SAMPLING COLUMN

There are standards—and there is *the* standard

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This series of columns has come to a natural half-way stage, at which it is worth reflecting a little. The basic principles for sampling of heterogeneous stationary lots, materials and systems have been covered—it is time start thinking of sampling from moving lots, dynamic systems and processes. But first: the Theory of Sampling (TOS) is proclaimed to be the only complete theory with which to address all the world's many types of materials with a view of guaranteeing representative samples, and the column makes an effort, hopefully appreciated and easy to follow, to explain all the elements and their relationships in this endeavour. However, many standards, guidelines and norm-giving documents (CEN, ISO) already exist, which include elements of prescriptions for "proper sampling", such as have been agreed upon by numerous task forces, committees etc. as being fit-for-purpose within the relevant scientific, trade and technological contexts addressed. There have been many such fits and starts towards a recommended sampling practice, but always in a partial sense only, indeed *none* cover the full breadth of all that is necessary to master representative sampling (with two spectacular exceptions: the iron ore and cement industry sectors). With so many partial recommendations available, when not in compliance with respect to TOS, there are objective, serious contradictions. *Que faire*?

The situation

The publication of DS 3077[1] represented the world's first standard dedicated exclusively to representative sampling. Hardly any other standard is in full compliance with the appropriate TOS requirements laid out here, although *partial* elements can be found in many places, e.g. see the bibliography in DS 3077.[1] Two notable exceptions exist, however, the cement and the iron ore industries, which have been well serviced with excellent standards in this context for many years.

Non-compliance issues regarding such standards, guidelines, good practices as well as regulatory and legal requirements must be handled with insight and patience. Where found not to comply with TOS' stipulations, it will be necessary to start a process of revision or updating of the relevant standards or norm-giving documents—which may be a lengthy process, and one that requires quite some logistical and organisational drive. While this is taking place, or when dictated by documented sampling variances that are too high (a key issue in quality control and assurance, QC/QA), it is always an option to employ more stringent quality criteria from a TOS-based approach than what is specified in today's imperfect standards. As there are serious economic and societal consequences of non-representative sampling, simply staying with "following the book" is never a sound strategy, scientifically as well as regarding the economic outcome of decisions which will then in reality be based on inferior, non-representative data. DS 3077 has the overall objective of establishing a comprehensive motivation and competence for taking the stand relying *only* on fully TOS-compliant sampling procedures and equipment irrespective of the theoretical, practical, technological, industrial or societal context under the law. No standard is a legal document on its own and is therefore not legally binding; all trade agreements ruled by international standards are based on a set of voluntary agreements. To the extent that international law on the subjects treated in standards dealing with sampling aspects has been adopted, this law must be adhered to. International law implemented in national laws also takes precedence to non-legal documents in case of conflict.

Be this as it may, there are very many advantages in not being complacent with the fact that sampling issues are mentioned in the existing body of relevant standards and norm-giving documents. Mentioning is not enough, only the principles guaranteeing representativity matter. A directed effort has been in place for some five years, involving a systematic critique of selected standards, specifically with respect to the full set of sampling errors outlined in TOS. Two examples of this work are presented below, which suffice to show how one should approach any part of a standard etc. that purports to recommend proper sampling procedures and equipment etc.

Analysis of sampling standards for solid biofuels

Assessment of all sampling procedures from CEN standards for sampling solid biomass (CEN/TS 14778 part 1 and part 2)[2,3] has shown that most of the recommended procedures do not lead to a fully satisfactory result, a representative sample. Correct delineation and extraction of many standardised methods as well as depicted, and thus

recommended, tools and equipment are *not* ensured. While for grab and shovelling methods, correct delineation and extraction is hardly ever possible, other recommended sampling methods lack sufficient specification regarding application conditions, which invariably increases the potential for incorrect sampling error effects. Table 1 gives an overview of the evaluation results with respect to potential *incorrect sampling errors* (ISE) caused by the methods stated, recommended or allowed in the standard for primary sampling CEN/TS 14778.[2,3] ISE comprise the three so-called bias-generating errors: Increment Delimitation/Delineation Error (IDE), Increment Extraction Error (IEE) and Increment Preparation Error (IPE), all concerning sampling equipment and sampling procedures. The full assessment of these sampling standards can be found in Wagner & Esbensen.[4]

