Theory of Sampling – what's next?

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ABSTRACT

The Theory of Sampling (TOS) has made remarkable strides and has witnessed a suite of achievements over its ~65-year history; especially in the last 15 years, since organised work began for the erstwhile fragmented sampling community (2003). Theory and practice have been very well exposed and disseminated, as witnessed in the successful series of the World Conference on Sampling and Blending (WCSB) events and other dedicated sampling and mining conferences in Australia and South Africa. At this WCSB8 occasion we fittingly devote a section of our conference to celebrate the life and monumental achievement of Pierre Gy, the originator of the Theory of Sampling; a special issue of TOS Forum was recently published to the same purpose. But TOS is most emphatically not a finished work.

Here I try to look into the future with a personal view: TOS - what's next? Commensurate with this objective there is offered some out-of-the-box thinking. The purpose is to try to zoom in on probable additional future aspects of TOS and its application compared to the first 15 years of our common journey. There are bound to be lacunae here-and-there across the vast application horizon delineated, which is as it should be, as this lecture is meant to initiate a broad(er) debate - and readers may agree with all, parts, or none of the topics presented. All is well that furthers this debate.

INTRODUCTION

Below I offer some thoughts that try to look into the future, a personal view: Theory of Sampling - what's next? Commensurate with this objective there is naturally *some* out-of-the-box thinking here. The purpose is to try to zoom in on the future horizon for TOS and its applicability. This lecture is meant to initiate a broad(er) debate - and readers may agree with all, parts, or none of the topics presented. All is well that furthers this debate.

THEORY OF SAMPLING – CONTINUALLY DEVELOPING

Although the TOS is rightly revered by all samplers as the ingenious product of Pierre Gy, it is a work-in-progress and several inspiring contributions have been made more recently, as presented at the world conferences by Pitard, François-Bongarçon, Minkkinen, Minnitt, Lyman. TOS is very fit and in a state of continuing development, and Pierre would have had it in no other way. There one will still find many intriguing 'nexts' for TOS! The role of this keynote lecture is not to review the seminal achievements made by the above-mentioned authors however, as all are on record in the relevant literature, primarily in WCSB Proceedings, *TOS Forum*.

An additional, very recent development that unfortunately could not be completed in time to make it to WCSB8 is indicated below (abstract) (see Figure 1):

A Generalised Constitutional Heterogeneity Formula (Gy's formula) – Applications to Contaminated Soils, Coated Particular Aggregates and Mixed Material Systems

Many trigger-happy estimations of the incompressible sampling variance based on Gy's classical $s^2(FSE)$ 'formula' reveal an uncomfortable lack of realism with respect to empirical estimates for real-world material systems. Recent inconsistent results from application to material systems that do not comply with the formula's assumptions have resulted in a new conceptualisation for mixed material systems *(mixtures of both analyte-enriched and coated particles, plus gangue). We here develop and illustrate a 'generalised constitution heterogeneity formula' for mixed material systems, which has the hallmark of being easier to apply in practice, and not necessitating measurements on all fragments in appropriate size and density fractions. The modified approach also does away with ill-reflected standard values of critical parameters, such as $g := 0.5$, and $s := 0.25$. As always, the evergreen 'liberation factor' issue (l) plays an important role, which is paid due attention. The generalised constitution heterogeneity formula is presented in a form that allows for (relatively) easy implementation – but the general shape factor, s, still lingers on, although we devise a possible way out by a forced normalisation. Extensive practical testing of this formula derivation is now underway* (Dubé and Esbensen, in prep).

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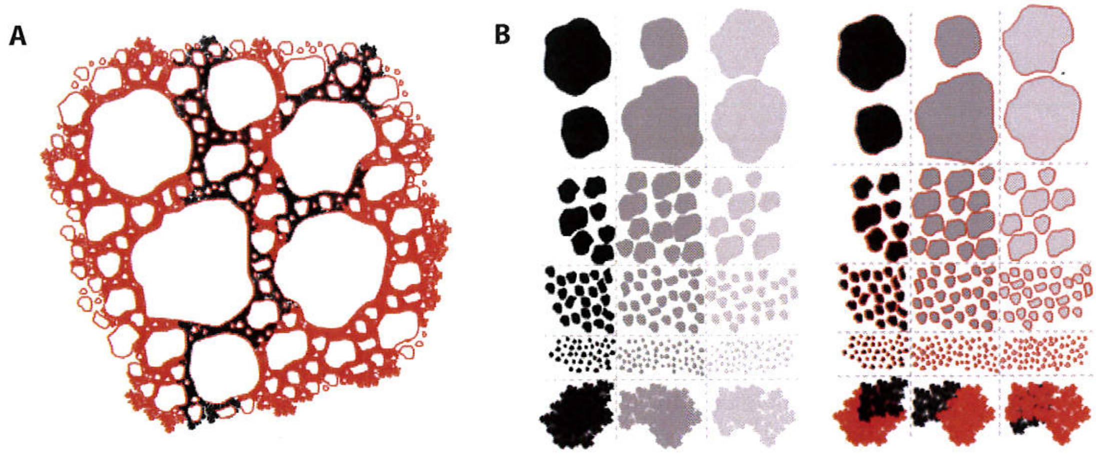

FIG 1 – (A) Soil model with typical particle size distribution. The analyte may be present as individual 100 per cent particles of any size and/or as particles embedded in larger gangue particles (usually considered for larger particle sizes only, but in reality this may be the case for all size classes). Red (colours not discernible in greyscale) represents coatings on all (or a fraction) of all grain size classes (pollutants, contaminants, mineralisations from percolating solutions). The coating illustrated can either be from an intrinsic or and extrinsic source, ie it may be original or it may be a result of a redistribution process. (B) Conceptual decomposition of the same soil matrix separated into both grain size classes (vertically separated classes) and density classes (separated horizontally – with grey tone intensity reflecting density). The right-hand illustration includes the coatings as deposited on the exact same number and size of particles. Illustration courtesy Dubé and Esbensen (2017).

