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The application of an integrated software library for controlling and monitoring ISO sampling systems

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There exists an industry need for an integrated system to monitor and control ISO compliant sample stations. This paper discusses the development and application of a control system toolbox to deliver complete ISO compliant functionality.

Traditionally automated sampling systems have often relied on generic equipment control standards to operate individual sampling components. The design of the sampling equipment in these systems may comply with ISO requirements but does the complete integrated system meet these same conditions?

The development of a software library that integrates ISO sampling standard requirements with customised equipment control units via a supervisory control module bridges this gap.

The application of standardised libraries, based on over 30 years of combined sampling, electrical and control engineering experience, has led to the seamless integration of standalone sampling devices into ISO compliant sampling systems. All components have well-defined interfaces, common functional control and reporting mechanisms. The result is a fully integrated sample station that performs as a cohesive quality system which harmonises with appropriate ISO standards.

The benefit of a standardised and integrated sample station is the consistent production of reliable and accurate results. Trustworthy sample data gives QA analysts, technicians and plant management a high degree of confidence that they have a full understanding of their material's properties and commercial worth.

Confidence in sampling results is essential as the quality of the material is inexorably linked to a company's reputation as a reliable supplier of quality products and, ultimately, to their bottom line.

Blast hole sampling in two areas of the same deposit

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The Rio Blanco ore deposit is a porphyry copper, located in the central zone of Chile, it is a planetary anomaly of copper and molybdenum. It contains resources identified in 20,000 million tonnes at 0.59% Cu and 0.016% Mo.

The deposit is divided in two zones exploited through open pit mining by two different companies, Codelco (Andina) and Anglo American (Los Bronces). In Andina and Los Bronces the blast hole grade is essential for mine planning. Each company has its own method for blast hole sampling.

This paper numerically compared and analyzes the blast hole sampling results in the two areas of the deposit, using as reference the grade of diamond drill holes composites for copper, molybdenum and arsenic.

Iron ore ROM sampling

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This study aims to evaluate bias and precision errors of the sampling process of ROM (Run-Of-Mine) from three pits of iron ore located in Minas Gerais, Brazil, Alegria Mine Complex. Firstly, during formation of ROM piles, it was done a manual sampling by little scoop (flatted with 5x5cm) inside a truck with about 140 tons. It was collected an increment for each twenty trucks during a period of 6 hours. During same period, about 120 trucks dump ore to compose piles that feed two ore dressing installations. Then, three increments with about 2kg each one were collect as a sample by installation. The partial results from piles formation are used to measure adherence of piles 'programming based on the geological model in order to achieve the specifications of process and product.Actually the results have shown a difference about 0.5% for monthly mean iron ore content (absolute error regarding a 90% confidence interval).To estimate the bias due to the finer material losing were collected two samples with about 5 kg each increment in the piles dumped by the trucks for

each twenty trips along six hours. During a month it was evaluated the error associated to the sample pairs (A= originals x B= duplicates) collected in different depths of the piles by a backhoe. One pair of duplicates was collected during six hours and forty during a month to supply information about accuracy of the method.

Evaluating Gy's formula accuracy for bauxite

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Quality control in the mining industry context is directed to the extraction of natural resources with reliability, effectiveness and minimum cost. The understanding and application of the Theory of Sampling (TOS), developed by Pierre Gy, integrated with process management, is essential for reaching this objective. Samples will never present exactly the same characteristics of the lot from which they are selected because of the constitutional heterogeneity, which generates the fundamental sampling error. Each stage of the sampling process produces an error which must be known for determining the reliability of the estimates. In this context, the heterogeneity tests are an essential tool for knowing the natural variability of the deposit, and to conduct sampling in a correct way in order to generate representative samples. The present work studies the heterogeneity of a Brazilian aluminum ore and compares the results obtained from Gy's formula calibrated by the heterogeneity test with the ones obtained from the formula using the factors for bauxite, i.e. shape, mineralogical, liberation and granulometric factors. Results validate the factors proposed by Pierre Gy for this particular Brazilian bauxite.

Relative efficiency of four common splitting procedures for bauxite

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According to Pierre Gy, all components of the overall sampling error result from the existence of one form or another of heterogeneity. The reliability of analytical results is often affected by uncontrolled sampling errors that result from the constitutional and the distributional heterogeneity, associated to the fundamental sampling error and the grouping segregation error, respectively. As a consequence, the probability of obtaining a sample which perfectly represents the parent distribution is remote. In addition, it is very rare for reduction from bulk to analytical sample to be carried out in just one operation. Laboratory assays have to be conducted using a small fraction from the whole lot. For this reason, sampling is a subject that should interest analysts, professors and students of analytical chemistry. In order to achieve the best possible mass reduction, the operator needs an appropriate technique to minimize the preparation error in the laboratory under the Theory of Sampling (TOS) sense. There are four sampling procedures commonly used for a representative mass reduction: cone and quartering; table sampling; rotating splitting and riffle splitting. This paper compares the relative efficiency of the four sampling methods using aluminum ore and presents the results in statistical terms.

The added-value of sampling

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Determination of the complete sampling distribution (Lyman, 2013 & 2014), as opposed to estimation of the sampling variance only as per current sampling practice, represents a leap in sampling theory. This is the link that has been missing for sampling results to be used to their full potential for quality assurance purposes. Indeed, access to the complete sampling distribution provides opportunities to bring all the concepts and risk assessment tools from statistical process control into the production and trading of mineral commodities.

The paper will present the way by which sampling theory, via the complete sampling distribution, interfaces with statistical process control theory and practice. Illustrative examples, through calculation of operating characteristic curves in particular, will establish the causal relationship between sampling precision and quality assurance trading risks. The case will be made that the added-value of sampling lies not only for production of minerals, but also, and perhaps more significantly for commercial risk assessment and trading. The paper foresees that sampling is expected to become a real source of competitiveness, hence revenue, for mining companies.

Placer gold sampling—the overall measurement error when using gravity concentration on particle size ranges during sample treatment

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Placer deposit are generally characterized by low grade of free gold. This is the case in French Guyana where the main placer deposit are in the river bed. Most of them have been already exploited by very small mining companies with sluices. If this

technology is efficient for coarse gold, it releases fine gold in the tailings. During these last years, studies have been performed on various sites and the recoveries have been estimated between 40 and 60% depending on the size distribution of gold particles and of the quality of the sluice configuration.

A lot of recent or ancient tailings are available with a non-negligible quantity of remaining gold, offering retreatment opportunities. They are generally disposed as a sand heap with the frame of a dejection cone at the discharge of the sluice. Due to the resulting large heterogeneity of distribution, it is necessary to take many samples at various locations. These samples have to be large enough to be representative of the neighbouring material. As gold is mainly liberated, traditional sample treatment with successive size reductions and sub-samplings is not efficient and can be very expensive. Another approach using sieving and gravity concentration per particle range have been preferred.

The paper, after the presentation of the sampling and measurement protocol, focuses on the calculation of the overall measurement error including sampling stages, mass measurement, and gold analysis errors. Various cases of tailings are presented for which the decision of retreatment depends on the obtained level of confidence in the estimate of the quantity of recoverable gold.

The overall measurement error – TOS and uncertainty budget in metal accounting

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Metal accounting is one of the main tools for financial and technical management of metal production industry. It is based on measurements and has to manage the uncertainty inherent to the measurement process. The uncertainty in the metal accounting generates financial risk. The accuracy of the metal accounting results is directly linked to the accuracy of the material balance and then to the accuracy of the mass and content measurements. Estimate the overall measurement error, through its probability distribution or its first and second moments (mean and variance), can contribute to the enterprise decision making.

The overall measurement error can be calculated and analysed by establishing the uncertainty budget. If this approach has been mainly introduced to calculate the analytical error (cf. ISO GUM), it has to take into account the sampling procedure. Even though it is not explicitly named “uncertainty budget”, the same approach is proposed in the Pierre Gy’s Theory Of Sampling, where the various components of the overall error are well identified and described with their properties and their relative weights.

The present paper propose a methodology to build such uncertainty budgets in the frame of the implementation of a metal accounting system. It can be applied to an existing measurement system, analysing the results in order to find some ways for improving the measurement accuracy. In addition, it can be used to define a new measurement procedure with an objective of accuracy. Various real examples illustrate both applications.

