



**Guidelines for the Requirements
for the Competence of
Reference Materials Producers**

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PREAMBLE

One of the key factors affecting laboratories' capabilities to produce reliable test data is the Availability of reference materials with property values that can be relied upon by their users. A number of international guides have been prepared through bodies such as ISO/REMCO to increase confidence in reliability of reference materials, including ISO Guide 34:1996 *Quality system guidelines for the production of reference materials*.

These *Guidelines* have been developed for evaluation of the competence of reference materials producers with a view to the eventual establishment of internationally accepted criteria.

They have been developed with the following major features:

- a) They cover producers of both *reference materials and certified reference materials (CRMs)*. (See separate definitions in ISO Guide 30:1992). Additional requirements are required to be met by producers of *certified* reference materials because, by definition, such CRMs require their certified property values to be traceable to an accurate realisation of the unit in which they are expressed and also require each certified value to be accompanied by an uncertainty at a stated level of confidence.
- b) There are many different arrangements in place for the supply and characterisation of, and assignment of property values to reference materials. These range from all functions being performed by a single organisation, through to various combinations of organisations conducting separate tasks leading to supply of a reference material. These tasks may include, for example, the planning; material preparation; homogeneity and stability assessment; testing (by suitably competent laboratories); assignment of property values and their uncertainties; packaging, labelling, and distribution of a reference material.

ISO Guide 30:1992 defines a certifying body as a: "Technically competent body (organisation or firm, public or private) that issues a reference material certificate which provides the information detailed in ISO Guide 31."

Guide 30 also notes that: "It [the certifying body] may be the same as, or different from, the issuing body (i.e. the body from which the certified reference material is available) and the testing body (i.e. the organisation that carried out the measurements leading to certification)".

For the purposes of these Guidelines, the organization which is responsible for supply of both the reference material and authorization of the data accompanying the reference material, whether in the form of a certificate (for a CRM) or in any other form of assignment of property values (for a reference material), is known as the producer.

Note: ISO Guide 30 only provides a definition for a "certified reference material producer". Therefore, for these Guidelines a more general definition of "reference material producer" is used. (See definition 1.3.1).

The organisation which is responsible for supplying a reference material should ensure that all sub-tasks leading to such supply have been performed competently, whether such tasks are carried out directly by the producer itself or in combination with various subcontractors. Subcontractors are defined as collaborators in ISO Guide 34).

Accordingly, it is the producer (and any sub-contracting arrangements it uses) which should be evaluated to establish its competence to supply reference materials of stated property values in accordance with these *Guidelines*.

- c) These *Guidelines* are based on existing ISO Guides relevant to the production, characterisation and use of reference materials and on the relevant elements of ISO/IEC 17025 (1999) applicable to tests and measurements involved in the assignment of property values to reference materials. Additionally, relevant elements of ISO 9000 are included in the *Guidelines* to eliminate the need for separate recognition of a producer of reference materials for compliance with ISO 9000.

Accordingly, the *Guidelines* have been prepared in two sections covering, respectively:

- (i) Management System Requirements, and
(ii) Technical Requirements

Producers are expected to comply with both the management system and technical requirements to achieve and maintain competence.

- d) The process used for evaluation of compliance with these *Guidelines* involves the use of assessment teams comprising technical experts and other full-time members of, for example, an accreditation body's staff. If the producer of the reference material is a laboratory, the assessment according to these *Guidelines* can be done by a laboratory accreditation body in connection with an accreditation according to ISO/IEC 17025. Otherwise, the assessment by a product certification body, accredited for this purpose, may be more appropriate.

Note: These *Guidelines* do not exclude the process of accrediting calibration laboratories to ISO/IEC 17025 for assigning values to reference materials (artefacts).

- e) Formal recognition of a producer of reference materials is granted in terms of defined categories of reference materials which describe the specific types of reference materials which an applicant is judged competent to produce. The scope of recognition will normally be negotiated with each producer. Distinctions are made in the scope between supply of reference materials or certified reference materials.

PURPOSE

These *Guidelines* have been developed for evaluation of the competence of reference materials producers with a view to the eventual establishment of internationally accepted criteria.

AUTHORSHIP

This publication was prepared by the ILAC Technical Accreditation Issues Committee and endorsed for publication by a decision of the ILAC General Assembly in 1999.

1 GENERAL

1.1 Scope

These *Guidelines* set out the criteria which a producer (and associated collaborators) must meet to be recognised as competent to supply specific types of reference materials of stated property values.

- 1.1.1 It is the responsibility of the producer to ensure that the total requirements (i.e. both technical and management systems) for competence are met by the producer and any associated collaborators.
- 1.1.2 It is recognised that there may be a number of alternative methods used by producers to comply with these Guidelines and throughout the document Notes provide information on possible sources of guidance. Such Notes do not form an integral part of the Guidelines.
- 1.1.3 Where Clauses of these Guidelines are considered to meet the existing requirements of relevant ISO Guides or ISO 9000:1994, these are cross-referenced in Annex 3.
- 1.1.4 Producers complying with these Guidelines are considered to comply also with the relevant requirements of the ISO 9000 Series:1994 for supply of specific types of reference materials. It is recognised that some producers do not directly supply their reference materials but provide them through separate distributors and, as such, some elements of ISO 9000 may not be relevant to their operations.

1.2 References

ISO/IEC 17025:2000, *General requirements for the competence of calibration and testing laboratories*.

ISO Guide 30:1992, *Terms and definitions used in connection with reference materials*.

ISO Guide 31:1986 (under revision), *Contents of certificates of reference materials*.

ISO Guide 34:1996 (under revision), *Quality system guidelines for the production of reference materials*.

ISO Guide 35:1989 (under revision), *Certification of reference materials - General and statistical principles*.

ISO/IEC Guide 65: 1999, *General Requirements for bodies operating product certification systems ISO 9000 series:1994, Quality management and quality assurance standards*.

ISO Guide to the expression of uncertainty of measurement (1993).

VIM:1993, (under revision) *International vocabulary of basic and general terms in metrology* (issued by BIPM, IEC, ISO and OIML).

EURACHEM document 1995: (under revision by Eurachem and CITAC). *Quantifying Uncertainty in Analytical Measurement*.

European Commission Guidelines for the Production and Certification of Reference Materials: 1997, Document BCR/01/97 Part A..

1.3 Definitions

For the purposes of these Guidelines, the following definition applies in addition to those described in ISO Guide 30:1992, ISO/IEC 17025:2000 and VIM:1993.

1.3.1 Reference material producer

Technically competent body (organisation or firm, public or private) responsible for the supply of reference materials or certified reference materials and authorises the property values assigned to reference material or certified reference material.

Other definitions relating to this document:

1.3.2 Accreditation

Procedure by which an authoritative body gives formal recognition that a body or person is competent to carry out specific tasks. [ISO/IEC Guide 2:1996].

1.3.3 Collaborator (Subcontractor)

Technically competent body (organisation or firm, public or private) that undertakes aspects of the supply and/or characterization of the reference material on behalf of the reference material producer, either on a contractual or voluntary basis. [Based on ISO Guide 34:1996].

1.3.4 Certified reference material (CRM)

Reference material, accompanied by a certificate, one or more of whose property values are certified Guidelines for the Requirements for the Competence of Reference Materials Producers by a procedure which establishes its traceability to an accurate realization 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 Guide 30:1992).

1.3.5 Reference material (RM)

Material or substance, one or more 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 for assigning values to materials. (ISO Guide 30:1992)

2 MANAGEMENT SYSTEMS REQUIREMENTS

2.1 Quality Management System

- 2.1.1 The reference material producer shall establish, implement and maintain a quality management system appropriate to the scope of its activities including the type, range and magnitude of reference material production which it undertakes.
- 2.1.2 The reference material producer shall define and document its policy, objectives and commitment to ensuring and maintaining the quality of all aspects of reference material production, including material quality (e.g. homogeneity and stability), characterization (e.g. equipment calibration and measurement method validation), assignment of property values (e.g. use of appropriate statistical procedures) and material handling, storage and transport procedures.

The quality policy shall include use of characterization studies employing laboratories which are active and competent in the respective field of measurement. The policy shall also include a commitment to produce reference materials which conform to the definitions in ISO Guide 30 and whose property values are assessed using accepted statistical techniques. (See Note below). The policy shall include commitment to comply with ISO Guide 31:1986 for the contents of reference material certificates and supply of associated information for users. The policy shall also specify the intended use of the range of materials supplied and shall commit the producer's organisation to ensure that clients are fully advised.

Note: ISO Guide 35:1989 Certification of reference materials - General and statistical principles provides some guidance and a bibliography covering possible techniques for assigning property values. Guide 35, however, is now under revision. Another source of guidance on this topic is given in the European Commission Document BCR/01/97 Part A, Guidelines for the production and certification of BCR reference materials.