APPLIED THEORY OF SAMPLING

There is currently a steadily growing presentation and publication rate of a multitude of TOS applications in an increasing number of technological and industrial sectors. It is not possible to pay *any* semblance of justice to this plethora of examples and case histories from our community - suffice to refer to the WCSB Proceedings and to the series of sampling and mining conferences under the auspices of the Australasian and South African Institutes of Mining and Metallurgy where most of these achievements are on record; there also exist key descriptions in several other, more scattered publications. This is of course the most certain 'next' of ever-growing dimensions.

TOS – MINING, MINERALS, GEOMETALLURGY

The history of TOS is inextricably linked to the mining and minerals industry as it provided Gy's initial impetus for development in 1949 (Gy, 2004). Sampling across the mine value chain covers broad aspects from exploration to production (eg resource and grade control), metallurgical (eg metal recovery and comminution), geo-environmental (eg acid and metalliferous drainage (AMD) and deleterious elements) and process plant sampling (eg mill balance).

Two negative themes can be found right across the mining and minerals sector. There is a preponderance of the thinking that simply stating that a sample is 'representative' is enough to make it so. No justification is given, merely a statement to this 'fact' (this attitude is certainly not unique for mining and minerals however, rather it is pretty universal …). In addition, some seem to think that QA/QC and TOS are independent of each other. Both of these themes are seriously misguided.

A critical development over the past ten years is that of geometallurgy, essentially a life-of-mine value chain optimisation process (Dominy and O'Connor, 2016). Geometallurgy seeks to integrate geoscientific disciplines with minerals and mining engineering. It aims to understand grade, metallurgical and mining variability based on information such as geochemistry, mineralogy, grade and lithology obtained from spatially distributed samples. Multiple spatially distributed small-scale tests are used as proxies for grade, mineralogy, process parameter and rock mass variability. These proxies need to be compared with larger metallurgical samples to establish correlations prior to modelling (eg rock quality designation and crush parameters versus Bond weight index: Kojovic, Michaux and Walters, 2010; Harbort, Lam and Sola, 2013).

The pregeometallurgical approach focused on plant design through testing of a number composite samples that are reported to be 'representative' of the orebody. Test work is carried out to determine factors such as grindability, floatability, leach recovery and/or other parameters. This test work is traditionally simply assumed to be appropriate. Subsequently, a process plant is constructed and commissioned - but at some point, often within the first year of operation, found *not* to be performing to design. The common reason for this relates to insufficient and unrepresentative samples and potentially inappropriate test work. This traditional approach generally fails to represent the orebody and its likely variability within (Guresin *et al*, 2012). Geometallurgy aims to resolve such variability, but still requires high-quality metallurgical test work, most emphatically based on representative samples collected/processed within the framework of TOS. Generalised recommendations for numbers of samples per domain are an effective guide, but require proper analysis on a case-by-case basis (Lishchuk, Lamberg and Lund, 2016). Traditional metallurgical sampling and test work is of course absolutely critical for the plant design process (Guresin *et al*, 2012). Scale-up work generally involves the use of pilot plant testing of large or bulk samples (see below). These need to be representative of both grade, spatial and population distributions within the ore zone.

Application of TOS to resource and grade control sampling is rather well-established in the mining and minerals sectors, though many practitioners still fail to realise its full potential (Dominy, 2016). Plant sampling for process control and reconciliation continues to be critical for the mining industry, with much good practice in existence (Holmes, 2010, 2015). To support corporate governance, it is now time for TOS to be integrated into international reporting codes (eg JORC, PERC, NI43-101). Development of the international sampling standard DS 3077 (2013) now provides a solid base for protocol documentation.

Many deposits (eg gold) display strong meso-scale grade and/or geological variability, and show significant *in situ* heterogeneity as well (eg high nugget effects). Underground or open pit bulk sampling provides an effective way to assess grades, producing samples in the order of hundreds or thousands of tonnes. Well-designed and planned programs provide extensive information across geology, geotechnical parameters and metallurgy (Dominy, O'Connor and Xie, 2016). It can effectively be seen as a risk reduction tool by verifying grades and metallurgical recovery as estimated from drill holes (Clark and Dominy, 2017; Dominy *et al*, 2017).

Prediction of AMD from mine waste materials is critically important during all stages of the mine value chain (Parbhakar-Fox and Lottermoser, 2015; Parbhakar-Fox and Dominy, 2017). However, to determine such geo-environmental characteristics, static and kinetic testing of waste units can only be performed on *bona fide* representative samples. The importance of sample selection cannot be underestimated also here. Poor sampling techniques and inadequate sample selection invariably lead to excessive variance, difficulties in interpretation and incorrect assessment. Current approaches to geo-environmental sampling do not fully realise the value of TOS nor apply proper QAQC (eg CRMs: Parbhakar-Fox and Dominy, 2017). More work is required to ensure representative and fit-for-purpose samples and test work.

Characterisation for sampling is an important activity, particularly with respect of the liberation diameter and particle size distribution determination of the critical phase(s). Beyond the by now well-known family of heterogeneity tests (Minnitt, 2016) and 'automated mineralogy' (QEMSCAN), recent use of high-resolution X-ray computer tomography (HRXCT) shows promise (Dominy *et al*, 2016). Continuous HRXCT scanning of drill core is the ultimate goal. Routine and continuous scanning of drill core would provide many advantages to a project across early stage commencement, automation and speed.