Comparing different heterogeneity tests for gold

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Estimating the heterogeneity of gold ores is one of the biggest challenges for the mining engineers and geologists who work with tonnage and grade estimation. The calculation of the minimum sample mass to represent a given ore at a given comminution size is based on the estimation of the constant factor of constitutional heterogeneity, IH_L, which can be derived by different heterogeneity tests. Two tests are well known in the mining industry: the Heterogeneity Test (HT) and the Duplicate Sampling Analysis (DSA). In 2011, Minnitt, Pitard and François-Bongarçon proposed a third test named Segregation Free Analysis (SFA), where the Grouping and Segregation Error (GSE) is to be eliminated. The tests often show different results, especially when it comes to gold ores. These differences are due to many reasons, but one of the main reasons is the analysis technique chosen for gold content estimation. Other reasons include the fragmentation mechanism when generating the fractions for the heterogeneity tests and, primarily, the difficulty of collecting a representative sample for the test, especially when dealing with high cluster and nugget effect deposits. This paper analyses and compares two different heterogeneity tests – HT and SFA – and two different fragmentation mechanisms for the same gold ore. The sample was comminuted using a jaw crusher whose main fragmentation mechanism is compression, as well as using the drop weight testing device whose main fragmentation mechanism is impact. First results show the complexity of estimating the heterogeneity of gold ores and highlight the importance of using different approaches to estimate the minimum representative sample mass for gold ores.

Validation of reverse circulation drilling rig for reconciliation purposes

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Taking representative samples of ores containing precious metals is a very difficult task. The more the grade decreases and the nugget and/or cluster effect increases, the higher the difficulty of selecting samples which are both accurate and precise. Reconciliation practices can be used as an effective tool to evaluate sampling accuracy throughout grade control processes. However, a proper reconciliation system must be based on reliable data and, therefore, the optimisation of sampling techniques is a must for the development of a reliable reconciliation system. This paper is a result of an extensive reconciliation study carried

out at a copper and gold mine in Brazil, where a significant reconciliation problem took place while using manual sampling for grade control and short-term modelling. After analysing several sampling equipment and sample selection techniques, the authors suggested the use of a reverse circulation (RC) drilling rig with automatic sampling system for grade control sampling. The samples generated by this automatic system were compared with the samples generated by the previous percussion rotary air blast drilling rig, with manual sampling after completion of the drill hole. Moreover, three pairs of twin holes were drilled in order to validate the reverse circulation drilling rig. Results allowed measuring the bias related to the Increment Weighting Error (IWE) generated by manual sampling and showed that the RC rig eliminates significant sampling biases, improving sample representativeness by increasing both sample precision and accuracy.

Improvement in short term mining planning with soft data

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Short-term mining planning typically relies on samples obtained from blastholes in mining operation. These samples may carry a large sampling error. The aim of this paper is to evaluate the impact of the sampling error in mining recovery. This paper presents a case study about a gold mine, where there were two different data types, Au grades collected by diamond drilling (hard data) and Au grades collected by reverse circulation (soft data). Two methodologies were investigated in order to estimate the Au grade of each block to be mined: Ordinary kriging considering data from diamond drilling and ordinary cokriging considering the two different data types. The results showed that samples with poor sampling protocols, even being biased, improved the estimates, compared to retaining only precise and unbiased samples (but in smaller number)s for estimation purposes.

A multi-parameters approach for process variograms

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In the theory of sampling, the variogram have proven to be a powerful tool to characterise the heterogeneity of 1-dimension-allots. Yet its definition and application in sampling for mineral processing have always been limited to one variable,typically ore grade. However this definition is not adapted to some cases,often encountered in mineral processing, where samples need to be representative for a large range of parameters,i .e. variables, such as multiple element grades, grain size, etc.For such cases the multivariable variogram, originally developed by Bourgault and Marcotte (1991) for spatial data analysis,can be used to summarise time variation of the multiple variables (e.g.ore characteristics which are important for theprocess) and highlights the multivariate time auto-correlation of these variables. Different metrics can be used to compute the multivariable variogram, commonly the identity matrix (Euclidean distance) or the inverse of the variance-covariance matrix (Mahalanobis distance) depending on the requirements of the sampler. A case study of a low grade ore show the potential of the multivariable variogram compared to the classical approach which do not take all the variables of interest into account.

Sampling considerations for characterization of radioactive contamination using geostatistics

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At the end of process equipment dismantling, the complete decontamination of nuclear facilities requires the radiological assessment of residual activity levels of building structures or remaining materials. Similar considerations also apply to remediation of contaminated land and groundwater. As stated by the International Atomic Energy Agency: “Segregation and characterization of contaminated materials are the key elements of waste minimization”.

In this framework, the relevance of the geostatistical methodology relies on the presence of a spatial continuity for radiological contamination, characterized through the variographic analysis. Geostatistics then provides reliable methods for activity estimation, uncertainty quantification and risk analysis, which are essential decision-making tools for decommissioning and dismantling projects of nuclear installations.

The objective of radiological characterization is to find a suitable balance between gathering data (constrained by cost, deadlines, accessibility or radiation) and managing the issues (waste volumes, levels of activity or exposure). Results should demonstrate sufficient confidence without multiplying useless data. Then the spatial structure of radioactive contamination advantageously enables the optimization of sampling effort (type, number and position of data points). Geostatistical methodology can help determine the initial mesh size and reduce estimation uncertainties, in particular with the false negative, in which an area is declared to be below the threshold using estimate results, but in reality exceeds the threshold.

In addition, the impacts of the information support (size of the measured or sampled area) and the estimation support (punctual, block or global estimates) are addressed, respectively on spatial structure interpretation and on waste classification.

Applications of sampling theory in bulk commodities: an iron ore case study

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Iron Ore supply is outpacing global demand, reinforcing the importance of product quality and reliability as critical factors that distinguish Iron Ore producers in a competitive market. This expectation calls for a dramatic shift in industry attitudes toward sampling in bulk commodities, beginning with a greater emphasis on optimisation of sampling protocols from Exploration to Port. Quantifying the Fundamental Sampling Error (FSE) of the sampling protocol is a minimum requirement to achieve this and should be preceded by experimental calibration of the sampling constant K and the exponent alpha (α).

Here, we present a case study in which calibration methodologies proposed by Francois-Bongarcon and Minnitt were comparatively used to determine K and α for a Channel Iron Deposit (CID) and a Brockman Iron Formation-hosted Bedded Iron Deposit (BID) from the Pilbara region of Western Australia. Following experimental calibration of K and α, liberation size was calculated for iron oxides and deleterious minerals using Gy's formula. The results were then critically compared against QEMSCAN analysis, thus the Qemscan approach to heterogeneity is put into context.

Although agreement was achieved between calibration methods, the QEMSCAN results raise questions about liberation size calculated using the experimentally derived K and α. Furthermore, the QEMSCAN results highlight uncertainty around mineralogical assumptions made in calculations of FSE. These observations emphasise the importance of validation when assessing FSE. The case study presents an industry perspective on the learnings, opportunities and caveats associated with applications of sampling theory in response to an increasingly competitive Iron Ore market.

Sampling protocol development in a coarse gold deposit

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The occurrence of coarse gold particles leading to grade complexity and sampling challenges is a common feature of many gold deposits. Poorly designed sampling protocols applied to these deposit types can lead to an excessively high Fundamental Sampling Error. Together with other errors, the Fundamental Sampling Error contributes to the nugget effect. Proper sample collection, preparation and assay protocols are required to minimise this error, and hence reduce the total nugget effect.

On a practical level, half core cutting of coarse gold mineralisation frequently leads to grade uncertainty given that the remaining half may or may not contain a material gold particle. This is frequently evidenced by the very poor precision between duplicate core half assays. In addition, further uncertainty is introduced where the assay of a half core sample is based on a single fire assay result.

A case study from the Ballarat East gold mine in Victoria, Australia is presented, which is characterised by notable quantities of coarse gold (>50% +100-micron gold) hosted in multiple-quartz veins. Diamond drilling provides a reasonable measure of gross geological continuity at drill a spacing of 15-25 m along strike and 5-10 m down-dip. On vein development with face sampling, together with detailed geological studies are undertaken during the resource delineation phase.

A number of different sampling and assay options have been trialled at Ballarat over its recent project history. The current drill core sampling protocol utilises whole core; using 2 kg LeachWELL assays after logging and photography.

This contribution describes the nature of the Ballarat East deposit, its gold particle size characteristics, how sampling and assay protocols have developed with time, application of Gy Sampling Theory and its calibration, and how the data is subsequently applied to grade estimation.