Insufficient specifications and the existence of incorrect sampling errors must under all circumstances be eliminated in sampling standards as the result will unavoidably be an inconstant *sampling bias*, always and for ever out of control; it is not possible to make any bias correction regarding the sampling bias, DS 3077[1] and Esbensen & Wagner.[5] Incorrect sampling methods, room for personal interpretation and the vertical standardisation approach of CEN specifying different procedures for each material group makes sampling a complicated issue with a highly uncertain and varying validity. Any procedure and standard that has not eradicated all such potential sampling bias elements, as illustrated above, does not comply with TOS' stringent and demands for sampling correctness. The result is always a biased sampling procedure—which is always unacceptable. The full assessment of CEN/TS 14778 has been published, but so far no reaction or response has been forthcoming.[4]

Analysis of grain sampling guide

The "Home Grown Cereals Authority" (HGCA) is a division of the "Agriculture and Horticulture Development Board" based in the UK, which is mainly responsible for research and knowledge transfer in the cereal and oilseed sector. In 2013 the HGCA published a guide on grain sampling to define key requirements for effective grain sampling at various process locations from harvest to storage until departure and arrival of the grain.[6] Besides physical extraction of a grain "sample", focus is also on monitoring moisture, temperature, pests and moulds, especially mycotoxins. The described sampling practices therefore must have an obligation to contribute to ensure procedures that reliably are able to assess harvested grain quality, to protect this quality level throughout the storage phase as well as to determine the quality level after storage (before transportation to buyer) and upon arrival at the buyer. For various commodities the latter two aspects (differences in quality level at departure vs quality level at arrival) have in the past caused major law cases, often due to inappropriate or inadequate sampling procedures. Besides these kinds of discrepancies which cause serious economic disputes, extraction of representative grain samples is also crucial with regard to impurity detection (e.g. GMO quantification, toxins), as regulated by international standards (e.g. ISO 24276:2006[7]). Table 2 gives an overview of the evaluation results for the HGCA[6] with respect to potential TOS-incorrect sampling errors. The full assessment can be found in *TOS forum*.[8]

This assessment shows that most of its recommended sampling procedures and equipment (for both primary sampling and sub-sampling) do *not* lead to a representative sample. The guide's sampling procedures have a high error potential for incorrect sample delineation and extraction, which unavoidably will lead to a significantly detrimental, or even fatal sampling bias.[1] Most of the guide's recommended sampling equipment, when rated with TOS criteria, reveal major incorrect sampling errors (ISE), vastly jeopardising grain control validity.
It is noteworthy that the body responsible for the HGCA guide undertook a

Table 1. Assessment of incorrect sampling errors of CEN/TS 14778.

	IDE	IEE	IPE
Three-dimensional lot	**Sampling from stationary lot**		
	High error potential	High error potential	Medium error potential
One-dimensional lot	**Conveyer belt**		
Manual sampling (stopped conveyer belt)	High error potential	Medium error potential	Medium error potential
	Low error potential		
Automatic sampling	High error potential	High error potential	Medium error potential
	Low error potential		
One-dimensional lot	**Falling source stream**		
Manual sampling	High error potential	High error potential	Medium error potential
Automatic sampling	High error potential	High error potential	Low error potential
	Low error potential	Low error potential	

Table 2. Assessment of incorrect sampling errors of HGCA sampling guide.