The current challenges for TOS in mining, minerals and geometallurgy are well identified – the future for TOS in mining, minerals and geometallurgy is therefore undeniably bright and exciting. Good new opportunities exist across ore/waste characterisation, and geometallurgical and geo-environmental sampling. It is recognised that much work still needs to be done to fully integrate these fields across the full mine value chain.

FROM UNIVARIATE TO MULTIVARIATE

A point to ponder: why is it that nearly everything regarding TOS is univariate (dealing with only one analyte/variable/parameter)? The real world in science, technology and industry is decidedly multivariate! While this latter approach is the *raison d'être* for chemometrics (see previous work by Pentti Minkkinen and Kim Esbensen), it is only very recently that Dehaine and Fillippov (2015) presented a proper multivariate variogram to our community (WCSB7), although the multivariate approach has been introduced and explored within geostatistics for a few decades. There is a challenging future in the multivariate realm also for the TOS community. This is a 'next' that has barely started.

Here is an example of a 'pure' multivariate data analysis of interest for the sampling community: 'Chemometric analysis of the efficacy of gene modified organism (GMO) test sites'. The purpose of this study would be to verify whether the global set of sites for GMO field trials are indeed capable of capturing multidimensional variability patterns. This endeavour will be undertaken using the largest number of 'end points' (variables) which are used to characterise GMO plants grown across a suite of climates and soil quality sites. Chemometrics allows characterisation of the covariance data structure for each site within the context of all sites modelled in a similar fashion. The relationship with TOS is clear in that end point averages and variances, the target for GMO field characterisation, is compromised by sampling bias errors if not eliminated properly; there is even the possibility that field sampling at different sites may not be performed by the same methods and approaches (crucially there is not yet an obligation to report on how sampling was carried out). Until now there have simply not existed sufficiently comprehensive guidelines with which to align the crucial sampling elements in GM plant risk assessment; see keynote lecture by Claudia Paoletti in this conference. These issues are outlined in detail in the recent European Food Safety Authority (EFSA) report (Esbensen and Wagner, 2016).

TOS – BREAKING OUT – GOING BEYOND

Taking the historical overview of TOS applications, for a very long time everything was *dominated* by the mining, minerals extraction and processing, coal and cement sectors. This is a historical fact reflecting the industrial sectors which immediately, or relatively quickly, accepted TOS as a key help in safeguarding economic profitability. For several decades these bulk commodity sectors continued to be the primary application sectors for TOS as the theory continued to be developed, mainly with challenges and examples from here. However there are (very) many other sectors for example food, feed, wine, pharmaceuticals, biomass and bioenergy in need of representative sampling for optimal process monitoring and control. In the very recent years significant inroads have been made re. TOS application in the food, feed, GMO areas (Esbensen, Paoletti and Theix, 2015; Esbensen, Paoletti and Minkkinen, 2012a, 2012b; Minkkinen, Esbensen and Paoletti, 2012). This is a 'next' that already began at WCSB2 (2005), and which is growing steadily at present.

TOS – PHARMACEUTICALS

Recently, a series of successful inroads have been made with respect to the pharmaceutical sector. This particular 'next' avenue has been very well covered and documented within our own circles (see proceedings of WCSB7 and WCSB8; see also the keynote paper by Rodolfo Romañach in these proceedings).

TOS – BIOLOGICAL SYSTEMS (TOSBS)

TOS has overwhelmingly been used on inorganic systems, materials, commodities etc. But one is lead to wonder, what about the organic, the biological world – not just in a commodity perspective? Are there objective sampling needs (scientific, technological) in the biological sciences for example? How do 'system biologists' acquire the analytical aliquots needed for the currently in vogue -omics techniques?

Sampling of biological systems is complicated, fraught with highly significant biological and environmental variability, read heterogeneity with a quixotic type of 'structure', of a quite different nature and magnitude than 'simple' lot heterogeneity! Biological organisms can be viewed as organised collections of cells that group into tissues, organs and subsystems that collectively form an organism. The human body for example contains about 200 different types of cells, organised into 20 different types of structures, or organelles. All told there are ~37×10^{12} human cells in a single human body. However, there are at least ten times a higher number of microbial cells, from at least 10 000 different species, in a single human body as well. Each and every of these human (and microbial) cells has a genome, which is transcribed into RNA (transcriptome), much of which is translated into proteins (proteome).

Considering that each human cell contains about three billion base pair genomes, encoding ~20 000 genes with each microbial genome containing ~5000 genes and add the desire to measure all of the small molecules (metabolites) involved in cellular function, this all adds up to enormously complex entities that systems biology are engaging today. Each cell is affected by/reacts with its environment, which itself changes over time. As such there is tremendous temporal and spatial variability at all scales as well. The characteristic in common for all biological systems is change (birth, growth, development, senescence, death), but this is just the natural complexity of a single organism.

In many modern studies there is the need to obtain samples from specific tissues across whole populations of individuals in order to do Genome Wide Association Studies (GWAS) or Phenome Wide Association Studies (PheWAS). Research avenues have been trying, and continue to focus on establishing functional links between genotype (ie the specific genetic makeup unique to every organism of any population) and phenotype (the morphological, behavioural or molecular characteristic of an individual organism). Given this complexity and the ability of all cells to rapidly respond to their environment (and even to have pre-wired temporal patterns), specific sampling problems abound at all physiological scales and biological levels – from the relatively simple collection of samples from an individual (albeit a growing and developing entity) to collection of samples across populations. Furthermore, there are critical sampling problems prevalent within the technologies used to measure omics data such as nucleotide sequencing for genomes, transcriptomes and microbiomes and chromatography coupled with mass spectrometry for proteomes and metabolomes.