- 2.1.3 The reference material producer shall establish and maintain a documented quality system appropriate to the type, range and volume of reference material production it undertakes to ensure that the reference materials produced conform to specified requirements.

As well as conforming to the relevant quality system requirements of ISO/IEC 17025:2000 and the ISO 9000:1994 series, the producer shall have a quality system that, in particular, covers the following:

- a) arrangements for ensuring the suitable choice (e.g. sample matrix, particle size, concentration range) of candidate reference materials;
- b) preparation procedures;
- c) assessment and quantification of the required degree of homogeneity of the reference material;

- d) assessment of the stability of the reference material, including ongoing assessment of stability where necessary;
 - e) procedures for undertaking characterization;
 - f) practical realization of traceability to national or international standards of measurement;
 - g) assignment of property values, including preparation of certificates or statements in accordance with ISO Guide 31 when appropriate;
 - h) provision of suitable production facilities;
 - i) arrangements for suitable identification, labelling and packaging facilities, packing and delivery procedures and customer service.
- 2.1.4 The documented system should specify which activities are undertaken by the producer and, where relevant, which activities are undertaken by collaborators and shall include policies and procedures used by the producer to ensure that all activities conducted by collaborators comply with the relevant clauses of these *Guidelines*.
- 2.1.5 The documented quality system shall define the roles and responsibilities of the technical manager (however named) and quality manager including their responsibilities for ensuring compliance with these *Guidelines*.

2.2 Organisation and Management

- 2.2.1. The producer, or the organisation of which it is part, shall be legally identifiable.
- 2.2.2. The producer shall:
- a) have managerial personnel supported by technical personnel with the authority and resources needed to discharge their duties and to identify the occurrence of departures from the quality management system or the procedures for producing reference materials and to initiate actions to prevent or minimise such departures;
 - b) have arrangements to ensure that its management and personnel are free from any commercial, financial and other internal and external pressures that may adversely affect the quality of their work;
 - c) have policies and procedures to ensure the protection of its clients' confidential information and proprietary rights;
 - d) have policies and procedures to avoid involvement in any activities that might diminish confidence in its competence, impartiality, judgement or operational integrity;

- e) define, with the aid of organisational charts, the organisation and management structure of the producer, its place in any parent organisation, and the relations between management, technical operations, support services, collaborators and the quality management system;
- f) specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the production of reference materials;
- g) have technical management, which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of production operations;
- h) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that these *Guidelines* are implemented and followed at all times. The quality manager shall have direct access to the highest level of management at which decisions are taken on production policy or resources;
- i) where possible, appoint deputies for key managerial personnel such as the technical and quality managers.

2.3 Document and Information Control

2.3.1. General

The producer shall establish and maintain procedures to control all documents (both internally generated and from external sources) and other information that forms part of its quality documentation.

2.3.2. Document approval and issue

2.3.2.1 All documents (including documented procedures) issued to personnel as part of the quality management system shall be reviewed and approved for use by authorized personnel prior to issue. A master list or equivalent identifying the current revision status of documents in the quality management system shall be established and be readily available to preclude the use of invalid and/or obsolete documents.

2.3.2.2 The procedures adopted shall also ensure that:

- a) authorized editions of appropriate documents are available at all locations where operations essential to the effective production of reference materials are performed;
- b) documents are periodically reviewed and where necessary revised to ensure continuing suitability and compliance with applicable requirements;

- c) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use;
- d) obsolete documents retained for either legal or information preservation purposes are suitably marked.

2.3.3. Document changes

2.3.3.1 Changes to documents (including documented procedures) shall be reviewed and approved by personnel performing the same function that conducted the original review and approval unless specifically decided otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval. Where practicable, the nature of the change shall be identified in the document or the appropriate attachments.

2.4 Request, Tender or Contract Review

2.4.1 Each request, tender or contract for production of a reference material shall be reviewed by the producer to ensure that:

- a) the requirements are adequately defined, documented and understood;
- b) the producer has the capability and resources to meet the requirements;
- c) Any differences between the contract or order requirements and those in a tender are resolved.

2.4.2 Records of such reviews including any changes shall be maintained. Records shall also be maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract or request.

2.4.3 The review shall include any work that has to be subcontracted by the producer.

2.5 Use of Collaborators (subcontractors)

2.5.1 The producer shall establish and maintain procedures to ensure that all tasks performed by collaborators (subcontractors) comply with specifications set by the producer for such tasks. The producer shall ensure also that collaborators comply with any clauses of these *Guidelines* relevant to the tasks performed by them for the producer.

2.5.2 The producer shall select collaborators on the basis of their ability to meet subcontracted requirements in terms of both their technical competence and any specific quality assurance requirements relevant to their tasks. The technical requirements to be satisfied by collaborators shall be equivalent to the technical requirements defined in Section 3 of these *Guidelines*.

Note: Where reference materials are intended for use for specific legal purposes, formal technical accreditation of collaborators (testing laboratories) may be required.

- 2.5.3 The producer shall maintain a register of all collaborators used in the production processes and include a record of any assessments made of their abilities to conduct subcontracted tasks.

2.6 Procurement of services and supplies

- 2.6.1 The producer shall have policies and procedures for the selection of services and supplies that affect the quality of its reference materials.
- 2.6.2 The producer shall use only those services and supplies that are of adequate quality to sustain confidence in its assignment of property values.
- 2.6.3 When no formal approval of the quality of services and supplies is available, the producer shall have procedures to ensure that purchased materials and services comply with specified requirements and records of actions taken shall be maintained.
- 2.6.4 The producer shall ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated or otherwise verified as complying with standard specifications or requirements defined in specifications for production, characterisation and assignment of property values to its reference materials.

2.7 Client Feedback

- 2.7.1 The producer shall have a policy and procedures for the resolution of complaints or other feedback received from clients or other parties. Records shall be maintained of all complaints and of the investigations and corrective actions taken by the producer.

2.8 Control of Non-conforming Reference Materials

- 2.8.1 The producer shall have a policy and procedures that shall be implemented when it establishes that any aspect of its production activities, or the property values provided for its reference materials, do not conform with its own procedures or the agreed requirements of a client. The policy and procedures shall ensure that:
- a) responsibilities and authorities for the management of nonconforming work are designated;
 - b) the actions to be taken when nonconforming reference materials are identified are defined;
 - c) an evaluation of the significance of the nonconforming work is made;
 - d) work is halted and certificates withheld as necessary;

- e) remedial actions are taken as soon as possible;
- f) where necessary, the results of nonconforming reference materials already released to clients are recalled;
- g) the responsibility for authorization of the resumption of work is defined.

Note: The identification of nonconforming reference materials or problems with the reference quality management system or with material production activities can occur at various places within the quality management system such as: customer complaints, quality control, checking of consumable materials, staff observations or supervision, certificate checking, management reviews and internal or external audits.

- 2.8.2 Where the evaluation indicates that the supply of nonconforming reference materials could recur or that there is doubt about the producer's compliance with its own policies and procedures, the corrective action procedures in 2.9 shall be promptly followed to identify the root causes of the problem and to eliminate these causes.

2.9 Corrective Action

2.9.1 General

The producer shall establish a policy and procedures and shall designate appropriate authorities for implementing corrective action when nonconforming reference materials or departures from the policies and procedures in the quality management system have been identified.

Any corrective action taken to eliminate the causes of non-conformances or other departures shall be to a degree appropriate to the magnitude of the problems and commensurate with the risks encountered.

The producer shall document and implement any required changes to the operational procedures resulting from corrective action investigations.

Note: A problem with the quality management system or with technical operations may be identified through a variety of activities within the quality management system such as control of nonconforming reference materials, internal or external audits, management reviews, feedback from clients or staff observations.

2.9.2 Cause analysis

Corrective action procedures shall include an investigation process to determine the root causes of the problem.

2.9.3 Corrective actions

The producer shall identify possible causes and potential corrective actions. It shall select the actions most likely to eliminate the problem and to prevent it recurring.

2.9.4 Monitoring of corrective actions

After having implemented the action plans, the producer shall monitor the results to ensure that the actions taken have been effective in overcoming the problems originally identified.

2.9.5 The results of corrective actions shall be submitted for management review.

2.10 Preventive Action

2.10.1 All operational procedures shall be systematically reviewed at regular intervals to identify any potential sources of non-conformances and any opportunities for improvement, either technical or within the quality management system. Action plans shall be developed, implemented and monitored, to reduce the likelihood of occurrence of such non-conformances and to take advantage of the improvement opportunities.

2.10.2 The results of preventive actions shall be submitted for management review.

2.11 Records (See also Clause 3.18)

2.11.1 General

2.11.1.1 The producer shall establish and maintain procedures for identification, collection, indexing, access, storage, maintenance and disposal of quality and technical records.

2.11.1.2 All records shall be legible and shall be stored and retained in such a way that they are readily retrievable, and in facilities that provide a suitable environment to prevent damage, deterioration or loss. Retention times of records shall be established and recorded.

