prograde PGX1350R sampling system. Field duplicates and fines samples were collected to assess the sampling performance during the testing. Results show that the fines have a different grade then the rest of the material, meaning it is essential for the sampling system to sample the fines as well, which the PGX1350R managed. Field duplicates cannot alone be used to assess sample quality as loss of material from the drill hole or sampling systems is not detected. "With sample analysis costs of US$25–30 per sample and annual total drilling assay costs of US$1–1.5 M confidence that sample quality is high is critical. The efforts and costs to produce these high quality samples are justified with the knowledge that the downstream effects of poor samples and the decisions made from them can result in the loss of profits and increase in production costs."
Gomes *et al.*[15]	Case study of the mine to mill reconciliation including analysis of possible BH sampling biases. Comparison was made between manual BH sampling using a canvas and using a drum fitted to the drill, with small opening for the drill rod. Results show that the normal method resulted in a loss of fines as the mass of the drum sample was 8.7% greater and the relative mass of the two finest fractions were much larger than for the canvas method. The study led to development of a BH sampler with cupola that further improved sample representativity.
Hapugoda *et al.*[16]	Comparison of DD, RC and RAB drill sampling methods. Conclusion is that DD is able to produce the best samples but is expensive and slow. RC has a better sample recovery and provide reasonably uncontaminated samples compared to the RAB sampling. Some identified problems with RC include damaged pipes, excessive dust generation. Advantages of RAB drilling include lowest cost, greater speed and large sample volume.
Hapugoda *et al.*[17]	More or less the same article as above, published in a different forum. Identical evaluation, results and conclusion.
Holmes[18]	Theoretical discussion about problems and solution with lack of representativity for BH sampling. Recommendation include taking sectorial or radial cuts from the BH cone, either using some sort of sectorial cutters placed prior to drilling, or using a shovel after the drilling is finalised. Using an automatic sample divider on a cyclone that collects the drill cuttings is also recommended but has many problems like loss of material around the blast hole as well as loss of fines in the dust filter. RC sampling is mentioned as a recent advance for drill sampling but not evaluated for representativity.
Holmes[19]	Theoretical discussion about problems with BH sampling similar to above publication. RC drill sampling is presented as being considered best solution for open pit mine sampling even due to the much higher cost. Presented solutions for accepted BH sampling is extracting radial sectors, vertical slices or channel cuts from the BH cone. Another suggestion is automated collection of drill cutting using compressed air and a cyclone. Best approaches for BH are, however, considered to be channel sampling or sectorial cutters.
Hoogvliet[20]	A case study of a gold and silver mine in Borneo where the grade control system was changed from BH sampling to RC sampling. The original sampling method was to collect samples over 2.5 m using a wedged pie sampler at the collar of the blast hole, any existing sub-drill is not sampled. After viability studies showing improved profits, the grade control was changed to RC drilling. Reconciliation studies show that the annual profit increased by approximately US$2.87 M after implementation of RC. Even after deducting the extra cost for drilling, over US$2 M remained. The authors conclude that desktop studies comparing different drill sampling methods are not sufficient and often overestimate possible profits. The best method to evaluate a new method is by reconciliation and actual produced ounces, i.e. profitability. Another conclusion is that even if RC in some cases has a large impact on profitability, the benefits over BH sampling may be minimal in some other situations.

Publication	Main conclusion from publication
Kirk *et al.*[21]	Evaluation of BH sampling in regards to RC sampling and evaluation of implementation of RC drill sampling systems as a substitute for the BH sampling. BH samples were collected every 2.5 m intervals using a wedge-shaped sampling tray placed radial to the drill string. Two previous studies had indicated that the BH sampling performed reasonably well for high-grade or and waste samples. For low- to medium-grade samples the BH sample was biased as the low grade mineralisation occurs predominantly in the fines that was lost in the BH drilling process and therefore not sampled. Some other BH sampling problems were high top size compared to sample size, as well as frequent collar collapses contaminating the samples. The main result of the biased BH sampling was that approximately 30% of low grade ore was misclassified as waste due to the loss of fines. The BH sampling was also showed to be less accurate than RC and DD sampling.
Magri *et al.*[3]	Theoretical simulation of the economic losses due to poor BH sampling as well as using kriging or polygonal estimation as estimation method. 10%, 20% and 30% fundamental sampling error for BH sampling was used in the simulation to approximate the losses. These numbers are derived from sample systems commonly used in the mining industry (not explained how). The study shows that both estimation methodology and sampling errors lead to losses of millions of dollars per annum.
Magri *et al.*[22]	Case study with comparison between BH and RC drill sampling as well as collection of complete BH cones. Duplicate samples from the BH manual sampling was also collected for analysis of precision. Results show that radial bucket BH sampling is biased compared to both complete BH cone and RC samples for $CaCO_3$. The study also compared previous results from DD, RC and BH which showed good correspondence between all methods and biases between BH and DD were lower then between BH and RC. Variograms were used to estimate nugget effect and these were very low for both RC and DD, but BH nugget effect was considerably larger in spite the larger support for BH. Conclusion is that "Higher quality samples and better short term planning could be achieved by replacing BH sampling with RC sampling, if an economic analysis which includes the hidden costs of misclassified blasted material supports the change."
McArthur[23]	Case study of manual BH sampling in flitch mining. Experiments were carried out to evaluate if the manual method to divide the BH cone in upper and lower flitch and sub-drill is representative. Result show large variability in the ratio between flitches and sub-drill causing problems when sampling. The sub-drill is over represented by an average of 10%. The conclusion is that despite the misallocation of some material between flitches and sub-drill, comparative assay result show that the manual sampling method produce and overall unbiased result.
Minkkinen *et al.*[24]	Case study of a new automatic sampler for BH drill rig. A sampling belt collects a sectorial sample from the drill cutting ejection and transfer the material to a rotating cone splitter. Full BH cone samples were used as reference and in general the new sampling method showed good agreement with this reference.
Niemeläinen *et al.*[25]	Test of an on-line XRF analyser for percussion surface drill rig. The conclusions are that the system is equally representative as DD and RC drill sampling, but much faster. Some deviations between results could be seen but is expected to come from calibration problems. The on line analyser does not collect the dust (similar to RC) as this is deviated by the dust collector.
Ortiz *et al.*[26]	The authors conclude in the introduction that BH samples have poor quality due to time and space constraints, that most BH sampling methods suffer from delimitation, extraction and segregation-related errors. The authors use a simulation methodology applied to three case studies to evaluate the performance of different sampling methods on different drilling grids. The relative error for BH sampling is evaluated by duplicate sampling to a range between 14% and 20% while the error for RC sampling is set from zero to 8%. The conclusion is that moving from BH to RC sampling provides significant economic benefits reaching millions of dollars per annum. "The case studies show that when operating conditions allow for a dedicated drilling rig, it is worth considering investing in a sophisticated sampling system mounted on an RC drilling rig to operate well in advance, thus providing timely data for building short-term models that can include several additional relevant variables."
Pitard[27]	Theoretical discussion regarding sampling, including RC and BH sampling methods. Problems with RC sampling is said to be down the hole contamination, preparation error, selective separation of coarse and fine particles and poor or excessive recoveries leading to extraction biases. BH sampling is presented as a monumental problem for the mining industry due to delimitation, extraction and preparation biases. The author also discusses three new automated BH sampling methods that are stated to be able to produce correct and representative samples. As the systems are not yet in production it is not clear which will be most reliable, but they do represent a major breakthrough in ore grade control for the mining industry according to the author.

