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Guidance for the in-house preparation of quality control materials (QCMs)

Lignes directrices pour la préparation interne des matériaux de référence utilisés pour le contrôle qualité

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/REMCO, *Committee on reference materials* (which has the task to prepare guidance documents for the preparation, characterization, certification and use of reference materials (RMs) and the competence assessment of reference material producers.

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Introduction

Reference materials (RMs) are widely used in measurement laboratories for a variety of purposes and it is important to recognize that the material most appropriate for a particular application should be used. Certified reference materials (CRMs), i.e. those which have property values and associated uncertainties assigned by metrologically valid procedures are primarily used for method validation and calibrations providing metrological traceability.

The preparation of reference materials for metrological quality control (i.e. control of the quality of measurements not products) is an important activity which provides materials suitable for the day-to-day demonstration that a particular (part of a) measurement system is under statistical control. Such materials do not require characterization by metrologically valid procedures, and can be prepared "in-house", i.e. by laboratory staff familiar with their behaviour, to fulfil specific quality control requirements.

Reference materials which are sufficiently homogeneous and stable are necessary for metrological quality control purposes, such as demonstrating a measurement system is under statistical control, performs as expected and provides reliable results; where the trueness of the measurement result is not critical. Different industries use various terminologies to describe such materials (e.g. in-house reference materials, quality control materials, check samples, etc.). For the purposes of this Guide, the term "Quality Control Materials" (QCMs) will be used to simplify repeated citation.

While CRMs are produced by established reference material producers and are commercially available, QCMs are often prepared by a laboratory for its own internal use. Frequently, QCMs are characterized only for a limited scope (a limited number of property values) and for specific laboratory applications.

The rationale for preparing quality control materials can be one or a combination of the following factors:

— to have an RM representing as closely as possible routine samples, suitable for quality control;

— to have a suitable day-to-day RM to complement a commercially available CRM;

— no suitable CRM exists;

— the application does not require a material having the full characteristics of a CRM (e.g. traceability and uncertainty for specified property values).

QCMs are RMs and as such the requirements of ISO Guide 34[1] for the production of RMs apply. However, if the material is only used in-house by the preparing laboratory, some requirements (e.g. for transport stability) can be relaxed. The preparation of a QCM is related to that of a CRM and those preparing QCMs may wish to consult ISO Guides 34[1] and 35[2] for further guidance. Where appropriate, this Guide will refer to relevant parts of these Guides.

It is recognized that the aim of many laboratories requiring QCMs is to minimize the time and effort needed to prepare the materials. To this end, many laboratories use samples of real products for which there is a body of analytical data available. A number of case studies are included as annexes of this guidance document to provide examples of how such data may be processed to confirm fitness for purpose of the materials.

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Guidance for the in-house preparation of quality control materials (QCMs)

1 Scope

This Guide outlines the essential characteristics of reference materials for quality control (QC) purposes, and describes the processes by which they can be prepared by competent staff within the facility in which they will be used (i.e. where instability due to transportation conditions is avoided). The content of this Guide also applies to inherently stable materials, which can be transported to other locations without risk of any significant change in the property values of interest.

The primary audience for this Guide is laboratory staff who are required to prepare and use materials for specific in-house quality control applications. Preparation of QCMs, where transportation is a necessary component of the supply chain, such as laboratory sites at different locations or for proficiency testing schemes, should conform to the relevant requirements of ISO Guides 34[1] and 35.[2]

The description of the production of reference materials (RMs), as detailed in ISO Guide 34[1] and ISO Guide 35[2] is also applicable to the preparation of quality control materials (QCMs). However, the requirements for "in-house" QCMs are less demanding than those for a certified reference material (CRM). The preparation of QCMs should involve homogeneity and stability assessments, and a limited characterization of the material to provide an indication of its relevant property values and their variation, prior to use. This document provides the quality criteria that a material should fulfil to be considered fit-for-purpose for demonstrating a measurement system is under statistical control. Guidance on uses of such materials, for example setting up a QC chart, is adequately covered elsewhere [3],[4],[5],[6] and is not included in this Guide.

The layout and structure of this Guide provides general information on the preparation of QCMs in the main chapters, with specific case studies covering a range of sectors in the annexes. The case studies are not complete "process manuals" but are included to highlight some of the key considerations when preparing QCMs. The case studies vary in complexity and detail, including sector specific terminology, but provide a range of information for laboratory staff to draw from.

It is expected that those involved in QCM preparation will have some knowledge of the type of material to be prepared and be aware of any potential problems due to matrix effects, contamination, etc.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO Guide 30, *Reference materials — Selected terms and definitions*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO Guide 30[7] ISO/IEC Guide 99[8] and ISO 3534-1[9] and the following apply.

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3.1
indicative value
value of a quantity or property, included in the certificate of a CRM or otherwise supplied, which is provided for information only (i.e. is not certified by the producer or the certifying body)

Note 1 to entry: Values assigned to quality control materials (QCMs) can only be indicative in that they have no metrological traceability. ISO Guide 30:1992[7] uses the term "uncertified value" to describe a value of a quantity provided for information only.

4 Quality control materials (QCMs)

The term "quality control material" or "QCM" has been devised for the purposes of this Guide solely to simplify repeated reference to materials used routinely to assess the precision of test procedures. It is not intended to define a new class of reference materials. Such materials are variously referred to in the open literature as "in-house reference materials", "quality control samples", "check samples", "set up samples", etc.

Where no suitable CRM exists, laboratories may use QCMs to provide an assessment of the repeatability / intermediate precision / reproducibility of a measurement result. QCMs cannot be used to establish metrological traceability or trueness of a measurement result.

QCMs should always comply with the basic requirements of any reference material, i.e. they should be sufficiently homogeneous and stable with respect to the properties of interest. The level of heterogeneity should be less than the expected standard deviation of the measurement process or an established criterion value against which the assessment of laboratory performance or the "normalization" of results is acceptable. The QCM should be stable for a period of time that is at least as long as that during which it is intended to be used.

5 Applications of quality control materials (QCMs)

The principal function of QCMs is to provide laboratories with an economical means of checking their routine test procedures for precision on a regular basis (e.g. daily, weekly or monthly).

While CRMs can in all cases replace QCMs, QCMs are not replacements for CRMs; they are complementary to them having a specific, limited purpose in the measurement process. CRMs produced according to the principles of ISO Guide 34[1] are essential to establish the concept of metrological traceability in a meaningful manner, and provide the highest standard with respect to reference materials. There is no requirement for QCMs to have metrologically traceable assigned values; consequently, QCMs cannot be used to establish metrological traceability or to estimate uncertainty. For method validation and uncertainty estimation, QCMs may be used to a limited extent (e.g. for establishment of a precision estimate as part of the total measurement uncertainty).

Uses of QCMs include (but are not limited to):

— preparation of QC charts – to demonstrate control of a measurement process within a laboratory or to confirm the effectiveness of a laboratory's quality control process or to demonstrate control of a measurement process over a period of time;

— comparison of results (e.g. from two or more series of related samples either in a short period of time or over an extended period of time when a measurement process is known to vary);

— method development – to establish consistency (for validation a certified reference material should be used);

— instrument performance checks;

— repeatability and reproducibility studies – repeated use over an extended period of time, instruments, operators, etc., to estimate long-term reproducibility or robustness of a measurement process or laboratory;

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