May 2010 Update for

COMPLYING WITH ISO/IEC 17025

A practical guidebook for meeting the requirements of laboratory accreditation schemes based on ISO/IEC 17025:2005 or equivalent national standards

Since the original preparation of this guide there have been developments in laboratory accreditation practice and in some of the associated standard documents. The electronic version of the guide has been fully updated and this update document has been prepared to be read alongside the hard copies of the original text to bring users of that text fully up to date. The main changes are in the sections dealing with certified reference materials and proficiency testing. In both of these areas there has been rapid progress involving the updating of the standard ISO documents and the move to accreditation of proficiency testing scheme providers and reference material producers. As a consequence of both of these developments we are now seeing accreditation bodies beginning to insist on the use of proficiency testing schemes which are ISO 17043:2010 accredited and on the purchase of reference materials which are from ISO Guide 34:2009 accredited organisations which have ISO 17025 accredited laboratories.

Section 7.5.1 Use of certified reference materials
An important way for methods to be calibrated by laboratories is the use of certified reference materials (CRM). A CRM (often referred to as a matrix certified reference material) is a sample for which the test results are firmly established and agreed, ideally on an international basis. They are sold by some national standards bureaux and similar organisations and usually verified by highly respected reference laboratories or by interlaboratory calibration.

Acceptable procedures for certification of reference materials are detailed in ISO Guide 35, *Reference Materials—General and statistical principles for certification*. This source also contains much information that can be equally applied in the production of in-house reference materials. At the time of writing, two new ISO guides aimed specifically at guidance for laboratories producing references for internal quality control purposes are in preparation. Guide 80 will deal with materials for quantitative analysis and Guide 79 with references for qualitative properties. The effective use of certified references and the evaluation of data generated by their use is covered by ISO Guide 33, *Guide to the use of certified reference materials*.

A laboratory which wishes to calibrate and validate its methods can check its performance by testing the CRM and so establish traceability. Laboratories which are able to achieve correct results with the CRM should, in theory, agree on any other test for the same parameters in the same matrix.

In order to be effective, a CRM must be typical of the samples which the laboratories are testing on a routine basis. A method for effluents will, ideally, require a CRM which is a typical effluent, and soils testing will need CRMs which are typical of the soils the laboratory normally tests.

Needless to say, CRMs must be stable and highly homogeneous as well as of established composition or properties. This is readily achieved in some areas, such as in the chemical analysis of alloys, the measurement of physical properties such as mass, dimension, etc., and with some geological samples, but things are not so simple in other areas of testing.

Materials testing, for example, often presents a particular difficulty since samples are usually totally destroyed in testing. The best that can be achieved here, therefore, is to have a large number of samples from the same source, for example cut from the same plate of metal or drawn from the same concrete mix, and to test a statistically significant number to arrive at an agreed figure for the whole batch.

Microbiology provides a different sort of problem since sample stability is virtually impossible. However, in microbiology there are, at least, certified reference cultures which provide a definition of particular organisms so that laboratories can verify that their test systems are adequately selective. Relatively recently, quantitative microbiology references have become readily available. These are generally based on the impregnation of cultures onto plastic supports of controlled surface porosity.

Where certified reference materials are not available, there are several alternative strategies, but the main approach is participation in interlaboratory proficiency exercises—see section 7.5.5. Such schemes, at least, give a laboratory a measure of its data relative to other similar laboratories and, if organised properly, provide a very effective addition to

the use of certified references. Accreditation bodies will always expect participation in appropriate proficiency schemes but, where certified references are available, these will be expected to be used as well.

Many basic test methods, especially in analytical chemistry, are intrinsically traceable. There is no need to have a certified reference for most titrations, for example. Here, traceability is provided via the calibrations of the balance and volumetric apparatus. The purist may argue that certification of the purity of the reagents which are weighed or measured is necessary but, provided the origin of the compounds is known and they are of known specification, it would be a harsh interpretation of the standard to insist upon this.