Publication	Main conclusion from publication
Pitard[2]	Theoretical discussions about problems associated with BH sampling and advantages with RC drilling. Examples of BH sampling problems presenter are: upward/downward contamination, upward material losses, refluxing, sub-drill material, pile segregation, pile shape irregularities, loss of fines, operator dependent sampling, sampling interfering with mining productivity, too small sample size, vertical drill holes and so on. A few presented advantages with BH sampling are: the same drilling technique for blasting and grade control, small visible cost, good lateral interpolation, less traffic in the pit. Presented advantages with RC sampling are: absence of sub-drill, possibility to drill several benches at once and to drill at an appropriate angle, limited contamination and losses, no interference with productivity, can drill months ahead of mining, possibility to drill less but better holes, smaller sample mass, information from lower benches, better vertical definition of ore and waste, automation is easy, and so on. The disadvantages with RC presented are: additional visible cost, increase in traffic in the pit. The conclusions are that BH sampling cannot provide representative samples and that RC sampling provide many advantages that may far outweigh the additional cost.
Séguret[28]	Case study of a copper mine in Chile. Comparison between BH sampler and DD using 3000 DD samples and 13,000 BH samples for the study. The authors use vertical and horizontal variograms, migration and cross variograms to evaluate the sampling methods. Conclusion show that DD sampling has errors and that both DD and BH variograms show approximately 50% nugget effect. Analysis of the BH error leads to conclusion that it is not the primary sampling step that generates the error, but it can rather be found later in the process. The authors suggest that DD and BH are used together for short term mine planning and that linear systems can be used to remove nugget effect from the data.
Spangenberg *et al.*[29]	The authors state RC drilling as preferred open pit mine sampling with no discussion regarding BH sampling. The authors discuss a few aspects of RC splitters that are biased and should be avoided. A specific sample mass reduction solution is mentioned as being representative and therefore correct.
Young[30]	Case study of a Zn/Pb/Ag mine where traditional BH sampling was replaced by RC sampling. BH sampling was conducted by using a PVC pipe, collecting eight increments from the BH cone. Problems with this method include: cone destruction by rigs, hole vs sample number mismatch, incorrect sampling technique, vertical drill holes in 75° ore body and time constraints. The BH sampling is, however, stated to have been relatively reliable when blasting benches of consistent height. Implementing RC sampling instead of BH did not increase cost for samples handling as the drill grid increased but the samples per hole increased. However, the cost of drilling increased due to the dedicated sampling drill holes that are not drilled when sampling blast holes. Comparison between the sampling methods (using RC as reference) show that BH sampling misclassified 18% of waste as ore and 13% of ore as waste. The cost of processing this waste without cost of lost opportunity (ore going to waste) more than covers the cost of RC drilling.
Ziegelaar *et al.*[31]	This publication only has an abstract and no prepared article for the conference presentation. The study is a comparison of different drilling techniques with DD used as reference. No conclusions are given by the abstract.

Figure 9. Left: drilling operations using a conventional cyclone and three-tiered splitter system. Right: drilling operations using the PGX1350R sampling system. Reproduced from Reference 14 with permission.

Table 3. Context and data collection methods in publications.

Publication	Theoretical discussion	Case study	Use of existing grade control data	Experiment generating new data
Total: 31 publications	10	22	12	16
Abzalov *et al.*[4]		✓	✓	✓
Abzalov *et al.*[5]		✓		✓
Alfaro[6]		✓	✓	
Caccioppoli *et al.*[7]		✓		✓
Carrasco *et al.*[1]	✓	✓	✓	
Chieregati *et al.*[8]	✓	✓	✓	
Chieregati *et al.*[9]		✓		✓
Chieregati *et al.*[10]		✓		✓
Crawford *et al.*[11]		✓		✓
El Hajj *et al.*[12]		✓	✓	✓
François-Bongarçon[13]	✓			
Goers *et al.*[14]		✓	✓	✓
Gomes *et al.*[15]		✓		✓
Hapugoda *et al.*[16]		✓		✓
Hapugoda *et al.*[17]	More or less the same article as above, published in a different forum.			
Holmes[18]	✓			
Holmes[19]	✓			
Hoogvliet[20]		✓	✓	
Kirk *et al.*[21]		✓	✓	✓
Magri *et al.*[3]	✓		✓	
Magri *et al.*[22]		✓	✓	✓
McArthur[23]		✓		✓
Minkkinen *et al.*[24]		✓		✓
Niemeläinen *et al.*[25]		✓		✓
Ortiz *et al.*[26]	✓	✓	✓	
Pitard[27]	✓			
Pitard[2]	✓			
Séguret[28]		✓	✓	
Spangenberg *et al.*[29]	✓			
Young[30]		✓		✓
Ziegelaar *et al.*[31]	Abstract only, no paper was prepared for this presentation			

to generate a large increase in profit when substituting BH sampling.

One major concern that is widely addressed is that the cost of RC drill sampling is "too high". Even when improved sampling performance can be proved, the increased cost for RC sampling is typically not accepted by the mining operation. This is most often due to the fact that it is more or less impossible to exactly quantify the possible value gain or economic losses due to inaccurate BH sampling.

There are several mining operations using RC drill sampling for short-term grade control despite the higher costs. Especially in precious metals mining, the improvements with RC drill sampling have proven to result in larger profit increase than the cost of drilling.[20]

However, the conclusion regarding representativity of RC drilling is not uniform in all publications. Some publications state as a prerequisite that RC is representative, while others conclude that RC, just as BH sampling, can be proven to be biased. Goers[14] evaluates different RC drill sampling systems and concludes that the choice of sampling system for the RC rig as well as the complete system for RC sampling and handling determines if sampling can be representative. Loss of fines, leading to sample bias, is for example a major problem with some RC sampling systems, see Figure 9.

Conclusions

The literature review collected a total of 31 publications (two were more or less duplicates and one consisted of an abstract only). The main source for publications on RC and BH drill sampling were dedicated sampling conferences, other mining conferences and some publications were found in peer-reviewed journals.

From the gathered publications, it is not possible to draw a general overall conclusion as to the superiority of one drill sampling method over another. Both RC and BH have advantages and disadvantages, and the choice of system needs to be related to the ore type and to the mining conditions. The overall conclusion is that it is always necessary to evaluate the specific sampling system to be used in the light of TOS (and with respect to the characteristics of the ore to be mined). It is always necessary to ascertain that the specific drilling sampling system contemplated does not lead to hidden losses that could have been avoided, or missed profits that could be gained with a more relevant and representative sampling system.

It would appear that the mining industry is doomed to continue to follow local, often economy-driven objectives and sampling solutions even if these can be documented as inferior when seen in the light of the representativity imperative. A call is made for universal adherence to the principles laid down by TOS for representativity in the

Table 4. Scope and reference methods used in publications.

