8.3 The measurement procedure

This section describes aspects of the measurement procedure which need to be considered in advance of actually making any measurements. Indeed, it is generally advantageous if this consideration starts well before any samples or test pieces are even taken. Factors to be taken into account whilst measurements are in hand are described in the following section.

It is important that the reasons for undertaking a measurement are clearly understood so that the measurement procedure can be properly planned. Good **planning** is vital in order to produce reliable data to time and to cost. The planning process should cover aspects such as the objectives of the measurement, sampling, selection of the method, the required level of accuracy and confidence, quality control requirements, and reporting.

Sampling is often considered to be outside the control of the measurement scientist but is frequently the major factor in determining whether the results of a measurement are fit for their purpose. Those responsible for the data must, therefore, make every effort to involve the measurement scientist in preparation of a sampling plan. Knowledge of the potential sampling error is also vital in selecting the measurement method; if the sampling error forms a high proportion of the total error it is futile to attempt to improve matters by further reducing the measurement error. Preparation of an effective sampling plan is essential if reliable data are to be obtained, yet in many areas of measurement this necessity is often overlooked. It is not uncommon for sampling to be undertaken on an *ad hoc* basis by unskilled staff with little if any knowledge of the purpose or the requirements of the measurement methodology. At the other extreme there are legal and statutory requirements governing sampling schemes for a wide variety of materials. Where such schemes do not exist the sampling plan should be based on two key elements: a valid statistical approach and adherence to good sampling practice. Statistical sampling theory underpins the methods used to select samples from a consignment. Common selection methods include probability sampling, non-probability sampling, bulk sampling and acceptance sampling. Good sampling practice is not a standard technique but embraces a number of generally recognised principles, for example:

visual inspection before sampling;
use of appropriate containers;
use of adequate precautions such as preservatives or special packing;

provision of suitable, safe and secure storage facilities;
maintenance of sample integrity;
provision of appropriate documentation.

Careful **selection of the measurement method** is essential if reliable data are to be obtained. A key factor in this process is determining whether the method is capable of providing a sufficiently reliable result, allowing for cost or other constraints such as time and safety considerations which may apply. There is no simple, established approach for doing this; it frequently comes down to the application of expert knowledge and judgement by the measurement scientist. The amount of work required to select the method varies widely. In some cases use of an existing, specific method may be dictated by company policy, by the customer, or by a regulatory body. At the other extreme an extensive experimental programme may be needed to develop a novel method. More generally, it will be necessary to seek guidance from previous work, from colleagues, or from the published literature and to carry out a modest experimental programme. In doing this it is advantageous to follow a few simple rules:

use familiar methodology rather than something completely new;
use the easiest method which meets requirements (i.e. which does not involve especially difficult manipulations or instrument adjustments);
use the simplest possible method (i.e. one which is not unnecessarily complicated and uses the smallest number of operations);
give preference to methods which have been tested or adopted by major national or international bodies or for which published validation data are available from a number of organisations.

Regardless of the amount of time and effort expended on method selection and/or development, it is essential to undertake formal method **validation** before using the method. Validation is the process of determining whether the chosen method can provide data meeting the measurement specification. It is essential that an organisation making measurements demonstrates satisfactory performance with the method even when extensive validation data have been produced by a major, external study. The effort required will depend on the extent of existing validation data, the relevant experience of the measurement scientist, and the degree of confidence required from the measurements. Common approaches include:

measurement of artificially generated signals or synthetic formulations;
comparison of results with those obtained from one or more established procedures which depend on different physical principles;
measurement of certified reference materials or standards.

This approach to method validation does not guarantee that the method is free from error; its aim is to confirm that the method is adequate to meet the agreed specification for the measurement. As such, method validation should address a range of relevant performance characteristics as well as bias and precision. These characteristics will vary with the type of measurement but include range, linearity, limit of detection or determination, repeatability, reproducibility, robustness (i.e. sensitivity to variations in the procedure, instruments, reagents, etc.) and selectivity. A particularly important, and useful, parameter is the measurement **uncertainty**. This is the range within which the true value is believed to lie with a specified (usually high) probability. As such it is an estimated quantity rather than a precisely measured factor. The difficulty of estimating uncertainty varies considerably between areas of measurement. It is commonly used for physical measurements but less so in the area of analytical

chemistry. Nevertheless, an effort commensurate with the purpose and value of the measurement should always be made, aiming to achieve a realistic estimate of the size of the uncertainty and to identify its contributing factors.

An important aspect of setting up any new measurement method within a quality system is the establishment of a well-defined **quality control** (QC) regime. This comprises a systematic series of checks to monitor day-to-day and batch-to-batch variation in performance. It is best achieved by periodic remeasurement of check specimens which resemble as closely as possible the test pieces or samples. These specimens need to be stable and available in sufficient quantity for measurements over an extended period of time. Good practice normally dictates the use of several specimens, some giving a similar response to the test pieces and others with zero or very low response (blank determinations). The level and type of QC must be agreed by the responsible scientist or engineer and be clearly defined in a quality manual or other documentation. It will depend on factors such as the nature and frequency of the work, the importance of the data, the reliability and robustness of the method, and the degree of automation. The data obtained from QC specimens should be archived with the relevant test data and also subjected to appropriate statistical analysis. The latter, such as the use of control charts, is designed to highlight deviations of the method from the specification. An essential element of every QC regime is the existence of clear, mandatory instructions setting out the steps to be taken when such deviations are detected.

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