And here is the crux: at present many sampling approaches used in current biology de facto amount to nothing more than grab sampling. Even application of the simplest form of replication experiments (RE) (DS 3077, 2013) will wreak havoc in these fields, revealing 'hidden' sampling variabilities of quite unsuspected magnitudes (Esbensen and Jacobson, in prep). The current scientific darlings, the -omics technologies, could greatly benefit from development of TOS, focusing on the challenges faced in systems biology. After having been in an emergent state for some five years, this work has finally started in a formal sense. Jacobsen and Esbensen's first focus is on deriving a genuinely new twist, TOSBS (Theory of Sampling for biological systems), which undoubtedly will be far more complex than standard TOS application at a certain development stage of a single organism. Much work lies ahead, all wonderfully challenging ... this is an interesting novel part of what's next for TOS.

TOS – PROCESS ANALYTICAL TECHNOLOGY

The technological backdrop for proper TOS application has overwhelmingly been oriented towards physical sample extraction, from stationary, as well as and from moving, lots (process sampling). While this is a technology basis that will continue to be with us at all times, there is now emerging a complementary technology base in the form of the powerful PAT approaches (process analytical technology) in which sensor technologies are brought to bear for the purpose of real-time, online multianalyte quantification, but based on 'remote sensing' in the so-called 'no physical sample extraction' mode. This is a development which started long ago within the process industry regimen, mainly in the mining, minerals processing and cement industries from which reports have emerged in the WCSB series and elsewhere, but quite a lot of this work is by its commercial and competitive nature proprietary and thus not well known. This work has in the main been restricted to one analyte (parameter), eg density, moisture etc at the time. There are undoubtedly many more endeavours in this context that have not been disclosed for proprietary reasons.

The present, broader analytical techniques involved in PAT are NIR, VIS, RAMAN, VIS, XRF, PFTNA in the traditional sensor probe modus, but also more dedicated spectral-acquisition arrays (cameras). The common hallmark is that most of these modalities are general-purpose multivariate sensors delivering multivariate spectral data which need to be calibrated with regard to their specific application purpose and context. This is the key area of chemometric multivariate calibration, which has been instrumental for phenomenal PAT successes in the last decade or more (Esbensen and Julius, 2009). PAT 'sensor sampling' is a very important 'next' for TOS with enormous potential. A much referred to modern textbook that undoubtedly can be a source of inspiration also for our community is Bakeev (2010). The combination of TOS with PAT and chemometrics will pave the way will pave the way for further PAT application further major industrial sectors, or clusters in the international process industry.

Across all of the above it is imperative to acknowledge that there is absolutely no difference physical sample extraction versus. PAT sensor signal acquisition when it comes to the impact of TOS sampling errors, incorrect as well as correct sampling errors (ISE, CSE), as was laid out in full detail in (Esbensen and Mortensen, 2010) and illustrated in Figure 2.

SAMPLING BIOSENSORS

A challenging parallel to 'conventional PAT sensor sampling' that has seen the light in the last five years only is the introduction of novel, highly specialised 'biosensors' often with a (more-or-less linear) accumulating effect, eg blue mussels (Dragsund *et al*, 2013) or egg yolk (Arkenbout and Esbensen, 2017). In this kind of application, standard TOS sampling errors abound (but are usually 'workable') while there is almost no limit to the levels of total analytical errors (TAE) intricacies involved, as is presented at WCSB8 for the first time in (Arkenbout and Esbensen, 2017). Backyard chickens are here introduced as novel pollution load averaging biosensors with a time-accumulating functionality, ending up with – eggs (Figure 3). Composite samples made up of the yolk from individual backyard chickens is shown to constitute highly sensitive biomarkers for dioxins and PCBs. But professional (biological, physiological and

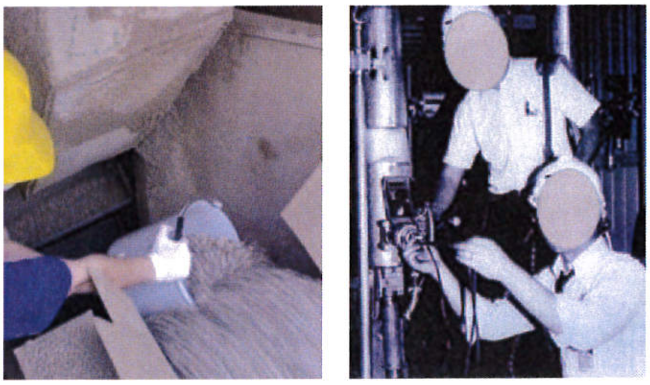

FIG 2 – There is a complete duality between physical 'sample' extraction and process analytical technology sensor signal acquisition – both examples shown here are fraught with fatal incorrect sampling errors. Photos courtesy of KHE Consulting (left) and unknown (right).

TOS) competence is needed when sampling in order to reduce or avoid significant incorrect and correct sampling errors before the specific TAE issues can be addressed meaningfully. While this 'sampling process' lies outside most traditional TOS experiences, its elements can never-the-less still be identified within the traditional sampling error framework. TOS is helping to focus the sampling process in this new application area (Arkenbout and Esbensen, 2017). The non-governmental organisation (NGO) Toxicowatch's 'novelty' emerged when it was demonstrated that dioxins in the carefully sampled eggs could be related directly to the incinerator source. After this breakthrough, this 'Colombus egg method' was presented all over the world, which inspired and empowered other researchers to further this approach. But something else also took place: this endeavour revealed remarkable gaps in European directives and in governmental enforcement. This story can be followed at Toxicowatch's homepages.

A HIGHER APPLICATION LEVEL

It is not difficult to think of at least ten good reasons why TOS is an opportunity – and not a burden! This goes for projects at all levels, from single projects to complex programs. Here is a proposal for a project that aims at taking TOS to a higher level: 'New risk analysis strategies for global trade of food and feed materials'. This project has the purpose of:

- improving current sampling strategies within food and feed
- improving safety survey and risk evaluation reliability
- harmonisation of consumers' protection standards
- early alert of new emerging risks.

