GUIDE 31

Reference materials — Contents of certificates and labels

Second edition 2000

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Guides are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft Guides adopted by the responsible Committee or Group are circulated to national bodies for voting. Publication as a Guide requires approval by at least 75 % of the national bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this Guide may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO Guide 31 was drawn up by the ISO Committee on reference materials (REMCO) and was approved by ISO member bodies.

This second edition cancels and replaces the first edition (ISO Guide 31:1981), which has been technically revised.

Introduction

The certificate which accompanies a certified reference material (CRM) should contain all the information which is essential to its use. Without the certificate, the material, however costly its production, is valueless. It follows, therefore, that producers of CRMs should pay very careful attention to the preparation of certificates. The ISO Committee on Reference Materials (ISO/REMCO) published the first edition of this Guide in 1981. During the past sixteen years there has been considerable growth in the number and variety of reference materials produced, and in their use. The increasing demand for reliability in the results obtained by analytical and metrological techniques, which has arisen especially from growing concern about pollution of the environment, has led to the demand for a widening range of CRMs of increasingly high quality for use in validation of measurement methods and as calibrants.

The definition of a CRM in ISO Guide 30 (see clause 2) requires all certified property values to be accompanied by an uncertainty at a stated level of confidence and for traceability to "an accurate realization of the unit in which the property value is expressed" to be demonstrated. These additional requirements must therefore be met in the certificate.

The *Guide to the expression of uncertainty in measurement*, published by ISO (see Bibliography), summarizes more recent international consideration of the subject of uncertainty in measurement and will require some modification of the definition of a CRM quoted above. Uncertainty should now be expressed as combined (type A + type B) standard uncertainty or as expanded uncertainty (with a coverage factor to be applied to the combined standard uncertainty). The concept of probability or level of confidence is now no longer central.

The first edition of this Guide discussed the difference between the information provided on the label, the certificate, and the certification report, and stressed the brief synoptic nature of the certificate. The past sixteen years, however, have seen a general decline in the issuing of certification reports and an increase in the information provided in certificates. This decline in the publication of certification reports is not necessarily to be condemned, provided all the information appropriate to a full certification report can always be obtained on application to the producers of the CRM. Production of certification reports is expensive and it is clearly unnecessary for one to be supplied to the same user every time a fresh sample from the same batch of material is purchased. At the same time, the information required from a certificate is usually more than the certified property value. Details concerning the way in which the container should be opened, the minimum sample size that 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.

Reference materials — Contents of certificates and labels

1 Scope

This Guide is intended to help producers to prepare clear and concise certificates to accompany certified reference materials. Such certificates, while maintaining their essential character, should help to provide, in summary form, all the information needed by the user of the reference material.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this Guide. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this Guide are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Terms and definitions

For the purposes of this Guide, the terms and definitions given in ISO Guide 30:1992 and the following apply.

3.1
certificate
document containing all the information which is essential to the use of a certified reference material

NOTE A CRM and its certificate should never be parted.

3.2
intended use
the use of a product, process or service in accordance with information provided by the supplier

[ISO/IEC Guide 51:1999, definition 3.13.]

3.3
hazardous situation
circumstance in which people, property or the environment are exposed to a hazard(s)

[ISO/IEC Guide 51:1999, definition 3.3.]

4 General

The amount of information which should be included in a certificate depends on the ready availability of a certification report. When a report is always supplied with the certificate, the amount of detail in the certificate can be limited, provided appropriate reference to the report is made. The details given in a certificate and certification report may permit the user to make a judgement of the quality and integrity of the CRM.

Information provided on the label or marked on the material 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, the batch number, and relevant health and safety warnings. It is recommended that the certified property value(s) **not** be included, in order to prevent use of the material without the information in the certificate having been studied.

5 Certificate headings

5.1 General

The various categories of information to be considered in the preparation of a certificate are indicated below. An explanation is given under each heading, together with examples where clarification is considered necessary. The headings are intended to cover the required information on the widest possible range of CRMs which may include reference materials certified for physical properties, amount concentrations of chemical species, isotopic composition (expressed in SI units); and for conventional and biological properties (defined in internationally agreed measurement scales based on internationally agreed measurement procedures, if not defineable in terms of the SI). Some information is considered obligatory and shall always be provided, even though it may not be important in every case, e.g. the stability of a metal alloy or of the isotopic composition of natural compounds will rarely be questioned.

A summary of the information which it is essential to include in a certificate is given in order to assist those organizations, e.g. accreditation bodies, which may wish to include some parts of this Guide in their requirements documents. Other details are optional and may be provided if they would enhance the usefulness of the CRM, e.g. the origin of materials prepared from natural sources.

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

— the general particulars of the certifying body and the reference material (5.2 to 5.5),

— a description of the material and its intended use (5.6 to 5.10), the certified values,

— their traceability and the period of validity of the certificate (5.11 to 5.16),

— other information (5.17 and 5.18), and a summary of the essential contents of a certificate (clause 6).