Publication	Evaluation of BH sampling	Evaluation of RC sampling	Comparison between RC and BH sampling	Full BH cone used as reference	RC drilling used as reference	DD used as reference	Plant feed or reconciliation used as reference	Prerequisite assumption of RC as being representative	Conclusion show RC to be more representative than BH drill sampling	Conclusion show that RC drill sampling methods can be non-representative	Conclusion show RC and BH drill sampling to be equally (non) representative	Conclusion show that BH sampling can be representative / fit-for-purpose	Conclusion show that BH sampling is not representative	Sampled ore used to draw conclusion
Total: 31 publications	12	4	15	6	5	9	3	7	11	5	5	13	6	
Abzalov *et al.*[4]			✓	✓		✓				✓	✓	✓		Fe
Abzalov *et al.*[5]			✓							✓	✓			Fe
Alfaro[6]	✓					✓							✓	Cu, Mo
Caccioppoli *et al.*[7]			✓		✓			✓	✓					Fe
Carrasco *et al.*[1]	✓					✓							✓	Cu
Chieregati *et al.*[8]	✓	✓									✓	✓		Au
Chieregati *et al.*[9]	✓						✓					✓		Au
Chieregati *et al.*[10]			✓	✓	✓			✓						Au, Cu
Crawford *et al.*[11]			✓	✓					✓			✓		Fe
El Hajj *et al.*[12]			✓	✓		✓		✓						Au, Cu
François-Bongarçon[13]			✓							✓	✓	✓		—
Goers *et al.*[14]		✓			✓					✓				Au
Gomes *et al.*[15]	✓						✓					✓		Au
Hapugoda *et al.*[16]			✓			✓			✓					Au, Cu
Hapugoda *et al.*[17]	More or less the same article as above, published in a different forum.													
Holmes[18]	✓											✓		—
Holmes[19]	✓								✓			✓		—
Hoogvliet[20]			✓				✓		✓					Au, Ag
Kirk *et al.*[21]			✓			✓			✓					Pt
Magri *et al.*[3]	✓						✓						✓	Au, Cu
Magri *et al.*[22]			✓	✓		✓			✓					Cu
McArthur[23]	✓											✓		Fe
Minkkinen *et al.*[24]	✓			✓								✓		Fe
Niemeläinen *et al.*[25]			✓		✓	✓					✓			Cu, Ni
Ortiz *et al.*[26]			✓					✓	✓				✓	Au, Cu
Pitard[27]	✓	✓								✓		✓	✓	—
Pitard[2]			✓					✓	✓				✓	—
Séguret[28]	✓					✓						✓		Cu
Spangenberg *et al.*[29]		✓						✓	✓					Au
Young[30]			✓		✓			✓	✓			✓		Zn, Pb, Ag
Ziegelaar *et al.*[31]	Abstract only, no paper was prepared for this presentation													

primary sampling stage, before economic, logistical or other (local) factors are allowed to intervene. What is the objective to analyse and to make decisions in the mining industry, based on samples that can be documented not to be representative?

References

1. P. Carrasco, P. Carrasco and E. Jara, "The economic impact of correct sampling and analysis practices in the copper mining industry", *Chemometr. Intell. Lab. Syst.* **74,** 209–213 (2004). doi: https://doi.org/10.1016/j.chemolab.2004.04.013
2. F.F. Pitard, "Blasthole sampling for grade control—the many problems and solutions", in *Proceedings Sampling 2008*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2008).
3. A. Magri, E. Magri and C. Neira, "Blasthole sampling validation at Mantoverde", in *Proceedings 5th World Conference on Sampling and Blending*, Santiago (2011).
4. M. Abzalov, B. Menzel, M. Wlasenko and J. Phillips, "Grade control at the Yandi iron ore mine, Pilbara Region, Western Australia—comparative study of the blastholes and reverse circulation holes sampling", in *Proceedings Iron Ore*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2007).
5. M.Z. Abzalov, B.B. Menzel, M.B. Wlasenko and J.B. Phillips, "Optimisation of the grade control procedures at the Yandi iron-ore mine, Western Australia: Geostatistical approach", *Trans. Inst. Min. Metallurg. B* **119(3),** 132–142 (2010). doi: https://doi.org/10.1179/1743275811y.0000000007
6. M. Alfaro, "Blast hole sampling in two areas of the same deposit", in *7th World Conference on Sampling and Blending*, Bordeaux (2015).
7. A.D. Caccioppoli and M.J. Candy, "Iron ore grade control for flitch mining – an alternative method for the Hope Downs Operation, Hamersley Province, Western Australia", in *Proceedings Eighth International Mining Geology Conference*, Queenstown (2011).
8. A.C. Chieregati and F.F. Pitard, "The challenge of sampling gold", in *Proceedings 4th World Conference on Sampling and Blending*, Cape Town (2009).
9. A.C. Chieregati, B.B. Frontini, D.B. Carvalho, F.F. Pitard and K.H. Esbensen, "Experimental validation of new blasthole sampler for short-term planning", in *Proceedings 5th World Conference on Sampling and Blending*, Santiago (2011).
10. A.C. Chieregati, T. El Hajj, C.F. Imoto and L.E. Pignatar, "Validation of reverse circulation drilling rig for reconciliation purposes", *TOS forum* **Issue 5,** 105–110 (2015). doi: https://doi.org/10.1255/tosf.58
11. D. Crawford, P. Leevers, S. Parsons, G. Solly and S. Nielsen, "Grade control in the Southern Middleback Ranges—a case study", in *Proceedings Iron Ore 2005*, Fremantle (2005).
12. T. El Hajj and A.C. Chieregati, "Illusory reconciliation: compensation of errors by manual sampling", *Proceedings 5th World Conference on Sampling and Blending*, Lima (2013).
13. D. François-Bongarçon, "Are blasthole samples that bad and reverse circulation samples that good for open pit grade control story of a trade-off", in *Proceedings Sampling 2010*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2010).
14. M. Goers and M. Almond, "Bit to bag—the importance of a total coordinated system in reverse circulation drilling", in *Proceedings Sampling 2012*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2012).
15. W.L. Gomes, A.C. Chieregati, H. Delboni and D.B. Carvalho, "Mine-to-mill reconciliation variability study of blast hole sampling in Serra Grande gold mine", in *Proceedings 5th World Conference on Sampling and Blending*, Santiago (2011).
16. S. Hapugoda and J.R. Manuel, "A comparison of drilling and sampling techniques as they relate to base and precious metal exploration in the Mt Isa inlier of North West Queensland and the Southern Lachlan Fold Belt in New South Wales", in *Proceedings Sampling 2010*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2010).
17. S. Hapugoda and J.R. Manuel, "Techniques used and problems encountered in normal and reverse circulation drilling at the Mt Roseby Project, Cloncurry, North West Queensland", in *Proceedings XXV International Mineral Processing Congress*, Brisbane (2010).
18. R.J. Holmes, "Correct sampling and measurement—the foundation of accurate metallurgical accounting", *Chemometr. Intell. Lab. Syst.* **74,** 71–83 (2004). doi: https://doi.org/10.1016/j.chemolab.2004.03.019
19. R.J. Holmes, "Sampling mineral commodities—the good, the bad and the ugly", *J. S. Afr. Inst. Min. Metall.* **110,** 269–276 (2010).
20. H. Hoogvliet, "Drill blast versus reverse circulation grade control sampling—a case study from the Mt Muro gold silver mine, central Kalimantan, Indonesia", in *Proceedings Fifth International Mining Geology Conference*, Bendigo (2003).
21. G. Kirk, T. Muzondo and D. Harney, "Improved grade control using reverse circulation drilling at Mogalakwena Platinum Mine, South Africa", in *Proceedings Eighth International Mining Geology Conference*, Queenstown (2011).
22. E. Magri and J.M. Ortiz, "Estimation of economic losses due to poor blast hole sampling in open pit mines", in *Proceedings of 6th International Geostatistics Congress*, Cape Town, Vol. 2, pp. 732–741 (2000).
23. G. McArthur, C. Jones and M. Murphy, "Blasthole cone sampling experiments for iron ore flitch mining", in *Proceedings Sampling 2010*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2010).
24. P. Minkkinen, I. Auranen, L. Ruosalainen and J. Auranen, "Comparison of sampling methods by using size distribution analysis", *TOS forum* **Issue 5,** 175–185 (2015). doi: https://doi.org/10.1255/tosf.63
25. E. Niemeläinen, J. Raatikainen, J. Siikaluoma and I. Auranen, "Comparison of traditional and novel on-line blast hole sampling in ore grade control", in *Proceedings 5th World Conference on Sampling and Blending*, Santiago (2011).
26. J.M. Ortiz and E.J. Magri, "Designing an advanced RC drilling grid for short-term planning in open pit mines: three case studies", *J. S. Afr. Inst. Min. Metall.* **114,** 631–637 (2014).
27. F.F. Pitard, "New sampling technologies for ore grade control, metallurgical accounting, and laboratory preparation", in *Proceedings MetPlant 2004*, Perth (2004).
28. S.A. Séguret, "Geostatistical comparison between blast and drill holes in a porphyry copper deposit", *TOS forum* **Issue 5,** 187–192 (2015). doi: https://doi.org/10.1255/tosf.51
29. I.C. Spangenberg and R.C.A. Minnitt, "An overview of sampling best practice in African mining", *J. S. Afr. Inst. Min. Metall.* **114,** 91–102 (2014).
30. B. Young, "From blasthole to RC sampling—changing grade control methods at Black Star open cut zinc-led mine, Mount Isa, Queensland", in *Proceedings Eighth International Mining Geology Conference*, Queenstown (2011).
31. B. Ziegelaar, P. Bleakley, G. Lee and R. Millington, "Grade variations from various drilling methods in Darling Range bauxite", in *Proceedings Sampling 2010*. The Australasian Institute of Mining and Metallurgy and CSIRO, Perth (2010).