Note: Records may be in the form of any type of media, such as hard copy or electronic media.

2.11.1.3 All records shall be held secure, and, where appropriate, in confidence to the client.

2.11.1.4 The producer shall have procedures to protect electronically-held data at all times and to prevent unauthorized access to, or amendment of, such data.

2.11.2 Technical records

The reference material producer shall establish and maintain a record system to suit its particular circumstances and to comply with any applicable regulations. It shall arrange for all individual measurement observations, appropriate calculations and derived data (e.g. statistical treatments and uncertainty budgets), calibration records and preparation reports to be retained until it is no longer probable that they will be needed.

Note: The period of retention should take into account the period for which the reference material remains valid.

The results of each calibration or measurement (or series of either) carried out by the reference material producer and its collaborators, where appropriate, shall be recorded accurately, legibly, indelibly, unambiguously and objectively, in accordance with any instructions in the calibration or measurement methods. The results shall normally be reported in a calibration or measurement report and shall include all information necessary for interpretation of the calibration or measurement results and a summary of the method employed.

Note: This is for internal reports of the reference material producer and should not be confused with a certificate which is supplied with a certified reference material.

2.12 Internal Audits

2.12.1 The producer shall, periodically and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the quality management system and these Guidelines. The internal audit program shall address all elements of the quality management system, including the technical and production activities leading to assignment of property values to a reference material. It is the responsibility of the quality manager to plan and organise audits as required by the schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

Personnel shall not audit their own activities except where it is necessary and it can be demonstrated that an effective audit has been carried out.

Note: The schedule for internal auditing should normally be completed in one year.

2.12.2 When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of reference materials, the producer shall take timely corrective action and shall notify, in writing, those of its clients whose activities may have been affected.

2.12.3 All audit findings and corrective actions that arise from them shall be recorded. The management shall ensure that these actions are discharged within an appropriate and agreed timescale.

2.13 Management Reviews

- 2.13.1 The senior management shall periodically conduct a review of the producer's quality management system and reference material production processes to ensure their continuing suitability and effectiveness and to introduce any necessary changes or improvements. The review shall take account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, feedback from clients, including complaints and other relevant factors.

Note: A typical period for conducting a management review is once every 12 months. Results should feed into the corporate planning program and should include the goals and objectives and action plans for the coming year.

- 2.13.2 Findings from management reviews and the actions that arise from them shall be recorded. The management shall ensure that those actions are discharged within an appropriate and agreed time-frame.

3 TECHNICAL REQUIREMENTS

3.0 General

This Section specifies the requirements that a producer of reference materials, and any of its associated collaborators, must meet to demonstrate that they are technically competent to produce specific types of reference materials.

In particular, laboratory work associated with the tests and measurements involved in the assignment of property values to a reference material must meet the relevant requirements of ISO/IEC 17025:2000.

Many factors affect the reliability of the property values contained in a certificate accompanying a reference material. As described in ISO Guide 34:1996 ".....it should be recognised that a reference material needs to be characterised mainly to the level of accuracy required for its intended purpose (i.e. appropriate uncertainty)". Accordingly, the degree of application of some of the requirements in this section will depend on the level of accuracy required for the types of reference materials covered by an individual producer. It is expected that all the technical criteria will need to be met by all producers, but it will be the responsibility of third-party assessment teams to judge the depth of application of individual criteria relevant to the types of reference material produced and the uncertainties quoted for their property values.

3.1 Management, Staffing and Training

- 3.1.1 The production, characterisation of, and assignment of property values to reference materials shall only be undertaken by producers and associated collaborators having experience with the particular type of reference material (or related material), as well as having experience and competence in their assigned tasks, i.e. the activity or activities in which they participate as collaborators. (Refer also to Clause 3.3 Collaborators).

- 3.1.2 The producer and associated collaborators shall have managerial staff with the necessary authority, resources and technical competence required to discharge their duties.
- 3.1.3 Measurement of the properties of interest shall be completed by, or under the supervision of, a technically competent manager possessing suitable academic qualifications and/or relevant work experience.
- 3.1.4 The producer's management shall define the minimum levels of qualification and experience necessary for the key posts within its organisation.
- 3.1.5 The producer shall have sufficient personnel having the necessary education, training, technical knowledge and experience for their assigned functions.

Note: For example, a staff member undertaking thermal expansion measurements as part of the process of assigning property values should have a degree, or appropriate level qualification, together with adequate experience in the field working with a more senior scientist making measurements at an equivalent level of accuracy.

- 3.1.6 The producer shall ensure that staff receive additional training, when necessary, to ensure competent performance of measurements, operation of equipment and any other activities which affect quality. Where possible, objective measures should be used to assess the attainment of competence through training.

Note: The need to periodically retrain staff should be considered (e.g. the reference material producer should have in place a policy for retraining staff when a method or measurement technique is not in regular use). Staff training and retraining policies should take account of technological change and aim at continuous skills upgrading.

- 3.1.7 The producer shall maintain an up-to-date record of the training that each staff member has received. These records shall provide evidence that individual staff members have been adequately trained and that their competence to complete particular types of material preparation and measurement has been assessed.

3.2 Producer's Laboratory

- 3.2.1 Where the producer's own laboratory is used (alone or in conjunction with collaborators) for testing for homogeneity, stability or characterization of a reference material, its laboratory shall be required to demonstrate competence for the performance of relevant tests or measurements for the relevant materials at the appropriate concentration levels, ranges etc.

Note: In evaluating the competence of a producer's laboratory, prior possession of laboratory accreditation to ISO/IEC 17025:2000 for appropriate tests and/or measurements will satisfy the requirement for demonstration of competence. In circumstances where the laboratory does not hold accreditation, other factors which should be considered

when evaluating the producer's compliance with these Guidelines will include satisfactory performance in appropriate proficiency testing schemes.

3.3 Collaborators (Subcontractors)

3.3.1 The producer shall be required to demonstrate that the collaborators' experience and technical competence are sufficient for their assigned tasks and comply with the relevant clauses of these Guidelines. (See also Clause 2.5.2).

3.3.2 Where a reference material producer subcontracts any part of the calibration or testing, this work shall be placed with a competent laboratory.

Note: In evaluating the competence of a subcontractor's laboratory, prior possession of laboratory accreditation to ISO/IEC 17025:2000 for appropriate tests and/or measurements will satisfy the requirement for demonstration of competence. In circumstances where the subcontractor's laboratory does not hold accreditation, other factors which should be considered when evaluating the subcontractor's compliance with these Guidelines will include satisfactory performance in appropriate proficiency testing schemes.

3.3.3 In assessing the competence of a collaborator, the producer shall require information on the collaborator's knowledge of the subject and details of past experience in the field, for example, by providing acceptable results for comparable measurements.

3.3.4 The producer shall ensure that all details of the methodology, results and all the outcomes of monitoring of any collaborators are available if required and that a register/database of all collaborators and their accreditation or other form of competence determination is maintained.

Note: The producer should ensure that a non-accredited collaborator's laboratory procedures and test records can be made available for inspection by an assessment team if so required. This would need to be provided for in the contract between the producer and the collaborator.

3.4 Production Planning

3.4.1 The producer shall identify and plan those processes which directly affect the quality of reference material production and subsequent assignment of property values and shall ensure that they are carried out in accordance with prescribed procedures.

3.4.2 The organisational and technical input of the different collaborators involved shall be identified, documented and regularly reviewed. A mechanism (e.g. a management/technical advisory group) shall be established to make recommendations on how to plan the production processes.

Note 1: These could include recommendations for production, setting up a monitoring system (to ensure timeliness and quality for each production phase) and having an evaluation procedure to assess the production processes retrospectively.

Note 2: When producing matrix reference materials, these should, whenever possible, have the same or nearly the same matrix as typical test material in order to simulate the measurement process as closely as possible. For example, they should not have artificially elevated levels of certain elements (if these elemental concentrations are to be assessed) due to contamination from handling and preparation (e.g. for a natural material which is to be characterized for chromium content, consideration should be given to avoiding grinding/mixing in a grinder/blender made from stainless steel).

3.4.3 In planning the overall process for production, characterisation, assignment of property values to (and, in some cases, distribution of) reference materials, the producer shall provide for, where appropriate, procedures and resources for:

- (a) material selection;
- (b) maintaining suitable preparation and testing environments;
- (c) material preparation;
- (d) measuring/testing;
- (e) calibration / validation of equipment / measurement methods;
- (f) assessing material homogeneity;
- (g) assessing material stability;
- (h) organising inter-laboratory studies with its collaborators;
- (i) assigning property values based on the results of measurements;
- (j) producing uncertainty budgets and assigning uncertainty intervals to the assigned property values;
- (k) ensuring adequate storage facilities and conditions;
- (l) ensuring adequate packaging facilities;
- (m) ensuring appropriate transport arrangements;
- (n) ensuring adequate post-distribution service;
- (o) ensuring adequate record storage facilities.