Analysis of granite's roughness using stratified random sampling for the evaluation of radon gas emanation

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There are three natural radioactive families according to their decay, which are: the uranium series (^{238}U decreasing to stable ^{206}Pb), the actinium series (^{235}U decreasing to stable ^{207}Pb) and thorium series (^{236}U $\rightarrow$ ^{232}Th decreasing to stable ^{208}Pb). The three series have radon gas as an intermediary element, each with a different atomic mass (^{222}Rn, ^{219}Rn and ^{220}Rn). The three isotopes are inert gases at ambient conditions and they are alpha particles emitters. Thus, soils naturally emanate these radioactive gases in variable concentrations depending on the location. The radon radioactive emanation is a mass flow composed of radionuclides emitted to the atmosphere from the surface of the material or transported to it. In the case of the elements on the surface, the emanation depends on the amount of radon atoms formed from the decay of radium and surface roughness. This study aims to analyse the roughness of granite plates using simple stratified random sampling of an area in order to minimize the analysis time. To validate the sampling method, stratified sampling results were compared to the measure made in the whole area, presenting a good adherence of both data. It is concluded that the measurements can be conducted just in a few points using stratified random sampling, significantly reducing the time for obtaining granite's roughness.

Handheld XRF analysis (hXRF) — field sensor sampling representativeness and development of a prototype FRAT (field rotary abrasion tool)

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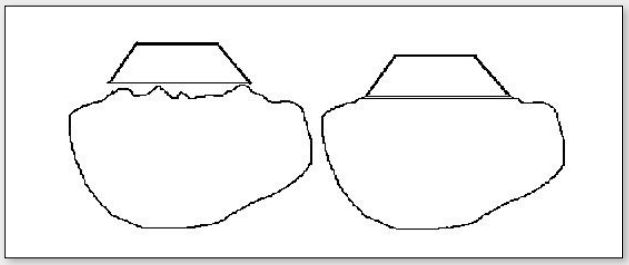
Handheld X-Ray Fluorescence instrumentation ('field hXRF') is used with increasing frequency within geology, mining, prospecting and metals processing providing real-time *in-situ* measurements that can aid scientific interpretations in the field, industrial processing and decision-making. hXRF is by its nature less powerful than laboratory XRF and a.o. precision is claimed to suffer as a result. We investigate to which degree this is the case, but much more importantly: what about accuracy (bias)? hXRF is subject to several types of sensor sampling errors as only a small field-of-view is measured (8 mm) and the specific outcrop morphology is critical, creating very uneven XRF path lengths. Multivariate (chemometric) prediction models have been created on the basis of a representative master training set, comparing the fidelity of measurements acquired from raw outcrop surfaces to those from cut (sawed) and *abraded* surfaces[1,2]. A new handheld, battery-operated "Field Rotary Abrasion Tool (FRAT) has been developed[2], intended to improve the field sensor signal acquisition. Abraded surfaces yield significantly more accurate and precise results (53% -73% improvements) compared to today's raw field surfaces[1]. When implemented these feasibility studies will allow *improved* field hXRF measurement quality for geological outcrops and other material surfaces with *similar* compositional characteristics, provided relevant and appropriate calibration data sets.



Schematic drawing of variable XRF path lengths on raw outcrop surfaces (left) compared to uniform path distances for abraded surfaces (right)[1]

Improved surface hXRF fidelity resulting from application of the newly developed Field Rotary Abrasion Tool (FRAT)[2]

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Proper sampling, total measurement uncertainty, variographic analysis and fit-for-purpose acceptance levels for pharmaceutical mixing monitoring

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Process monitoring in technology and industry in general, in pharmaceutical batch and continuous manufacturing in particular, is *incomplete* without full understanding of all sources of variation. Pharmaceutical mixture heterogeneity interacts with the particular sampling process involved (by physical extraction or by PAT signal acquisition) potentially creating four Incorrect Sampling Errors (ISE), two Correct Sampling errors (CSE) plus the analytical error (TAE). In the highly regulated pharmaceutical production context it is essential to eliminate, or reduce maximally, all unnecessary sampling contributions (TSE) to the total measurement uncertainty (MU) in order to be able to meet the regulatory stringent blend - and dose uniformity requirements. Current problems a.o. stem from inadequate understanding of the challenges in sampling of powder blends. In this endeavor the Theory of Sampling (TOS) forms the only reliable scientific framework from which to seek resolution. We here present a variographic approach with an aim to conduct problem-dependent TSE error variance decoupling and to show how to develop *fit-for-purpose* acceptance levels in powder blending process monitoring. The key issue is shown to be the nugget effect, which contains all non-optimised [ISE, CSE] plus TAE contributions to the total Measurement Uncertainty (MU_{total}). A large nugget effect w.r.t. the sill is a warning that the measurement system is not tuned in, and must be improved. Regulatory guidances have called for sampling from within blenders, leading to sampling errors associated with the insertion of sample thieves (a.o. spear-sampling from blenders). Instead of blender sampling we call for on-line variographic characterisation of the

blender outflow stream. Practical case histories are described in a parallel contribution to WCSB7 ("TOS to the rescue").

Calculating the sampling constant of particulate ore samples — examples from base metal ores

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The challenges presented in the sampling of particulate materials in mineral processing operations was one of the drivers for Pierre Gy to develop his sampling theory and this challenge continues today. Gy's simplified formula is frequently used in designing appropriate sampling protocols for the representative sampling of fine particulate ore materials from process streams. One of the key parameters required in Gy's simplified sampling equation is the Liberation Factor and the variable nature of ores means that the value of this factor can change significantly as the ore texture changes. Ideally the liberation factor should be calculated for each ore and Lyman and Schouwstra[1] have developed a method to do this which uses the mineralogical data for individual particles in polished sections, data which are readily available from automated mineralogy measurement systems.

This paper applies the method developed by Lyman and Schouwstra[1] to a number of base metal ores with a range of textural characteristics and examines how the sampling constant varies across these different ore types over the range of particle sizes typically found in mineral processing operations. The work also calculates the liberation factor for each particulate sample and examines how this changes as particle size changes.

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Pre-crusher stockpile modelling to minimise grade variability

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The use of pre-crusher stockpiles for storage and buffering to control quantity variations is usually well recognised and managed. However, a third function of these stockpiles, to reduce variations in the grade of ore entering the crusher, is often given insufficient attention. Well designed stockpiles and a disciplined approach to building and reclaiming are essential to systematically reduce short- term grade variation.

Pre-crusher stockpiles are commonly built and reclaimed with little or no regard to where the ore is dumped to or reclaimed from. Consequently, the full potential to reduce short-term grade variability is diminished as well as losing the ability to reconcile mine/blast block grades with crusher grades.

It is important to acknowledge that the pre-crusher stockpiling procedures referred to in this study are not those used for long-term storage of ore but those which facilitate the short-term buffering and flow-through of ore.

This paper reports the conclusions from studies simulating the reduction of grade variability by a range of alternative models, and quantifies the benefits achievable in reducing the grade variance by building stockpiles of appropriate dimension using systematic methodologies. The models considered cover a range of stockpile configurations, including stockpiles built with single and double layers, and reclaiming in directions perpendicular or parallel to the build direction.

Building confidence intervals around the true value of a sample

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Common practice in sampling for the TOS erudite consists of using the sampling variance obtained from Gy's numerical theory to build confidence intervals around the true sample value. This is usually done to characterize the 'precision' of the sample, and, by centering that interval on the sampled value, one states for instance that *"the true value has 95% chances of being between values x and y"*, those two values usually being centered on the sampled value". The somewhat naïve rationale behind this practice is reviewed in some details and criticized. It is suggested the confidence interval of real interest to the user of the sampled value, is more difficult to define and more delicate and indirect to build. Some methods for doing so are examined and a methodology is recommended.

Gy's theory of sampling: applications and limitations

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The achievements of modern Theory of Sampling (TOS) to date are reviewed and the focus is then put on their limitations, examining the question: "Are we always using TOS within its domain of validity?" Specific examples from common practice are examined with the aim of producing a best practice guide for using TOS, with emphasis on the meaning and epistemology of using mathematical models.

Proper field sampling and laboratory processing for archæometric discrimination between cultivated and fallow Bronze-age fields on Bornholm, Denmark — TOS meets chemometrics meets archeology

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Archeologically it is interesting to be able to predict whether a particular Bronze-age field has been cultivated or not based on soil chemistry characteristics augmenting traditional archeological evidences. A field sampling campaign was carried out in the summer 20014 on the island of Bornholm with the objective to discriminate between documented cultivated and fallow Bronze-age agricultural fields based on multivariate data analysis (chemometrics) of metal concentrations (ICP-MS) in soil. The experimental design (Fig. 1) was directed towards investigating the degree to which *proper* field sampling (TOS) plays a significant role a.o. including replicate sampling at three levels. Fig. 1 shows the geographical layout of one cultivated field (JP01-JP10) and a nearby fallow field (JP11-JP19), termed "D1" and "Ud" respectively (all samples originate from the same depth corresponding to the paleo-cultivated layer or the equivalent depth in fallow fields). Applying Principal Component Analysis (PCA), the t_1-t_3 scores plot (right) corresponds to 54% of the most discriminative variance in the 11 variable/19 sample X-matrix containing the ICP-MS metal concentrations. Another cultivated field (D2), located 1 km away, also appears in Fig.2. It is highly satisfying that the first and third PC-component is able to delineate a complete discrimination [Ud] vs. [D1,D2]; PC component no. 2 models other, general soil chemistry features that are *compensated* for in the PCA solution.

