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DS 3077 Horizontal—a new standard for representative sampling. Design, history and acknowledgements

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July 2013 saw the conclusion of a five-year project, design, development and quality assurance of a new generic sampling standard: DS 3077 Horizontal. DS 3077 Horizontal is published by the Danish Standardisation Authority (DS). Development of this standard was carried out by task force DS F-205. This contribution summarises the history of this endeavour, focuses on a few salient highlights and pays tribute to the taskforce and to a group of external collaborators responsible for initial proof-of-concept and the final practical quality assurance. DS 3077 describes the minimum Theory of Sampling (TOS) competence basis upon which any sampler must rely in that sampling can be documentable as representative, both with respect to accuracy and reproducibility. It represents a consensus based on industry, academe, official regulatory bodies, professionals, students and other interested individuals.

Introduction

The primary objective behind DS 3077[1,2] was to develop a fully comprehensive, yet short, easy-to-understand introduction to the minimum principles necessary for sampling all types of materials and lots, at all scales. The overarching goal was to be able to reach absolutely all sampling novices or persons who perhaps earlier had been overwhelmed by the oft-quoted (but wrongly so) impression that the Theory of Sampling is “difficult”. This undertaking was ambitious—it took an accumulated 12 core participants in the task force a total of five years to reach a consensus and a product acceptable to all parties (industry, academe, regulatory bodies, students and professionals). Part of this work necessitated development of partially new didactic approaches, some of which are illustrated below. This contribution is only allowed to quote a few salient highlights for copyright reasons, but this is enough for an appreciation of the result achieved. The standard has benefitted significantly by valuable input from a large group of external reviewers, assessors, standard writers, sampling consultants and “users” from science, technology and industry, most of whom are thanked explicitly.

Ever since WCSB1, it has dawned upon the international sampling community that there is a serious lacuna in the arsenal with which we try to reach out to *new* communities in science, technology and industry regarding a simple, short, easy-to-understand sampling standard. Many attempts have been made but to date a truly universal standard has not yet seen the light—while very valuable achievements are on record regarding sampling standards with a restricted target, e.g. commodities, major raw materials, manufactured goods etc. These are highly significant such achievements, all of which have also served as inspiration for the present work regarding DS 3077. Setting the scene can best be done with a few selected quotes (indicated by the blue text), brought here with permission from the Danish Standardisation Authority, the publisher of DS 3077.

DS 3077 foreword

DS 3077 outlines a practical, self-controlling approach for representative sampling with minimal complexity, based on the Theory of Sampling (TOS). The generic sampling process described and all elements involved are necessary and sufficient for the stated objective, in order to be able to document sampling representativity under the conditions specified. It is always necessary to consider the full pathway from primary sampling to analytical results in order to be able to guarantee a reliable and valid analytical outcome. This standard, including normative and related references, annexes and further, optional references constitute a complete competence basis for this purpose. The present approach will ensure appropriate levels of accuracy and precision for both primary sampling as well as for all sub-sampling procedures and mass-reduction systems at the subsequent laboratory stages before analysis.

A sampling process needs to be structurally correct in order for the essential accuracy requirement to be fulfilled, with no exceptions allowed. For the process also to be sufficiently precise it is often necessary to proceed through iterative stages, until the effective sampling variance has been brought below an *a priori* given threshold; this is also known as ‘fit-for-purpose’. In this endeavour the key feature is the heterogeneity of the target lot, which shall be identified and quantified. Heterogeneity characterisation forms one key element of the present standard. Only when both the accuracy and precision demands have been met properly, can all types of solid lots and two-phase (solid–liquid) materials be sampled representatively (gasses are excluded from the present standard), and the derived quality assurance of the sampling process is hereby subject to open public inspection. Without informed commitment to such an empirical heterogeneity characterization, all prospects of being able to document representativity will remain out of reach.

This standard outlines a systematic scientific basis for improving sampling procedures, which will lead to increased reliability for decision-making based on measurement results. Not all existing standards are in compliance with the appropriate TOS requirements, although partial elements can be found in many places (2.1 and Bibliography). Relationships to other standards, guidelines, good practices as well as regulatory and legal requirements shall be handled with insight. Where found in opposition to other, less TOS-compliant 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. While this is taking place, or when dictated by documented sampling variances that are too high (a key issue in the present standard), it is always an option to employ more stringent quality criteria from a TOS-based approach, than what may be presently codified. As there are serious economic and societal consequences of non-representative sampling, these are appropriately described and illustrated in this standard, which also outlines impacts caused by inferior analytical results and related non-reliable decision making.

