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Quality Assurance and Accreditation in the Analytical Laboratory

A REAGECON GUIDE

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1.0 Quality Assurance & Accreditation in the Analytical Laboratory

1.1. Introduction

The value of the chemical measurement depends upon the degree of confidence that can be placed on the results. Increasingly , quality assurance principles are being applied. These do not actually guarantee the quality of the data produced, but increases the possibility of these results being soundly based.

Appropriate quality assurance can enable a laboratory to show that is has adequate facilities, and equipment for carrying out chemical analysis. It also enables the laboratory to show that competent staff carried out the work in a controlled manner, following a documented validated method. Quality assurance should focus on the key issues, which determine quality.

There are a number of formalised principles of quality assurance. Those most widely recognised and used in chemical testing fall into three groups and are then applied according to laboratories individual needs. The 3 main groups are:

A) ISO9000 - relates primarily to quality management, for facilities carried out in production, or providing services including chemical analysis.

B) ISO17025 (previously ISO Guide 25)- specifically addresses the technical competence of laboratories to carry out specific tests. This is the new standard adopted by the National Accreditation Board (ILAB) in Ireland.

C) OECD Guidelines for Good Laboratory Practice (GLP)- relates to the organisational processes and conditions under which laboratory studies related to regulatory work are carried out.

D) There are also Total Quality Management (TQM) approaches to QA in the analytical laboratory, which place emphasis on continual improvement.

A laboratory may decide to design its own quality assurance procedures or it may follow one of the established standards. In the latter case it may claim informal compliance against the standard or ideally may undergo independent assessment from an official expert body, with the aim of gaining independent endorsement of its quality system.

This independent endorsement/assessment is variously known as accreditation, registration or certification depending on which standard the assessment is made against. The independent assessment route has recognised advantages, particularly where the laboratory's customers require objective evidence of the quality assurance measures in place in the laboratory.

1.2 Definitions

Definition of some important terms used in quality assurance.

AUDIT:
"A systematic and independent examination to determine whether quality activites and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives" (ISO8402-1994,3:10)

In practice quality audits take two forms. An audit carried out by an independent external body, as part of the accreditation process is more usually known as an *assessment*. Quality audits are carried out within the laboratory, by the laboratory, are sometimes subdivided into *audit* (which checks that the quality procedures are in place and are effective, and fully implemented) and *review* (which checks to ensure that the quality system is in line with the organisation's quality policy and meets the requirements of both the laboratory and the accreditation body, given that operating circumstances may have changed. The review is carried out by top management with responsibility for the work of the laboratory).

ACCREDITATION:
" Procedure by which an authoritative body gives formal recognition that a body or person is competent to carry out specific tasks" (ISO/CASCO 193 (rev 2), 1.111 & ISO Guide 2-1991,13.7). In the context of compliance with quality management systems, accreditation is used in two contexts.

A) In the context of a laboratory making measurements, accreditation is formal recognition that a laboratory is competent to carry out specific calibrations or tests or specific types of calibrations or tests.
B) Accreditation is also used in the context of ISO9000 based activities to describe whereby a national organisation formally recognises certification bodies as competent to assess and certify organisations as being compliant to the ISO9000 series of standards. In this case it is the certification body (NSAI) which is formally recognised as competent to perform certification activities.

CERTIFICATION:
"Procedure by which a third party gives written assurance that a product, process or service conforms to specified requirements" (ISO/CASCO 193 (Rev. 2), 4.1.2 & ISO Guide 2-1991)

QUALITY ASSURANCE:
"All those planned and systematic actions necessary to provide a product or service will satisfy given requirements for quality (ISO 8402-1994,3.6).

QUALITY CONTROL
" The operational techniques and activities that are used to fulfil requirements for quality" (ISO 8402-1994, 3.7)

REFERENCE MATERIAL (RM):
"Material or substance one of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or of assigning values to materials." (ISO/IEC Guide 30- 1992,2.1)

CERTIFIED REFERENCE MATERIAL (CRM):
"Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure, which establishes its traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence" (ISO/IEC Guide 30-1992. 2.2)

TRACEABILITY:
"Property of the result of a measurement or the value of a standard (ie. measurement standard) whereby it can be related with a stated uncertainty, to stated references, usually national or international standards (i.e. measurement standard) through an unbroken chain of comparisons." (ISO/IEC Guide 30-1992,3.8).