We present the TOS-specifics pertaining to the critical field sampling procedure, including the hierarchical three-level experimental design used for quantifying all contributions to the total Measurement Uncertainty (MU) budget in the field-to-analysis pathway (grab vs. composite field sampling, local replicate site-sampling, replicate sub-sampling in the laboratory, replicate test portion extraction as well as σ^2(TAE), in order to better understand the discrimination achieved. Five elements appear to be particularly involved in the discrimination [P, Fe, Mn, Zn, Pb], currently undergoing paleo-agricultural/geochemical interpretation. Based on these first results we plan a full *test-set validation* campaign in 2015 – which will be the ultimate performance test for this type of archæo-chemometric discrimination. This contribution illustrates the versatility and power of multivariate data analysis (Chemometrics) applied to data with a substantial proportion of potential sampling errors.

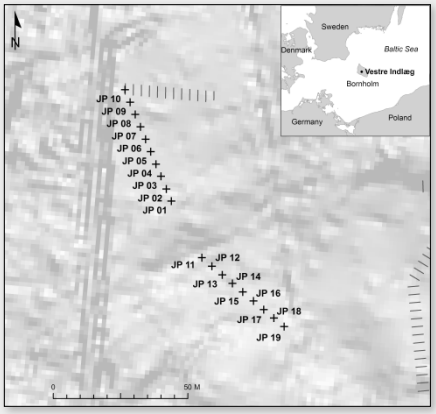

Figure 1.

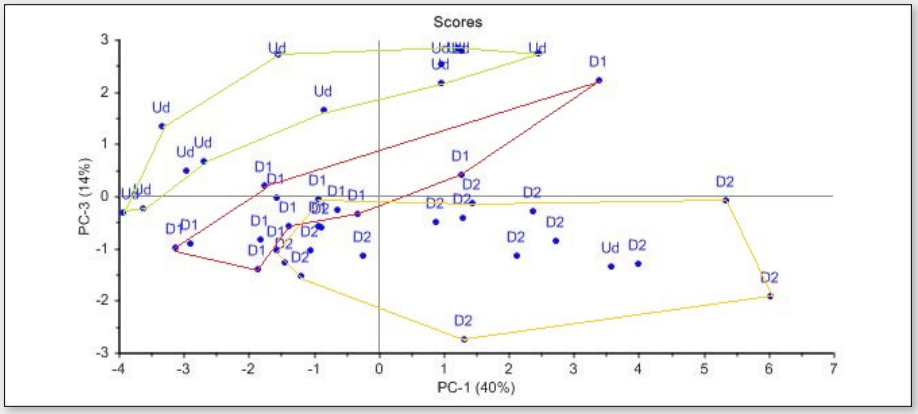

Figure 2.

Using an optimisation algorithm to determine a stockpiling and blending strategy for iron ore

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WorleyParsons RSA conducted a feasibility study on the development of an open-cast iron ore mine in South Africa. As part of the study the team needed to determine how many stockpiles of run-of-mine ore should appear before the crushing section, how the mined material should report to the stockpiles and what is the maximum mass of saleable iron ore that could be blended from the stockpiles. The specifications obtained from the client stated that the blended iron ore product has to contain at least 58% iron (Fe), with a maximum content of 10% silica (Si), 3.5% aluminium oxide (Al_2O_3), 0.07% phosphorous (P), 0.05% sulphur (S), 0.3% alkali and 0.5% manganese (Mn), respectively. A mine plan model showed that batches of run-of-mine (ROM) ore would report to the surface on a weekly basis over the life of mine and also indicated the associated concentrations of Fe and other contaminants. This data was obtained from a geological model of the ore body and showed a great variety in the concentrations

of all elements. These varying characteristics necessitated the development of a strategy to ensure that the maximum amount of saleable product and minimum amount of waste will originate from the operation.

A numerical score was calculated for each batch of ROM ore that reported to the surface, based on its Fe and Si concentrations, as predicted in the mine plan. Based on this numerical score the material was allocated to different stockpiles, where the most ideal stockpile had the highest Fe and lowest Si concentrations, respectively. Conversely, the least ideal stockpile had the lowest Fe and highest Si grades, respectively. At each time step, a multivariable optimisation algorithm in Matlab® (fmincon) calculated the maximum amount of saleable product that could be formed through the blending of material from all available stockpiles, while adhering to constraints on the concentrations of all elements. Another set of constraints were formed by specifying that neither the masses taken off the stockpiles nor the actual stockpile levels can take on non-negative values. The result of performing the stockpiling and blending algorithm for a selection of between four and 12 stockpiles resulted in estimated weekly production data over the life of mine, as well as the associated stockpile levels and the concentrations of Fe and all other contaminants. Out of all scenarios it was found that 12 stockpiles allow for the greatest flexibility for blending run-of-mine ore which leads to the highest mass of saleable material with the least amount of waste material left over at the end of life of mine. The algorithm also allows the user to lift any or all of the restrictions on the concentrations of contaminants and it was found that production over the life of mine can be increased by 11% if only Fe and Si constraints are considered in the optimisation algorithm.

Sample station design and operation

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Accurate sampling practices in the mineral industry are critical to determining the chemical, mineralogical and physical characteristics of ores and mineral products for resource evaluation and utilisation, feasibility studies, process design and optimisation, quality control, metallurgical accounting, and ultimately commercial sales. Sampling is the first step in the measurement chain and is where the measurement process all begins, so if the sample that is collected is not representative, then the whole measurement chain is compromised at the outset. However, frequently the responsibility for sampling is entrusted to personnel who do not fully appreciate the significance and importance of collecting representative samples for analysis, and everyone seems satisfied as long as some material is collected and returned to the laboratory for analysis. In the case of sample stations, cost is often the main consideration rather than sampling correctness. Clearly this is unacceptable and needs to change.

The paper will provide guidelines for the correct design of sample stations, which will be illustrated with a range of examples of good and bad designs, eg, the design of primary cutters, the operation of secondary cutters, crusher performance, sample mass and ease of inspection. It is important that sampling experts are involved at the design stage to avoid design flaws and the subsequent need for expensive retrofits to address major problems. Furthermore, ongoing audits of performance need to be conducted to ensure sample stations are adequately maintained and continue to conform with correct sampling principles. Provision also needs to be made for duplicate sampling to monitor the precision achieved in practice on an ongoing basis for quality assurance purposes.

PFTNA logging tool and its contributions for boreholes in situ elemental analysis

P. Jeanneau and V. Flahaut
Sodern

Nowadays, nuclear geophysical techniques are used extensively for boreholes characterization with main demand from oil well logging exploration. The Oil and gas industry has made large efforts to develop high tech components that can withstand great depths severe conditions. PFTNA (Pulsed Fast and Thermal Neutron Activation) is one of those nuclear elemental analysis techniques that gained great benefits from innovation in scintillator detectors and reliable and safe electrical neutron sources introduced in oil well logging. Sophisticated methods, sustained by ever more powerful computers, also enable simulation for optimum instrument design and advanced data processing.

Relieved from major challenges as high stress in temperatures and shocks, the technology can be simplified, and reasonably priced, for the mining industry where boreholes elemental analysis receives increasing interest either in exploration or mining. PFTNA method is currently applied for in situ measurements of the elemental composition of the rock surrounding the hole. It can also derive some physical properties as density. Traditionally, borehole material is collected in various size and shapes upon drilling techniques from solid continuous cores to cuttings surface recovery and then sent to laboratory for assay. This provides desired information about boreholes, but the whole process usually extends over several weeks, delaying as much any decision to proceed. PFTNA Nuclear logging is able to provide information almost instantaneously for major elements. Proposing different tradeoffs in response time and performance, laboratory analysis of samples and PFTNA in situ elemental logging should be implemented accordingly. The two approaches should also not be opposed in regards with theory of sampling (TOS). If physically inaccessible, samples volume measured by nuclear PFTNA can be yet investigated in details by Monte Carlo simulations. Effective measured samples with this contactless methods is yet found rather complex to establish. Nuclear signals depth of penetration in the material is usually roughly defined and submitted to the influence of material properties themselves. But physical sampling methods could also be severely hindered by classical source of errors attached with samples recovery or handling operations. Representativeness of those samples remains also limited to infer surrounding material composition especially in heterogeneous deposits. The volume of rock sampled each meter by nuclear PFTNA borehole logging is in the cubic meter range and thus provides very good sampling statistics.

