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MEETING REPORTS

Uncertainty from sampling: workshop to launch a Nordtest handbook on sampling uncertainty estimation and control

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Abstract A workshop on uncertainty in sampling was held in Hillerød, Denmark, on 12–13 April 2007 to launch a new handbook on sampling quality assurance and uncertainty estimation. The participants of the workshop were approximately 60 delegates from 15 European countries, representing institutions performing sampling, users of the data, research institutions, as well as accreditation bodies. Materials from the workshop, including examples, tools, and calculation aids for the work can be found at http://www.samplersguide.com. The Nordtest handbook *Uncertainty from sampling* will be made available on the Nordtest web site at http://www.nordicinnovation.net/nordtest.cfm under NT technical reports, report number NT tec 604. Until the final report is available on the Nordtest web site, an advance draft of the Nordtest handbook is available from http://www.samplersguide.com.

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Introduction

The main purpose of most measurements is to enable decisions to be made. The credibility of these decisions depends on knowledge about the uncertainty of the measurement results. Uncertainty in measurement can be defined as being made up of two components: uncertainty derived from sampling a target and the uncertainty derived from the analytical process. If the uncertainty of measurements is underestimated, for example, because the sampling is not taken into account, then erroneous decisions may be made that can have large financial, health, and environmental consequences. For this reason, it is essential that effective procedures are available for estimating the uncertainties arising from all parts of the measurement process. These must include uncertainties arising from any relevant sampling and physical preparation, as well as variability arising from material heterogeneity within the sampling target.

The Nordtest[1] handbook was developed in parallel with a similar guideline prepared by Eurachem and several other organizations [1]. This Eurachem document is the master document, whereas the Nordtest handbook [2] is shorter and is intended for practical applications of the principles. This workshop was held to launch the Nordtest handbook

[1] Nordtest is part of the Nordic Innovation Centre (NICe) under the Nordic Council of Ministers, who initiates and finances activities that enhance innovation collaboration and develop and maintain a smoothly functioning market in the Nordic region. Measurement quality plays an important role for Nordtest.

and a similar conference will be held to launch the Eurachem guide in April 2008 in Berlin, Germany.

The overall objective of the workshop was to present a set of tools for the calculation and control of the uncertainty arising from sampling and to make these tools available and understandable, outside as well as inside the world of analytical chemistry.

The workshop was intended for those involved in the planning or the evaluation of sampling for monitoring or control programmes and for those evaluating data that might have been affected by the sampling step. In other words, the programme was designed for people involved with sampling from several different perspectives.

Estimating sampling contribution to measurement uncertainty

In order to estimate the measurement uncertainty, the measurand, *the quantity intended to be measured* [3], has to be properly specified. The specification of a measurand requires knowledge of the kind of quantity (e.g., the mass fraction of total iron in iron ore) and a description of the substance carrying the quantity, the sampling target (e.g., a produced batch of material). In the Nordtest handbook, the sampling target is defined as the *portion of material, at a particular time, that the sample is intended to represent.* The sampling uncertainty is *the part of the total measurement uncertainty attributable to sampling* (IUPAC 2005) [4]. It is important to properly define the sampling target, including its location in space and time. If, for example, there are time variations, different sampling targets are possible, for example, contaminant concentration at a factory outlet at the time of sampling, or the average contaminant concentration at the outlet over a year. An overview of a typical measurement including sampling, sample preparation, and analysis is given in Fig. 1.

For a measurement according to Fig. 1, the expanded measurement uncertainty, U, for a result referring to the sampling target can be calculated in most cases according to the following equation:

$$U_{\text{measurement}} = 2 \cdot \sqrt{u^2_{\text{sampling}} + u^2_{\text{analysis}}}$$

where u_{sampling} is the sampling standard uncertainty and u_{analysis} is the analytical standard uncertainty. The organization responsible for the sampling should be able to estimate the uncertainty of sampling, just as the organization responsible for analysis, the laboratory, should be able to estimate the analytical uncertainty. However, in many cases, this is a complex process, since we cannot estimate sampling uncertainty without performing any analysis. It may also be that the composition of the samples under investigation is outside the normal range considered by the laboratory, which means that the prior estimate of analytical uncertainty may have to be revised. Therefore, a close cooperation between samplers and analysts is the best way to obtain a reliable estimate of the measurement uncertainty for the actual sampling target.