3.5 Production Control

3.5.1 The producer shall identify the verification procedures necessary to ensure the quality of each stage of reference material production and shall assign adequate resources and personnel for such activities. These activities shall include inspection, testing and monitoring of all stages of production.

3.6 Environment

3.6.1 The producer shall ensure that all laboratory accommodation, calibration and measurement areas, material preparation and packaging areas, energy sources, lighting, temperature, humidity, pressure and ventilation are such as to facilitate proper material preparation and packaging as well as proper performance of calibration and measurements.

- 3.6.2 Where appropriate, the environment in which these activities are undertaken shall be monitored with appropriately calibrated equipment, controlled and recorded, such that results and processes are not adversely affected by unsuitable environmental conditions.
- 3.6.3 The producer shall also ensure that, in addition to its own laboratory possessing technical competence relevant to the production and characterisation of reference materials, these environmental requirements are met by any collaborator involved.

Note: It is imperative that all possible precautions are taken against possible contamination of the reference material during its production, characterisation and assignment of property values. All reference material production and testing areas, in addition to satisfying requirements for humidity and temperature, should be protected from vibration, airborne dust and microbiological contamination, magnetic fields and electromagnetic radiation (as appropriate). For example, the packaging of a cement material will require conditions of low humidity, and the preparation of a material in which the content of traces of lead is to be measured will require clean room conditions to prevent contamination from airborne lead particulates due to car emissions. Clean room conditions may be required for reference materials intended for trace analysis.

- 3.6.4 Appropriate health, safety and environmental protection precautions shall be implemented where necessary, e.g. when handling pesticides, sera or body fluids.

3.7 Material Preparation

- 3.7.1 The producer shall establish whether the item or material has received adequate preparation for its intended use. Procedures for material preparation shall include, where appropriate:
- (a) qualitative analysis for verification of material type;
 - (b) machining, grinding, blending, sieving and riffing (i.e. dividing into representative samples);
 - (c) determination of particle size distribution;
 - (d) cleaning of sample containers;
 - (e) drying (including lyophilization), sterilisation;
 - (f) packaging (e.g. bottling, etc) representative samples from the batch;
 - (g) homogeneity testing;
 - (h) stability testing over a range of conditions which may influence the property values and/or matrix composition of the reference materials being produced, e.g. different levels of humidity, temperature, light, magnetic fields, etc, and establishment of product shelf life.
- 3.7.2 The producer shall be able to demonstrate that the candidate reference material is sufficiently homogeneous, that is, the difference, if any, between representative sample measurements must be smaller than the overall uncertainty limits of the measurements.

Note: A relatively inhomogeneous material may be the best available, and may therefore still be useful as a reference material provided the uncertainty of the assigned property values takes due account of this.

3.8 Assessment of Homogeneity and Stability

- 3.8.1 The producer shall use a statistically random selection of a representative number of samples from a batch of candidate reference of the material to assess the homogeneity of the material. This assessment procedure shall be documented and be conducted in accordance with acceptable statistical designs.

Note 1: It is recognised that different experimental designs may be used for evaluation of homogeneity. Some guidance on possible techniques is given in ISO Guide 35 (under revision) and in BCR/01/97 Part A.

Note 2: For materials which can reasonably be expected to be physically homogeneous, systematic sampling (e.g. 1 of every 50 samples produced in a continuous process; sampling at regular intervals for each sub-batch in those cases where the sub-batch can be defined) may often be a better way to detect inhomogeneity than random sampling, for example, segregation of fine and coarse particles in a powder. A statistical trend analysis may also be helpful in detecting inhomogeneity.

- 3.8.2 If a material is produced in several batches, it will be necessary to test the uniformity of the batches (or to assign property values to each batch separately).
- 3.8.3 The assessment of homogeneity shall be performed after the material has been packaged in its final form unless stability studies indicate that it should be stored in bulk form. In some cases, an intermediate homogeneity check may be necessary, for example, before sealing into ampoules.
- 3.8.4 Where appropriate, the property values to be assessed shall be measured periodically, preferably over a range of conditions under which the material is to be stored prior to distribution to the user. The effects of light, moisture, heat and time shall be quantified in order to provide advice on storage location and lifespan (and hence a suitable shelf-life or expiry date).

Note 1: Stability testing can only be performed after sufficient homogeneity has been demonstrated. Then any sample (assuming that it is not smaller than the samples used to test homogeneity) can be considered representative; there is no constraint on the number of samples required, nor any requirement to randomly choose them. However, there will be variation in results depending on the repeatability and intermediate precision measure of the technique and so replicate tests should be performed.

Note 2: When the intended use of a reference material is for the calibration of a method requiring a small quantity of test sample, for example, analysis using graphite furnace AAS or ICP techniques (which use 10?g of sample), it will be necessary to assess the homogeneity on similar-sized portions of reference material.

Note 3: The sample size at which the homogeneity of the reference material has been established should be specified on the certificate of analysis, and the certificate should state, in the instructions for use, the minimum sample size for use.

- 3.8.5 Where appropriate, an assessment of the stability of assigned property values of the reference material shall be performed at specified intervals after characterization to confirm that all values are maintained from production until the end of shelf life.

Note: If the material has a shelf life of several years, it may be necessary to continue these checks for several years after characterization to confirm that all values are maintained from production to the end of shelf life.

- 3.8.6 Wherever appropriate, the producer shall provide an expiry date for the useable life of the reference materials produced based on initial and ongoing stability studies, as recommended in ISO Guide 35. The basis for an expiry date should be made clear on the certificate of analysis (e.g. the date of issue, the date of shipment or the date of opening the packaging).

Note: Some certificates may have more than one expiry date. For example, the date of issue of the certificate, or a date from opening of the container by the user.

- 3.8.7 The producer shall, upon request by a purchaser or supplier, provide details of the homogeneity and stability studies carried out.

3.9 Measurement Methods

- 3.9.1 The producer and collaborator(s) shall use appropriate documented methods or procedures, which include a protocol defining the means to be adopted for different analyses, calibrations, measurements and related activities within their responsibility, and including preparation of items, sampling, handling, preservation, storage, packaging, transport to collaborators, estimation of measurement uncertainty and analysis of measurement data.

Note: These activities should be consistent with the required accuracy of the assigned values of the reference material, and with any standard specifications relevant to the measurement concerned.

- 3.9.2 Measurement methods developed in-house by the producer, or by any collaborator, shall be validated and authorised (e.g. by a management/technical advisory group or appropriately designated person).

Such methods shall have been thoroughly investigated and the results documented. They shall clearly describe the necessary conditions and procedures for which the measurement of the property values of interest are valid at the level of accuracy commensurate with the intended use of the reference material.

Note: In some cases, reference materials will be characterised for method-dependent properties, e.g. leachable metals, pH or flash point.

- 3.9.3 Where sampling is carried out as part of the measurement method (e.g. sub-sampling a representative quantity from a batch of material), the producer and/or collaborator(s) shall use documented procedures and appropriate statistical techniques to take test portions.

3.10 Measuring Equipment

- 3.10.1 Measuring equipment used in the production, characterisation, and assignment of property values to reference materials shall be properly calibrated, verified and maintained with all procedures being documented and the results recorded.
- 3.10.2 Where appropriate, periodic performance checks shall be carried out (e.g. to check the response, stability, linearity, resolution, alignment, repeatability and separating efficiency) to ensure measuring equipment is performing adequately. The intervals between such performance checks shall not be more than the calibration intervals specified by the national laboratory accreditation body.
- 3.10.3 Any item of equipment that has been subjected to overloading or mishandling, shown to provide suspect results, or shown by verification or otherwise to be defective, shall be clearly identified, withdrawn from service and, wherever possible, stored at a specified location until repaired and shown by calibration, verification or test to perform satisfactorily. The producer shall review the implications for results obtained using such equipment, with particular regard to the extent of the calibration deviation, the results involved and the allowable tolerance on the results.
- 3.10.4 Where results may have been in error, the producer shall have the results checked and shall take appropriate remedial action. Records of the review and any checks or remedial action shall be maintained.
- 3.10.5 Each item of equipment, including any measurement standard, that is used in the calibration of equipment or validation of measurement methods used for the production or characterization of a reference material, shall be labelled, marked or otherwise identified to indicate its calibration status and expiry date. Reagents used in chemical analysis and in microbiological testing, etc., shall be appropriately identified.
- 3.10.6 All measuring and testing equipment having an effect on the accuracy or validity of calibrations or measurements shall be calibrated and/or verified before being commissioned into service. The producer and its collaborators shall have an established program for the calibration and verification of measuring and test equipment.

- 3.10.7 The overall program of calibration and/or verification of equipment must be designed and operated to ensure that, wherever applicable, measurements made by the producer and its collaborators are traceable to national and/or international standards of measurement through an unbroken chain of comparisons (with stated uncertainties).
- 3.10.8 Calibration certificates of measurement instruments shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated total uncertainty of measurement.