DS 3077 has the overall objective to establish 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.

Scope

DS 3077 is based exclusively on the Theory of Sampling (TOS).

DS 3077 is a matrix-independent standard for representative sampling. Compliance with the principles herein ensures that a specific sampling method (procedure) is representative.

DS 3077 sets out a minimum competence basis for reliable planning, performance and assessment of existing, or new sampling procedures with respect to representativity.

DS 3077 invalidates grab sampling and other incorrect sampling operations, by requiring conformance with a universal set of seven governing principles and unit operations.

DS 3077 specifies two simple quality assurance measures regarding:

- Sampling of stationary lots, the Relative Sampling Variability test (RSV)
- Sampling of dynamic lots, Variographic Analysis (VA), also known as variographic characterisation, with an analogous RSV$_{1\text{-dim}}$. [DS 3077 contains a variographic software program (freeware) making simple variographic characterisation available to all readers]

DS 3077 stipulates maximum threshold levels for both these quality assurance measures.

DS 3077 enforces professional self-control by stipulating mandatory disclosure of one of two comprehensive quality assurance approaches as produced by RSV or variographic characterisation to all parties involved.

DS 3077 specifies documentation and reporting of sampling representativity and efficiency for each analyte in combination with a specific class of materials respectively. Any deviation from this standard's quality objectives (QO) shall be justified and reported.

DS 3077 employs a dual acceptance approach: items not mentioned are not acceptable as modifications in any sampling procedure or sampling plan, unless specifically tested and assessed by the QO's described herein—while all modifications successfully passing this test requirement are acceptable.

We can only bring you a small quotation from clause 3 "definitions and terms"; it will suffice here to concentrate on the didactic presentation which has been developed in order to comply with the aspirations re. a "short, simple, easy-to-understand ..." standard.

3.11 grab sample

increment resulting from a single sampling operation (literally "grabbing"), almost always emphasizing alleged efficiency, inexpensiveness, effort-minimizing desirability. (Figure 1).

Note: Grab sampling can result in representative samples only in the rarest of instances. If a grab sampling procedure is contemplated, it is mandatory to test and document it by one of the two heterogeneity characterization methods in DS 3077, RSV or variographic characterization.

Grab sampling constitutes the world's most misused sampling operation. All single-sample approaches for heterogeneous materials are in conflict with the Fundamental Sampling Principle (FSP) and militate against the necessary heterogeneity counteraction.

Note: Grab sampling is applicable at all sampling scales, from the field, in the industrial plant to the analytical laboratory, but fails totally to comply with the fundamental sampling principle. DS 3077 mandates composite sampling for all situations in which grab sampling has not been approved by a pertinent validation, either RSV or by variographic analysis."

3.6 composite sample

sample made up of a number, Q, of increments (Figure 2)

Note 1: The ISO equivalent of a composite sample is the bulk sample. There is full conceptual consistency between the definition of composite (TOS) and bulk sample (ISO), but a composite sample shall either be representative or not, according