Through the case of a recently developed instrument, this paper describes first the latest in design of PFTNA nuclear

logging tool. If the heart of the physics of the instrument lies in a high resolution gamma scintillator detector coupled with a recent compact electrical neutron source, the whole system is discussed such as the benefit of modern digital electronics, surface to tool data transfer and user interface. The paper will then discuss the outcome of elemental analysis with PFTNA and how it is interrelated and mutually supportive with traditional sampling method

Improved counteracting soil heterogeneity sampling designs for environmental studies – TOS meets chemometrics

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This project aims at development of an improved soil heterogeneity characterization methodology for 'next generation' sampling/monitoring and spatial modeling practices a.o. allowing consideration of realistic pesticide variability in environmental contaminant assessment studies. Within the environmental sciences there is a strong need for an integrated understanding of chemical contaminant transformations (e.g. pesticide degradation), spatial modeling and multivariate data analysis. All critical parameters are in need of effective counteraction of the variability related to inherent soil heterogeneity. This study evaluate a series of improved designs of field and laboratory sampling experiments at all stages from the primary field sampling to the final analytical sample preparation. The effect of soil heterogeneity at different scales critically affects the sampling/monitoring procedures involved. Soil samples were collected from the topsoil (A-horizon; 0-25 cm) of a typical sandy soil with an equidistance of 1 m along a 100 m long profile oriented parallel to plough lines intended for *large scale* variographic analysis; results for clayey soils will also be presented. Each sample contains 20-30 gram moist soil; the profile center included a *short scale* replication experiment. A large suite of 38 inorganic elements in addition to moisture, loss on ignition and bacteria counts (CFU) were analyzed, plus carbon-14 measurement of MCPA sorption and mineralization and glucose respiration. Contemporary sampling approaches in environmental/soil sciences makes little or no allowance for soil heterogeneity, often resulting in significant between-pot heterogeneity which unnecessarily endangers the discriminating power in laboratory pot experiments. Variographic analysis shows the advantage of using composite sampling locations with a distance below the pertinent minimum half-range for both organic and inorganic compounds based on the empirical soil sample autocorrelations. Synoptic overviews of the correlation data structure between 40+ chemical parameter, as well as between their variograms, were analysed by multivariate data analysis (chemometrics), allowing to quantify the combined covariance-spatial soil heterogeneity.

Review of a non-probabilistic sampler versus a vezin sampler on low weight percent solids slurries

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The Hanford Tank Operations Contractor (TOC) and the Hanford Waste Treatment and Immobilization Plant (WTP) contractor are both engaged in demonstrating mixing, sampling, and transfer system capability using simulated Hanford High-Level Waste (HLW) formulations. This work represents one of the remaining technical issues with the high-level waste treatment mission at Hanford – the TOCs' ability to adequately sample high-level waste feed to meet the Waste Treatment and Immobilization Plant (WTP) Waste Acceptance Criteria Data Quality Objectives. A full scale sampling loop was used at a cold test facility to evaluate sampler capability. The sampler under investigation for deployment is non-probabilistic but radioactive environment friendly. A Vezin sampler (probabilistic) was used to obtain reference samples and accurately characterize the simulant as it flowed through the test loop. The two samplers are located in series, allowing for multiple samples to be taken from both samplers over the same time period (sample pairs) and direct sample comparison. The Vezin sampler was modified to minimize material build up allowing for steady-state operation. This report discusses modifications made to the Vezin sampler and the results of sampler comparison.

A method to evaluate the possible sampling error of a multiple cutter metallurgical sampler

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The head loss caused by metallurgical sampling for the slurry stream can be reduced by metallurgical sampler design. When the process stream volumetric flow is sampled by vertical static cutters before the equal number of moving cutters, the installation requires less installation head space and is easier to accommodate at a suitable location in the process. Low head loss reduces the building costs for the processing plant and operational costs during the life time of the plant.

The presence of a possible systematic bias in the particle size distribution or the chemical composition between the vertical static cutters caused by segregation in the metallurgical sampler can be estimated by a sampling campaign where sub samples are cut from each of the moving cutter sample streams simultaneously. The sub sample assay results can be evaluated by the F test to reveal if there exists significant variance between the cutter assays.

The heterogeneity and minimum possible error caused by the sampling and analysis system can be estimated by performing a sampling campaign where spot samples are collected at

equal intervals to perform a variographic experiment to study the heterogeneity of the process stream and a minimum possible error by estimating the V_0 intercept. V_0 is the variability of a single measurement and is an indication of the minimum practical error (MPE) and also the minimum sampling variance expected in practice.

MPE includes the fundamental sampling error, the grouping and segregation error, the analysis error including preparation errors and the possible incorrect sampling errors.

In this paper we present how above mentioned methods can be used to evaluate the possible errors of an economical metallurgical sampler.

Complete sampling distribution for primary sampling, sample preparation and analysis

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Following from the author's recent paper at Sampling 2014 which presented a method for calculation of the sampling probability density function due to the particulate heterogeneity (density function of the fundamental sampling uncertainty), it is possible to apply the same characteristic function method to arrive at the overall sampling distribution for any sampling protocol and analysis method.

This paper develops the application of the method of characteristic functions to the overall sampling problem including the uncertainty which derives from the primary sampling from a process stream. The assay distributions in a process stream or of impurities in the flow of a final product can be governed by non-Gaussian, serially correlated distributions. The paper shows how such circumstances can be dealt with to arrive at robust solutions.

The paper represents an end-point in the theory of sampling as it provides a means of determining the entire distribution function for a sampling system. Such a determination has not previously been possible and having determined the entire distribution function, the statistics of the sampling process are completely determined.

Determination of the precision of sampling systems and on-line analysers

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There is a simple and inexpensive way of determining the precision of sampling systems and on-line analysers when a data base of output values from the sampling system or on-line analyser can be accessed and there exists serial correlation in the data sets. Basically, if it is possible to construct a variogram for the data set, it is possible to extract the precision estimate as the variance is simply given by the intercept (nugget variance) of the variogram.

The method is much superior to doing interleaved sampling, which gives incorrect estimates of the precision when serial correlation exists. It is rare to find that there is no serial correlation in the data.

This paper outlines the method and illustrates the procedure with a number of data sets from various areas. It also demonstrates by simulation why the interleaved sampling method is incorrect. When variogram estimation is made using maximum likelihood methods, a confidence interval for the precision can be calculated.

The weighting error: a study by simulation

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In Gy's theory of sampling, estimation of sampling error due grade variations in a process stream is carried out under the assumption that the mass flow is constant. Gy's work does not provide quantitative analysis of the impact of variations in the mass flow. In the same way as the grade variations are treated as a random function characterised by a variogram or covariance function, mass flow variations can be characterised.

A formal analysis of the impact of mass flow variations uncorrelated with the grade variations leads to complex expressions. Armed with a means of efficiently creating realisations of random functions, it is a simple matter to study the impact on sampling variance by simulation. The simulation method provides exact results for a given set of conditions as long as a sufficient number of simulations are used. Simulation can also deal with cross- correlation between the mass flow and the grade variations.

The paper explores the analysis of the weighting error both analytically and by simulation with the objective of indicating at what magnitude the flow variations begin to cause the sampling uncertainty to increase significantly. Bias in sampling due to cross-correlation is also investigated.

Sampling of cereals : development of a protocol for mycotoxins analysis

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European directives (UE) 401/2006 and (UE) 691/2013 for official controls of some contaminants such as mycotoxins set methods for sampling and analysis.

The composition of batches of cereals is rarely homogeneous and, in particular, certain contaminants like Fusarium-mycotoxins are distributed in a non-uniform way. Sampling therefore is a procedure which requires a great deal of care, and it is necessary to get a representative sample before initiating any analysis.

In order to harmonize sampling procedures and to determine the best way to prepare a homogenous and representative laboratory sample, studies have been undertaken by a French working group associating storage organizations and suppliers of sampling devices.

The aims of these studies were :

- To evaluate the mycotoxines distribution in cereal batches,

- To compare different sampling protocols including the European directive,
- To determine the relationship between the number of samples and the uncertainty of the analysis result, and
- To define an acceptable sample weight for the laboratory.

The results obtained concerned different toxins (DON, fumonisins, zearalenone). They came from trials in large storage silos on flowing or static grains. They showed that the heterogeneity of the batch increases with the level of contamination.

According to the data, it is possible to reduce the number of samples to be taken during sampling without a significant impact on the result. Finally, the studies showed that the weight of the sample sent to the laboratory for the detection of Fusarium-mycotoxins could be reduced.

These results supported the standard EN ISO 24333, Cereals and cereal products – Sampling and more recently allowed an evolution of the European directive.