ACCREDITATION

Accreditation is granted to a laboratory for a specified set of activities (i.e. tests or calibrations) following assessment of the laboratory. Such assessments will typically include an examination of the quality system, a review of the quality documentation, and the analytical procedures in use.

The latter will be examined to ensure that they are technically appropriate for the intended purpose and that they have been validated. The performance of tests are usually witnessed to ensure documented procedures are being followed, and indeed can be followed.

The laboratories performance in external proficiency testing schemes may also be examined. This procedural assessment is often called a "system audit". A more rigorous assessment may additionally include a "performance audit", where the laboratory is required to analyse samples supplied by the accrediting body and achieve acceptable levels of uncertainty. This performance audit is effectively a form of proficiency testing and is covered under the Quality Control section.

For accreditation purposes, it is the laboratories responsibility to ensure that all procedures are appropriate for their intended purposes. Accreditation requires the assessment process to examine this fitness-for purpose.

Every accreditation body has established procedures against which it operates, assesses laboratories and grants accreditation. Likewise, assessors are chosen against specified criteria and in general, usually have technical expertise in the area of operation for which the company is going for accreditation.

The benefit of accreditation is that it enables potential customers to have a degree of confidence in the likely quality of the work performed by the laboratory for the accredited activities. Various international developments mean that the endorsement conferred by accreditation and other assessments may have world-wide recognition. For example, the European national accreditation bodies , who are members of EAL (European Accreditation of Laboratories) have signed a multilateral agreement to recognise the compatibility of each others accreditation schemes ie ILAB (Irish accreditation is recognised in UK etc).

The guidance in the below sections will be of use to laboratories seeking accreditation against ISO17025, ISO9000 standards and GLP principles. There are differences between these three operations, however the applicability of the guidance is broadly the same.

1.3.1. SCOPE

A laboratory may apply quality assurance to all or part of its operations. Where a laboratory claims compliance against, or certification or accreditation to, a particular standard, it is important to be clear what this compliance, certification or accreditation applies to. The formal statement of activities which have been accredited against a quality management standard is known as the "scope". ISO9000 and GLP require only a brief description of the activities covered by the organisation but the standard ISO 17025 advocate a detailed list of tests covered by accreditation.

For ISO 17025, the scope may be typically be defined in terms of:

i) The range of products, materials or sample types tested or analysed;
ii) The measurements (or types or measurements) carried out;
iii) The specification or method/equipment/technique used;
iv) The concentration range and uncertainty as appropriate.

Where a laboratory carries out routine testing, definition of a scope in rigid terms is easily applied. However, where non-routine testing is carried out, a more flexible approach to scope is desirable, but the scope must be as specific as is feasible and the QA system maintained by the laboratory must ensure that the quality of the results is under control.

If a laboratory wishes to change its scope, either by adding additional tests or changing the methodology of existing tests, they will require the approval of the accreditation body. The accreditation body will have a specified policy for such situations. This policy should be as flexible as possible. In general, it should be possible to grant simple changes by examination of documentation. For more complex changes, particularly where new techniques are involved, additional assessment may be required.

1.3.2. QUALITY OF THE ANALYTICAL RESULT

This is being dealt with in Section 1.0 of this booklet.

1.3.3. NON-ROUTINE ANALYSIS

Non- routine analysis can be considered as either routine tasks, where reliable methodology is established, but which are carried out infrequently, or tasks where every sample requires a slightly different approach, and methodology has to be established at the time.

The costs of chemical measurement reflect the costs associated with the various stages of method development, validation, instrumentation consumables, ongoing maintenance, staff input, calibration, quality control etc. Many of these costs are independent of the number of samples subsequently analysed using that method.

Measurements in non-routine analysis, usually involve the isolation of the analyte and then a measurement of this analyte. Once the analyte has been isolated, it may now be possible to have a single generic method to cover the measurement of a wide variety of analytes ie UV –vis spectrophotometry.

The documentation of these generic methods should be designed so that it can easily accommodate the small changes which relate to the extraction, clean-up or measurement of different analytes, for example by the use of tables.

The value of such methods for non-routine analysis is that where a new analyte/matrix combination is encountered, it is frequently possible to incorporate it within an existing generic method with appropriate additional validation, measurement uncertainty, calculations and documentation.