WCSB8

8th World Conference on Sampling and Blending

9–11 May 2017, Perth, Western Australia

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Invitation

The 8th World Conference on Sampling and Blending (WCSB8) is being held in Perth, Australia, on 9-11 May 2017, and follows on from the previous successful conferences in the series held in Denmark, Australia, Brazil, South Africa), Chile, Peru and France. WCSB8 is being combined with the Australian Sampling conference normally held every two years, and hence is being jointly organised by The Australasian Institute of Mining and Metallurgy (The AusIMM) and CSIRO, with the support of FLSmidth as a Platinum Sponsor, Thermo Fisher Scientific as a Gold Sponsor, and IMP Innovative Solutions and Multotec as Silver Sponsors. The conference aims to bring together all parties involved in the many aspects of sampling and blending in the mineral, pharmaceutical, food, feed, agricultural, biomass and recycling industries, including environmental monitoring. Sadly, Pierre Gy passed away in Bordeaux, France, in November 2015, so WCSB8 will be dedicated to his memory and lifetime achievements in sampling mineral commodities.

Despite the wealth of knowledge available on correct sampling principles and practice, it is surprising how little attention and resources are sometimes dedicated to extracting representative samples. Quite often, everyone appears satisfied as long as some material is collected and delivered to the laboratory for analysis. Yet, unless the samples are representative, the whole measurement process is flawed at the outset and no amount of re-analysis can fix the problem. Consequently, companies stand to lose millions of dollars in terms of poor investment decisions, wasted resources, poor plant performance, poor product quality and income from product sales. Sampling, therefore, needs to be given the attention it deserves to ensure that the samples extracted are representative so that meaningful decisions can be made based on their analyses.

In addressing this need, WCSB8 will provide unparalleled opportunities for updating your sampling knowledge, benchmarking sampling and QAQC practices, networking, meeting respected international sampling experts, sharing ideas and catching up on the latest developments in sampling, sample preparation and blending of a wide range of different commodities. A number of sampling, grade control and data analysis workshops will also be held both before and after the conference, as well as a technical tour to inspect the latest in sampling and analysis equipment.

On behalf of the Organising Committee, The AusIMM and CSIRO, I invite you to register for WCSB8 in Perth on 9-11 May 2017. I look forward to seeing you in the beautiful city of Perth and trust that you find the conference a rewarding experience.

Yours sincerely,

Dr Ralph Holmes
Conference Chair,
8th World Conference on Sampling and Blending 2017

CSIRO Mineral Resources

ORGANISING COMMITTEE

Conference Chair

Dr Ralph Holmes FAusIMM(CP), CSIRO Mineral Resources

Committee Members

Andre Balt MAusIMM, IMP Group
Jim Docherty, SGS Australia Pty Ltd
Oscar Dominguez MAusIMM, BHP Billiton Iron Ore Exploration
Simon Dominy FAusIMM(CP), Exchange Minerals / WA School of Mines
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Claudia Paoletti, European Food Safety Authority - EFSA
Rodolfo J Romañach, University of Puerto Rico - Mayaguez
Antonia Riley, CSIRO Mineral Resources
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Mia Wotherspoon, Coordinator, Publishing

WHY ATTEND WCSB8 2017

- WCSB8 is the leading industry event dedicated to providing practical advice and knowledge sharing to enhance operations across sampling and blending.
- WCSB8 will bring together all parties involved in sampling and blending in the mineral, pharmaceutical, food, feed, agricultural, biomass and recycling industries, including environmental monitoring.
- Engage in valuable discussions with the specialised operators who will be presenting real and recent case studies and valuable experiences.
- Network with industry providers during the trade show who are keen to share their knowledge with you.
- Challenge yourself and attend related professional development workshops to further your knowledge.

CONFERENCE THEMES

- Tributes to Pierre Gy
- Theory of sampling and blending
- Geostatistics
- Sampling and blending mineral commodities
- Quality control and metallurgical accounting
- Sampling of feed, agricultural and biomass products
- Sampling and quality control in the pharmaceutical and food industries
- Sampling of wastes and recyclable materials
- Environmental sampling and monitoring
- New developments in sampling, sample preparation and blending equipment
- Future technologies
- Development of national and international standards
- Case studies

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PROGRAM AND KEYNOTE SPEAKERS

The Organising Committee is currently developing an exciting technical program that will feature a range of topics presented during various keynote and plenary sessions.

The following is a preliminary program outline to assist in your planning. Please note that the program is subject to change. A detailed program will be available shortly.

PRELIMINARY TIMETABLE

Sunday 7 May	Monday 8 May	Tuesday 9 May	Wednesday 10 May	Thursday 11 May	Friday 12 May
W4: Sampling Theory, Sampling Practices, and Their Economic Impact	W4: Sampling Theory, Sampling Practices, and Their Economic Impact	Day 1 WCSB8 Conference Exhibition	Day 2 WCSB8 Conference Exhibition	Day 3 WCSB8 Conference Exhibition	Post-conference Tour
	W2: Theory of Sampling and Multivariate Data Analysis	Welcome Reception	Networking Hour		W1: A Practical Guide to Designing and Running Effective Sampling Programs
	Exhibition setup		Conference Dinner		W3: Grade Control in Underground Gold Operations
	Pre-conference Registration				

WELCOME RECEPTION

This opening networking event sets the tone for the conference as the industry congregates at this first function. The Welcome Reception is where you can catch up with old colleagues and form new contacts.

Date: Tuesday 9 May 2017
Venue: Pan Pacific Perth Hotel
Time: 5.30 pm – 6.30 pm
Cost: Complimentary for all delegates
Guests: A$66

NETWORKING HOUR

Following the second day of the conference, the Networking Hour is a superb networking opportunity for all delegates and exhibitors. Held amongst the exhibition, it's a chance to walk through the stands, relax and mingle.

Date: Wednesday 10 May 2017
Venue: Pan Pacific Perth Hotel
Time: 5.30 pm – 6.30 pm
Cost: Complimentary for all delegates
Guests: A$33

CONFERENCE DINNER

The social highlight of the conference! Join your fellow colleagues and enjoy a fabulous three course meal, beverages and entertainment.

Date: Wednesday 10 May 2017
Venue: Pan Pacific Perth Hotel
Time: 7.00 pm for a 7.30 pm start
Cost: Complimentary for all delegates
Guests: A$132

KEYNOTE SPEAKERS

Dr Isobel Clark FAusIMM
Director and Principal Consultant, Geostokos Limited, Scotland

Isobel has taught, researched and consulted in the field of geostatistics for over 42 years. Possibly best known as the author of the introductory text *Practical Geostatistics* (1979), and co-author of a more complete textbook, *Practical Geostatistics 2000*.

Dr Clark lectured at the Royal School of Mines, London, at the University of the Witwatersrand in Johannesburg, was visiting professor for Camborne School of Mines and is currently visiting professor of practice at the University of Johannesburg.

To subsidise these academic engagements, she has been a Director of Geostokos Limited, an international consultancy company based in Central Scotland for 35 years.