This Guide, however, is concerned only with the information contained in certificates, and the order or titles of the headings may be changed to suit the preference of the producer.

5.2 Name and address of the certifying body

The name (usually given in prominent type at the head of the certificate) should be that of the body or organization that accepts responsibility for the information in the certificate, i.e. the certifying body. The name should always be accompanied by the full postal address, telephone and fax numbers, and, where available, e-mail address.

5.3 Title of the document

There should be a distinct title, such as *Certificate of Analysis* or *Certificate of Measurement*. The occasional practice of issuing provisional certificates can lead to confusion, with the existence of more than one certificate for the same batch of material, and is to be discouraged.

5.4 Name of material

As far as possible, the name should describe the type of reference material in sufficient detail to distinguish it from other similar materials. Thus the name of the rock or ore, followed by its locality or a compositional characteristic, gives more individuality to geological materials; for example, “Syenite (Phalaborwa)” or “Nepheline syenite". For trace analysis of pollutants in natural matrices it is important to state the nature of the matrix and, if several similar reference materials are available, the level of contamination, for example, "Aflatoxin M1 in whole milk powder

(medium level)". (It may, however, be advisable to avoid a detailed description on documentation supplied to carriers and customs authorities where the naming of toxic pollutants may introduce unnecessary problems with dispatch). For metallurgical samples, it is appropriate to indicate the concentration of the important elements, for example "6Al-4V titanium alloy".

5.5 Reference material code and batch number

Every CRM should have a unique alphanumeric code by which it is distinguishable from any other CRM issued by the same or any other producer, e.g. BCR CRM 186, LGC 7016, NIST SRM 41. In addition, the batch number should be stated, even when it is the first batch of a particular CRM. This is to avoid confusion which may arise when a user laboratory has material from more than one batch in use at the same time. Some producers incorporate the batch number in the alphanumeric code for the material, for example NIST SRM 41c.

5.6 Description of the CRM

The general description of the material should, in effect, amount to a more detailed explanation of the name. The source of the material will be largely irrelevant to its use in most cases but for substances certified for their chemical composition, whether they be prepared from individual constituents, such as alloys, or obtained from natural sources, such as rocks, waters, or products of animal or vegetable origin, the composition of the matrix, especially the presence or absence of substances which may interfere with analytical procedures, may be of considerable importance in the selection of appropriate analytical methods. Even if the source is not disclosed, the approximate composition of the material should always be stated. Certifying bodies should always avoid the situation where failure to disclose relevant information about the source may result in the CRM being used to validate an analytical method applied to the same batch of material as the CRM.

The physical description of the material should also be given, where appropriate, for example, sample size, particle size, dimensions of metal cylinders or discs, and the nature of the container in which it is supplied. The presence of preservatives, such as mercury(II) chloride added to aqueous solutions of ethanol, shall also be stated. Where the same material is also available in alternative forms and sample sizes, this information may also be included.

5.7 Intended use

The primary purpose for which a CRM is issued by the producer should, as far as possible, be stated but, provided the certified properties do not relate only to a particular analytical or measurement procedure (for example, elemental analysis of a mineral after a prescribed leaching procedure or measurement of flash point by a carefully defined method), this statement is not intended to restrict the use for other purposes. The certificate, or additional information provided in the certification report or otherwise supplied, must, however, provide sufficient information for the user to be able to decide whether the proposed application of the CRM is justified.

Examples of intended use are:

— for the realization of a fixed-point temperature on the international temperature scale (ITS-90) and, therefore, for the calibration of temperature-measuring equipment;

— for the calibration of instruments for determining the amount concentration of precious metals in ore sample;

— for the validation of analytical methods used to determine the amount concentration of pollutants in natural matrices;

— as an arbitration sample for commercial transactions;

— for the preparation of "working reference samples" for use in routine laboratory analyses.

5.8 Instructions for the correct use of the reference material

It is essential that the reference material is used under the conditions prescribed in the certificate. For example, if drying is necessary the exact conditions, for example for 2 h at 105 °C, shall be stated. It may be necessary to open the container under prescribed conditions. For example, materials stored at sub-ambient temperatures should be allowed to warm to laboratory temperature before the container is opened in order to prevent contamination by condensed water vapour; material stored under an inert atmosphere should be opened only in a glove-box containing a similar inert atmosphere. Appropriate cautions should be given in such cases. Unless prescribed in the certificate, CRMs should not be subjected to further comminution procedures such as grinding, and, especially in the case of materials certified for their content of trace elements, a warning should be given against the use of any equipment containing these elements.

Special instructions may be necessary when the use of a CRM involves reconstitution of a solution from a solid CRM, particularly in the field of clinical chemistry. The nature of the solvent, temperature, mixing procedure, delay before use, and stability of the solution shall be carefully specified.

It is essential to state the conditions under which the material shall be stored (e.g. temperature, exposure to light) in order to maintain the validity of the certificate. In the case of materials which are inherently unstable, such as radioactive substances, the appropriate mathematical expression for calculating the value of the property at the time of use should be included as part of the statement of the certified value.