It is usually possible to accredit such activities and accreditation bodies will have a policy of assessing such methods and describing them in the laboratory's accreditation schedule. The onus is on the laboratory to demonstrate to the assessors that in using these techniques, it is meeting all of the criteria of the quality standard. In particular, the experience, expertise and training of the staff involved will be a major factor in determining whether or not such analyses can be accredited.

1.3.4. STAFF

Normally, it is the responsibility of the laboratory management to define the minimum levels of qualification and experience necessary for the key posts within the laboratory. Chemical analysis must be carried out by, or under the supervision of a qualified and experience analyst, qualified to degree level in chemistry or equivalent and probably holding a relevant professional qualification. Alternative qualifications may be acceptable when staff have extensive experience and/or the scope of activities is limited. Staff qualified to degree level will normally possess at least 2 years relevant work experience before being considered as experienced analysts. Staff undergoing training or with no relevant qualifications may undertake analyses provided they have demonstrably received an adequate level of training and are adequately supervised.

In certain circumstances the minimum requirements for qualifications and experience of staff carrying out particular types of analysis may be specified in the quality manual.

All staff must receive training adequate to the competent performance of the tests and operation of equipment. Where appropriate this will include background training in particular techniques. Where possible, objective measures should be used to assess the attainment of competence during training and continued competence should be measured. Retraining staff periodically should be considered where a technique/method is not used on a regular basis. Although laboratory management are responsible for ensuring that adequate training is provided, it must be emphasised that a strong element of self-training takes place, particularly among experienced analysts.

It is of crucial importance that an up-to-date record of the training that each member of staff has received is maintained. This is to provide evidence that individual members of staff have been adequately trained and their competence to carry out particular accredited tests has been assessed. Training records should typically include:

i) Academic qualifications
ii) External and internal courses attended
iii) Relevant on-the-job training (and retraining where necessary)
iv) Also where applicable - participation in proficiency testing schemes, with associated data and technical papers published.

In some cases it may be necessary to record competence in terms of particular techniques.
Access to these training records will be necessary in the course of everyday work. However, access to other staff records are usually restricted.

1.3.5. ENVIRONMENT

It is of great importance that samples, reagents, measurement standards and reference materials are stored as to maintain their integrity. The laboratory should guard against deterioration, contamination and loss of integrity. The laboratory must also be able to demonstrate how it carries out this.

The laboratory environment should be sufficiently uncrowded, clean and tidy to ensure the quality of the work carried out is not compromised. This may involve the division of the laboratory into "non-chemical" areas for sample reception and storage, weighing and instrumental measurements and " chemical" areas such as for sample preparation, extraction, reagent and measurement standard preparation and other chemical reactions. The laboratory must also comply with any safety regulations, regardless of its quality requirements.

In some cases it is necessary to restrict access to certain areas of the laboratory due to the work being carried out there. Where such restrictions are in force, staff should be made aware of:

i) The intended use of a particular area
ii) The restrictions imposed on working within such areas
iii) The reasons for imposing such restrictions
iv) The procedure to follow when such restrictions are breached

It is also necessary for the laboratory to provide appropriate environmental conditions and controls necessary for particular tests or operation of particular equipment, including temperature, humidity, freedom from vibration, special lighting etc. Critical environmental conditions must be monitored.

Decontamination procedures may be appropriate where environment or equipment is subject to changes of use or where accidental contamination has occurred.

1.3.6. EQUIPMENT

All equipment used in laboratories should be of a specification sufficient for the intended purpose AND kept in a state of maintenance and calibration consistent with its use. Equipment normally found in the chemical laboratory can be categorised as follows:

i) *general service equipment* not used for making measurements or with minimal influence on measurements (e.g hotplates, stirrers, non-volumetric glassware) and laboratory heating or ventilation systems;
ii) *volumetric equipment* (e.g. flasks, pipettes, burettes etc) and measuring instruments (e.g hydrometers, thermometers, timers, chromatographs, spectrometers, electrochemical meters, balances etc)
iii) *physical measurement standards* (weights, reference thermometers)
iv) computers and data processors

i) *General service equipment* will typically be maintained by cleaning and safety checks as necessary. Calibrations or performance checks will be necessary where the setting can significantly affect the test or analytical result (e.g. the temperature of a furnace or constant temperature bath).
ii) *Volumetric equipment and measuring instruments*- the correct use of this equipment is critical to analytical measurements and therefore it must be correctly used, maintained and calibrated in line with environmental considerations. The performance of some volumetric glassware is dependent on particular factors , which may be affected by cleaning methods etc.