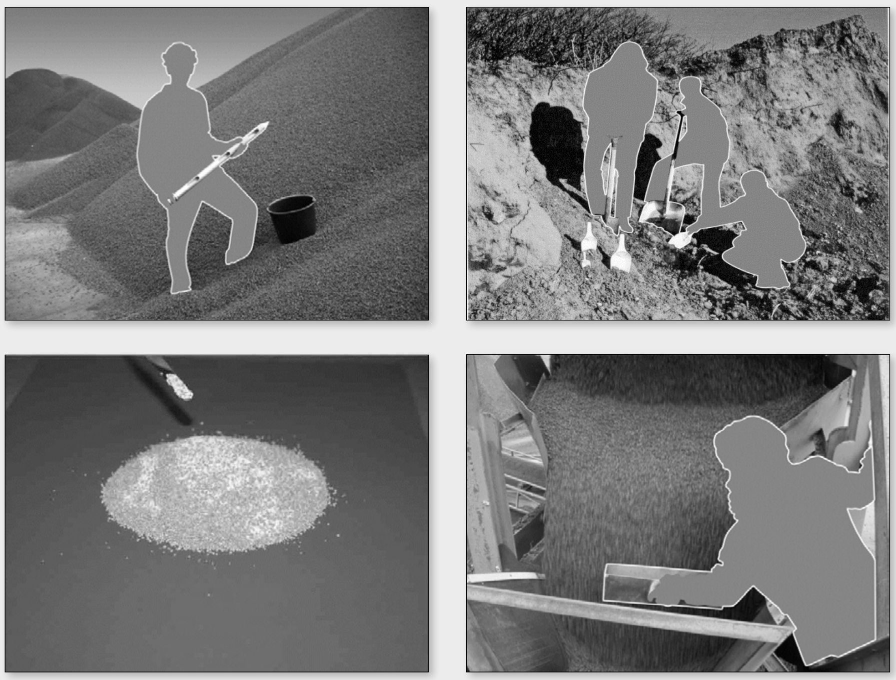

Figure 1. Grab sampling illustration across all scales of interest (from macroscopic stacks to powder piles) for both stationary and dynamic lots. The possibility for any single-increment extraction operation to achieve representativity is virtually zero since the lot cannot be covered with respect to its intrinsic spatial heterogeneity (DH).

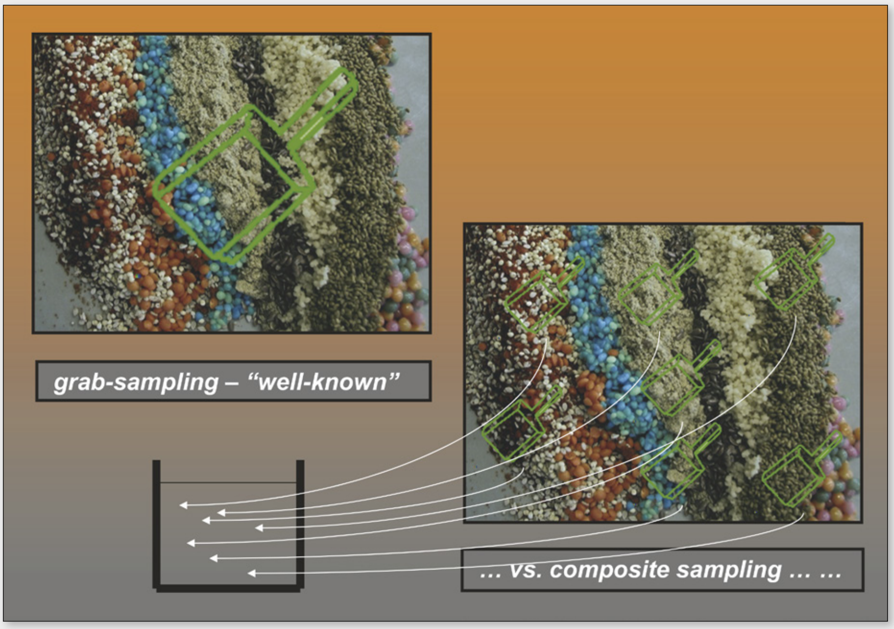

Figure 2. Composite sampling of significantly heterogeneous material. Irrespective of scale, a composite sample (Q increments) is able to "cover" the spatial material/lot heterogeneity far better than a sample originating from a single extraction operation (grab sampling).

to the characteristics of how its increments were extracted, a distinction only made in TOS.

Note 2: The primary purpose of composite sampling is to cover spatial and/or compositional heterogeneity of the lot as best possible subject to given logistical and practical conditions and a specific sampling procedure. The same sampling tool (e.g. scoop) can be used significantly better as a provider of a composite sample than when used for grab sampling (single sample operation). In principle, and in practice, informed and competent use of composite sampling will result in a considerably reduced sampling variance (TSE) compared to grab sampling; the average will in general also lie closer to the true lot composition for composite sampling.