This is not to be taken to mean that methods which use basic measurements such as titrations need never be tested against certified references. In many methods there are preparation steps prior to the end measurement which need to be challenged. For example, a nitrogen determination in a soil by a Kjeldahl procedure will involve a digestion and distillation prior to titration. Such a method will need to be validated using a suitable certified reference soil where available.

Section 7.5.2 Use of spikes [5.9]
Spikes are widely used for method validation and calibration in chemistry and microbiology. They provide a reasonable alternative to certified references, if the spiking material is adequately authenticated, ideally by certification of its purity. On the face of it, a spike has the advantage that the laboratory can spike into a matrix which is absolutely typical of its normal sample stream. The counter argument is to question whether a material spiked into the sample artificially is really present in the same distribution and speciation as the actual target. The strength of this argument depends on the matrix. A metal ion spiked into a water sample might well be regarded as a valid approach, but a pesticide spiked into a food sample may be questioned on the grounds that the pesticide in real samples was, perhaps, systemically absorbed by the crop used to make the food and so may be bound into the cell structure. However, in complex matrices the spike may be the only alternative, however imperfect it may be suspected to be.

A spike is generated by taking a real sample and adding a known amount of the target in question. Ideally, the base sample for the spike should have little or none of the target present before spiking. If this is not possible, the spike level should be large compared to the natural level present. Of course, the natural level must be known in this instance. The spike must be thoroughly mixed and distributed homogeneously throughout the matrix.

The spike does not provide true traceability but it can be reasonably assumed that laboratories which are able to demonstrate good recoveries of spikes have good accuracy and hence will tend to agree.

The use of spikes is especially important where laboratories are carrying out tests in complex matrices which may affect the results. Examples are water analysis, where matrix effects are common, and microbiology, where components of the sample may well affect the viability of organisms. The spike, at the very least, demonstrates that the laboratory would detect the material or organism being sought if it were present.

As more certified reference materials become available, accreditation bodies are, however, increasingly expecting method validation to involve their use and are less inclined to accept validations based on spiking.

7.5.5.1 Selection of proficiency testing schemes by laboratories (New Section)
Provision of proficiency testing is now essentially a commercial activity, and laboratories subscribe to suitable schemes. Accreditation of proficiency testing operators against ISO Guide 43, recently revised as ISO 17043, *Conformity Assessment—General requirements for proficiency testing* is now becoming extensive and, although ISO 17025 does not currently insist that an accredited proficiency test scheme be used by laboratories, accreditation bodies are increasingly insisting that, where an appropriate accredited scheme exists, it should be used. Annex 3 to ISO 17043 is a useful reference for laboratories as it gives guidance on the issues to be considered when choosing a PT provider.

Section 9.4.3 Understanding the hierarchy and validity of reference materials
A reference material is any material or substance where one or more property values are sufficiently homogeneous and well accepted that they can be used for checking methods or apparatus. Two key types of reference materials are a) single compounds or items of established purity or properties and b) matrix references which are specific types of sample where accepted values of one or more determinands have been established.

The key to the reference material is in the acceptance. The highest level of acceptance is a certified reference material (CRM) but even this term is somewhat variable in meaning. Strictly speaking, the only certified reference material of impeccable pedigree is one complying with the definition of a CRM in ISO Guide 30, which means one produced

according to ISO Guide 35 by an organisation complying with ISO Guide 34 and where the certificate complies with ISO Guide 31

Up until relatively recently, there was no accreditation system for reference materials producers so any claims which might be made regarding a reference material and its producer were effectively self-certified.

What happened in practice was that suppliers of commercial reference materials made an evaluation, and purchasers relied on the credibility of the supplier and on the content of the certificates provided to give confidence that the values quoted for the reference material were reliable. Whether the reference material came with enough information to enable it to be classed as a certified reference material (CRM) or only as a reference material (RM) was a matter of interpretation. The basis of any particular supplier's interpretation of the terms can normally be found in their catalogue. Note that in the US the terms NIST Reference Material or Standard Reference Material (SRM) are generally regarded as equivalent to CRM.