Contamination must be avoided at all costs as this can affect the analytical result. Cleaning procedures must be implemented to ensure that the risk of contamination or cross-contamination remains minimal.

Correct use combined with periodic servicing, cleaning and calibration will not necessarily ensure that an instrument is performing adequately. Where appropriate, periodic performance checks should be carried out, e.g. check response, stability and linearity of sources, sensors and detectors.

The frequency of these performance checks may be specified in manuals or operating procedures. If not, it is necessary to determine these based on experience and on need, type and previous performance of the equipment. Intervals between checks should be shorter than the time the equipment has been found in practice, to drift outside acceptable limits.

It is often possible to build these performance checks – system suitability checks - into test procedures. These checks must then be satisfactorily completed before the equipment is used.

iii) *Physical measurement standards* - the laboratory must have access to the relevant measurement standard where a physical parameter is critical to the correct performance of a particular test.

In some cases, a test and its performance is actually defined in terms of a particular piece of equipment and checks will be necessary to confirm that the equipment conforms to the relevant specification.

Measurement standard materials and any accompanying certificates should be stored and used in a manner consistent with preserving the calibration status. Particular consideration should be given to any storage device given in the documentation supplied with the measurement standard.

iv) *Computers and data processors* - In today's laboratory, computers have a wide variety of uses, from control of critical environmental conditions to the capture, storage, retrieval, processing of data. It is important that interfaces and cables are chosen to suit the particular application since they can seriously affect the speed and quality of data transfer.

As the chemical testing environment creates particular hazards for the operation of computers and storage of computer media, advice should be obtained from their manuals.

Initial validation should verify as many aspects of a computer's operation as possible. Similar checks should be carried out if the computer's use is changed, or after maintenance, or revision of software. Where a computer is used to gather and process data associated with chemical testing, for validation of that function, it is usually sufficient to assume correct operation if the computer produces expected answers when input with known parameters.

Computer programs performing calculations can be validated by comparison with manually generated results, ie check calculations on an auto-titrator manually to see if software is functioning correctly. It should be noted that some faults will occur only when a particular set of parameters is input. In chemical testing, suitable checks on the data gathering and handling functions could be made using a Certified Reference Material for the initial validation, with a secondary measurement standard such as a quality control material used for regular repeat checks.

When the computer processor is linked to a piece of analytical equipment, it is usually the whole system that is validated, as it is more difficult to validate these systems in isolation.

1.3.7. REAGENTS

The quality of reagents and other consumable materials must be appropriate for their intended use. Preferably, reagents and consumables should be purchased from manufacturers who have a quality system such as ISO9000.

The grade of any reagent used(including water) should be stated in the method together with guidance on any particular precautions which should be observed in its preparation, storage and use. Reagents and reference materials prepared in the laboratory should be labelled to identify substance, strength, solvent (where not water), any special precautions or hazards, restrictions of use, and date of preparation and/or expiry. The person responsible for the preparation shall be identifiable either from the label or from the records.

The correct disposal of reagents does not directly affect the quality of sample analysis, however it is a matter of good laboratory practice and should comply with national environmental or health and safety regulations.

Where the quality of a reagent is critical to a test, the quality of a new batch should be verified against the outgoing batch before use, provided that the outgoing batch is known to be still serviceable.

1.3.8. METHODS/PROCEDURES FOR CALIBRATIONS & TESTS

Any method may be used providing it is suitable for its intended purpose, adequately validated and documented. It is the laboratory's responsibility to use methods which are appropriate for the required application.

Methods developed in-house must be adequately validated, documented and authorised before use. Where they are available, CRM's should be used to determine any bias, or where this is not possible, results should be compared to other techniques, preferably on different principles of measurement. Estimation of uncertainty must form part of this validation process and is essential for ongoing quality control.

Documentation of methods, shall include validation data, limitations of applicability, procedures for quality control, calibration and document control.

Developments in methodology and techniques will require methods to be changed from time to time and therefore method documentation must be subject to adequate document control. Each copy of the method

should show issue number/date, issuing authority and copy number. It must be possible to determine from the records which is the most up-to-date version of each method, authorised for use.

Obsolete methods should be withdrawn but must be retained for archive purposes and clearly labelled as obsolete. The difference in performance between the revised and obsolete methods should be established so that it is possible to compare new and old data.