3.11 Traceability

- 3.11.1 The producer and collaborator(s), where testing is subcontracted, shall provide documentary evidence of the traceability of the results of their measurements to national or international standards of measurement. Where this is not possible, the producer shall provide satisfactory evidence of the correlation of results with the values of other reference materials either by exhaustive evaluation of the measurement process or by correlation with the values of known and accepted national and/or international certified reference materials.

Note 1: Ideally, the latter approach should include the use of certified reference materials the values of which are themselves traceable.

Note 2: Although it is usually possible to establish the traceability of the value of a property by a series of successive comparisons back to the value of an appropriate SI base unit, traceability in the pure metrological meaning can be more difficult when chemical composition quantities [e.g. amount of substance (concentration), specific amount of substance, mass fraction and mass concentration] of complex materials are considered.

In these cases the main contribution to the uncertainty of the measurement result is not the lack of traceability of the measured values of, for example, mass, volume, electric current or amount of substance to the SI units kilogram, metre, ampere or mole respectively, but from the more or less limited selectivity of the measurement procedure for the component of interest which in many instances is accompanied by other components of similar chemical behaviour. (See Annex 5 Measurement Traceability)

- 3.11.2 For certified reference materials, a full uncertainty budget for the values obtained by each measurement procedure shall be prepared, according to the principles of the ISO Guide to the Expression of Uncertainty of Measurement, which identifies contributions to uncertainty and assesses their relative significance. This should be stated in full, with uncertainty components identified prior to the final evaluation of uncertainty for particular measurements. (See Annex 5, Measurement Traceability)

3.12 Procedures for Characterisation of Reference Materials

- 3.12.1 The producer and its collaborators shall use and document technically valid procedures to characterise its reference materials.

Note 1: Assessment teams will evaluate the acceptability of submitted techniques for characterisation of specific types of reference material, taking into account the types of material, the uncertainties proposed for property values, and the availability and cost-effectiveness of use of competent resources to characterise the materials.

Note 2: It is recognised that there are various combinations of techniques available to characterise reference materials. For example, ISO Guides 34 and 35 describe some commonly used techniques.

The particular approach selected will depend on the type of reference material, its matrix, its intended use, the analytical facilities and accredited scopes of technical competence for the laboratories involved and the capabilities of methods employed.

- 3.12.2 The producer and its collaborators shall monitor and assure the quality of all tests and measurements which lead to the characterisation and assignment of property values to the reference materials which it produces.

This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

- (a) internal quality control schemes using statistical techniques;
- (b) participation in interlaboratory comparison or proficiency testing schemes;
- (c) regular use of certified reference materials and/or internal quality control using secondary reference materials;
- (d) replicate tests or calibrations using the same or different methods;
- (e) re-testing or recalibration of retained items;
- (f) correlation of results for different characteristics of an item.

Note: The selected methods should be appropriate for the type and volume of work undertaken.

3.13 Assignment of Property Values and Their Uncertainties

- 3.13.1 The producer shall use documented procedures based on accepted statistical principles for assignment of property values. These should include, as appropriate:

- (a) details of the experimental designs and statistical techniques used;

- (b) policies on treatment and investigation of statistical outliers and/or use of robust statistics;
- (c) whether separate, method-dependent property values are assigned when significant differences are established using different methods;
- (d) whether weighting techniques are used for contributions to assigned property values derived from different methods with different uncertainties;
- (e) the methods used to assign uncertainties to property values; and
- (f) any other significant factors which affect the assignment of property values.

Note 1: ISO Guide 34:1996 discusses issues related to assignment of property values and assessment of uncertainty. The ISO (1993) publication Guide to the expression of uncertainty in measurement is of relevance, particularly for physical measurements. The EURACHEM (1995) document Quantifying Uncertainty in Analytical Measurement also provides guidance.

Note 2: The reference material producer should not rely entirely on a statistical analysis of the characterisation data when assessing the property values of interest. Outliers should not be excluded on purely statistical evidence until they have been thoroughly investigated and, where possible, the reasons for the discrepancies identified. The use of robust statistics may be appropriate in some cases.

Note 3: When several methods have been used to characterize a reference material, difficulty may arise when the results show significant differences. In such a case, a property value based on the mean is inappropriate. It is essential in such cases that the producer and its collaborators should have considerable experience with the different methods and be able to give more or less weight to the results from the use of a particular measurement method. For example, the means of two or more measurement methods may differ statistically, but the results from both methods may agree within the uncertainty of each method. In this case the results may be weighted according to the inverse of the variance of each method. In some cases, measurement methods will produce irreconcilable results and it may be necessary to assign separate property values according to the methods used (i.e. a method specific approach).

- 3.13.2 The producer shall ensure that calculations and data transfers are subject to appropriate checks, including those from its own sources or those from its collaborators.

- 3.13.3 Where computers or computer controlled systems are used for the capture, processing, evaluation, recording, reporting, storage or retrieval of calibration or test data, the producer shall ensure that for itself and collaborator(s):
- (a) computer software is validated, particularly when developed in-house, and is adequate for use;
 - (b) procedures are established and implemented for protecting the integrity of data; such procedures should include, but are not limited to, integrity of data entry or capture, data storage, data transmission and data processing;
 - (c) equipment is maintained to ensure proper functioning and provide environmental and operating conditions necessary to maintain data integrity;
 - (d) appropriate procedures are established and implemented for the maintenance of data security including prevention of unauthorised access to, and amendment of, computer records. Hard copies of all computer records and computer disk copies of programs should, where possible, also be retained in order to overcome potential difficulties in comparing new data with data obtained using outdated or replaced software.
- 3.13.4 All technical data related to production of reference materials should be retained as required in Clause 2.11.2.

3.14 Certificates and Supporting Information

- 3.14.1 The producer shall ensure that clients are provided with certificates and supporting documentation for each supplied reference material or batch of reference material containing all information needed for the proper use of the reference material.

Note: The producer may acknowledge on such certificates and/or supporting information that it is accredited for such certifications, provided that the certificates and supporting documentation comply with Clause 3.14.2 below.

- 3.14.2 Certificates and other supporting information accompanying reference materials shall together contain all information (appropriate to the type of materials concerned) described in ISO Guide 31:1996 (Contents of certificates of reference materials.)

3.15 Materials Handling and Storage

- 3.15.1 In order to avoid contamination of the reference material, the producer shall identify, preserve and segregate from all chemicals and samples all candidate materials and reference materials from the time of preparation through to their distribution to users.

- 3.15.2 The producer shall ensure adequate packaging of all reference materials (e.g. where appropriate use air-free, moisture-free or inert gas packaging) and provide secure storage areas and/or stock rooms which prevent damage or deterioration of any item or material between characterisation and distribution. Appropriate methods for authorising dispatch to, and receipt from, such areas shall be defined.
- 3.15.3 Where appropriate, the condition of all stored or stocked items and materials shall be assessed at specified intervals during their storage life in order to detect possible deterioration.

3.16 Packing

- 3.16.1 The producer shall control packing and marking processes to the extent necessary to ensure conformity with relevant regional, national and/or international safety and transport requirements.

Note 1: The proper distribution of samples can present severe problems for some types of material, for example, those which require uninterrupted storage in a freezer or which should not be exposed to X-rays, shock or vibration. Most types of chemical materials would benefit from air-tight packaging to avoid contamination by atmospheric contaminants, for example, fuel vapours or engine exhaust gases which may be encountered during transport. The user should be provided with information on the correct means of storage and minimisation of the risk of contamination of the packaged material.

Note 2: The producer has a responsibility to ensure that the integrity of the reference material is maintained until the seal is broken, or up to the point where presented for analysis. The producer cannot be held responsible for goods once the seal is broken. This may require, in some cases, that the reference material be packaged in unit quantities sufficient for a single use.

3.17 Labelling and Dispatch

- 3.17.1 The producer shall ensure that material labels are securely attached to the product packaging of individual reference material units and are designed to remain legible and intact within the period of validity of the material.
- 3.17.2 The label must identify the material, the certifier, its batch and catalogue numbers and any other information necessary to enable the material to be uniquely distinguished and referenced, where appropriate, to its statement or certificate.
- 3.17.3 The producer shall arrange for maintaining the integrity of each reference material throughout the entire production process. Where contractually specified, this protection shall be extended to include delivery to destination.

3.18 Internal Records and Reports (See also Clause 2.11)

- 3.18.1 The producer shall establish and maintain a record system to suit its particular circumstances and comply with any applicable regulations.

The record system should encompass measuring instruments, staff, collaborators and suppliers, and, for laboratory activities, should be established and maintained according to these Guidelines.

- 3.18.2 The producer shall arrange for the retention of all individual measurement observations, appropriate calculations and derived data, calibration records and preparation reports pertaining to a reference material for a defined period beyond the expected use of its property values.