In practice, a reference material obtained from a reliable organisation, such as NIST, the EU Community Bureau of Reference (BCR) or the United Kingdom Laboratory of the Government Chemist (LGC), would be very widely recognised and could usually be regarded as a reliable basis for checking the accuracy of methods.

Now, however, the situation is changing rapidly. Accreditation of reference materials producers against ISO Guide 34, *General requirements for the competence of reference materials producers*, is now increasingly widespread and many accreditation bodies are beginning to insist that only results from reference materials from accredited producers are acceptable as demonstrating traceability of measurement. In practice reference materials producers will also form part of the certification chain and will need to have laboratories to monitor preparation of materials and to participate in the setting of certified values. This means that not only must the materials producer show compliance with ISO Guide 34 as regards certification but they must also have an ISO 17025 accredited laboratory.

Guidance on the use of certified reference materials in analytical chemistry specifically can be found in ISO Guide 32.

It should also be noted that reference materials as interpreted by most ISO 17025 accreditation bodies include materials such as standard solutions and buffers which are frequently purchased by laboratories from laboratory chemicals suppliers. Increasingly, accreditation bodies are insisting that laboratories should use only those from IS0 Guide 34 accredited sources.

In summary, it is now very important to check with your accreditation body on its attitude to acceptance of materials as references before committing to buy any particular item.

Add this extra paragraph at end of Section 1.4, Scope of Accreditation
The ILAC Document, *The Scope of Accreditation and Consideration of Methods and Criteria for the Assessment of the Scope in Testing*, which is included in the bibliography, provides a basis for the specification of scope of accreditation, but laboratories should always check carefully with the accreditation body which they intend to use as not all follow this document. This is particularly important when flexible scope is being considered as some bodies may not even permit accreditation on this basis at all.

Bibliography Updated May 2010

ISO and other standards

These publications are normally available from your national standards body, assuming it is ISO affiliated.

ISO/IEC 17025: 2005, General requirements for the competence of testing and calibration laboratories.

ISO/IEC 17011: 2004, Conformity assessment—General requirements for accreditation bodies accrediting conformity assessment bodies.
This is the international standard to which assessment bodies are expected to adhere.

ISO 10012-1: 2003, Measurement Management Systems—Requirements for measurement processes and measuring equipment.
Provides a model for setting up a system for controlling measuring equipment and specifies the records to be kept; also gives guidance on strategies for setting calibration and confirmation intervals.

ISO/IEC 17043: 2010, Conformity Assessment—General requirements for proficiency testing.
Effectively the quality management system standard to be complied with by operators of proficiency testing schemes. Like ISO 17025 it has both technical and management requirements. Appendix 3 provides specific guidance to users needing to select appropriate PT schemes.,

ISO Guide 30: 1992/Amd 1:2008, Revision of definitions for reference materials and certified reference materials.
Provides a formal statement of the reference materials hierarchy.

ISO Guide 31: 2000, Reference materials—Contents of certificates and labels.
Useful to laboratories trying to decide whether a particular certificate or label is consistent with the material having been properly sourced.

ISO Guide 32: 1997, Calibration in analytical chemistry and use of certified reference materials.
Title is self explanatory. In practice the book referenced in the next section, Applications of Reference Materials in Analytical Chemistry, by Barwick et al, is a more easily understood and comprehensive reference on this topic.

ISO Guide 33: 2000, Guide to the use of certified reference materials.
Covers the use of certified reference materials in assessing the performance of a test.

ISO Guide 34: 2009, General requirements for the competence of reference materials producers.
Essential the quality management standard for reference materials producers and the basis for their accreditation. ISO Guide 35, see below, complements ISO Guide 34 in that Guide 35 is effectively a statement of the technical approach required for reference materials certification.