Comparison between samples with constant mass and samples with constant fragment population size

by G. Matheron
Translated from French to English, clarified and further commented by Dominique François-Bongarçon[1] and Francis Pitard[2]
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In his essay "l'echantillonnage des minerais en vrac" that could be translated as "sampling of particulate ore" published in 1967 in France by the Revue de l'Industrie Minerale, Pierre Gy suggests a calculation of the variance associated with samples with a constant number of fragments. In practice, samples with a constant mass are instead collected, which may seem at first like a contradiction. In this mathematical development it is clearly demonstrated that these two kinds of samples lead to variances that are similar within well-established mathematical limits.

Comparison of sampling methods by using size distribution analysis

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Pierre Gy [1,2] has derived an equation, which can be used to estimate the relative variance of the fundamental sampling error of size distribution results given as mass fractions for each size class. This theory is used in this study. Heterogeneity invariant, ***HI***, is the relative variance of the fundamental sampling error extrapolated to a sample size of a unit mass (usually 1 g). ***HI*** can be estimated from a sieve analysis for each size class ***i*** from Eq. 1.

$$HI_i = \left(\frac{1}{a_i} - 2\right) v_i \rho_i + \sum_{i=1}^{n} \rho_i a_i v_i \qquad (1)$$

Here a_i is the mass fraction of size class i, v_i the average particle size in class i and ρ_i the density of particles in size class i. Given HI_i, the relative variance of the fundamental sampling error, S^2_{FSE}, can be estimated for different sample sizes to be sieved from the test material:

$$s^2_{FSE} = HI_i \left(\frac{1}{m_s} - \frac{1}{m_L}\right) \qquad (2)$$

Here m_s is the sample size to be sieved and m_L the size of the lot from which the sample is taken.

If the sampling methods performs correctly and is able to minimize the segregation effects, always present when material consisting of fragments or particles having a wide size distribution, the observed variance of replicate samples should be close to that obtained by using the above equations and it is possible to calculated confidence interval for a given size distribution.

In this study a newly developed sampler was tested by sampling blast hole drill cuttings and the results were compared to other sampling methods currently in use. Part of the samples were also sent for chemical analysis to see if the analytical results correlate with the size classes. A convenient way to summarise and compare size distribution results and analytical results is carry out Principal Component Analysis (PCA) on both size data and analytical data. These results will be presented WCSB7.

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Empirical evidence for a simplified version of Gy's qquation for low grade gold ores

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Classically Gy's equation for the Fundamental Sampling Error takes the form:

$$\sigma^2_{FSE} = \frac{Kd^3_N}{M_S} \qquad (1)$$

Later work by Francois-Bongarçon (1995) led to a modification of Gy's formula, particularly for low grade gold ores that takes the form:

$$\sigma^2_{FSE} = \frac{Kd^{\alpha}_N}{M_S} \qquad (2)$$

Where the exponent to d_N is replaced by alpha (α) rather than 3 as given in the original equation by Gy. Francois-Bongarçon (1995) further showed that the sampling constant K and the exponent alpha can be calibrated for a given ore-type and that the calibrated constants could then be used to provide the ideal solution for stages of comminution and mass reduction in the sampling nomogram for that particular ore.

The calibration method suggested by Francois-Bongarçon (1995) has been carried out numerous times by various sampling practitioners and is a well-established procedure in the literature. The procedure is referred to in the literature as the Duplicate Sampling Analysis (DSA) method and is clearly described by authors who have undertaken this type of calibration. Another type of calibration procedure referred to as the Segregation Free Analysis (SFA) method produces results similar, if not identical, to that suggested by the DSA method of Francois-Bongarçon (1995). All the calibration experiments carried out in low grade gold ores have produced values for K that vary between 70 and 170 and have yielded values for alpha in the range 0.97 to 1.30. The range of values for alpha suggests strongly that the average value for alpha in low grade good ores may in fact be 1, rather than 3 as originally proposed by Gy (199?) (Equation 1) or the 1.5 proposed by Francois-Bongarçon (1995) (Equation 2).

It is suggested that for low-grade gold bearing ores that the equation for the Fundamental Sampling Error should take the form:

$$\sigma^2_{FSE} = \frac{Kd_N}{M_S} \qquad (3)$$

Such an equation for the variance of the Fundamental Sampling Error greatly simplifies the characterisation of gold ores which now only required the calibration of K for a given mass and established fragment size.

Food and feed safety assessment: proper sampling is imperative

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The general principles for safety and nutritional evaluation of foods, feed and associated hazardous compounds have been developed by FAO and WHO and further elaborated in the EU funded project SAFE FOODS. Nevertheless, the crucial role that sampling has in foods/feed safety assessment has never been explicitly recognized. High quality sampling should always be applied to ensure the use of adequate and *representative* samples as test materials for hazard identification, toxicological and nutritional characterization of identified hazards, as well as for estimating quantitative and reliable exposure levels of foods/feed or related compounds of concern for humans and animals. The importance of representative sampling is underlined through examples of risk analyses in different areas of foods/feed production. The Theory of Sampling (TOS) is recognized as the only frame to ensure accuracy and precision of all sampling steps involved in the field-to-fork continuum, necessary to monitor foods and feed safety. As such, it is emphasized how TOS must be integrated in the well-established FAO/WHO risk assessment approach in order to guarantee a transparent and correct frame for the risk assessment and decision making process.

Practical case: representative sampling for full-scale incineration plant test

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Today all impregnated wood waste in Denmark is exported. The Danish Environmental Protection Agency needed knowhow in order to investigate whether wood waste could be incinerated in an environmentally safe way in Demark.

Therefore, the Danish Technological Institute was commissioned to execute a full-scale test, including the measurement of all waste streams on an already existing incineration plant.
For the test, 600 tons of impregnated wood waste was gathered. For mass balance purposes, a representative sample of this wood waste was necessary.

Prior to the incineration test, a procedure for sampling was prepared. The plan involved successive steps of sample mass reduction in the waste deposit site, including shredders and front loaders. The reduction of the 600 tons of wood waste resulted in a 10 kg sample. The further sample preparation, including further mass reduction, was handled in the laboratory.

A project funded by the Danish Environmental Protection Agency

Sample system designs for the new NSPS standards

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In 2015 all flares in the United States which have sour gas feeds will be required to meet the requirements of 40 C.F.R. Part 60 Subpart Ja. The need to meet this rule has required companies with flares that fall under the guide lines to determine how best to implement the rule at their facility.

Triad Control Systems designs and builds analytical systems for the petrochemical industry. For the past year we have been very involved in the design, construction and implementation of systems designed to meet the requirements of Subpart Ja.

This paper will review several different ways companies in the Gulf Coast region of the United States have chosen to address the requirement of Subpart Ja. Focus will be on the analytical equipment chosen and the sampling techniques devised to meet the stated requirements of the ruling. Additional emphasis will be give to the safety issues involved in working with high concentration H2S samples.

Layout, sampling methods and equipment used in the Continuous Environmental Monitoring systems (CEM's) to monitor the amounts of Hydrogen Sulfide (H2S) and other sulfur compounds sent to the flare will be reviewed. Additionally, methods to monitor the flow through the flare will be addressed.
H2S is a very toxic chemical and must be handled very carefully in order to prevent injury to personnel. Many of these flares during upset conditions can see H2S concentrations go from 0 to +60% very quickly. How to address these safety issues will also be covered.

The decision unit – a lot with objectives

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Sampling is more than shoveling material into a bucket. It is even more than using adequate mass, increments, and tools. Sampling is a systematic process that incorporates everything from development of objectives through final decision-making. Many sampling protocols currently in use focus only on the physical sample collection and ignore the preceding steps in the sampling process. The ignored steps include the development of the objectives, integration of sufficient quality control, inferences from test portion to lots, and final decision making, statistical or otherwise. Without this supporting framework, it is impossible to ascertain the validity of the sampling protocols when needs or objectives change. Often, the same sampling protocol is implemented year after year without any consideration to its appropriateness.

Proper Sample Quality Criteria (or Data Quality Objectives) are to be determined from the objectives of the project and must be an integral part of any sampling campaign. The major components of the Sample Quality Criteria are 1) Question, 2) Decision Unit, and 3) Confidence. The Decision Unit is the specific material to which an inference from the analytical result is made and ultimately to which a decision is made. If the Decision Unit is not precisely determined and integrated into the development of the sampling protocol, the resulting decisions will be incorrect or, at a minimum, not cost effective. This contribution will address development and integration of the Decision Unit into the sampling protocol framework.

The role of inference in food safety

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People have been trying to determine the safety of their food since ancient times. In ancient times, people themselves were the ultimate test of food safety, but as humans progressed other techniques such as sensory perception and experimentation on animals were used. Today sophisticated analytical techniques and models are available to measure and predict food safety. These sophisticated techniques and models are dependent not only on the quality of samples that are collected and analyzed but also on how inferences are made from the analytical results to the food being sampled. Unfortunately, the Theory of Sampling and the role of inference have not been fully integrated into prediction of food safety.