Note: Where the stability of the material allows, part of the records should consist of one or more specimens of the material. This archived material should be kept after the expiry of its shelf life.

- 3.18.3 The results of each calibration or measurement (or series of either) carried out by the producer and its collaborators, where appropriate, shall be reported accurately, legibly, indelibly, unambiguously and objectively, in accordance with any instructions in the calibration or measurement methods.

3.19 Post-distribution Service

- 3.19.1 Where the producer is also responsible for distribution of reference materials to end users, it shall:

- (a) establish, document and maintain procedures for ensuring that corrective action is undertaken whenever a product is found not to be conforming to the specified requirements. Any resulting changes, for example, in procedures or data, shall be recorded and all purchasers or distributors of the reference material notified if there is a change to its assigned property values (for example, as a result of additional measurement studies) within the period of the validity of the material; and
- (b) maintain a mailing list of purchasers of every reference material in order to advise any users who may need to be informed of a problem concerning the reliability of assigned property values of a particular reference material.

Note: It is recommended that the producer also have an advisory service to provide guidance and technical services to users. Where the goods are subject to resale through a distributor, the producer should make arrangements with the distributor to keep records of purchasers and end-users of the reference materials.

APPENDIX A

BIBLIOGRAPHY

1. *NATA Requirements for Accreditation for Competence of Producers of Reference Materials*, February 1998.
2. *AS 3780:1991, Certified reference materials - General guide to material selection, preparation, testing and certification.*
3. *ISO/IEC Guide 2:1996, Standardization and related activities - General vocabulary.*
4. *ISO Guide 32:1997, Calibration in analytical chemistry and use of reference materials.*
5. *ISO Guide 33:1989 (under revision), Uses of certified reference material.*
6. *ISO 3534 Series : 1993, Statistics - Vocabulary and symbols.*
7. *ISO 8402:1994, Quality management and quality assurance - Vocabulary.*

APPENDIX B

CATEGORIES OF REFERENCE MATERIAL

Introduction

The following list of reference material categories has been derived following consideration of the existing entries in the reference material catalogues of bodies such as the National Institute of Science and Technology (NIST), the Laboratory of the Government Chemist (LGC), UK, COMAR, and the European Community Bureau of Reference (BCR).

The purpose of classifying reference materials is to enable the laboratory accreditation body and the reference material producer to identify and define those types of material for which the producer has been found by assessment to be competent.

The System of Classification

The principal headings or categories under which it is suggested that the reference materials are to be listed are:

Category A: Chemical composition

Reference materials, being either pure chemical compounds or representative sample matrices, either natural or with added analytes (e.g. animal fats spiked with pesticides for residues analysis), characterised for one or more chemical or physicochemical property values.

Category B: Biological and clinical properties

Materials similar to Category A, but characterised for one or more biochemical or clinical property values.

Category C: Physical properties

Materials characterised for one or more physical property values, e.g. melting point, viscosity, density.

Category D: Engineering properties

Materials characterised for one or more engineering property values (e.g. hardness, tensile strength, surface characteristics, etc).

Category E: Miscellaneous

These principal categories are subdivided into subcategories as indicated in the following draft list. It should be noted that these sub-categories are indicative only. Other sub-categories can be added at any time to address the needs of applicants seeking recognition of competence in producing types of reference materials not currently listed.

CATEGORY A: CHEMICAL COMPOSITION

A1: METALS

- A1.1 Ferrous**
Steels
 carbon steels
 low alloy steels
 high alloy steels
 cast steels
 speciality steels

- Irons
 white cast irons
 ductile irons

Gases in metals

- A1.2 Nonferrous**
Aluminium alloys
Copper base alloys
Lead base alloys
Tin base alloys
Brasses
Bearing alloys
Titanium base alloys
Zirconium base alloys
Gases in metals

- A1.3 Special alloys**

- A1.4 Refractory metals and alloys**

- A1.5 Rare earth metals**

- A1.6 High purity metals**
Solid forms
Spectrochemical materials
Spectrochemical solutions

A2: INORGANIC REFERENCE MATERIALS

- A2.1 Ores and minerals**

- A2.2 Cements, clays and related products**

- A2.3 Ceramics, glasses and refractory oxides**
Carbides
Glasses

- A2.4 Agricultural chemicals and fertilisers**

A2.5 Solid fuels

Coal and coke
mineral content
major elements
trace elements

A2.6 Pure chemicals

Stoichiometry standards
primary standards
working standards
secondary standards
Chromatography standards
Pharmaceutical materials
Cosmetic materials

A2.7 Stable isotope materials**A3: ORGANIC REFERENCE MATERIALS****A3.1 Pure organic compounds**

Compounds for elemental analysis
Compounds for molecular weight
Chromatography standards
Illicit drugs and their metabolites - (See also A8 Forensic Reference Materials)
Illicit drugs
 delta-9-THC and other cannabinoids
 amphetamine
 methamphetamine
 3,4-methylenedioxyamphetamine
 3,4-methylenedioxy-methylamphetamine
 3,4-methylenedioxyethylamphetamine
 diacetylmorphine
 morphine
 cocaine
 lysergic acid diethylamide and isomers
Therapeutic drugs
Veterinary drugs
Steroids
Pesticides, herbicides, acaricides, etc
Metabolites of any of the above
Priority pollutants
 PCBs, PAHs, etc
Fine chemicals
Pharmaceutical materials
Cosmetic materials
Isotopically labelled compounds

A3.2 Agricultural materials, fertilisers

A3.3 Foodstuffs

Proximate analysis
Nutritional properties
Vitamins
Other food additives
 antioxidants
 emulsifiers
Toxins
 animal origin
 plant origin
 other biological origin
Trace elements
Trace organics
 pesticide residues
 other organic contaminants

A3.4 Plastics and rubbers

Hardness
Natural rubber content
Identity
 copolymers
 plasticisers
 vulcanising agents
 blowing agents
 antioxidants
 fillers

A3.5 Petroleum products

Fuels and lubricants
 lead
 vanadium
 nickel
Transformer oils
 moisture
 PCBs
Heat exchange fluids
 moisture
 PCBs

A3.6 Vegetable oils and fats

Fatty acid profile
Triglyceride composition

A4: ENVIRONMENTAL REFERENCE MATERIALS

A4.1 Soils and sludges

Trace elements
Mineral content
Trace organics
TCLP leachate

- A4.2 Ashes**
 - Fly ash from coal and coke
 - Incinerator ash

- A4.3 Waters**
 - Potable water
 - routine analytes
 - trace elements
 - organic pollutants
 - other analytes
 - Fresh water
 - major elements
 - trace elements
 - other analytes
 - Sea water
 - major elements
 - trace elements
 - other analytes
 - Industrial waste water
 - routine analytes
 - trace elements
 - organic pollutants
 - other analytes
 - Treated sewage
 - routine analytes

- A4.4 Plant material**
 - Trace elements
 - Mineral content

- A4.5 Marine**
 - Fish) trace elements
 - Molluscs) mineral content
 - Plankton) organics

- A4.6 BOD reference compounds**

- A4.7 Miscellaneous biological materials**
 - (e.g. Human hair)

A5: HEALTH AND INDUSTRIAL HYGIENE

- A5.1 Clinical laboratory materials**

- A5.2 Ethanol solutions**

- A5.3 Toxic substances in urine**
 - Toxic metals
 - Fluoride
 - Mercury

A5.4 Drugs of abuse in urine

A5.5 Drugs of abuse in hair

A5.6 Materials on filter media

A5.7 Trace elements in blank filters

A5.8 Lead in paint (powder and sheet forms)

A5.9 Respirable silica

A6: ENGINE WEAR MATERIALS

A6.1 Metallo-organic compounds

A6.2 Wear metals in oil

A7: ANALYSED GASES

A7.1 Gas mixtures

A7.2 Trace volatile organic compounds

A8: FORENSIC REFERENCE MATERIALS

A8.1 Ethanol reference standards

Ethanol

Ethanol, aqueous solutions containing 0.050, 0.150, 0.250 g/100mL

A8.2 Drugs (individually named) and metabolites*

In whole human blood and urine (*metabolites to include glucuronides).

See also A3.1 Pure Organic Compounds.

