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8.4 The measurement

This section describes the quality assurance principles which should be applied during day-to-day work, i.e. once samples or test pieces are in hand and measurements on them are being undertaken. These principles also need to be taken into account when developing and validating the measurement procedure, as described in the preceding section.

Responsibility for samples or test pieces begins when they are handed over by the client or customer. Hence the quality system should include procedures for **registering** samples on arrival and allocating unique identifiers. In many organisations this will involve the use of a computerised registration system such as a LIMS (Laboratory Information Management System). In most cases registration will require a record of origin, date and time of receipt, physical description and parameters to be measured, although many organisations add additional information for their specific purposes. It is important to include provisions for reconciling sample details with any documentation and to deal with problems such as damage, loss or ambiguity. In many cases a suitable area will need to be designated for unpacking and/or storing samples taking into account factors such as instability or hazard. Where materials are particularly unstable, provision may be needed for immediate measurement or pre-processing.

Many samples will require **sub-sampling** in order to obtain an appropriate quantity or portion of the material for the measurement procedure. Sub-sampling of heterogeneous materials requires particular care, noting any requirements specified in the documented measurement procedure. In some cases the specification may necessitate selection of components of the sample whereas in others it must be made sufficiently homogeneous. In this respect 'homogeneous' implies that there is no significant variation of the measurand or other relevant parameter at the level used in the procedure. It is important to confirm that equipment and procedures used for homogenising and/or sub-sampling do not introduce significant errors through factors such as loss, contamination or change of physical properties. The requirements noted above for identifiers and appropriate storage will also apply to sub-samples unless they are to be further processed immediately.

The measurement procedure will often entail quite extensive **processing** prior to the final measurement step, using an electronic instrument or other calibrated apparatus. All such processing must be regarded as an integral part of a validated method. As such it should be documented and followed precisely as set out. Appropriate precautions such as those noted above for sub-sampling must, of course, be taken. It is important to remember that in many measurement systems one or more preparatory steps may contribute a far larger proportion of the total error than the

final, instrumental determination. Where a measuring instrument is used for several procedures there should be clear, unambiguous written instructions for its operation in addition to the documented measurement procedures. These instructions should include provision for checking instrumental performance at appropriate intervals and a note of factors which may affect its response.

Proper and regular **calibration** of measuring instruments is essential in order to obtain reliable data. It is mandatory under all formal quality assurance schemes. The term 'calibration' is used in several slightly different ways which may sometimes cause confusion. Many measuring instruments have one or more semi-permanent calibrations which require checking and perhaps slight adjustment from time to time, for example a wavelength scale in a spectrometer or a voltage meter. There are also many instances where the response of an instrument is calibrated for a specific application using appropriate calibration standards. This is the case, for example, with many instruments used for analytical chemistry where calibration solutions are often prepared from reference or other suitable materials. Finally, it is sometimes possible to calibrate an entire measurement procedure using standards or reference materials which are similar in nature to the test samples. Regardless of the approach used, it is essential to follow a properly documented procedure, record all relevant parameters, and archive the calibration data with the sample data. An important aspect of calibration standards is the extent to which their assigned properties or values are traceable to reference measurements made by a recognised national or international organisation. These reference measurements should, where possible, be established through realisation of the appropriate SI unit. The traceability of a measurement allows comparison between sets of data from different organisations, from different countries, or even from different periods of time. It can be achieved directly if the measurement procedure has no significant systematic errors and the calibration standard can be related to an appropriate national or international standard through an unbroken chain of comparisons. This is possible with many physical measurements but rarely feasible for analytical chemistry. Traceability of chemical measurements can, however, be obtained through comparative measurement of a traceable certified reference material having similar composition to the samples.

It is current practice in many areas of measurement to apply complex **signal processing** or **computation** to the raw data obtained from an instrument. These techniques are an integral part of the measurement procedure and should be treated accordingly, i.e. they must be fully documented and not changed arbitrarily. Computerised systems should be properly validated before use, either by the manufacturer or by the user. Validation by the former is becoming more common in some areas of measurement as a result of requirements set out by regulatory or other official bodies. It is also important that appropriate measures be taken to prevent tampering or deliberate falsification of data stored on electronic media. Where data are available only in electronic form, specific provision will be required to ensure that data can be stored and subsequently retrieved over the same period of time as archived written or printed material. Archived material should include all data and other information required to evaluate the entire measurement process for a specific sample if subsequent problems arise. All such material should carry the sample identifier and be signed and dated as evidence of its authenticity. The period of storage will depend on the policy of the relevant laboratory, customer, regulatory body, etc., but can be five years or even longer.

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