The goal is to introduce and integrate reliable sampling strategies in the European Union (EU) food and feed safety assessment approach to minimise/control all sampling errors along the industrial food and feed pathways, providing a science-based, representative, or fit-for-purpose framework for risk assessment and safety decision-making processes (Kuiper and Paoletti, 2015; Paoletti and Esbensen, 2015). The project will deliver new generic EU sampling standards applicable to all types of food/feed commodities, which would lead to proper sampling for reliable toxicity assessment, pollution and pollutant/residue characterisation/thresholding and reliable allergenicity evaluation. Project tools include TOS, food/feed risk analysis, exposure (how, how much, dietary and food composition databases), fit-for-purpose representativity, safety assessment and consumer protection.

This will be a TOS-oriented project at the EU level (EU/27 member states). The general principles for safety and nutritional evaluation of foods and feed and potential health risks associated with hazardous compounds (microbial or chemical contaminants, natural toxins and anti-nutrients) have been developed by FAO, WHO, and EFSA, and further elaborated in the EU funded project 'Safe Foods'. This approach is internationally well accepted and used for safety assessment of food/feed related issues, but it does *not* address the fundamental issue: how to ensure validated representative sampling methods throughout the field-to-analysis pathway of the very many different food/feed materials, at different stages of processing, in order to assess their safety. Inadequate sampling plans will obviously impact negatively on the validity of the target risk assessments. Sampling is up against a series of inherent difficulties because of the great diversity in food and feed sources and commodities, the different kinds and degrees of food contaminations, and the many different types of intentional food/feed alterations. The sampling frameworks currently used rely on specific statistical distributional assumptions (ie homogeneous distribution of compounds/test materials), which are typically never verified in practice as the current protocols are not even obliged to characterise heterogeneity patterns stemming from the specific properties of the test material. Certain statistical distributions are simply just assumed! Moreover the current protocols do not provide estimates of the risk associated with the sampling surveys themselves (Kuiper and Paoletti, 2015), nor address uncertainties associated with spatially irregular distributions (Paoletti and Esbensen, 2015). Representative sampling is key in order to reduce the possibilities of either misestimating actual exposure levels for humans and animals or, worse, underestimating the risks for consumers to exceed tolerable intake levels. This is also important in the case of foods and feed with nutritional benefits, where under – or over-estimating intake levels may lead to potentially dangerous nutritional imbalances. Furthermore the ever increasing globalisation of trade in the area of food/feed production results in new logistics in transport pathways with a clear danger of accidental admixture of food/feed materials from different sources and origin, which may pose new emerging contaminant risks, possibly exacerbated by their interactions. This will require new comprehensive and flexible approaches

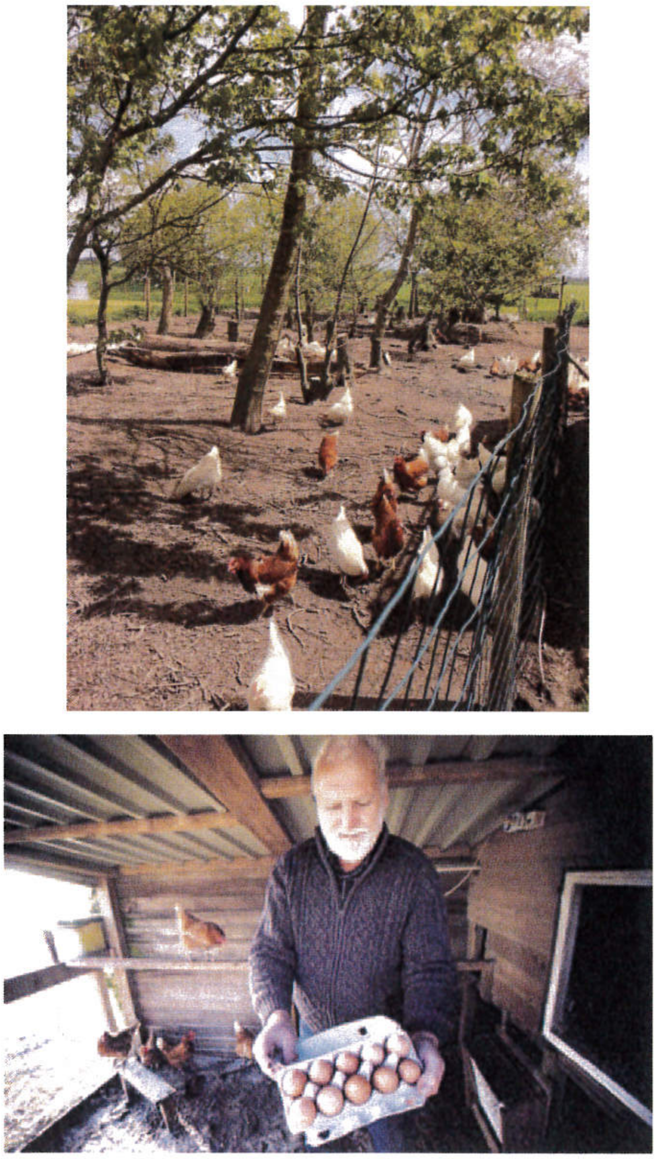

FIG 3 – Non-traditional 'sampling' based on accumulating/processing biosensors: free range chicken eggs. Photos courtesy of Toxicowatch.

for sampling of the extracted test materials and their risk assessment, approaches capable of taking spatial and/or time-dependent factors into account.