A8.3 Glasses

bottle

window

automotive

spectacle

A8.4 Paints

Automotive

Architectural

A8.5 Accelerants

Flammable liquids and residues thereof

A8.6 Explosives and primers

A8.7 Gunshot residues

- A8.8 Noxious substances**
 - Crowd control agents
 - capsaicin
 - o-chlorobenzalmalononitrile (CS)
 - chloroacetophenone (CN)

- A8.9 Document examination**

A9: ION ACTIVITY

- A9.1 pH standards**
- A9.2 Ion selective electrode calibrants**
- A9.3 Conductivity standards**
- A9.4 Buffer systems**

CATEGORY B : BIOLOGICAL AND CLINICAL PROPERTIES

B1 General Medicine

- B1.1 Human serum materials (powder and solution forms)

B2 Clinical Chemistry

- B2.1 Proteins
- B2.2 Apolipoproteins
- B2.3 Enzymes
- B2.4 Hormones
- B2.5 Trace elements
 - lead and cadmium

B3 Tissue Pathology

B4 Haematology and Cytology

- B4.1 Blood serum

B5 Immunohaematology

B6 Immunology

B7 Parasitology

B8 Bacteriology and Mycology

- B8.1 Reference cultures
- B8.2 Antibiotics

B9 Virology

B10 Other biological and clinical reference materials

B11 Forensic Reference Materials

Purified DNA of known and continuing genetic composition
Human, primate and animal blood
Animal hairs
Fibres (see also C7.1 to C7.3)

CATEGORY C : PHYSICAL PROPERTIES**C1 Reference Materials with Optical Properties**

C1.1 Optical rotation
C1.2 Refractive index
C1.3 Spectral absorbance
visible
ultraviolet
infrared
C1.4 Specular reflectance
C1.5 Colour
white reference material (opal glass)
ceramic tiles

C2 Reference Materials with Electrical and Magnetic Properties

C2.1 Dielectric strength
C2.2 Resistivity
C2.3 Magnetic susceptibility

C3 Reference Materials for Frequency Measurements**C4 Reference Materials for Radioactivity**

C4.1 Radiation dosimetry
C4.2 Radiopharmaceuticals
C4.3 Labelled compounds
C4.4 Natural matrix materials
C4.5 Carbon-14 dating

C5 Reference Materials for Thermodynamic Properties

C5.1 Calorimetry
C5.2 Thermal conductivity
metals
pyrex glass
resin-bonded fibre board
C5.3 Vapour pressure
C5.4 Thermal expansion
C5.5 Thermal resistance
C5.6 ITS-90 temperature fixed point
C5.7 Curie point
C5.8 Boiling point
C5.9 Melting point
C5.10 Thermal analysis standards

C6 Reference Materials for Physicochemical Properties

- C6.1 Density
- C6.2 Viscosity
- C6.3 Surface tension
- C6.4 Molecular weight

C7 Reference Materials for Fibre Identification

- C7.1 Natural fibres
 - animal hairs
 - plant fibres
- C7.2 Synthetic fibres
 - organic polymers
 - inorganic
- C7.3 Asbestos fibres
 - crude fibres
 - mounted specimens for fibre counting

C8 Reference Materials for other properties

- C8.1 Shear testing of powders
- C8.2 Minerals for x-ray diffraction

CATEGORY D : ENGINEERING PROPERTIES**D1 Surface Finish**

- D1.1 Surface roughness
- D1.2 Corrosion
- D1.3 Microhardness
- D1.4 Abrasive wear
- D1.5 Properties of films and surfaces
 - Nominal thickness
 - x-ray fluorescence
 - B particle backscattering
 - ion beam sputtering

D2 Sizing

- D2.1 Particle size
 - Particulate materials
 - Latex sphere suspensions
- D2.2 Surface area

D3 Nondestructive Testing

- D3.1 Dye penetrant test blocks
- D3.2 Artificial flaw for eddy current
- D3.3 Magnetic particle inspection

D4 Hardness

- D4.1 Rockwell hardness
- D4.2 Izod hardness

D5 Impact Toughness

D5.1 Charpy V-notch test blocks

D6 Tensile Strength

D7 Elasticity

D8 Creep

D9 Fire Research

D9.1 Surface flammability

D9.2 Smoke density

CATEGORY E : MISCELLANEOUS PROPERTIES

(Sub-categories to be developed as required).

APPENDIX C

CROSS-REFERENCES TO ISO 9000, ISO GUIDE 34 AND ISO/IEC 17025

Cross-references between elements of the *ILAC Guidelines for the Requirements for the Competence of Reference Materials Producers* and, where relevant, ISO 9001:1994, ISO Guide 34:1996 and ISO/IEC 17025:2000

ILAC Requirements	ISO 9001:1994	ISO Guide 34:1996	ISO/IEC 17025:2000
2.1.1	-	4.2	4.2
2.1.2	4.1.1	4.2	4.2.2
2.1.3	4.2.1 & 4.2.2	4.2	4.2.1
2.1.4	4.6.2	4.4	-
2.1.5	4.1.2	4.3	4.1.4
2.2.1	-	-	4.1.1
2.2.2	4.1.2	4.3	4.1.4
2.3.1	4.5.1	-	4.3
2.3.2.1	4.5.2	-	4.3.2.1
2.3.2.2	4.5.2	-	4.3.2.2
2.3.3.1	4.5.3	-	4.3.3.1
2.4.1	4.3.2	-	4.4.1
2.4.2	4.3.3	-	4.4.2
2.4.3	-	-	4.4.3 & 4.4.4
2.5.1	4.6.1	4.4	4.5.1 & 4.5.4
2.5.2	4.6.2	4.4	4.5.2
2.5.3	4.6.2	4.4	4.5.4
2.6.1	4.6.1	-	4.6.1
2.6.2	4.6.1 & 4.6.2	-	4.6.2
2.6.3	4.6.4	-	4.6.3
2.6.4	4.6.4	-	4.6.3
2.7.1	4.14.2	-	4.7 & 4.8
2.8.1	4.13	-	4.9.1
2.8.2	4.14.2 & 4.14.3	-	4.9.2
2.9.1	4.14.1	-	4.9.2 & 4.10
2.9.2	4.14.2	-	4.10.2
2.9.3	4.14.2	-	4.10.3
2.9.4	4.14.3	-	4.10.4 & 4.10.5
2.9.5	4.14.3	-	4.14.1
2.10.1	4.14.3	-	4.11.1
2.10.2	4.14.3	4.6	4.11.2 & 4.14.1
2.11.1.1	4.16	4.6	4.12.1.1
2.11.1.2	4.16	-	4.12.1.2
2.11.1.3	-	5.7	4.12.1.3
2.11.1.4	-	4.6	4.12.1.4
2.11.2	-	-	4.12.2.1 & 4.12.2.3
2.12.1	4.17	-	4.13.1
2.12.2	4.17	-	4.13.2
2.12.3	4.17	-	4.13.3 & 4.14.3

GUIDELINES FOR THE REQUIREMENTS FOR THE COMPETENCE OF REFERENCE MATERIAL PRODUCERS

ILAC Requirements	ISO 9001:1994	ISO Guide 34:1996	ISO/IEC 17025:2000
2.13.1	4.1.3	-	4.14.1
2.13.2	4.4.3	-	4.14.2
3.1.1	-	4.3	5.2.1
3.1.2	-	4.3	5.2.5
3.2.3	-	4.3	5.2.5
3.1.4	-	4.3	5.2.2 & 5.2.4
3.1.5	4.1.2.2	4.3	5.2.1
3.1.6	4.18	4.3	5.2.1 & 5.2.2
3.1.7	4.18	4.3	5.2.1
3.2.1	4.6.2	4.4	4.5 & 5.2
3.3.1	-	4.4	4.5.1
3.3.2	-	4.4	4.5.1 & 4.5.2
3.3.3	4.6.2	4.4	4.5.3
3.3.4	-	-	4.5.3
3.4.1	4.9	5.1	-
3.4.2	4.9	5.1	-
3.4.3	-	5.1	-
3.5.1	4.10	5.1	5.4
3.6.1	-	4.1	5.3.1
3.6.2	-	4.1	5.3.2
3.6.3	-	4.1	-
3.6.4	-	4.1	5.3.5
3.7.1	4.10.2	5.2	-
3.7.2	-	5.2	-
3.8.1	4.10	5.3	5.7.1
3.8.2	4.10	5.3	-
3.8.3	4.10	5.3	-
3.8.4	4.10	5.3	-
3.8.5	-	5.3	-
3.8.6	-	5.3	-
3.8.7	-	5.3	5.7.3
3.9.1	-	5.4	5.4.1 & 5.4.2
3.9.2	-	5.4	5.4.3 – 5.4.5
3.9.3	-	5.4	5.7
3.10.1	4.11	5.5	5.5.1 & 5.5.2
3.10.2	4.11	5.5	5.4.6 & 5.5.2
3.10.3	-	5.5	5.5.8
3.10.4	4.11	5.5	4.9
3.10.5	4.11	5.5	5.5.4, 5.5.5 & 5.5.9
3.10.6	4.11	5.5	5.5.2 & 5.5.12
3.10.7	4.11	5.5	5.6.1 & 5.6.2
3.10.8	-	5.5	5.5.2 & 5.6.1
3.11.1	4.11.2	5.6	5.6.1 & 5.6.2
3.11.2	-	5.6	5.10.3
3.12.1	4.9	5.8	-
3.12.2	-	-	5.9
3.13.1	4.20	5.9	5.4.7