Dr Kathy Ehrig MAusIMM
Principal Geometallurgist, BHP Billiton Olympic Dam, Australia

Kathy graduated with a BSc in Geology in 1984 and conducted geophysical surveys in the search of geothermal energy resources for the US Navy in the mid-1970s to early 1980s. She then completed a PhD in Geology from the University of California in 1991 and left San Francisco in 1992 to join the former WMC as a research geologist to work on the genesis of the Olympic Dam (OD) deposit and to provide mineralogical support to metallurgy.

Kathy worked and lived at OD from 1992-2006 and held various roles as research geologist, senior research geologist, and chief research geologist. Kathy then joined BHP Billiton in 2005 when WMC was acquired by BHP Billiton and moved to Adelaide in 2006 as Principal Geometallurgist, leading the team who developed and implemented the massive geometallurgy program which is used to support the future expansion of OD.

Her current work consists of optimising the OD geometallurgy models for use in short, medium, and long-term mine planning and developing geometallurgy testing programs to be used in exploration.

KEYNOTE SPEAKERS – CONTINUED

Dr Kim Esbensen,
Professor, Consultant, KHE Consulting, Denmark

After 35 years as a research professor in Geoscience Data Analysis and Sampling at GEUS (National Geological Surveys of Denmark and Greenland), chemometrics professor with the ACABS research group, Aalborg University, Denmark and professor (Process Analytical Technologies) at Telemark Institute of Technology, Norway, in 2015 he phased out this institutional career and established an international consultancy: www.kheconsult.com

A geologist/geochemist/data analyst by training, since 2001 he has devoted his time to the theme of representative sampling of heterogeneous systems and processes (Theory of Sampling, TOS), PAT (Process Analytical Technology) and chemometrics (multivariate data analysis). In 2003 he organized the first World Conference on Sampling and Blending conference.

Dr Claudia Paoletti
Deputy Head GMO Unity, European Food Safety Authority – EFSA, Italy

Claudia did her Masters in Biological Science at the University of Rome (Italy) and her PhD in Plant Genetics at the University of Connecticut, USA. She spent three years at Dalhousie University (Canada) studying plant population genetics and biometry. She continued her research activity at the Research Institute for Industrial Crops in Bologna (Italy) where she focused on the evaluation of the risks associated to the use of transgenic crops. She has been the Italian expert nominated by the European Commission, for definition of sampling plans for GMO detection in conventional seeds. She was responsible for the European Commission sampling research projects on GMOs and she was the biometric officer of the EU Community Reference Laboratory for GMOs. In January 2006 she joined the GMO Unit of the European Food Safety Authority (EFSA). Currently, she is Deputy Head of the GMO Unit and leader of the molecular characterisation, food and feed risk assessment team, focusing on food and feed safety assessment and on the development of statistical and sampling approaches suitable for risk assessment. She has over 75 contributions either as book chapters or scientific papers.

Dr Anita Parbhakar-Fox MAusIMM
Research Fellow in Geoenvironmental Studies, University of Tasmania, Australia

Anita completed her degrees at the Royal School of Mines, UK and CODES, UTAS. Currently she is a postdoctoral research fellow/lecturer in Geoenvironmental Studies at the ARC Transforming the Mining Value Chain Research Hub, UTAS. Anita is focused on mine waste characterisation for improved mine planning and waste management. She has developed new tests and protocols in this area, particularly for acid rock drainage prediction. Anita is also involved in identifying remediation options for abandoned/historical mine sites. Most recently, she has been characterising a range of mine waste materials to re-evaluate their economic potential.

Dr Francis Pitard
President, Francis Pitard Sampling Consultants, USA

Francis has over 40 years of progressive, technical and management experience in the natural resources industry and atomic energy. Accomplished in teaching short courses on the sampling of particulate materials for several universities and numerous companies around the world, in consulting, in directing the activities of a production oriented research analytical facility with emphasis on innovation and cost effectiveness. Versatile in applying talents in a variety of areas including nuclear chemistry, analytical chemistry, geochemistry, and statistical process control. Outstanding expertise in all aspects of sampling accumulated during a 20 year association with Dr C O Ingamells and Dr Pierre M Gy. Dr. Pitard is the author of many papers, three books on sampling and a gold medal recipient from the World Conference on Sampling and Blending.

Prof Rodolfo J Romañach
University of Puerto Rico, Mayaguez

Dr Romañach is Professor of Chemistry at the University of Puerto Rico – Mayagüez Campus, and site leader for the Engineering Research Center on Structured Organic Particulate Systems (http://ercforsops.org). He worked in the pharmaceutical industry for over 12 years before joining the Chemistry Department in 1999. His research is focused on developing a thorough understanding of the uncertainty of real time and at-line sensor measurements and sampling error in pharmaceutical analysis, and includes over 60 publications. Five graduate students have completed their PhD studies in his research group, and 14 students have completed their MS in Chemistry.

He has worked in the sampling and analysis of pharmaceutical blends for over 25 years. In 2010, he read Pierre Gy's *Sampling for Analytical Purposes* and found that TOS provides a much needed insight for the development of real time analytical methods. He is currently collaborating with Kim Esbensen on the application of TOS to pharmaceutical applications.

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PRELIMINARY LIST OF PAPERS

Case Studies

Sampling of the mineralised tailings dumps – case study of the Mount Morgan project, Central Queensland, Australia — *M Abzalov and C Newman*

Validation of a modified cross-belt sampler for reconciliation purposes — *A C Chieregati, E A Amaral Jr and J C O Souza*

Blasthole sampling (replicate and variographic experiments) in LKAB open pit iron ore mines – fit-for-purpose representativity? — *K Engstrom and K H Esbensen*

Pitfalls in vezin sampling of finely crushed materials — *C J Kruger and E le Roux*

Variographic case study for designing, monitoring and optimising industrial measurement systems – the missing link in LEAN and Six Sigma — *E Thisted, U Thisted, O Bøckman and K H Esbensen*

Heterogeneity tests and core logging – a final reconciliation — *F L S P Villanova, A Heberle and A C Chieregati*

Development of National and International Standards

A practical guide to sampling in coal preparation plants — *B W Atkinson*

Optimising sampling protocols for aluminium ore – a new approach for international standards — *D A Bortoleto, A C Chieregati and R C Oliveira*

Environmental Sampling and Monitoring

Sampling, monitoring and source tracking of dioxins in the environment of an incinerator in the Netherlands — *A Arkenbout and K H Esbensen*

Geostatistics

Bailer uncertainty evaluation in a lithium salar deposit — *S A Séguret, P Goblet, E Cordier and A Galli*

New Developments, Sample Prep and Blending Equipment

Proposed workflows for portable XRF and NIR instruments for soil and drill hole sampling — *H L Bridgwater and N H Jansen*

An automatic sampling methodology to determine contractual PSD in rapid load out stations — *S C Labram and A Stabile*

New on-line/at-line splitter designs for laboratory automation – first feasibility results — *M Lischka, A Hollweg and K H Esbensen*

Quantifying segregation of minerals and metals in particulate materials using computed X-ray tomography and variography — *R C A Minnitt, T Jashashvili and G Gilchrist*

Process Analytical Technology

Inline analysis of washability parameters for process control — *J F Bachmann, H B Wurst, C C Bachmann, M P Cipold and J Ha*

Near real time assay with down hole assay tool (FastGrade 100) — *R A Maddever, A Mahanta, B Chi and O Dominguez*

On-line X-ray fluorescence spectrometer for accurate process monitoring and improving blending sharpness — *M G C Zoontjes, T van der Maten, L Kempenaers and U König*

Quality Control and Metallurgical Accounting

Online quality control of a coal blending yard — *J F Bachmann, C C Bachmann, M P Cipold and J Ha*

Optimised sampling protocols for the grade and metallurgical evaluation of selected vein gold deposits — *S Dominy*