Process location (HGCA)	IDE	IEE	IPE
Sampling at harvest			
Method 1: Sampling before cleaning/drying—Sampling of trailer as it is tipped into store	High error potential	High error potential	Low error potential
Method 2: Sampling after conditioning—Sampling from the cleaner/dryer outlet	High error potential	High error potential	Low error potential
Sampling in store			
Sampling spear (3–5 apertures)	High error potential	Medium error potential Low error potential	Low error potential
Sampling at outloading			
Sampling from loading bucket	High error potential	High error potential	Low error potential
Automatic bucket sampler	High error potential	High error potential	Low error potential
Sampling from spout loading Jug/Bucket Interrupter plate	High error potential Medium error potential	High error potential Medium error potential	Low error potential
Sampling from grain heap	High error potential Medium error potential	Medium error potential Low error potential	Low error potential
Sampling at commercial intakes			
Manual or automatic sampling spear	High error potential Medium error potential	Medium error potential Low error potential	Low error potential

careful response to the above critique, which was published in *TOS forum* (see box section).[9]

It is in the interest of the science of sampling to bring this kind of discussion to the attention of everybody interested in representative sampling. While the present authors of the critique of the HGCA[6] do not agree with most of the "reasons for lowering the standard w.r.t. representativity" in the rebuttal (see above), both science and industry *will* benefit from the clearly stated argumentation vs the original critique. It is, as always, up to the reader to form his/her own conclusions based on the evidence presented *pro et con*.

Sampling for GMO risk assessment

Currently an EFSA-funded project is a.o. engaged in a similar critique of all standards and norm-giving documents governing sampling for GMO risk assessment. The project reports will, after approval by EFSA, be available on the appropriate homepages within the EFSA portal.

Examples of too glib recommendations

For want of space, we end this column by showing a few examples "from undisclosed standards" of a few "recommended" sampling procedures/equipment, which would not under any circumstances find acceptance under the systematics of the Theory of Sampling, TOS (Figures 1–5).

The reader is invited to try to determine which sampling error(s) are compromised in each specific example. It is not relevant to refer to the specific standards from which the examples originate; they are shown here in complete anonymity with the sole purpose of illustrating that sampling is not a game in which anything goes… More seriously, they are examples of what can happen when committees are guided by a regimen of consensus where truly *anything goes*, as long as it is unanimously voted and agreed on… Pierre Gy often used to deliver a wry comment on this state of affairs in his lectures and courses: "With this approach a committee could vote that Newton's second law no longer applies". The few examples are a vivid illustration to this dictum—very many "recommended" sampling procedures and equipment are nothing but a showcase of not having invested the necessary effort to investigate the basics of TOS principles. But, there is always room for improvement.

Summary

There is no need for unnecessary confrontations, but there is a need for absolute clarity with respect to the responsibility carried by international (and national) standardisation authorities. There is no excuse for recommending non-compliant sampling procedures

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A critical assessment of the HGCA grain sampling guide

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HGCA's grain sampling guide is assessed with respect to the principles for representative sampling as set forward in the Theory of Sampling (TOS). Sampling correctness, which requires the elimination of all Incorrect Sampling Errors (ISE), constitutes the only guarantee for valid, representative grain quality control; presence of ISEs causes a varying, uncontrollable sampling bias that cannot be corrected for. Contrary to a first superficial observation ("grain is grain"), many different species and varieties, as well as differences caused by soil types, availability of local nutrients, make "grain" a significantly heterogeneous commodity, which requires special attention when sampled at various process locations (from harvesting, storage until commercial intake). The present appraisal shows that most of the respected HGCA grain guide's recommendations do not comply with TOS principles of sampling correctness. The suggested sampling procedures constitute major error potentials, which strongly compromise sample representativity.

Introduction

The "Home Grown Cereals Author
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impurity detection (e.g. GMO quar
toxins), as regulated by internation
ards (e.g. ISO 24276:2006).[3]

The following critical assessm
HGCA's grain sampling guide serves to evaluate whether

Dear *TOS Forum*,

Thank you for publishing the recent critique of the HGCA *Grain Sampling Guide*, which raises some interesting and thought-provoking issues for anyone involved with practical on-farm sampling.

We thought it might be helpful for your readers to explain HGCA's approach as set out in the Guide, which is focused on providing growers with a practical and cost-effective means of sampling—particularly at very busy times such as during harvest.

The methods outlined were developed to be suitable for growers in real, on-farm situations where time is constrained and resources are often limited.