Note 3: Composite sampling can also be used for more local purposes, i.e. for minimizing the effect of local heterogeneity (segregation or otherwise) of a single localized sample - for example when expressing or modeling concentration changes in 1-D, 2-D or 3-D geometrical contexts, e.g. trend surface analysis."

3.40 theory of sampling, TOS

a body of theoretical work starting in 1950 by the French scientist Pierre Gy, who over a period of 25 years developed a complete theory of heterogeneity, sampling procedures and sampling equipment assessment (design principles, operation and maintenance requirements). TOS was subsequently further elaborated into a coherent didactic framework in the next 25 years by Gy, as well as also added to by newer generations especially in the last two decades. Gy's personal account of TOS and its development history can be found in the note reference immediately below.

NOTE Pierre Gy has published c. 275 papers and seven books on sampling, in later years joined by several other international sampling experts (Pitard, Bongarcon, Minkkinen, Holmes, Lymann, Smith, Carrasco). A tribute to Pierre Gy's scientific *oeuvre* can be found in the reference below.[3]

TOS, synoptic overview

The figure below (Figure 3) shows a didactic flow path of relationships between sampling stages, sampling errors, four practical sampling unit operations (SUO) and three Governing Principles (GP).

Empirical heterogeneity testing, RSV (heterogeneity characterisation) is universally applicable, both for the total sampling process as well as for specific sampling stages. Process sampling relies on variographic analysis (VA) for heterogeneity characterization, sample mass (composite sampling, Q) and sampling rate optimization. There are two additional sampling errors especially related to process sampling (trend process sampling error; cyclic process sampling error), which can be brought under control relatively easily. Within the framework of this standard, sampling from either stationary or dynamic lots, covers a necessary basis with which to address very nearly all sampling issues...

Freeware; Variogram

DS 3077 Horizontal contains an appendix which is comprised by a stand-alone software package, designed to be able to perform basic variographic data analysis for an entry of up to 100 measurements. This software calculates a relative variogram on the basis of user input (two spreadsheet columns: concentration, increment weight—if no weight is assigned, the software assumes identical weights for all increments arbitrarily set to 1.00). Variogram calculation is the only option, indeed the only task included. This freeware is in no way

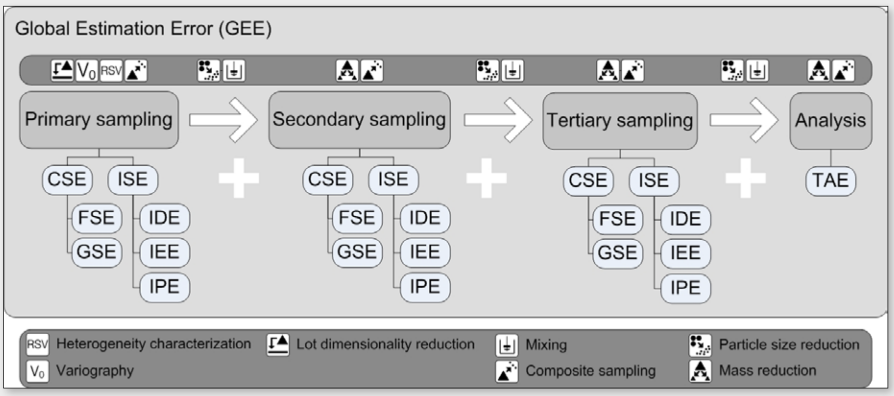

Figure 3. A minimum sampling competence encompasses FSP, TOS' paradigm of sampling correctness, five sampling errors (CSE/ISE) and four Sampling Unit Operations (SUO).