Use of variography to measure improvement of sampling practices for the quality control of graphite consistency in marble used for carbon-sensitive applications — *C Haughty and F F Pitard*

A new approach to implement QAQC to technological innovations – quality of spectral data capture and processes in the minerals industry — *D Mittrup, O Dominguez and M Haest*

Does process control sampling always have to be a compromise? — *R Steinhaus*

The benefits of automated metallurgical accounting for plant sampling — *J P Vagenas and D W Wall*

Sampling and measurement for percentage moisture in the iron ore industry — *B Ziegelaar and M Fritz*

Sampling and Blending Mineral Commodities

Sampling coarse gold mineralisation – developing effective protocols and a case study from the Ballarat East mine, Australia — *S Dominy*

Modelling ore flow in the design of high capacity sampler cutters — *M Hidding and R Shaw*

Common pitfalls in sampling iron ore — *R Holmes*

Comparative evaluation of manual sampling and Outotec MSA 2/50 metallurgical slurry sampler — *J Loimi, P Minkkinen and T Korpela*

Sampling data validation using twin holes in a niobium mine — *M T G C Marques, T M El Hajj, J M Braga Jr and A C Chieregati*

Access, select, include – a review of the commercial sampling of traded bulk commodities in the context of Gy theory of sampling — *D A Vogel*

Sampling and Quality Control in Pharmaceutical and Food

Theory of sampling meets the NSF I-corps™ program — *R J Romanach, C P de la Rosa, V Rodriguez and M Hormaza*

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Optical sieve analysis for online quality control and real time monitoring of the granulation process — *R Waggeling and J Ha*

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The sampling characteristics of grains contaminated by mycotoxins — *G J Lyman and S A Tittlemier*

ISO TC 34/SC 16 horizontal methods for molecular biomarker analysis – molecular biomarker analysis and sampling management system — *M D Sussman*

Sampling of Wastes and Recyclable Materials

Plastics recycling and sampling — *H J Glass and S Dominy*

The theory of sampling applied to the sampling of municipal solid waste for their characterisation – a case study in France — *Ph Wavrer*

Theory of Sampling and Blending

Combining multivariate variographic approach to process modelling to predict metallurgical performance variability — *Q Dehaine and L Filippov*

Sampling hall of fame and sampling hall of shame — *K H Esbensen*

The evolution of the concept of liberation factor and a surprising new result — *D Francois-Bongarcon*

Theory of sampling and geostatistics – the ultimate link — *D Francois-Bongarcon*

The impact of the weighting error — *G J Lyman*

Dependence of the variance of lot average on the sampling mode and heterogeneity type of the lot — *P Minkkinen*

A simplification of Gy's equation for low-grade gold ores – empirical evidence — *R C A Minnitt*

From errors to uncertainty – a clarification for proper use by the theory of sampling — *F F Pitard*

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CONFERENCE WORKSHOP PROGRAM

W1: A Practical Guide to Designing and Running Effective Sampling Programs
Friday 12 May 2017

This one-day workshop has been developed for those involved in the sampling process; laboratory and metallurgy; project evaluation; and resource and exploration geologists.

This workshop focuses on the importance of best sampling practices providing a hands on guide to designing and running effective sampling programs. All concepts are explained using case studies and practical examples.

By the end of the workshop you will:

- be able to critically assess and design sampling and sample preparation systems, from drilling through to process sampling
- understand the importance of good sampling practice
- understand the sources of sampling error and the cost of poor sampling
- understand and apply Gy's sampling theory to sample size selection and the design of sampling protocol.

Workshop presenter:

John Graindorge MAusIMM(CP), Principal Consultant, BSc (Hons) (Geology), Grad. Dip. Geostatistics,

Cost:	AusIMM member: A$1060 per person (GST inclusive) Non-member: A$1249 per person (GST inclusive)
Time:	8.30 am – 4.30 pm
Venue:	TBA
Includes:	Includes lunch and refreshments. Participants will be required to bring a laptop for practical activities

SNOWDEN

7 PD HOURS Ausimm

W2: Theory of Sampling (TOS) and Multivariate Data Analysis (Chemometrics)
Monday 8 May 2017

This one-day workshop is an introduction to chemometrics, sufficient to initiate a professional competence level, in combination with an introductory software package. Participants will be able to perform Principal Component Analysis (PCA), as well as PLS-regression on multi-analyte data matrices, using the UNSCRAMBLER software. The workshop features practical examples and cases, focusing on the interaction between chemometrics and TOS. Participants receive a copy of the new 6th edition of the textbook: *Multivariate Data Analysis in Practice – an Introduction* (USB).

Course background

Why is it that TOS is almost exclusively univariate, ie dealing with one analyte, one variable or one parameter only? This is in stark contrast with the real world in science, technology and industry, which is decidedly multivariate! There have not been many studies, case histories or projects in which the overlapping realm between TOS and multivariate data analysis (chemometrics) is in focus – indeed it is only very recently that Dehaine & Fillippov presented a proper multivariate variogram to our community (WCSB7). There is a challenging future in the multivariate realm, also for the TOS community, even though it is often times believed that 'all one needs to know for appropriate sampling' is to be able to identify the singular analyte with the most heterogeneous distribution. While this may be true in some or even many cases, there never-the-less also exist situations in which a multivariate approach to multi-analyte issues will be of significant value for complete insight into complex materials and systems. The completely updated course textbook has to date helped ~33000 new data analysts and professional scientists, engineers and laboratory technicians to an efficient start.

Workshop presenter:

Dr Kim H Esbensen

Cost:	AusIMM member: A$990 per person (GST inclusive) Non-member: A$1210 per person (GST inclusive)
Time:	8.30 am – 4.30 pm
Venue:	Pan Pacific Perth Hotel
Includes:	Includes course textbook (USB stick) lunch and refreshments

7 PD HOURS Ausimm

W3: Grade Control in Underground Gold Operations
Friday 12 May 2017

This course will demonstrate how to control ore in underground gold mines from a perspective of correct sampling. It has been designed for anyone involved in or about to become involved with the design, implementation and monitoring of an underground gold mine grade control system.

You will learn about:

- what is grade control and introducing ore control?
- importance and value of good sampling
- applied theory of sampling and notion of representivity
- how to sample correctly in an underground environment
- sampling QAQC
- designing and implementing ore control systems
- interfacing between geology and mine design.

The presenter will make extensive use of case studies to illustrate the development of relevant topics. In particular, discussions on how to deal with coarse gold/high nugget ore bodies will be presented.

Workshop presenter:

Dr Simon Dominy FAusIMM(CP)

Cost:	AusIMM member: A$990 per person (GST inclusive) Non-member: A$1210 per person (GST inclusive)
Time:	8.30 am – 4.30 pm
Venue:	Pan Pacific Perth Hotel
Includes:	Includes course notes on USB and refreshments

7 PD HOURS Ausimm

CONFERENCE WORKSHOP PROGRAM – Continued

W4: Sampling Theory, Sampling Practices, and Their Economic Impact

Sunday 7 May & Monday 8 May 2017

The objective of this course is to reach a modern understanding of the theory of sampling of particulate materials and how it can be implemented in practice in a friendly way at mines and processing plants. At the end of the course, attendees will be better equipped to present the economic advantages of good sampling practices. Thus, the course is a prerequisite for bank investment: bankers must listen and trust the theory of sampling.

Note: a scientific calculator and a laptop computer are recommended.

Workshop Presenters:

Dr Dominique Francois-Bongarcon and Dr Francis F Pitard

Cost:	AusIMM member: A$1450 per person (GST inclusive) Non-member: A$1595 per person (GST inclusive)
Time:	8.00 am – 5.00 pm
Venue:	Pan Pacific Perth Hotel
Includes:	Includes course notes on USB, lunch and refreshments for both days

14 PD HOURS AusIMM

For further workshop information, please visit www.wcsb8.com/workshops

CONFERENCE TOUR

Sampling and Laboratory Tour

Friday 12 May 2017, 7.30 am – 5.00 pm

Delegates on this tour will visit the FLSmidth, MinAnalytical and SGS laboratories.