Workshop on 12–13 April 2007

The workshop took place in Hillerød, Denmark, at the Pharmakon Conference Centre and about 60 participants from 15 different European countries participated. The participants were representatives from national environmental agencies (e.g., the Latvian Environmental Geology and Meteorology Agency and the Flemish Environmental Agency), accreditation bodies (e.g., DANAK, SWEDAC, and LATAK), research and educational institutions, as well as public and private enterprises, such as Vodovod-Kanalizacija Public Utilities, Ljubljana (a public enterprise for water and waste water from Slovenia).

Workshop content: first day

The workshop presentations started with a general introduction to measurement, sampling, and analytical uncertainty. Methods for estimating the uncertainty were listed and discussed. Emphasis was placed on empirical, rather than modeling methods. Examples of one empirical method, the duplicate design, were presented more thoroughly and considerations were made to the interpretation and fitness for purpose of the estimations.

After an introduction to the Nordtest handbook, the main general points in the handbook were highlighted:

- Design of sampling program, including sampling purpose, sampling target, and quality objectives
- Uncertainty in measurements—sources and types of uncertainty
- Principles of quality assurance in sampling—validation and quality control

In the Nordtest handbook, examples of the estimation of uncertainty are given that use an empirical approach to the sampling design and the calculations. In the workshop, the following examples were presented:

- *Ground water monitoring*. In this example, the quality objectives and quality control was highlighted. Uncertainty was estimated using range statistics.
- *Industrial production of iron ore*. This example looked at the production of iron ore pellets and again used range statistics for the estimation of sampling uncertainty.

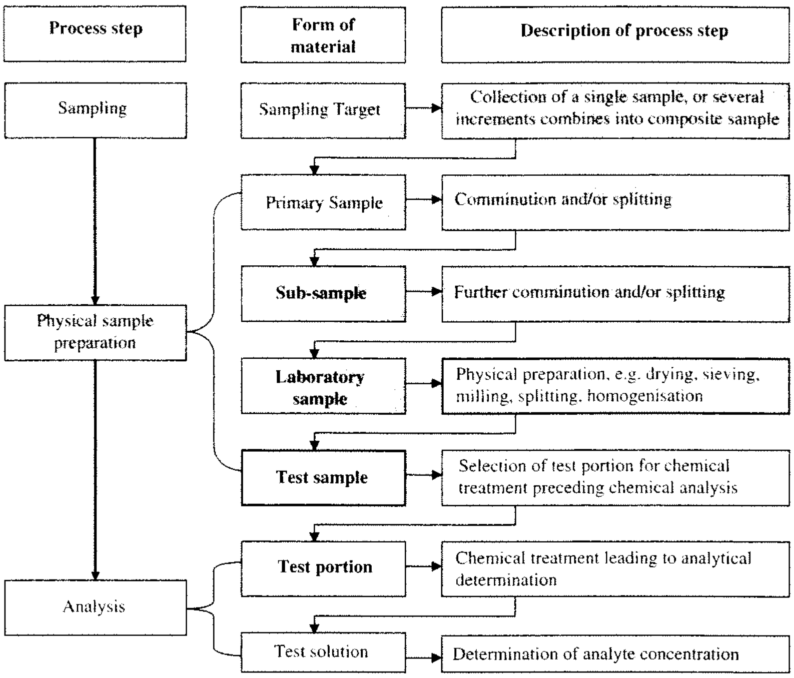

Fig. 1 Schematic diagram of a typical measurement process including sampling, physical sample preparation (including transport), and analysis [1]. The analytical steps are *white* and the sampling and physical sample preparation steps are *shaded*

- *Food sector.* This example demonstrated the estimation of measurement uncertainty of vitamin A in baby porridge using analysis of variance (ANOVA). This example was a case where it was possible to estimate both the random as well as the systematic contribution to uncertainty. Quality control of measurement uncertainty, including the sampling, was described, in addition to the initial validation.
- *Waste water.* This example demonstrated the use of variography as a tool for determining variations in time or space within the sampling target (presented on the second day).

Workshop content: second day

The second day of the workshop consisted partly of presentations and partly of group work, in which the participants had a chance to work with their own data or data provided by the organizers. Two presentations were given where the sampling uncertainty is the dominant source of uncertainty—one concerning the sampling of waste water using variography and another concerning the sampling of contaminated soil (e.g., contaminated land). For contaminated soil, it was shown that the evaluation of the contamination level of the site was significantly different when sampling uncertainty was considered. The calculation tool used was robust ANOVA, and a calculation program to achieve this, called ROBAN, was demonstrated.