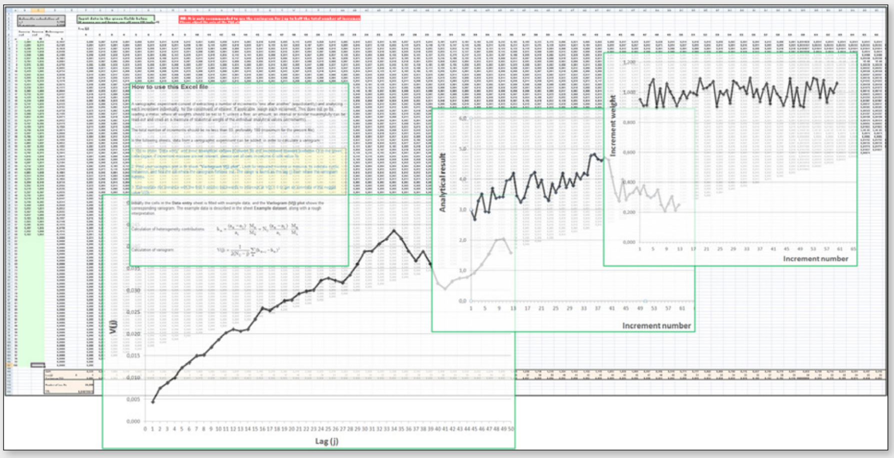

Figure 4. DS software VARIOGRAM (Appendix): Brief program manual, input spreadsheet, plotting routines for concentration and weight values and the resulting variogram (bottom). This software is designed for an input of up to 100 entries.

intended as a competitor to existing professional and commercial variographic software programs or packages on the market, all of which perform several more essential functions for in-depth usage, e.g. decomposition of variance components originating from periodicity and trends, estimation of TSE. The role of the freeware appendix is solely to allow standard readers an initial familiarisation with variographic modelling.

The VARIOGRAM freeware (Figure 4) was programmed by the second author of the present report (LPJ).

Discussion and conclusion

DS 3077 Representative Sampling—Horizontal has been discussed at innumerable occasions in the period since its gestation (which covers all the 10 first years of the existence of the WCSB conferences), where it was unanimously concluded that there is a serious need for such a standard. There is no doubt that the present ver. 1.0 is but the beginning on a new journey. As any other international standard it will be subject to regular revision in agreement with the pertinent stipulations (CEN/ISO). It is hoped that many will feel compelled to contribute towards its continuing development and improvement. DS 3077 Representative Sampling—Horizontal represents an intense five-year taskforce project, solely guided by the prospect of being able to contribute towards better teaching and dissemination of the Theory of Sampling (TOS). It represents a consensus based on industry, academe, official regulatory bodies, professionals, students and other interested individuals.

Attribution

The core taskforce behind DS 3077 (DS F-205) consisted of the following members: KHE (chairman), LPJ, Hans S. Møller, Christian Riber, Anders Larsen, Martin Thau, Jette Bjerre Hansen, Lars K. Gram, Jørgen G. Hansen and Bodil Mose Pedersen. Merete Westergaard Bennick and Lone Skjerning served as able secretaries. DS 3077 benefitted significantly by valuable corrective and additional input from a large group of external reviewers, assessors, standard writers, sampling consultants and "users" from science, technology and industry. The following individuals are gratefully acknowledged for their major contributions in this work—but are in no way responsible for perceived errors, omissions or declarative issues in the standard: Francis Pitard, Ralph Holmes, Pentti Minkkinen, Claudia Paoletti, Anna de Juan, Kaj Heydorn, Ulla Oxenboll Lund, Loren Mark, Melissa Gouws, Claas Wagner, Peter Thy, Peter C. Toft, Anders Larsen, Henri Sans and Mark O'Dwyer. In addition to this attribute, the first author would like to express his sincere gratitude to a core group of colleagues: **Hans S. Møller, Francis Pitard, Christian Riber, Pentti Minkkinen and Claudia Paoletti** for their steadfast personal support in this endeavour from the very beginning. It is equally pleasing to acknowledge PhD fellow **Claas Wagner** for extraordinary inspiration during the last three years of this work.

THANK YOU PROFOUNDLY ALL for contributing to the ever-increasing dissemination of TOS.

Acknowledgements

Tracing illustrations in Figure 1 are included with permission from the future publisher Wiley-VCH, to appear in Esbensen & Minkkinen: *Representative Sampling—In Science, Technology and Industry* (publication expected in 2014). Quotes from DS 3077 are reproduced with permission of Danish Standards.

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Historical snapshot from the inaugural meeting of taskforce F-205 (Danish Standards), 2008. From the right: Christian Riber, Merete Westergaard Bennick, Lars Petersen Julius, Jan Hinnerskov Jensen, Martin Thau, Lars K. Gram, Jette Bjerre Hansen, Kirsten Jebjerg Andersen. Photo: chairman KHE.