The basis for many “modern” food sampling protocols was developed prior to the development of the Theory of Sampling. Many of these sampling protocols were based on concepts of acceptance sampling procedures and associated inference. The Theory of Sampling enables the representative sampling of bulk materials and eliminates the reliance of acceptance sampling as the only method for the characterization of food and utilizes a different type of inference than for acceptance sampling. This contribution addresses the differences between inference for acceptance sampling and inference for the sampling of bulk materials and the implications of these differences for food safety.

TOS to the rescue: estimating TSE for near infrared spectroscopic analysis of pharmaceutical blends

Rodolfo J. Romañach, Barbara Alvarado, Andres Román Ospino, Kim H. Esbensen

The results of a **replication experiment**[1] of a five component pharmaceutical blend are presented. Replication experiments represent a new approach to the analysis of pharmaceutical blends and to estimating sampling and measurement uncertainty. The current protocol for routine analysis for pharmaceutical blends preclude use of replication experiments, they being either too labor-intensive or they may alter the powder mixture every time that samples are extracted from a lot or batch. The blends analyzed here are similar to many pharmaceutical formulations since they include lactose monohydrate and microcrystalline cellulose, the two most common excipients in the pharmaceutical industry and include acetaminophen (APAP), a widely used active ingredient. APAP is a very cohesive powder and pharmaceutical formulators are challenged to break drug agglomerates to achieve the desired drug distribution. This experiment also sheds light on current ‘mixing efficiency’ issues, which is a subject of intense discussion in pharmaceutical manufacturing. In this present research sampling is performed with near infrared spectroscopy (NIRS), which brings a number of advantages to the fore. The principal advantages of NIRS stem from its non-destructive nature and rapid analysis times, which may range from a few milliseconds to 30 seconds, depending on the spectrometer used. These features facilitate the use of a replication experiment. Measurement repeatability was determined by focusing the NIR beam on a single analysis footprint of the powder mixture, taking many consecutive measurements at identical conditions. This part of the experiment furnishes an estimate of the Total Analytical Error (TAE). Subsequently the replicate experiment is also deployed analyzing one extracted test portion of the blend multiple times; subsequently several aliquots were extracted as close to one-another as possible. Finally, similar analysis of multiple aliquots from the lot were performed to determine the Total Sampling Error in a pattern dictated by the current regulatory protocols. The resulting hierarchical variances are subtracted from one-another allowing a complete decomposition of the individual error contributions in the chain primary sampling errors, secondary ditto, test portion extraction error and var(TAE) respectively. This experiment was repeated but now carried out on the output material stream exiting the mixture blender upon mixing completion (on its way towards tableting), i.e. conducted as a *variographic experiment*. These experiments provide critical information to develop adequate regulatory expectations based on a quantitative comparison between these two principal approaches – always subject to the degree that the laboratory simulations can be shown to be identical to routine manufacturing practice (scaling-up issues). In the pharmaceutical industry there is today a strong desire to move towards ‘sampling-free’ approaches such as PAT (Process Analytical Technologies), but NIR is not a panacea, the main limitation being that the mass analyzed by the NIR radiation is not known – this is conventionally *assumed* to be comparable, if not constant. The analyzed volume, i.e. sampled volume, or mass, can be estimated however; we present a new approach for this critical part of the NIR

analytical approach. The sampling volume may also be reduced or increased as a function of the spectral acquisition parameters. At this time a complete understanding of PAT 'sampling' and the associated analytical error for NIR analysis of powder blends is not available however - TOS and the **replication experiment** provide a critically needed contribution forward.

Reference

[1]DS 3077 Horizontal. Representative Sampling.

When "homogeneity" is expected – TOS in pharmaceutical manufacturing

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Pharmaceutical manufacturing contains an expectation, indeed a regulatory demand that powder blends that precede tablets and capsules be "homogeneous". This term is a first collision between TOS and pharmaceutical industry quality control (QC) practices. Here "homogeneous" does not imply a perfect mixture where the distribution of particles is strictly identical throughout the lot however, but is used to communicate that heterogeneity is sufficiently low that patients will receive a product with the strength "it purports or is represented to possess". These "homogeneous" unit doses are usually required a relative standard deviation (RSD) of less than 5%. Quality control units in pharmaceutical manufacturing have a strong interest both in determining the average concentration of a blend, and an equally strong interest in determining how the drug varies throughout a lot (*so much for homogeneity in TOS' fashion*). The pharmaceutical industry has traditionally relied on *grab sampling* (using sampling spears) to obtain the desired information in this context, which to any informed TOS-entity appears as Mission Impossible; indeed a significant number of publications have described the problems that have resulted. Industrial strategies have sought to find "dead spots" in blenders with incomplete mixing that could lead to an over or under-dose. This traditional pharmaceutical QC area is now being addressed with a new TOS-based approach: i) It is futile to continue searching for documentable heterogeneity in blenders, which are emptied onto conveyor belt immediately after sampling anyway; ii) variographic analysis (for both batch and continuous mixing) are being brought to bear. Real-time analysis of drug concentration is now performed by near infrared spectroscopy, a non-destructive analytical method applied to blender output streams (what else?). Replicate experiments will be presented and evaluated. The quest only to rely on *relevant* total Measurement Uncertainty systems (MU) of pharmaceutical blends has started: TOS meets pharma!

Geostatistical comparison between blast and drill holes in a porphyry copper deposit

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Diamond drill holes grades are known to be of a better quality than blast holes ones, is it true? We present a formal study of a porphyry copper deposit in Chile where the 3 meter length drill hole samples variogram is compared to the 15 meter length blast hole ones and we show that the blast holes can be considered as a regularization of the punctual information deduced from the drill holes, up to a nugget effect proper to the blasts.

In fact the blast drilling length is approximately 17m and to restore the requested 15m, 5 cm of the material is removed by hand from the envelop of the blast cone, leading to another question: could the error proper to the blasts be due to the arbitrary removal of this material and the blast length variability? Extension variances show that the answer is no and the blast error belongs to the set of Pierre Guy sampling errors. In the study, the drill information is taken as reference because:
Referring to the blast is not possible, the blast nugget effect is too important,

We are in an full heterotopic case where there is no spatial location where blast and drill are both known and could make possible the calculation of cross variograms,
The variograms present a linear component which forbids the use of cross co-variances.

Consequence is that we could not model an eventual error proper to the drills and the blast error pointed out is a minimum. The maximum could be 50% more important if there is no "natural" nugget effect (i.e. micro structure).

The first conclusion is that the blast holes in this mine are of much better quality than one generally believes, justifying the short term planning and the ultimate selection based on them.

Design advances and operational studies for the True Pipe® Sampler: a symmetry based unit for reliable sampling of pressurised particulate streams

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Obtaining representative samples with minimised sampling errors, is critical for calculating accurate metallurgical mineral balances on process plants. A challenging situation exists, where no acceptable, robust or economically viable sampler has been commercialised for sampling of one-dimensional pressurised slurry pipelines yet. The design of the True Pipe® in-line sampler is based on the principle of symmetry, as described by Dominique François-Bongarçon (2005), and operates on a fail-safe principle for control on the synchronous opening and closing of valves for the sample chamber. Previous test work on the True Pipe® in-line sampler indicated that the prototype sampler is reliable within certain tolerances, initially indicating the concept could well be a viable design option. This paper presents the results from further test work, which mainly investigated three sampling phenomena in more detail, by examining classical one dimensional sampling, with the aid of an automated valve actuator. Firstly, the transient effect, which originates from the disruption in laminar particle flow. Secondly, the effect of split sampling, where the portion of the stream is sampled as well as the full stream. Thirdly, the effect of symmetry is confirmed. The expected accuracy level of the True Pipe® in-line sampler is also evaluated for varied material conditions. Advances on the design include the ability to sample the entire pressurised particulate stream in a safe

operating condition, by making use of a mechanical actuator for synchronous opening and closing of the sample chambers, as well as improved control on the valve opening and closing cycles.