GUIDELINES FOR THE REQUIREMENTS FOR THE COMPETENCE OF REFERENCE MATERIAL PRODUCERS

ILAC Requirements	ISO 9001:1994	ISO Guide 34:1996	ISO/IEC 17025:2000
3.13.2	-	5.9	5.4.8
3.13.3	4.11.1	5.7	4.12.1.4 & 5.5.11
3.13.4	4.10.5	-	5.4.7 & 5.7.3
3.14.1	4.12	5.9	5.10.1
3.14.2	-	5.9	-
3.15.1	4.12 & 4.15	4.5	5.8
3.15.3	4.15.3 & 4.15.4	4.5	-
3.16.1	4.15.3 & 4.15.4	4.5	-
3.17.1	4.12 & 4.15.4	4.5	-
3.17.2	4.12 & 4.15.4	4.5	-
3.17.3	4.15.6	4.5	-
3.18.1	4.16	4.6	4.12.2.1
3.18.2	4.16	4.6	4.12.2.1
3.18.3	-	4.6	4.12.1.2; 5.9 & 5.10
3.19.1	4.19	4.7	-

APPENDIX D

CONTENT OF CERTIFICATES ACCOMPANYING REFERENCE MATERIALS

Introduction

ISO Guide 31 provides guidance on the information which should be contained in certificates accompanying reference materials. The current edition of ISO Guide 31 was published in 1981 and is at present under revision.

ISO Guide 31 suggests that: “The past 15 years, however, have seen a general decline in the issuing of certification reports and a corresponding increase in the information provided in certificates. This is not necessarily a problem for users of RMs, provided that the information appropriate to a full certification report is always readily available on application to the providers of the CRM.”

At the same time, the information required from a certificate is often more than the certified property value or assigned property value. Details concerning the way in which the container should be opened, the minimum sample size which should be taken for a measurement, the stability of the material, the way in which it should be stored, and, in the case of CRMs where the certified value is method-dependent, the method used to determine the certified value, are all essential information for the user.

ISO Guide 31 states that the information provided on the label should serve only to identify the CRM and should be confined to the name of the producer, the name of the material, the producer’s code for the material, and the batch number. It recommends that, in order to prevent use of the material without the information in the certificate having been studied, the verified property value(s) should not be included on the label.

Scope

A series of headings is given in Clause 2 of ISO Guide 31 which indicate the various categories of information to be considered in the preparation of a certificate. An explanation is given under each heading, together with an example where clarification is considered necessary. The headings are intended to cover the required information on the widest possible range of CRMs, including reference materials certified for physical properties, chemical composition, and conventional and biological properties (expressed in units not definable in terms of the SI).

Some information is considered obligatory and should always be given; other details are optional and may be provided if they would enhance the usefulness of the CRM. Not all the obligatory information will be of equal importance in all cases, e.g. the stability of a metal alloy will rarely be questioned, but none of the obligatory items should ever be omitted.

The headings are given in a logical order for presentation of the information which may be summarised as:

- the identity of the producer body and identification of the reference material (Clauses 2.1 to 2.4);
- a description of the material and its intended use (Clauses 2.5 to 2.9);
- the certified values, their traceability and the period of validity of the certificate (Clauses 2.10 to 2.15);
- other information (Clauses 2.16 to 2.18).

The certificate information headings are as follows:

- 2.1 # Name and address of certifying body
- 2.2 # Title of the document
- 2.3 # Name of material
- 2.4 # Reference material code and batch number
- 2.5 # Description of the material, origin and history
- 2.6 Intended use
- 2.7 # Instructions on the correct use of the material
- 2.8 # Safety
- 2.9 Level of homogeneity
- 2.10 # Certified values and their uncertainty intervals at a stated confidence level
- 2.11 # Traceability
- 2.12 Values obtained by individual laboratories or methods
- 2.13 # Uncertified values
- 2.14 # Date of certification or assignment of property values
- 2.15 # Stability
- 2.16 Further information
- 2.17 # Legal considerations
- 2.18 # Signatures and names of certifying officer(s), or officer(s) signing an analytically certificate

Note 1: The items marked # above indicate the minimum information which must be provided, where relevant, on a certificate for reference materials covered by a producer's scope of recognition.

Note 2: Clauses 2.10 and 2.11 are not relevant to reference materials (RMs) and would be replaced by assigned property values or characteristics shown under Clause 2.13.

Note 3: The definition of a CRM in ISO Guide 30 requires property values to be certified by a procedure that establishes traceability of the measured value to an accurate realisation of the unit in which the property values are expressed. The revision of this definition in the second edition of ISO Guide 30 requires all certified property values to be accompanied by an uncertainty interval at a stated level of confidence, and for traceability to "an accurate realisation of the unit in which the property value is expressed" to be demonstrated. These additional requirements should therefore be considered in the preparation of a certificate.

The remainder of the information headings listed in ISO Guide 31 should be provided to users of CRMs in other supporting documentation supplied with the CRM and its certificate.

APPENDIX E

MEASUREMENT TRACEABILITY

Traceability can be understood as the demonstration of quantified links and their uncertainties (evaluated according to the principles of the ISO Guide to the Expression of Uncertainty in Measurement) between the results of a measurement and the value of national or international measurement standards.

Note: ISO Guide 34:1996 (under revision) discusses the assignment of property values and the assessment of uncertainty in reference material production, and the EURACHEM document *Quantifying Uncertainty in Analytical Chemistry 1995* provides guidance on the application to chemical measurements of the ISO Guide to the Expression of Uncertainty in Measurement.

Traceability is not an end in itself. The purpose of establishing traceability of the results of measurements is to ensure that the results can be stated with quantified uncertainties in the appropriate measurement units (usually SI measurement units) so that they are in fact what they are purported to be, and are accurate, are comparable with the values obtained by other measurements made by other methods in other domains, are stable in the long run, and are not subject to systematic errors or extraneous factors.

Note: When measurements are made for legal purposes, the appropriate measurement units are prescribed as legal units of measurement. Demonstration may be required in a court of law of the traceability linkages between the result of a measurement tendered as evidence and the values of national measurement standards.

The smallest uncertainty to SI units in chemical analysis (or in any other field of measurement) is achieved by measurements made using primary methods of measurement, which are correctly applied and stated with evaluated uncertainties. However, in practice, there may be other indirect means of measurement (with their evaluated uncertainties), the results of which are traceable to values of other stated references.

Note 1: Assessors will evaluate traceability of values with regard to the uncertainties and coverage factors required for the intended uses of the reference material.

Note 2: A primary method of measurement is a method having the highest metrological qualities, whose operation can be completely described and understood, for which a complete uncertainty budget can be written down in terms of SI units, and whose results are therefore accepted without reference to a measurement standard of the quantity being measured.

Recognised examples of primary methods of measurement for chemical composition in analytical chemistry include titrimetry, gravimetry, coulometry, isotope dilution mass spectrometry, and certain colligative or physicochemical property determinations, such as freezing point depression.

Note 3: A primary reference material is one having the highest metrological qualities and whose value is determined by means of a primary method of measurement.

Note 4: *indirect methods may require:*

- (a) *use of atomic masses, molar masses or various fundamental constants (e.g. Faraday and Avogadro constants) which have evaluated uncertainties and which link the values of pure materials or well-defined systems to the value of SI units;*
- (b) *combinations of methods for which all uncertainties have been evaluated, incorporating all links to the values of national and international standards expressed in the appropriate SI unit involved;*
- (c) *use of reference materials of the same or similar substance, themselves linked to SI units through a chain of comparisons culminating in a measurement using a primary method and for which all uncertainties, including those due to matrix effects, have been evaluated;*
- (d) *comparison with the values of other standards, methods or instruments which realise, represent or generate accurate chemical composition values which are themselves linked to SI units.*

Note 5: *Approaches using a single method should only be used where appropriate equipment and expertise is available. It is often preferable that property values be confirmed by several collaborators working independently and using more than one method, for each of which a full uncertainty budget and linkages to SI units have been well-established.*

Establishing traceability can sometimes be difficult for the values of reference materials characterised for chemical composition. *Annex A of ISO Guide 34:1996* discusses some of the complexities which may arise in the certification of the values in particular classes of reference materials and states that it is essential to specify to what “appropriate standard” traceability has been established. In many cases it will be possible to establish traceability to the values of national standards of measurement (and hence to SI units) as described above, but in others it may be to a defined scale or a described method.

Note: *ISO Guide 34:1996 and ISO Guide 35:1989 (under revision) make reference to the use of a “single definitive method” as one of the possible procedures for certification of reference materials and defines a “definitive method” as one where the property “is either directly measured in terms of the base units of measurement or indirectly related to the base units through physical and chemical theory expressed in exact mathematical equations.”*

Thus “definitive methods” as understood in these two ISO Guides include primary methods of measurement as described above for the particular case where property values are determined in terms of SI units, but may also include defined scales or described methods where SI units are not appropriate.