TOS – NICHE AND BRAND PRODUCTS

From an industry point of view, proper sampling for food and feed products capable of conquering a share of the market at the global level is often perceived as a threat because the only effect this may have, it is claimed, is delays for marketing efforts or, in the worst case, product removal from the market. There is an interesting implicit admission in stating the last option! Despite the professed interest for high quality, in reality the necessary steps to verify this (which of course includes proper sampling) often translates into a financial demand that many industrial business plans are not willing to take on. Following this 'logic', the food and feed industry will only rarely invest proactively in optimisation of sampling strategies. To change this current scenario one of two things (or better a combination of both) must happen: either sampling is perceived as the tool with which to guarantee quality standards or sampling it is seen as the tool to protect the uniqueness of a product for example. What type of product? Niche products, designed to capture the interest of an elite portion of consumers. In this latter case, sampling has the potential to be appreciated by industry as a tool to *enhance* business interests in other words sampling becomes a money maker and not a money spender. Producers have aggregated into consortia with the objective of facilitating their business. One of the top consortia priorities, especially in the food area, is to protect the compositional, nutritional and organoleptic properties of a given product. As but one example of TOS potential role in the niche and brand product market, representative sampling is the critical tool necessary to protect from fraud.

From the regulator/managers point of view, there *should not* possibly be any difference of opinion with respect to such a constructive TOS involvement. Still the distributional rationale prevails in (very) many places. Here is a 'next' of formidable proportions, but a breakthrough starting point *is* at hand.

TOS – DEVELOPING COUNTRIES

Leaving the problems of non-representativity in the developed world for the moment (there are still plenty of issues to contend with), the focus shall be directed to developing countries and their specific need for representative sampling. Food is the singular common factor in all societies. 'On a global scale the food and feed sector economies dwarf even mining' (*sic*). While food safety plays a vital role in protecting consumers in the developed world, the fact that the population of the developing world is larger by far, makes it clear food and feed safety will become a critical success factor in all economies – very soon. While TOS alone will not save the world, food and feed risk assessment is now increasingly entering the agenda on developing countries and without a modicum of competent representative sampling risk assessment can never be reliable. As an example, Namibia recently requested expert advice and help to establish a food and feed agency and a national laboratory. It is an obvious fact that developing countries will not have access to high-level technology in this emerging endeavour, nor will there ever be economic means in the foreseeable future anywhere near like in the developed world. There is a need for effective training programs and expert help regarding the basic start-up competence and know-how transfer, including suitable curricula with which to take on this task. Industry has clearly understood the situation and is already proposing developing countries products with a clear market interest in the local environments (eg drought-resistant crops in dry African areas) *for free*, ensuring themselves increasing dependence on their products from the local economies (shades of the 'green revolution' in the 1980s here). This does not necessarily need to end as badly as did the green revolution however – on the contrary this could turn into a win-win situation: while helping society, companies with this insight and attitude gain legitimate market advantages – the crucial issue is of course that the goals of the commercial interests are fully disclosed upfront. Developing the technical tools and instruments needed is not a particularly difficult task, but how to organise this kind development aid is often a much more complex undertaking: what is the responsible organisation to organise such projects? While these issues are being resolved the experts already in place in EFSA, FAO and other governmental or inter-governmental organisations in collaboration with the TOS community, are perfectly able and ready to put together the needed training programs. A lot of good can be done in this regimen. This is a noble and progressive next!

TOS – NON-GOVERNMENTAL ORGANISATIONS

NGOs often (although not always) contribute to society with critical advocacy on behalf of voices, issues and causes not sufficiently heard; this plays out mainly on the political level (eg climate change, ecological awareness, ecological organisations and degrowth[5]).

But NGOs also contribute on the scientific and technological scene, in which TOS can play a constructive and perhaps even disruptive role, for example by introducing representative sampling procedures and equipment where none have been deployed before; the fields of environmental science, hydrology and environmental toxicology are but a few examples. There is a formidable 'next' challenge for TOS in surveying and remedying sampling methods, procedures and the ubiquitous demand for 'sampling plans' (most often in relation to sampling from a 2-dimensional lot) without any regard for initial heterogeneity characterisation ('one sampling plans fits all purposes'). This latter is an almost universal defect across so many sectors … TOS has an important role to play in non-traditional societal sectors and application areas, facilitated by many new partners. Good examples would be Toxicowatch and the environmental toxicology association, the International Network of Environmental Forensics (INEF). A full-day workshop entitled 'Introduction to representative sampling' was presented at the Third International Environmental Forensics Conference, University at Örebro, Sweden, in June 2016.

It is a distinct pleasure that the Toxicowatch Foundation is behind a presentation at WCSB8.

> *Toxicowatch* – Knowledge is power; to measure is to know. The Toxicowatch foundation has set a goal for connecting public concern and industrial and governmental issues on topics related to dioxins (POPs), hormone disruptors (EDCs) and other environmental pollutants. In general Toxicowatch aspires to raise public awareness on the topics of toxic hazards and the resulting health risks to our environment, food and lifestyle products. The Toxicowatch foundation wants to catalyse the transition towards sustainable development, defined as the enhancement of peace, social justice and well-being, within

5. Degrowth – a political, economic, and social movement based on ecological economics, anti-consumerist and anti-capitalist ideas.

and across generations. Toxicowatch is an independent scientific society dedicated to contributing to the society while maintaining its integrity by staying independent from commercial businesses and government interests. Toxicowatch wishes to increase its scientific impact by serving the needs of Toxicology, with respect to the health of human and animal life and the environment and in determining the risks of chemical substances. Toxicowatch works to promote increased recognition of toxicology and increased implementation of advanced toxicology into chemical risk assessment (see: http://toxicowatch.wixsite.com/toxicowatch/foundation-c2lc).