Practical use of variographics to identify losses and evaluate investment profitability in industrial processes

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The first example concerns production of light-weight expanded clay aggregates ('LECA'), produced in cement-like rotary kilns. Clay raw material is heated to 1150°C to be expanded. A periodicity was observed in a specific plant cooler regarding fluctuations in the material height (level). This influences the amount of air passing through the cooler and thereby amount of air and the pressure in the kiln. Periodicities in the pressure in the kiln cause periodicities in the level of expansion and thereby output from the kiln. A lower expansion means a smaller amount (m^3) of material produced from the same amount of raw material, i.e. higher production costs (sales are valued by m^3). This problem can be resolved by a more stable level in the cooler, which could be engineered by a small investment of about 20,000 Euro. A variogram characterization was carried out to evaluate the amplitude of the periodicity, and thereby the quantities involved (losses), which were finally used to calculate the pay-back time of the investment. From the variogram it was observed that the lowered kiln output was at least 0.4%. During one year with improved cooler level control this translates into savings of about 50,000 Euro, i.e. a pay-back time will be less than 6 months. The other example is from a LRM-project (Loss and Reduction Model) at a plant producing pre-mixed mortars, in which the variance of the weight of the produced bags was found to be consistently too large. A variographic analysis is applied with an aim to identify the root cause(s) of this problem (previous work has identified several potential factors contributing to this variance). Investment caution rules the day, e.g. it is futile to invest in expensive equipment to level the filling degree if the main problem turns out to be a high nugget effect v(0) caused by inaccurate scales. In such a case, investment should instead be directed at upgrading the scales or to change the scale control routines. This investigation is currently ongoing; final results will be presented at WCSB7. Variographic analysis is a powerful tool for industrial technicians, for process engineers - and industrial managers as well, in the present case for evaluating investment profitability in industrial processes.

JAOAC special guest editor section: representative sampling for food and feed materials: a critical need for food/feed safety

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A special collection of papers on all aspects of food and feed safety sampling - to be used in risk assessment, process control in a food/feed manufacturing environment, foodborne disease outbreaks, and regulatory compliance -is now available as open access on the Journal of AOAC INTERNATIONAL's website. Visit http://aoac.publisher.ingentaconnect.com/content/aoac/jaoac for 11 fully refereed papers in J. AOAC Int.'s March/April 2015 issue.

These papers, with unrestricted online access, are the result of a ground-breaking trans-Atlantic collaboration between researchers, samplers, and regulators from Europe and the United States, a true first within the sampling world. Indeed, the authors gathered in Windsor, Colorado in October 2014 to collaborate and write. The authors brought strong opinions to the meeting and worked hard to reach a consensus. (jokingly referred to as the 'shootout'.)

The papers in this Special Section introduce the Theory of Sampling (TOS), which is relevant for all aspects of food and feed safety sampling, as the principles governing representative sampling apply universally. The papers are not independent; they were written and composed to integrate with each other, thus providing a comprehensive overview of the criteria that must be followed to ensure representative sampling.

The guest editors were: Kim Esbensen, Geological Survey of Denmark and Greenland and Aalborg University, Denmark, Claudia Paoletti, European Food Safety Authority Parma, Italy and Nancy Thiex, Thiex Laboratory Solutions, and Agricultural Materials section editor for the Journal.

The target audience for this Special Section includes all food/feed protection personnel: field sampling operators, academic and industrial scientists, laboratory personnel, companies, organisations, regulatory bodies and agencies that are responsible for sampling, as well as their project leaders, project managers, quality managers, supervisors, and directors. In the United States alone, there are an estimated 45,000 federal, state and local food/feed regulatory personnel, not including industry or laboratory personnel.

"We hope to trigger a scientific discussion and awareness towards global harmonisation of representative sampling approaches for food and feed commodities," it is stated in the section's introduction. "As a collection, these papers represent a leap forward with respect to a valid analytical methodology for the discipline."

The Special Guest Editor Section includes the following contributions:

- "Food and Feed Safety Assessment: Proper Sampling is Imperative" by Harry Kuiper and Claudia Paoletti. A free online appendix of terms used in key sampling standards and documents is included.
- "Towards a Unified Sampling Terminology: Clarifying Misperceptions" by Nancy Thiex, Kim H. Esbensen and Claudia Paoletti
- "A Systematic Approach to Representative Sampling" by Charles Ramsey and Claas Wagner
- "Sample Quality Criteria" by Charles Ramsey and Claas Wagner
- "Materials Properties: Heterogeneity and Appropriate Sampling Modes" by Kim H. Esbensen
- "Theory of Sampling—Four Critical Success Factors Before Analysis" by Claas Wagner and Kim H. Esbensen
- "Quality Control of Sampling Processes—A First Foray; From Field to Test Portion" by Kim H. Esbensen and Charles Ramsey
- "Considerations for Inference to Decision Units" by Charles Ramsey
- "Distributional Assumptions in Agricultural Commodities—Development of Fit-for-Decision Sampling Protocols" by Claudia Paoletti and Kim H. Esbensen
- "Critical Practicalities in Sampling For Mycotoxins in Feed" by Claas Wagner
- "Considerations for Sampling Contaminants in Agricultural Soils" by Charles Ramsey
- "Considerations for Sampling of Water" by Charles Ramsey

The Special Guest Editor Section is now available online at http://aoac.publisher.ingentaconnect.com/content/aoac/jaoac

The Special Guest Editor Section is scheduled for the March/April 2015 print issue of J. AOAC Int. For additional information on publication of this special section, please contact Catherine Wattenberg at cwattenberg@aoac.org

Figure 1. Transatlantic Special Section taskforce, October 2014, Windsor, Colorado (left to right): Nancy Thiex, Thiex Laboratory Solutions; Kim H. Esbensen, Geological Survey of Denmark and Greenland and ACABS Research Group, University of Aalborg; Charles Ramsey, EnviroStat, Inc.; Claas Wagner, Wagner Consultants; and Claudia Paoletti, European Food Safety Authority, Parma, Italy.

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Claudia Paoletti is employed by the European Food Safety Authority (EFSA). The positions and opinions presented in this special session are those of the author alone and do not necessarily represent the views or scientific works of EFSA.

Critical practicalities in sampling for mycotoxins in feed- heterogeneity characterisation

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The presence of mycotoxins, in particular aflatoxin B1, can cause significant health problems as well as severe economic loss, and are therefore regulated with respect to maximum acceptable concentration for various feed- and foodstuffs. International regulatory authorities have recognized the importance of representative sampling, and sampling guidelines have been formulated which only partly comply with the Theory of Sampling (TOS). In particular, practical guidance regarding sampling, including correct design and operation of sampling devices and explanation on how to develop sufficient sampling protocols are lacking in current guidelines. These are critical practicalities of main importance, especially when dealing with trace concentrations and/or concentrations that are irregularly distributed - as is the case for mycotoxins. Furthermore, heterogeneity characterization, which is a necessary requirement to be able develop valid sampling protocols or validation assessments of existing sampling operations, is currently not mentioned in the existing guidelines. The present paper focuses on heterogeneity characterization with respect to sampling of mycotoxins for 1–D and 3-D feed decision units. Structural guidelines for correctly designing experimental heterogeneity characterizations are presented, allowing evaluation of sampling representativeness and determination of optimal number of increments per composite sample.

A European standard for sampling of waste materials: EN 14899

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Wastes are materials, which the holder discards, or intends or is required to discard, and which may be sent for final disposal, reuse or recovery. Such materials are generally heterogeneous and the testing of them allows informed decisions to be made on the appropriate way in which they should be treated or not, recovered or disposed. In order to undertake valid tests a representative sample of the waste may be required.

The European Standard EN 14899, developed for the characterisation of waste by the European Committee for Standardisation and published in December 2005, specifies the procedural steps to be taken in the preparation and application of a waste Sampling Plan. The Sampling Plan describes the method of collection of the laboratory sample necessary for meeting the objective of the testing programme. The principles or basic rules outlined in this European Standard, provide a framework that can be used by the user:

- to produce standardised sampling plans for use in regular or routine circumstances (elaboration of daughter/derived standards dedicated to well defined sampling scenarios),
- to incorporate the specific sampling requirements of European and national legislation,
- to design and develop a Sampling Plan for use on a case by case basis.

This European Standard is accompanied by five Technical Reports dealing with sampling techniques and procedural options and providing essential information and instructions for its application.

Innovative sampling solutions for the mining industry

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While online analytical systems are continuously improving the mine site laboratory remains the benchmark. The laboratory is expected to produce high quality information, so the sampling process is critical. Process managers demand high quality, timely produced results from the laboratory. Mine managers and shareholders are demanding that the process, analytical results and productivity is optimized maximize return on investment. These demands conflict with traditional sampling and laboratory routines which are frequently slow, labor intensive and frequently involve potentially dangerous, not to mention unscientific methods and work practices.

For more than a quarter of a century, working with the world's largest mining companies. Over this time, IMP has teamed with its partners and likeminded customers, to challenge conventional sample collection and processing techniques. In doing so we have developed ground breaking innovative automated sampling and laboratory solutions for the mining industry. This paper will introduce you to a selection of the automated sampling and laboratory solutions that we have developed. It will achieve this by presenting case studies, including but not limited to, a time based and mass based automated sampling and laboratory solutions for lump and fines, powder sampling and analysis techniques and slurry sampling and analysis solutions.