5.9 Hazardous situation

Where information is available on the safety of a CRM, this should be included in both the label and the certificate. Details concerning the nature of any hazard and appropriate precautions to be taken should be provided in a data sheet accompanying the material.

5.10 Level of homogeneity

Most CRMs are supplied in amounts from which sub-samples are taken for chemical analysis or measurement of some physical property, and the sub-sampling presupposes that the supplied sample is sufficiently homogeneous for the intended use. The certificate should state the minimum size of sub-sample which should be taken by users of the CRM. This should be accompanied by a warning that taking a smaller sub-sample will invalidate the use of the certified value of the property and the associated statement of uncertainty. Where appropriate, the certificate may require agitation of the container by a specified procedure to ensure sufficient homogeneity of the sample.

Details and results of the procedure by which the level of homogeneity of the material has been evaluated and on which the statement of minimum sub-sample size is based should be available to users, either included in the certificate (or certification report) or provided on request by the certifying body.

5.11 Certified values and their uncertainties

There should be a clear statement of the property and its certified value, together with the uncertainty of the certified value, according to the procedures in the *Guide on the expression of uncerainty in measurement* [8], and Eurachem [2]. The method of evaluating the certified value and assessing its uncertainty should be stated. Where a purely statistical treatment of the results of a measurement has been used, the method of treatment should be indicated. Frequently, however, especially when several measurement techniques have been employed, the specialist experience and expertise of the certifying body can be used to derive the certified value and its uncertainy from the results of the measurements. The reasons for weighting some results more heavily than others should always be given.

5.12 Traceability

The definition of a CRM in ISO Guide 30 requires the certified property values to be certified by a procedure that establishes traceability to an accurate realization of the unit in which the property values are expressed. Traceability is defined as the property of the result of a measurement whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated

uncertainties [3]. The characterization of a CRM should, therefore, ideally be traceable to the SI or to an internationally agreed measurement scale.

In the case of CRMs for physical properties, it is usually possible to establish an unbroken chain of instrument calibrations which relate the measurement to base units of the SI, realized in a national metrology laboratory. The problem is greater for chemical measurements where the methods used may range from primary methods of measurement (defined as those having the highest metrological qualities and whose operation can be completely described and for which a complete uncertainty statement can be written down in terms of SI units [4], (e.g. isotope dilution mass spectrometry, coulometry and gravimetry), to well-defined methods for which traceability to an internationally agreed measurement scale can be demonstrated. Every certificate should, however, contain a statement where the principles of the measurement procedures are clearly stated, together with evidence of their validity and the measurement scale to which they are traceable. Traceability of chemical measurements is more fully discussed in references [5], [6] and [7].

5.13 Values obtained by individual laboratories or methods

Where several methods have been used to characterize the reference materials, these should be stated. It is often sufficient to refer to a general method, such as atomic absorption spectroscopy or X-ray fluorescence, but where a less general method or modification of a standard method has been used, a complete literature reference or description should be given. Where several laboratories or independent analysts have contributed to the characterization of a reference material, their names should be listed, together with the methods they have used. The property values obtained by each method, and by each practitioner of the method where several laboratories or analysts have used the same method, may, in some cases, also be separately reported. Opinions differ on the advisability of this practice, which is optional and may be left to the discretion of the producer. On the one hand it enables users to assess the quality of the CRM on the basis of the concordance of the individual results and of their knowledge of the measurement techniques involved but, on the other hand, it may tempt users to make their own assessment of the property value, but without the experience and expertise of the certifying body. Such misuse of the information in a certificate would invalidate the use of the material as a CRM. Full details of the results from each method shall, in any case, be available from the producer in the form of a certification report or otherwise provided.

Some reference materials are certified for properties whose values depend on the method used to measure them, e.g. the elemental content of a mineral where values depend on the method of leaching, or flash point determinations or values of hardness which are wholly dependent on the method used. In such cases the certificate should give an explicit warning that the certified values are method-dependent, and give full details of the method used or a reference to a publication in which the method is fully described.

5.14 Uncertified values

During the course of characterization of a reference material, producers often obtain values of other properties of the material which do not fulfil the criteria required of certified properties. Such information, for example the approximate amount concentration of other elements in a complex matrix, may be useful to users of the CRM and may be included in the certificate. It should, however, be clearly distinguished from certified values and, to avoid possible confusion, not included in the same table as the values of certified properties.

5.15 Date of certification

The date on which the certificate was first issued should be stated. Where a certificate contains revised values for the same batch of a CRM, the date of the original certificate and of all revisions should be given.

5.16 Period of validity

The certification may contain an expiry date after which the certified value is no longer guaranteed by the certifying body. This should be the practice for all materials where instability has been demonstrated or is considered possible. When an expiry date is given the certificate should contain an assurance that the certified value(s) will be monitored at appropriate intervals and that purchasers will be notified of any significant changes resulting in re-certification or withdrawal of the CRM during the stated period of validity of the certificate. Even when no expiry date is given and unexpected changes in certified value(s) are detected, purchasers should be informed when