FLSmidth Perth Supercenter

Certified to ISO 9001 the Perth Supercenter is an integrated office, workshop, laboratory, training and warehouse facility delivering a complete package of equipment, services and support to customers under a single roof.

The Supercenter workshop and facilities tour will provide an opportunity for delegates to inspect a range of the Essa® sampling and laboratory equipment as well as any other FLSmidth mineral processing equipment that may be in various stages of fabrication or repair in the 10,000 m² workshop.

This visit is a great opportunity for you to talk to the people responsible for designing and engineering world class sampling solutions.

MinAnalytical Facility

This visit will include a tour of the MinAnalytical facility and automated sampling and XRF laboratory and demonstration of new Ausdrill Rock Commander drill rig and sampling device.

SGS Australia Perth Minerals Laboratory

The final tour location is a visit to SGS Australia's major international Minerals laboratory located at the Perth Airport. The facility includes sample preparation, automated XRF fusion, fire assay for precious metals, instrumental techniques such as ICPOES and ICPMS for base metals analysis, classical techniques for shipment and umpire analysis along with MMI soil geochemistry. The Perth facility is a hub laboratory to the SGS group

Cost:	A$165 per person, including transport and lunch
Time:	7.30 am – 5.00 pm
Requirements:	Attendees are required to wear full long sleeve shirts and full length trousers with steel capped boots. Additional PPE requirements will be provided onsite.
Cancellation Policy:	30-7 days before the tour = 50% refund 7 days or less before the tour or non-attendance = No refund (no exceptions)

GENERAL INFORMATION

CONFERENCE VENUE

Pan Pacific Perth Hotel
207 Adelaide Terrace
Perth Western Australia 6000
Telephone: +61 8 9224 7777
Email: reserve.ppper@panpacific.com
Website: www.panpacific.com/en/Perth/Overview.html

ACCOMMODATION

The Pan Pacific Perth Hotel is pleased to offer delegates a discounted rate of A$210 per night for a Deluxe room and A$230 for a Premier room.

To book this conference rate, please use the following link:
https://aws.passkey.com/e/49044749

EVENT MANAGEMENT: The AusIMM

Miriam Way MAusIMM, Acting Chief Executive; Director, Events
Eliza Sanneman, Team Leader, Senior Coordinator, Events
Mia Wotherspoon, Coordinator, Publishing

The Australasian Institute of Mining & Metallurgy (The AusIMM)
PO Box 660, Carlton South, Victoria Australia 3053
Telephone: +61 3 9658 6105
Email: esanneman@ausimm.com.au
Website: www.ausimm.com

CONFERENCE PROCEEDINGS

All delegates will receive either a printed hard copy or USB version of the conference proceedings. Please nominate your preference when registering. Additional copies of the proceedings may be purchased via the registration form.

Sponsored by HERZOG

- Additional USB Memory Stick Proceedings Cost: A$88
- Additional Printed Proceedings Cost: A$110

REGISTRATION DESK

The registration desk will be open during the following hours:

Monday 8 May 2017	4.00 pm – 6.00 pm
Tuesday 9 May 2017	7.30 am – 5.00 pm
Wednesday 10 May 2017	7.30 am – 5.00 pm
Thursday 11 May 2017	8.00 am – 2.00 pm

CONFERENCE NAME BADGES

All participants at the conference will be issued with a name badge upon registration. Your name badge is the official pass to all sessions and must be worn at all times. Lost name tags can be replaced at the registration desk.

Sponsored by PROCESS IQ

SPECIAL REQUIREMENTS

Every effort is made to ensure people with special requirements are catered for. Should you require any specific assistance or have dietary requirements, please include a notation with your registration form to enable us to make your visit a pleasant and comfortable experience.

DRESS CODE

The dress code for the conference, social functions and workshops is smart business casual.

JUSTIFICATION OF ATTENDANCE

We know that travel and training budgets are tight and it can be difficult to get approval to attend events and conferences. A letter that helps to justify your attendance can be downloaded from the conference website. It details the reasons why attending the Eighth World Conference on Sampling and Blending is beneficial for you and your company.

VISA INFORMATION

All travellers to Australia must have a valid visa before boarding their plane. Travellers to Australia cannot apply for a visa on arrival, with the exception of New Zealand and Norfolk Island passport holders, who will be issued a visa on arrival in Australia.

We strongly advise all participants to apply for a visa at least eight weeks in advance of their intended date of travel. Please see the Department of Immigration and Border Protection's website for further information: www.border.gov.au

WCSB8 8th World Conference on Sampling and Blending
REGISTRATION FORM

All participants are required to officially register to attend the 8th World Conference on Sampling and Blending 2017. Please complete the below registration form or visit the conference website to register online.

1 PERSONAL INFORMATION

Title – Prof / Dr / Mr / Mrs / Miss / Ms (Please circle)

Last Name*

First Name*

Preferred Name*

AusIMM Number (if applicable)

AusIMM Member Post-nominals (if applicable)

Organisation*

Position*

Address*

City* State*

Post Code* Country*

Telephone* Mobile

Email*

☐ Please indicate (✔) if you do **NOT** wish to appear on the list of participants provided to all delegates at the event containing name, position, company and email address

☐ Please indicate (✔) if you do **NOT** wish to receive future AusIMM event information, professional development opportunities and discount offers

Special Requirements

Please advise any special requirements regarding diet and mobility below

TERMS AND CONDITIONS

ATTENDANCE
Only pre-registered, pre-paid registrants will be guaranteed access to the event. Upon receipt of your registration and payment, The AusIMM will send registration confirmation.

REGISTERING ON-SITE
On-site registrants, with payment only, will be admitted on space availability.

AusIMM MEMBER RATES
To qualify for the special rates of 'AusIMM Member' as quoted on the registration booking form, you must be a financial (paid) member. AusIMM 2017 Membership Fees were due by 1 January 2017. Non-member registration fees apply to all non-members and non-financial AusIMM members.

METHOD OF PAYMENT – CREDIT CARD ONLY
Payment must accompany all registrations. We accept the following credit cards: Visa, AMEX, Diners and MasterCard. All enquiries regarding payments, please telephone +61 3 9658 6120

STUDENT REGISTRATION
A student must be currently enrolled full-time at a tertiary institution. Proof of full-time status must be submitted with the registration form.

REGISTRATION ENTITLEMENTS
Full registration:
- Access to all conference technical sessions (excluding workshops)
- Lunch, morning and afternoon teas daily
- Conference name badge and satchel
- Conference printed or USB memory stick proceedings
- One (1) ticket to the Welcome Reception
- One (1) ticket to the Networking Hour
- One (1) ticket to the Conference Dinner*

** Student registration excludes attendance at the Conference Dinner and additional tickets must be purchased to attend.*

Day registration:
- Access to all conference technical sessions on designated day (excluding workshops)
- Lunch, morning and afternoon teas on designated day
- Conference name badge and satchel
- Conference printed or USB memory stick proceedings
- Attendance at the networking function on the evening of your registration

PRIVACY POLICY
The AusIMM is a professional institute that relies on the use of personal information to provide support and services to members and non-member stakeholders and customers of AusIMM services. We rely on comprehensive and accurate personal information about our members and non-members who engage with the AusIMM.

The main purposes for which we collect, hold, use and disclose personal information are to provide services and benefits for our members and to maintain and extend our membership. We collect information from members and non-members so that we can provide services, manage our professional relationships, manage our business, comply with our legal obligations, communicate effectively and enhance the level of service being offered. Please visit the conference website to view the policy.

At the time of registering for this event, you have the option to indicate whether or not you agree to the AusIMM contacting you for promotion of future events, professional development opportunities and discount offers.