INEF – The International Network of Environmental Forensics was founded in 2008 to provide a forum for scientists, environmental consultants, regulators and lawyers to share information regarding the use of environmental forensics. Environmental forensics is the use of scientific techniques to identify and apportion the source(s), age and timing of a contaminant into the environment. INEF is a non-profit, interest group within the Royal Society of Chemistry (RSC). An indicative list of INEF2016 topics includes: 1) Analytical methods: screening or confirmatory methods for organic or inorganic contaminants; approaches for emerging compounds and instrumental techniques. 2) Design and sampling for targeted or non-targeted analysis, emergency response, long-term monitoring and biomonitoring. 3) Occurrence and fingerprints of contaminants in various media, impacted or remote sites, wildlife, indoor environments, food and feed contamination; human exposure. 4) Contaminated Sites, case studies, remediation, emission prevention and control; risk assessment and policy. 5) Teaching, interpretation of results and communication (see: https://www.oru.se/english/research/research-environments/ent/mtm/inef-2016).

TOS – CIRCULATORY ECONOMY, WASTE MANAGEMENT

Another beginning to venture outside the core industrial application areas for TOS can be found in the editorial for the journal *Waste and Management Research* (Esbensen and Velos, 2017), from which a few quotations suffice to outline the scope:

Setting the scene – the role of TOS in waste management and circular economies

A circular economy involves the closing of material loops or cascading used resources, transforming waste into new (secondary) resources, or even preventing waste from occurring in the first place. Here, we analyse the interface between the concepts of circular economy, 'uncertainty' and solid waste, looking at multiple approaches. We emphasise that effective and scaled up transition to a circular economy is fundamentally unfeasible without understanding and quantifying both uncertainty and variability. This quantification, in turn, depends on the existence and availability of relevant theoretical and practical tools, such as the standard description of variability for solid flows, the so-called TOS, initially developed for practical needs of the mining industry as well as on the skill set and funds required to operationalise these tools. The vital reason behind this prerequisite is that waste streams are almost always considerably heterogeneous and will therefore always exhibit high (even extreme) levels of variability.

Educational needs for uncertainty quantification: TOS

When we try to quantify via a measurement process (eg measure the concentration of a potentially toxic element, such as cadmium, via an analytical determination in the laboratory), we cannot just report a single value, but also the error associated with this measurement, a factor of equal importance. The scientific fields of metrology and statistics address such aspects via the concepts of accuracy *and* precision. *The field of measurement uncertainty has traditionally been used to quantify the 'measurement error'. Any subsequent characterisations or calculations involving properties identified via analysis should, in principle, involve the use of pertinent error propagation approaches. There are still considerable training needs for researchers and investigators into correct and complete assessment of uncertainty. It is unsettling that the vast majority of the research studies report only MUanalysis, almost completely neglecting the dominating MUsampling (MU = Measurement Uncertainty) as laid out in detail in (Esbensen and Wagner, 2014).*

Recent publications have assessed current approaches to MU with respect to the complete ensemble of sources affecting the measurement process, in particular the extent to which sampling errors, as set out in TOS, are appropriately considered in the international standards, guides and norm-giving documents. All preanalysis sampling steps are important, collectively dominating the MUtotal estimate. In addition, when it comes to applying TOS in the realm of solid waste, many more empirical examples are needed in order to get a fundamental database on how to correctly quantify (calibrate) the various parameters involved. In this regard, we are still on a steep learning curve. The emergence of sensor-based, in-line, real-time characterisation equipment (eg near infrared-based (NIR)) does not negate the need for TOS – on the contrary, TOS is needed for the correct interpretation of the results, for example in combination with techniques such as variographics.

TOS FORUM

The current international economic situation undoubtedly puts constraints of the possibility for full interaction between members of the international sampling community, among other witnessed by reduced attendance and even retraction of submitted (and accepted) presentations/papers to our biannual conferences for purely economic reasons. A small remedial action that is very easily implemented could be to step up the status of our communication platform *TOS Forum*, so as also to develop towards a more regular, peer-reviewed scientific journal (without losing its role as the premier communication platform between the sampling community and its members). An alternative could be to survey the interest, need and willingness to contribute to a new journal with a scope that can be characterised as 'holistic process monitoring'. A first step will be to secure a group of experienced board members and reviewers from our own ranks as well as from outside peers.

TOS – TEACHING AND DISSEMINATION

It is emblematic that the Pierre Gy Sampling Gold Medal is awarded for: 'Excellence in teaching and application of the Theory of Sampling'. The most prominent role of the peers within our community is that of teaching and dissemination of TOS – the pursuit of great individual careers notwithstanding.

A notable lacuna in this context is the sparsity, or downright *lack*, of formal TOS courses at very many science and technological universities offering scholarly education in, as but examples, analytical chemistry, geology/geochemistry, mining, geo-technology, process engineering, process technology, pharmaceutical and environmental sciences; the list is legion. Happily there are notable exceptions - a towering giant in this context is Pentti Minkkinen who singlehandedly, as early as 1976, started giving introductions and courses in TOS the very day he started his professorial career at Lappeenranta University of Technology, Finland (Minkkinen, 2016). It has been the greatest privilege of the present author to have been inspired by Pentti's omnipotent achievements throughout my own academic career (Pentti Minkkinen is the only recipient of *both* the Pierre Gy Sampling Gold Medal and the Herman Wold Gold Medal, the exact equivalent for the science of chemometrics). This didactic outreach offers the greatest challenges imaginable - how often have we not all witnessed the constant lament of how difficult it is to 'break through' in this or that academic discipline or technological/industrial context? But this activity also offers the absolutely greatest satisfaction!

The good news, It is safe to say, is that there has been a highly significant increase in this work since the sampling community started regular gatherings as from WCSB1 (2003), the record of which is well documented in WCSB Proceedings and in TOS Forum. Be this as it may, there is still a virtually unlimited challenge in increasing and improving this crucially important work - undoubtedly the noblest tribute to Pierre Gy's entire life and work. Please join in this pursuit, all contributions large or small adds valuably to our joint project.