ISO Guide 35: 2006, Reference materials—general and statistical principles of certification.
Deals with the role of reference materials in measurement science and the procedures for their certification. Many of the procedures are equally applicable to the calibration of secondary references against certified references, so it provides useful guidance for laboratories preparing their own in-house quality control standards. It is particularly useful in the area of homogeneity testing. However ISO Guides 79 and 80, currently in preparation, will address in-house reference preparation directly.

Other useful publications

Practical statistics for the analytical scientist, Trevor Farrant, 1997, Royal Society of Chemistry (ISBN 0-85404-442-6).
A really good introduction to statistics for the analyst, whilst covering advanced topics as well; a useful bench guide. Purchase directly from the United Kingdom laboratory of the Government Chemist or RSC.

Statistics and Chemometrics for Analytical Chemistry, J N Miller and J C Miller, 4th Edition, 2000,Prentice Hall (ISBN 0 130 22888 5).
The standard guide to statistics for analytical chemistry; a really good introduction and continuing source of reference. Covers everything a chemist needs to know about statistics. Earlier editions are also useful. Titles of editions 1 to 3 do not mention chemometrics. Purchase through bookseller.

Applications of Reference Materials in Analytical Chemistry, V Barwick, S Burke, R Lawne, P Roper, and R Walker, 2001, LGC, Teddington, (ISBN 0 875404 448 5).
In spite of the title, this book covers other issues, including uncertainty of measurement and statistics of comparison of data. Explanations assume a minimum of familiarity with statistics and are very clear. Purchase directly from the United Kingdom laboratory of the Government Chemist.

Traceability in Chemical Measurement, 2003, Eurachem.
Covers key issues of method validation and also deals with uncertainty of measurement estimation. Can be downloaded free from Eurochem site.

Quantifying Uncertainty in Analytical Measurement, 2000, 2nd Edition, Eurachem.
A useful general guide but does lose the plot occasionally; contains several detailed worked examples of uncertainty estimations which provide a useful pattern which will be appreciated by laboratories working in a much wider area than analytical chemistry. Can be downloaded free from Eurochem site.

Evaluating Uncertainty for Laboratories, Dr Alan G Rowley, Alan Rowley Associates.
An introduction to the subject specially designed to meet the needs of the chemist with little in the way of statistical background. Available from the author.

ILAC G18:04/2010 Guideline for the Formulation of Scopes of Accreditation for Laboratories
The purpose of this publication is to provide information on how to define the scope of accreditation and to identify some criteria and ways of assessing the scope in order to provide practical guidance for an effective and harmonised application of the relevant international standards. Can be downloaded from ILAC site, see below.

Useful websites

'www.ilac.org'
This website describes fully the workings of international recognition of accreditation and also has a wide range of guidance documents for laboratories and accreditation bodies, which can be downloaded, including the guidance document on scope above.

'www.ukas.com'
UKAS is the United Kingdom laboratory accreditation body. They have all of their technical guidance documents available for free download. These are a useful source of information for laboratories needing to achieve technical compliance with ISO 17025 in a wide range of areas of expertise.

'www.lgc.co.uk'
Laboratory of the Government Chemist, United Kingdom; has links to a collection of downloadable catalogues of reference materials and a searchable database of reference materials. The database is not comprehensive, however, and the catalogues should also be consulted. Also has contact information about where you can buy the publications mentioned above.

There are also links to the site dealing with the proficiency testing schemes operated by LGC.

'www.eurachem.org'
Useful source of guidance documents which can be downloaded free. These include the guides to uncertainty of measurement and to traceability in chemical measurement mentioned above.

'www.eptis.bam.de'
Searchable database of proficiency testing schemes in Europe. Many schemes operate globally. Note that the EPTIS database is not screened in any way, so look carefully as some schemes are neither frequent nor very active.

'http://www.fasor.com/iso25/'
Very useful page with links to all kinds of ISO 17025 related issues, helpful sites and national accreditation bodies.