Occasionally, it may be necessary to revise a method. The revision may be of a minor nature, involving different sample sizes, different reagents. Alternatively, it may involve significant changes, such as the use of radically different technology or methodology. In either situation, it will be necessary to carry out further validation to ensure that the performance capabilities of the revised method are fully understood.

1.3.9. CALIBRATION

Calibration is the process of establishing how the response of a measurement process varies with respect to the parameter being measured. The usual way to perform calibration is to subject known amounts of the parameter (eg using a measurement standard or reference material) to the measurement process and monitor the measurement response.

The overall programme for the calibration of measuring equipment in the chemical laboratory should be designed to ensure that, where the concept is applicable, all measurements are traceable through certificates held by the laboratory, either to a national or international measurement or to a CRM. Where no such measurement standard or CRM is available, a material with suitable properties and stability should be selected or prepared by the laboratory and used as a laboratory reference. The required properties of this material, should be characterised by repeat testing, preferably by more than one laboratory and using a variety of methods.

Analytical tests can be sub-divided into 3 general classes depending on the type of calibration required:

A) Measurement of physical properties, such as gravimetry which involves weight measurement, and titrimetry which involves volume, and sometimes weight measurement. These properties usually have a significant effect on the results of a measurement and so a suitable calibration programme is necessary.

B) Where a test is used to measure an empirical property of a sample, such as flashpoint, equipment is often defined in a national or international standard method and traceable reference materials should be used for calibration purposes.

C) Instruments such as chromatographs and spectrometers, which require calibration as part of their normal operation, should be calibrated using reference materials of known composition.

In some cases, calibration of the whole analytical process can be carried out by comparing the measurement output from a sample with the output produced by a suitable reference material that has been subjected to the same full analytical process as the sample. The reference material may either be a synthetic mixture prepared in the laboratory from materials of known (and preferably certified) purity, or a purchased certified matrix reference material.

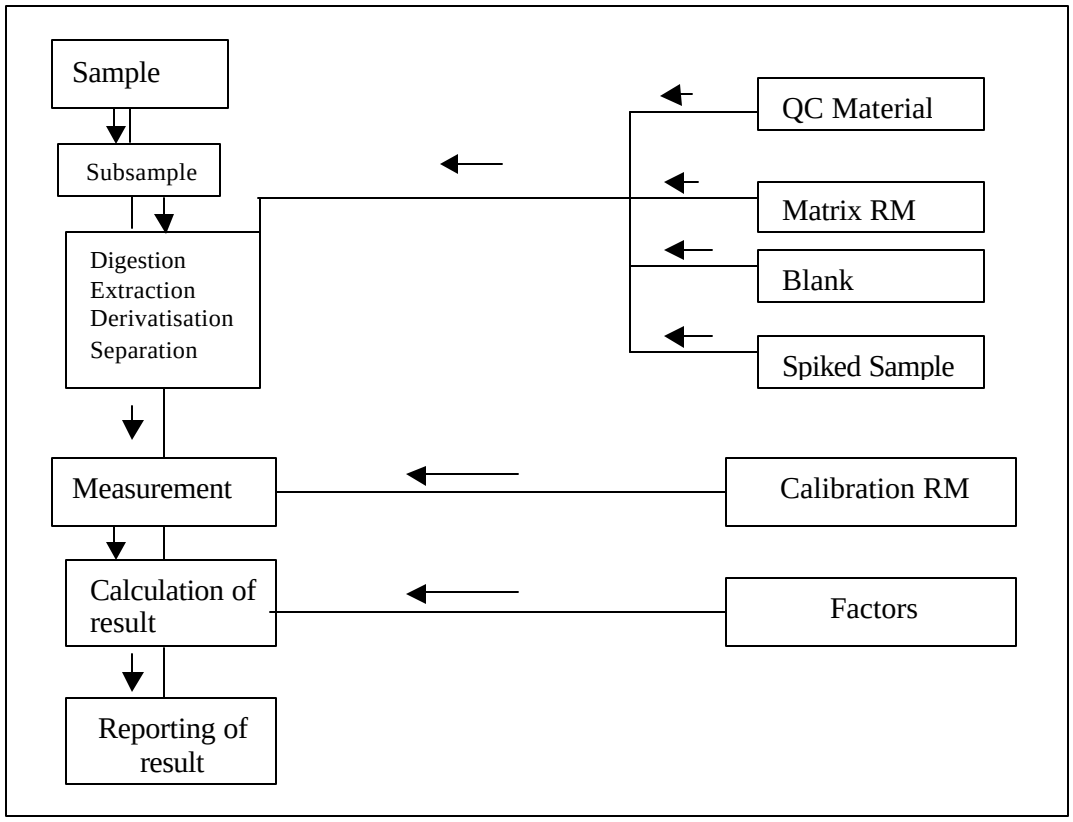
However, in many cases, calibration is only performed on the final measurement stage. This calibration does not take into account factors such as contamination or losses that occur during the sample preparation and extraction stages. It is therefore essential during the method validation process to explore the potential samples through the whole measurement process, and design the day-to-day calibration procedure and quality control checks accordingly.