INTERNATIONAL PIERRE GY SAMPLING ASSOCIATION

There has been discussion for several years whether our presently completely informal community attending the series of WCSB conferences would benefit from the establishment of a formal organisational body with the aim to represent the interests of the international sampling community. An *ad hoc, pro tem* committee took it upon itself to test this concept. Thus a proposal for the 'Constitution of the International Pierre Gy Sampling Association (IPGSA)' was published in *TOS Forum* Issue 6[6]. An attempt has been made to keep the association organisation as simple as possible. The outcome of the inaugural assembly at WCSB8 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. If the association is established this is certainly taking the sampling community business to the next level.

A BROADER SOCIETAL PERSPECTIVE

This keynote speaker wishes to end by proposing a multithreaded broader perspective. I almost don't know how to say this to this group of distinguished peers and colleagues, but ...: 'It is *not* only about sampling'. While we are trying to help this world a tiny little bit by solving some of its sampling issues, we also have a higher responsibility. It is *not* only about reducing costs (it certainly is also about that, but *not* exclusively), it is *not* only about reducing the inevitable economic consequences of bad decision-making from non-representative sampling (and the resulting unreliable analytical results), *nor* is it only about getting higher, or the highest, profitability - an area where proper sampling indeed can make a highly significant contribution.

It is just as much about putting TOS in the broader societal and economic perspective. TOS is also a key player in securing representative samples within the environment monitoring and control arena, and for helping to improve proper process control, which is not only about optimising this and that product or economic parameter - it is equally as much about helping to reduce material, heat and energy *waste* (all of which should alternatively better be viewed as new, under-used resources with but the smallest twist to traditional thinking). TOS in fact has many other societal roles as well. Here is a close parallel, just published as an editorial within the geoscience literature by a far-sighted author, Marcia K McNutt, with the title: 'Convergence in the geosciences' (McNutt, 2017). The abstract of the editorial follows:

> *The 21st century presents unprecedented challenges if society is to continue to provide abundant energy, water, and food and high-quality housing and medical care to a growing global population. Deforestation and aquifer depletion are at unsustainable rates, and use of fossil fuels is leading to unprecedented climate change. Geoscientists can confront these challenges by expanding partnerships with other disciplines. 'Convergence', the integration of engineering, physical sciences, computation, and life sciences to benefit health, energy and the environment, has been successfully developed for biomedical research. It is time for the geosciences to embrace* convergence, *as our future depends upon it.*

TOS – FUTURE FOCUS

The present keynote paper ends with a reflected vision, mission, scope and 'how to get there' (the reader may respectfully agree, or disagree, with all, or parts, in the below):

TOS' vision is to be a key international resource providing state-of-the-art sampling, PAT and specific consulting and competence for reliable decision-making in science, technology, industry, individual businesses, regulating authorities and society.

TOS' mission is to provide competence for optimal decision-making, efficiency and profitability in the form of:

- didactic outreach
- competence-building
- targeted consulting
- providing the critically necessary science-based input to new types of, or contribute to the continued development of, representative sampling equipment.

TOS' scope - analytical results forming the basis for important decision-making in science, technology, industry and society must be relevant, valid and reliable. But analytical results cannot be detached from the specific conditions under which they originate. Sampling comes to the fore as a critical success factor. Analysis should only be made on documented *representative* samples. Representative sampling, including process analytical technologies (PAT) constitutes a complement of necessary competences for reliable decision-making. The TOS is of vital importance for process industry (eg mining, exploration, minerals processing, bioenergy production, pharmaceuticals, food, feed, beverage, plastics, dairy, geo-science, environmental monitoring and emerging recycling industries) as well as in technical and commercial laboratories. Disregarding TOS competence results in a significantly larger decision uncertainty than necessary, compromising reliable decision-making in international corporations, companies, regulating bodies, authorities and governmental institutions (eg GMO testing). TOS and PAT can contribute towards more fully developed corporate social responsibility regarding unwanted environmental, process and resource utilisation side-effects.

How to get there - the TOS is the only professional, science-backed framework for representative sampling - for all materials, at all scales, under all conditions. This is a truth

6. See: <https://www.impublications.com/content/tos-forum-table-contents?issue=17_7>

so persuasive that the term 'sample' is used across all these sectors, to indicate the basis upon which decisions are taken; but only representative samples can lead to reliable and defensible decisions. Disregarding TOS is a violation of due diligence be this in science, technology, industry and commercial enterprises.

While we are getting there – in this broadest possible context, nobody has put just the right *human* perspective on all aspects of the modern world's chimera, or yoke (depending on one's personal understanding and viewpoint) *growth*, better than Uruguay's president José Mujica (see: http://www.youtube.com/watch?v=cCEgcd7G9Bg).

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This paper expresses the ideas of the keynote author only – there is nothing in this publication that in any way reflects the opinions or stands of WCSB8, nor the scientific organisations of which he is a member or is affiliated with. I am grateful for continuing discussions on the role of science in society in general, of TOS in particular, from three of my closest companions-in-arms in this troubled world, Claudia Paoletti – a kindred scientific spirit and a very clear-headed friend since WCSB2 who manages to temper my writings like no one else, and Jørgen Riis Pedersen who, although we have only known each other for about a year, is a relatively late-in-life tremendous inspiration and a source and catalyst of much thought out-of-*my*-box. I thank Simon Dominy profusely for joint endeavours into many old and especially new aspects of TOS application, with continually augmented insights; I am particularly indebted to him for his expertise and his willingness to share the complexities of geometallurgy. While kindly having reviewed this feature internally before submission, indicating a fair bit in serious need of clarification and improvement in an earlier version of the manuscript, they are of course not responsible for the specific framework of thoughts, stands and opinions expressed here.

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