The Guide was drawn up in close conjunction with the UK arable industry to reduce errors as far as practically possible and to provide growers with a realistic and basic level of information about the physical properties of their grain.

This information will help growers understand whether their grain meets contractual specifications on attributes such as moisture, protein levels, specific weight and Hagberg Falling Number.

The Guide's working assumption is that these attributes will follow a normal distribution, so the protocol is sufficient to give a basic, but useful, level of information about the farmer's crop.

In addition, grain coming from a single field can be regarded as reasonably homogenous because it is a single variety that has largely received the same agronomic management and has been exposed to the same soil and weather conditions.

This context is somewhat different to the Theory of Sampling principles to which you compare the HGCA Guide. These principles are very rigorous and are more suitable for finding contaminants present at a low inclusion rate, and is not necessarily what is required on-farm.

All the information within the guide was written to adhere to:

- BS EN ISO 24333:2009 Cereals and cereal products – sampling
- BS EN ISO 542:1990 Oilseeds – sampling

Growers and the UK grain industry will continue to work towards the common objective of providing an improved understanding of grain quality which meets both contractual and due diligence requirements.

As the UK industry moves forward, HGCA will ensure its *Grain Sampling Guide* is reviewed regularly and we will continue to look at how issues such as those raised in your article can be better reflected in our on-farm advice.

Yours sincerely

Dr Dhan Bhandari (HGCA) and Dr Ken Wildey (Technology for Growth)

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SAMPLING COLUMN

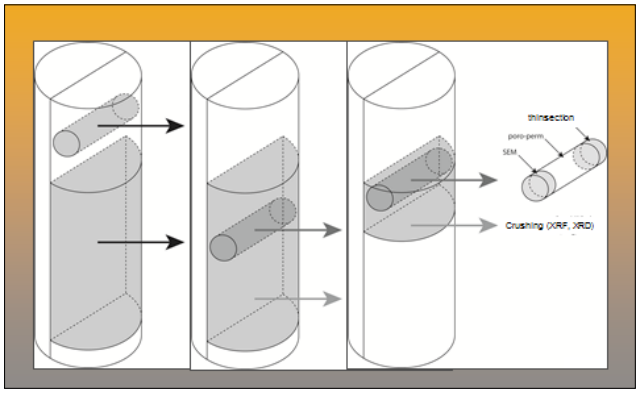

Figure 1. From the geological realm. Sub-sampling a drill core (vertical) with an aim of providing analytical samples for diverse analytical modalities [SEM, porosity and permeability measurements (poro-perm), thin section (petrological microscopy), XRF, XRD]. Although a serious attempt at creating a standardised sub-sampling scheme (left: original recommendation), there is clearly room for improvements as seen in the centre and right-most evolutions towards the smallest coeval sample volume, i.e. the maximum possible joint sample support. There are plenty of arguments from geologists to the tune: "the rocks in the drill core are pretty well homogenous, so some 15–30 cm vertical separation does not matter much… " or endless variations on this theme, which, however, completely misses the point: There is no need deliberately to create significant IDE and IEE errors in the sampling process (which is tantamount deliberately to create the fatal sampling bias). Also, homogenous materials do not exist in the real world, especially not in the poly-phase heterogeneous worlds of rocks… While there may very well be rocks of particular low vertical heterogeneity that need drilling (e.g. limestone/chalk oil reservoir rocks), setting of a standardised sub-sampling scheme for all reservoir rocks based on this scenario can only lead to significant sampling bias. Allowing for this is not the role of a standard.