Individual calibration programmes should be established depending on the specific requirements of the analysis. It also, may be necessary to check instrument calibration after any shutdown, whether deliberate or otherwise, and following service or other substantial maintenance. The level and frequency of calibration should be based on previous experience and should be at least that recommended by the manufacturer.

Procedures for performing calibrations shall be adequately documented, either as part of specific analytical methods or as a general calibration document. The documentation should indicate how to perform the calibration, how often calibration is necessary, action to be taken in the event of calibration failure. Frequency intervals for re-calibration of physical measurement standards must also be indicated.

The calibration of volumetric glassware normally relates to a particular solvent at a particular temperature. The calibration is rarely valid when the glassware is used with other solvents because of other densities, wetting characteristics, surface tension, etc. For the highest accuracy, measurements can often be made by mass rather than volume.

The following flow chart shows a typical analytical process and illustrates the role of calibration in relation to quality control.



1.3.10. REFERENCE MATERIALS

This is being dealt with in section 2.0 of this booklet, under the heading of standards.

1.3.11 LABORATORY AUDIT AND REVIEW

An important aspect of quality management is the periodic re-examination of the quality system. In general, all aspects of the quality management system should be examined at least once a year. The system should be examined in two ways.

Firstly, it should be examined to ensure that it is sufficiently well documented to enable adequate and consistent implementation, and that staff are actually following the system described. This examination is commonly known as (internal) auditing (as opposed to external auditing or assessment carried out by accreditation or certification bodies).

Secondly, the system is examined to see whether it meets the requirements of the laboratory, its customers, and if appropriate, the quality management standard. Over a period of time, the needs of the laboratory and its customers will change and the quality system needs to evolve to continue to fulfil its purpose. This second type of examination is commonly known as a review. It is carried out by the laboratory management and draws on information from a number of sources, including results from internal audits, external assessments, proficiency testing scheme participation, internal quality control studies, market trends, customer complaints and compliments etc.

The programme of audits and review is normally co-ordinated by the laboratory quality manager, who is responsible for ensuring that the auditors have the correct training, guidance and authority necessary for their work. Audits are normally carried out by laboratory staff who are independent of the area they are examining.

Audits may be carried out in two basic ways. In the horizontal audit, the auditor will examine in detail single aspects of the quality system, for example calibration or reports. In the vertical audit the auditor will select a sample and follow its progress from receipt to disposal, examining all aspects of the quality system relating to its testing.

1.3.12. SAMPLE, SAMPLE HANDLING AND PREPARATION

Analytical tests may be required for a variety of reasons, including establishing an average analyte across a material, establishing an analyte concentration profile across a material, or determining local contamination in a material. In some cases, for example in forensic analysis, it may be appropriate to examine the entire material. In others, it is appropriate to take some sort of sample. Clearly the way samples are taken will depend on the reason for analysis.

The importance of the sampling stage cannot be over emphasised. If the test portion is not representative of the original material, it will not be possible to relate the analytical result measured to that in the original material, no matter how good the analytical method is nor how carefully the analysis is performed. The final result **may** be dependent on the analytical method, it will **always** be dependent on sampling process. Sampling errors **cannot** be controlled by the use of measurement standards or reference materials. Sampling is always an error generating process.

Selection of an appropriate sample or samples, from a larger amount of material is a very important stage in the chemical analysis. It is rarely straight-forward. Ideally, if the final results produced are to be of any practical value, the sampling stages should be carried out by, or under direction of, a skilled sampler, with an understanding of the overall context of the analysis. It is a very common pitfall to underestimate the importance of the sampling procedure and delegate it to an unskilled and untrained employee.

