amc technical brief

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cal Methods Committee

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Terminology – the key to understanding analytical science. Part 2¹: Sampling and sample preparation

Sampling is a topic where analytical chemists have been accustomed to use words rather loosely, which is fine in conversation but can lead to confusion in written work. In particular, we tend to use the word "sample" itself incorrectly: it is confusingly used to signify many quite different entities. The following is a list of key words from sampling, some with established meanings, and some where the meaning is in the process of definition. Only generally applicable words are listed here: synonyms used in specific application areas are omitted.

What is a sample?

Sample: portion of material selected from a larger quantity of material^{2,3}.

Representative sample: sample resulting from a sampling plan that can be expected to reflect adequately the properties of interest in the parent population.^{2, 3}

Sampling target: portion of material, at a particular time, that the sample is intended to represent.

Primary sample: the collection of one or more increments or units initially taken from a population.²

Increment: individual portion of material collected by a single operation of a sampling device.²

Composite sample: two or more increments/subsamples mixed together in appropriate proportions, either discretely or continuously (blended composite sample), from which the average value of a desired characteristic may be obtained.³

Sub-sample: portion of a primary sample obtained by splitting.

Laboratory sample: sample or sub-sample sent to or received by the laboratory.²

Test sample: sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis.²

Test portion: quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample²

AMC comments

The hierarchy of sample types is illustrated overleaf (Figure 1). **Sampling Target** is becoming established because of a requirement for a general term to indicate the whole of such entities as: consignments or lots of commodities; batches of manufactured or processed materials; crops or topsoil in a particular field (or at an exact point in a field); a particular metal ingot; or the particulate matter in a specified volume of air during a specified time interval.

A **representative sample** is meant to reflect the composition of the target adequately, but not exactly. Samples from a heterogeneous target will never be identical in composition, and the variation in composition gives rise to (usually) the greater part of the uncertainty from sampling. So "adequate reflection" mean that the uncertainty from sampling is fit for purpose. A **test portion** is the part of the test sample that is taken for digestion and chemical analysis, by procedures specified in the analytical method

Sampling plan etc

Sampling plan: predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample^{2, 3}.

Sampling procedure (or **protocol**): operational requirements and/or instructions relating to the use of a particular sampling plan³ (*i.e.*, the instructions for the implementation of the plan)

Terms relating to sampling quality

Replicate (duplicate) sample: one of two or more samples obtained separately by the same sampling procedure from the same target. **Sampling precision:** the part of the total measurement precision attributable to the sampling.

Sampling bias: the part of the total measurement bias attributable to the sampling.

Sampling uncertainty: the part of the total measurement uncertainty attributable to the sampling¹

Random sample: sample taken so that all equalsize sectors of the target have equal probability of being selected to form the sample.

AMC comments

There are two independent sources of uncertainty in the result of a measurement, namely the sampling procedure and the analytical procedure⁴. Sampling uncertainty can usefully be considered as one component of the measurement uncertainty, with contributions from both random and systematic errors. Sampling precision quantifies the random errors arising from the sampling method. It is quantified as its converse, sampling standard deviation (or variance), which indicates the magnitude of the random errors introduced by (a) the heterogeneity of the target and (b) variation in the execution of the sampling plan. Sampling bias gives an estimate of the systematic error introduced by the execution of the sampling plan. This systematic error might result from (a) failure to address the target exactly, (b) failure to execute the protocol exactly, and (c) misinterpretation of the protocol. Sampling bias is difficult to detect, and techniques for its estimation (such as a reference sampling target) are still in their infancy. Only a random sample can be guaranteed to give a sampling plan that is unbiased. However, in many circumstances other types of sample plan will be almost as good, but the execution of any plan may introduce bias inadvertently. The definition of replicate (duplicate) sample has been modified from the ISO definition³ to remove inconsistencies and unnecessary restrictions.

Finally...

Perhaps one of the most important messages in this document is that we should avoid using the word "sample" when we mean any of the following: "test portion", "test material", "analyte", "aliquot", "test solution", "matrix", etc.

References

- 1. AMC Technical Brief No 13.
- 2. W, Horwitz. *Pure Appl.Chem*, 1990, **62**, 1193-1208.
- 3. ISO11074-2: 1998. (ISO), Geneva.
- 4. AMC Background Paper No 1

This Technical Brief was prepared for the Analytical Methods Committee by the Subcommittee for Sampling Uncertainty and Quality (Chairman M H Ramsey), which is supported by the Food Standards Agency.

The Subcommittee for Sampling Uncertainty and Quality:

- encourages analysts to include primary sampling in uncertainty budgets for the whole measurement process;
- provides information on methods for the evaluation of the uncertainty associated with sampling protocols;
- assesses existing procedures of evaluating methods of sampling and sample preparation, and encourage the development of improved procedures;
- encourages best-practise in ensuring sampling quality that is fit-for-purpose;
- ensures international applicability of guidance from the Sub-committee by collaboration with appropriate organisations;
- clarifies who needs to be responsible for the quality of sampling, and enable their effective communication with the analysts.



Figure 1. Key terminology of sampling, together with typical activities involved in producing the hierarchy of sample types. In many circumstances some or all of the intermediate sample types and their corresponding activities may be omitted or combined.