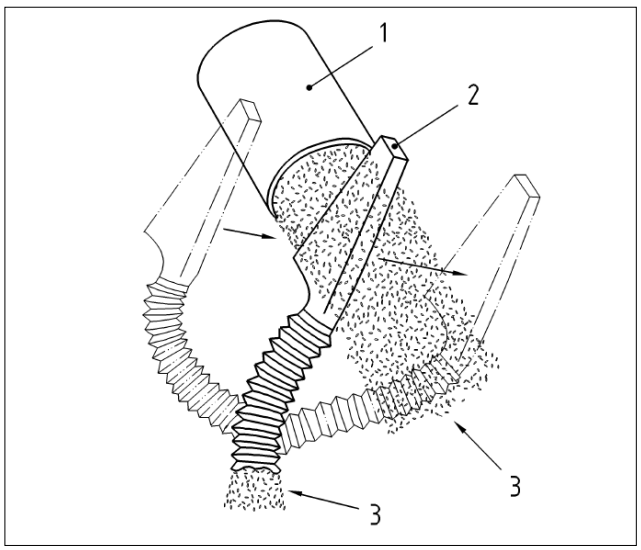

Figure 3. A recommended sampler with a much better chance of being approved by TOS—although the conditions under which this equipment is to operate are totally missing, making it open to several fundamental uncertainties.

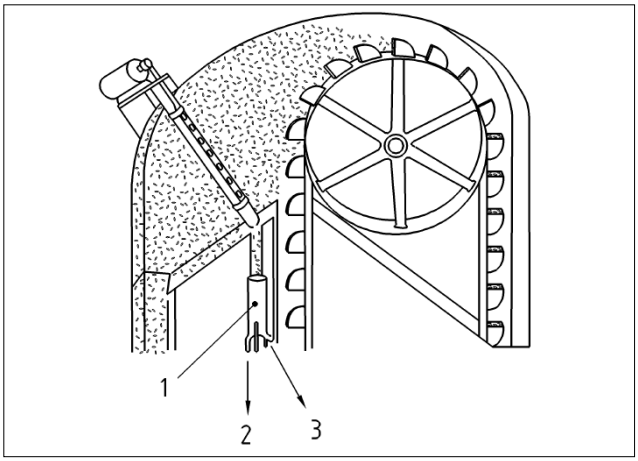

Figure 2. A particularly ill-conceived recommendation of a "grain stream sampler". When this example was used as a basis for an exam question in a PhD course on "Representative Sampling, TOS" a student wrote: "The mind boggles!".

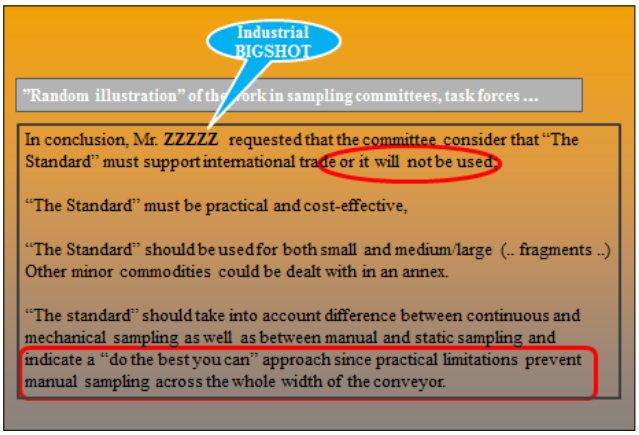

Figure 4. A potpourri of verbatim quotes from discussions in sampling committees and fora. The mind boggles at the lack of respect for representativity, while logistics, practicality and economics would appear to be the only drivers. The effect of letting such proxies dictate sampling procedures, operations and equipment alone was discussed thoroughly, and dismissed, by Esbensen *et al.*[10,11] and Minkkinen *et al.*[12]

Figure 5. Using a cylindrical coring tool for cheese sampling (left) does not allow a representative sample of the highly irregularly distributed components of a mature blue cheese. Only the two right-hand approaches will pass muster for TOS.

and equipment; the result can only be inferior sampling and inferior, indeed compromised, decision making. A chain is only a strong as its weakest link. TOS-compliance is the *missing link* in very many standards etc. There is only one remedy—get involved, get TOS literate!

There are plenty of relevant courses, lectures, consulting companies, experts on the subject matter of representative sampling, all contributing and doing a remarkable job in the last 15 years (for some up to 40 years), but none will receive specific identification here. All the reader needs is a willingness to start looking for the singular operative characteristic: representativeness—as in representative sampling and the Theory of Sampling (TOS).

Que faire?

Start here: DS 3077![1]

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