PHOTOGRAPHY
By attending this event, I consent to my image being taken and used at the discretion of the AusIMM.

CONFIRMATION OF BOOKINGS
Conference registrations will be acknowledged as they are received with payment in full. Please check the confirmation letter and advise of any alterations immediately.

CANCELLATION POLICY
Cancellations of registration must be in writing only. Refunds will apply as follows:
- More than 28 days before the conference – **Full refund**
- 28–7 days before the conference – **Refund (less A$500 administration charge)**
- 7 days or less before the conference or non-attendance – **No refund (no exceptions)**

An organisation may send an alternative delegate if registration has been paid and the registered person is unable to attend due to unforeseen circumstances. In such cases, Event Management must be advised of the change prior to the conference.

WAIVER OF LIABILITY
The AusIMM and CSIRO accepts no liability to any persons or body for any loss, injury or damage caused, organised, promoted or sponsored by the AusIMM and CSIRO

2 CONFERENCE REGISTRATION Please indicate (✔)

Conference fees are quoted in Australian dollars and include 10% Goods and Service Tax (GST).

FULL REGISTRATIONS		Total
AUSIMM MEMBER	☐ $1320	$
AUTHOR	☐ $1210	$
NON-MEMBER	☐ $1760	$
INTERNATIONAL DELEGATE	☐ $1320	$
NEW PROFESSIONAL MEMBER	☐ $1210	$
STUDENT AUSIMM MEMBER	☐ $330	$
STUDENT NON-MEMBER	☐ $450	$

DAY REGISTRATIONS				Total
AUSIMM MEMBER	☐ $660			$
NON-MEMBER	☐ $990			$
Day attending	☐ Tues 9 May	☐ Wed 10 May	☐ Thurs 11 May	
			TOTAL	**$**

3 NETWORKING FUNCTIONS

Please indicate your attendance at all functions and advise if additional tickets are required. **Boxes not ticked indicate you will not be attending.**

Function	Complimentary Attendance	Guest/Additional Tickets	Total
Welcome reception	☐ Yes	$66 pp ( ) ticket/s	$
Networking hour	☐ Yes	$33 pp ( ) ticket/s	$
Conference dinner	☐ Yes	$132 pp ( ) ticket/s	$
		TOTAL	**$**

4 CONFERENCE PROCEEDINGS

The choice of either one USB proceedings or one printed proceedings is included in full- and single-day registration. Please indicate below which format you would like to receive. Additional copies of both formats can be purchased by indicating below.

Please select your preferred proceedings format:

☐ Printed proceedings ☐ USB proceedings

Proceedings	Additional proceedings	Total
Printed proceedings	$110 ( ) copies	$
USB proceedings	$88 ( ) copies	$
	TOTAL	**$**

5 WORKSHOPS

Workshop	AusIMM Member	Non-member	Total
W1: Effective Sampling	☐ $1060	☐ $1249	$
W2: Theory of Sampling	☐ $990	☐ $1210	$
W3: Grade Control	☐ $990	☐ $1210	$
W4: Sampling Practices	☐ $1450	☐ $1595	$
		TOTAL	**$**

6 CONFERENCE TECHNICAL TOUR

Tour	Cost	Tickets	Total
Sampling and Laboratory Tour	☐ $165	( ) ticket/s	$
		TOTAL	**$**

7 PAYMENT – TAX INVOICE (INC 10% GST) ABN 59 856 002 494

CREDIT CARD ONLY – Please (✔) debit my:

Visa ☐ Mastercard ☐ AMEX ☐ Diners Card ☐

Card No.

Expiry Date: CSV Number:

Signature:

Please print name of cardholder:

How to register

Telephone: +61 3 9658 6120 | Facsimile: +61 3 9662 3662 | Email: conference@ausimm.com.au | Online: www..ausimm.com.au

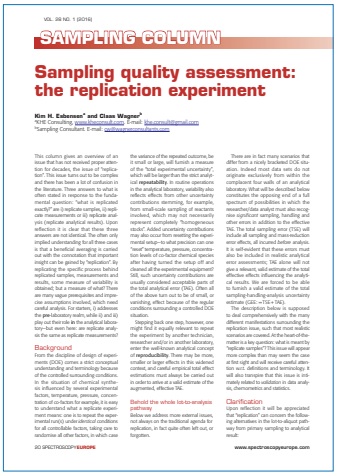
Sampling Resources

Sampling Columns in *Spectroscopy Europe*

SPECTROSCOPY europe

The essential magazine for spectroscopists in Europe

The Sampling Columns published in the free magazine, *Spectroscopy Europe*, and edited by Kim Esbensen and Claas Wagner are a valuable introduction to representative sampling and the Theory of Sampling (TOS).


SAMPLING COLUMN

Sampling quality assessment: the replication experiment

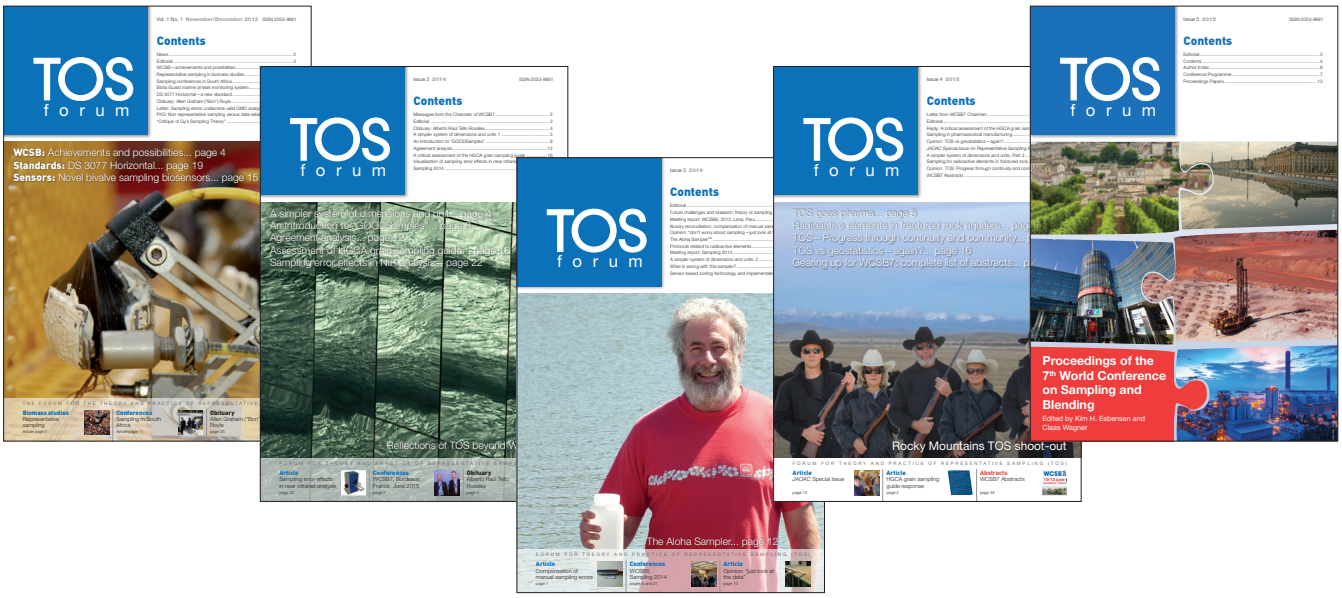
All can be read free-of-charge in print, web and digital editions, as well apps for iOS and Android devices.

Starting with an introduction to TOS, the columns have continued by looking at heterogeneity, composite sampling, a sampling quality assessment and sampling quality criteria.

Read all the Sampling Columns at:

http://www.spectroscopyeurope.com/articles/sampling

TOS forum



Read all the back issues of *TOS forum* at:

http://www.impublications.com/tosf