The group work was one of the last activities of the workshop. The aim of the group work was to give the participants a chance to get "hands on" experience of the tools presented before leaving the workshop. The participants were divided into four groups working with different sampling objectives and calculation tools as summarized below:

- Sampling of fresh water from a canal—a sampler proficiency test. The aim of the sampling proficiency test (SPT) for trace elements determination was to determine the sampling uncertainty by analyzing different samples from a sampling target taken by different persons using different equipment.
- Sampling of baby porridge. The aim was to estimate the measurement uncertainty and the contributions from both sampling and from analyses in the determination of vitamin A. The uncertainty was calculated using ANOVA.
- Sampling of ground water. The aim was the determination of the measurement uncertainty for dissolved iron in a sampling validation study and the subsequent control of sampling uncertainty during monitoring. The uncertainty was calculated using range statistics.
- Sampling of waste water. The data were evaluated using variographic analysis. The aim was to estimate the measurement uncertainty as well as individual uncertainty contributions from inherent heterogeneity, the automatic sampling, the pre-treatment, and from the analyses of waste water in the measurement of electrical conductivity.

The participants provided feedback indicating that they were generally satisfied with the outcome of the group work. It is, however, difficult to satisfy all of the participants because of their very variable background knowledge and expectations on this subject.

Important outcomes

The important outcomes of the workshop were an increased appreciation by the participants on several issues, including:

- Written guidance, and supporting software, is available on the methodologies that can be used to estimate the contribution of measurement uncertainty arising from sampling, from both Eurachem and Nordtest
- Estimates of measurement uncertainty should include the contributions from the primary sampling and physical sample preparation, unless specifically precluded by a definition of the measurand as based solely upon the laboratory sample
- The most appropriate method of those available for the evaluation of uncertainty from sampling can be selected by careful consideration of their relative suitability for a particular sampling target, as described in the guidance
- Practical experience of estimating uncertainty from sampling, and the experimental design required to achieve it, are essential to provide a reliable evaluation of measurement uncertainty
- Knowledge of reliable estimates of measurement uncertainty, from all of its sources, gives rise to:
  - Improved reliability of decisions that are based upon measurements
  - Rational and transparent means of allocating appropriate funding to the measurement process overall, and also in the subsequent division of funding between sampling and chemical analysis
  - A new tool for estimating and monitoring the quality of field sampling, and the physical preparation of samples

How to continue this work

In the laboratory sector, the implementation of analytical uncertainty evaluation based upon method validation and quality control was driven by requirements set as part of the laboratory accreditation according to ISO 17025. These requirements cannot be expected to have the same impact with sampling for several reasons:

- Samplers that do not work for laboratories (and, thus, are not accredited) dominate sampling in several sectors
- Accredited sampling is only required in very few regulations, national as well as European
- The quality assurance requirements set by accreditation bodies for the accreditation of sampling is, in most cases, far less stringent than for analysis

Conversely, the increasing demand for developing compliance rules in, e.g., environmental control based upon probabilistic principles sets a requirement for knowing the measurement uncertainty including that from sampling, as well as the material heterogeneity before allowing this. In order make this information available, two major routes seem to be open:

- For commercial purposes (e.g., the control of product quality), the cost efficiency of uncertainty based decisions need to be demonstrated in order to "pave the road" for a more common use of uncertainty evaluation and control
- For regulatory purposes (e.g., monitoring in accordance to the European Water Framework Directive), the way forward is to set regulatory requirements for the estimation and consideration of the sampling uncertainty

A number of activities were suggested in order to facilitate the implementation of the concepts of sampling uncertainty in legislation:

- Derivation of a popular style Eurachem guide presentation (two-page leaflet)
- Development and presentation of more practical examples (demonstration) with detailed documentation
- Workshop/courses for regulators introducing the concepts and their benefits
- Support to the European Commission in the process of developing guidelines for the implementation of EU directives including, where relevant, uncertainty concepts

Finally, there is a need for the further development of tools for estimating and controling the contribution of systematic errors to sampling uncertainty. The most obvious approach would be to develop sampling proficiency tests equivalent to the analysis proficiency tests that are readily available.

This workshop was the first of its kind held at a European level, but it is to be expected that it has laid the foundation for many further workshops. These could be on the dissemination of these existing methodologies, but also on new research that improves these methods, compares their effectiveness, and considers the benefits that are gained by the users of measurements by having this new information.

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