It is important when documenting a sampling procedure that all of the terms used are clearly defined, so that the procedure will be clear to other users.

A sampling strategy should be designed taking into consideration some or all of the following parameters:

i) If the sample is not heterogenous, it may be appropriate to mix the material to ensure a representative particle size distribution.

ii) The properties of the analyte should be taken into account, ie the volatility, sensitivity to light, thermal lability, chemical reactivity etc. These parameters may also need to be taken into consideration when deciding what container the sample is going to be put into. Essentially the packaging should be inert and that no contamination from the packaging (i.e leaching) can occur into the sample. There should be no leakages from the container, and preferably the sample should be sealed.

iii) The sampler must keep a clear record of the procedures followed in order that the sampling process may be repeated exactly.

iv) A diagram could be included to indicate the pattern of sampling for extra clarification

v) If the laboratory, is not responsible for the sampling stage it should be documented that the samples were analysed as received.

vi) The sample label is an important aspect of the documentation that should be involved in the sampling strategy. This should unambiguously identify the sample to related plans or notes. Labelling is particularly important, further into the analytical process, where the sample may have been divided, subsampled or modified in some way. Labelling must be firmly attached to the sample packaging and where appropriate, be resistant to fading, autoclaving, sample or reagent spillage and reasonably resistant to extremes of temperature and humidity.

Samples should be stored so that there is no hazard to laboratory staff and the integrity of the samples is preserved. Storage areas should be kept clean and organised so that this is no risk of contamination or cross-contamination, nor packaging and any related seals being damaged. Extremes of environmental conditions should be avoided, and where necessary environmental monitoring should be used. An appropriate level of security should also be exercised to restrict unauthorised access to the samples.

All staff concerned with the administration of the sample handling system should be properly trained and this should be documented. The laboratory should have a documented procedure for the retention and disposal of samples.

1.4. QUALITY CONTROL

The meaning of the terms “quality control” and “quality assurance” often vary according to the context. In practical terms quality assurance relates to the overall measures taken by the laboratory to regulate quality, where quality control describes the individual measures which relate to the quality of individual samples or batches of samples.

Laboratories, as part of their day-to-day activities, must operate an appropriate level of internal quality control (QC) checks and participate wherever possible in appropriate proficiency testing schemes(external QC). The level and type of QC will depend on the criticality, nature of analysis, frequency of analysis, batch size, degree of automation, and test difficulty and reliability.

Internal QC - may take a variety of forms including the use of ;blanks, measurement standards, spiked samples, blind samples, replicate analysis and QC samples. The use of control charts is recommended and is of great benefit , particularly for monitoring QC control samples.

The level of QC adopted must be demonstrably sufficient to ensure the validity of the results. Different types of QC may be used to monitor different types if variation within the process; analysis at different intervals will indicate drift, use of blanks can indicate what contributions, if any, can be attributed to the instrument used, duplicate analyses give a check of repeatability.

QC samples are typical samples which are sufficiently stable and available in sufficient quantities as to be available for analysis over an extended period of time. The random variation in performance of the analytical process can be monitored over time. This is usually carried out by plotting the controls on a control chart, assigning limits to these control charts and as long as the control samples are within these limits it is likely that the results from samples are reliable.

It is the responsibility of the analyst to set and justify an appropriate level of quality control, based on a risk assessment taking into account the reliability of the method and the criticality of the work. It is widely acceptable for QC analysis that 5% is a reasonable amount, i.e. 1 in every 20 samples analysed should be a QC sample. For analyses performed less frequently, a full system validation should be performed on each occasion.

Proficiency Testing (External QC)- one of the best ways for an analytical laboratory to monitor its performance against both its own requirements and the norm of other laboratories is to participate regularly in proficiency testing schemes. Proficiency testing and other types of inter-comparison are accepted as being an important means of monitoring quality at national and international levels. Accreditation bodies recognise the benefit of these schemes and strongly encourage laboratories to participate in proficiency testing as an integral part of their quality assurance protocols. It is important to monitor proficiency testing results as a means of checking quality assurance and take action as necessary. In certain instances, accreditation bodies may specify participation in a particular testing scheme as a requirement for accreditation.

1.5. MEASUREMENT UNCERTAINTY AND VALIDATION

Both of these topics are covered in section 1.0 and 4.0 respectively in this booklet.