PD ISO GUIDE 33:2015

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Reference materials — Good practice in using reference materials

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National foreword

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Contents

Page

Foreword ... **v**
Introduction ... **vi**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms and definitions ... **2**
4 Symbols ... **3**
5 Conventions ... **3**
6 RMs and their role in measurement ... **4**
6.1 Common applications of RMs ... 4
6.2 Property values ... 6
6.2.1 General ... 6
6.2.2 Specification of the property ... 6
6.3 Uncertainty statement ... 6
6.4 Traceability statement ... 8
7 Handling of RMs and CRMs ... **9**
8 Assessment of precision ... **9**
8.1 General ... 9
8.2 Number of replicate measurements ... 9
8.3 Requirements with respect to the RM ... 10
8.4 Measurement ... 11
8.5 Data treatment ... 11
8.6 Calculation and assessment of precision ... 12
9 Bias assessment ... **12**
9.1 General ... 12
9.2 Approach to bias checking ... 13
9.3 Utilizing bias data ... 13
10 Calibration ... **14**
10.1 General ... 14
10.2 Establishing metrological traceability ... 15
10.3 Calibration models ... 15
11 Assigning values to other materials ... **15**
11.1 General ... 15
11.2 Pure materials ... 16
11.3 Gravimetry and volumetry ... 17
12 Conventional scales ... **18**
12.1 General ... 18
12.2 pH-scale ... 19
12.3 Octane number ... 19
13 Selection of CRMs and RMs ... **20**
13.1 General ... 20
13.2 Selection of a CRM ... 20
13.3 Selection of RMs ... 23
13.4 Relevance to the measurement system ... 23
Annex A (informative) **Key characteristics of a reference material with respect its common applications** ... **25**
Annex B (informative) **Calibration models and associated uncertainty models** ... **26**
Annex C (informative) **Decision errors** ... **29**

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Bibliography **30**

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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*.

This third edition cancels and replaces the second edition (ISO Guide 33:2000), and ISO Guide 32:1997 which have been technically revised.

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Introduction

The aim of this Guide is to provide general recommendations on the use of RMs. These recommendations are exemplified by real-world examples, which to some degree also reflect the level of complexity associated with RMs. This level of detail is deemed to be useful for anyone who has a responsibility in the quality management in laboratories, such as drafters, reviewers, managers, and assessors of procedures, working instructions, standard operating procedures and the like.

The main applications of reference materials are calibration, establishing traceability, method validation, assigning values to other materials, and quality control.

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Reference materials — Good practice in using reference materials

1 Scope

1.1 This Guide describes good practice in using reference materials (RMs), and certified reference materials (CRMs) in particular, in measurement processes. These uses include the assessment of precision and trueness of measurement methods, quality control, assigning values to materials, calibration, and the establishment of conventional scales. This Guide also relates key characteristics of various types of RMs to the different applications.

1.2 For CRMs, the metrological traceability of the property values to international scales or other measurement standards has been established. For RMs not being CRMs, this kind of traceability of property values has often not been established. Nevertheless, these RMs can still be used for assessing parts of measurement procedures, including evaluating various levels of precision

1.3 Mainstream applications of RM include precision control (Clause 8), bias assessment (Clause 9), calibration (Clause 10), preparation of calibration RMs (Clause 11) and maintaining conventional scales (Clause 12).

NOTE Not all types of RMs can be used for all indicated purposes.

1.4 The preparation of RMs for calibration is also part of the scope of ISO Guides 34[1] and 35[2]. The treatment in this Guide is limited to the fundamentals of small-scale preparation of RMs and the value assignment, as used by laboratories to calibrate their equipment. Larger scale production of such RMs, with the possible aim of distribution is beyond the scope of this Guide. This type of activity is covered in ISO Guides 34[1] and 35[2].

1.5 The development of working standards, as used in, e.g. natural gas analysis, clinical chemistry, and the pharmaceutical industry is not covered in this Guide. This type of activity is covered in ISO Guides 34[1] and 35[2].

2 Normative references

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

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

NOTE The "Guide to the expression of uncertainty in measurement" is referred to as "GUM", whereas the "International vocabulary of basic and general terms in metrology" is referred to as "VIM".

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3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO/IEC Guide 98-3, ISO/IEC Guide 99 and ISO Guide 30 and the following apply.

NOTE Further definitions can be found in the ISO online browsing platform, accessible through https://www.iso.org/OBP/ui/

3.1
reference material
RM
material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

Note 1 to entry: RM is a generic term.

Note 2 to entry: Properties can be quantitative or qualitative, e.g. identity of substances or species.

Note 3 to entry: Uses may include the calibration of a measurement system, assessment of a measurement procedure, assigning values to other materials, and quality control.

Note 4 to entry: ISO/IEC Guide 99:2007, has an analogous definition (5.13), but restricts the term "measurement" to apply to quantitative values. However, Note 3 of ISO/IEC Guide 99:2007, 5.13 (VIM), specifically includes qualitative properties, called "nominal properties".

[SOURCE: ISO Guide 30]

3.2
certified reference material
CRM
reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

Note 1 to entry: The concept of value includes a nominal property or qualitative attribute such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities or levels of confidence

Note 2 to entry: Metrologically valid procedures for the production and certification of RMs are given in, among others, ISO Guides 34 and 35.

Note 3 to entry: ISO Guide 31[17] gives guidance on the contents of RM certificates.

Note 4 to entry: ISO/IEC Guide 99:2007 has an analogous definition (5.14).

[SOURCE: ISO Guide 30]

3.3
property value
<of a reference material> value corresponding to a quantity representing a physical, chemical or biological property of a reference material

[SOURCE: ISO Guide 30]

3.4
certified value
value, assigned to a property of a reference material (RM) that is accompanied by an uncertainty statement and a statement of metrological traceability, identified as such in the RM certificate

[SOURCE: ISO Guide 30]

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3.5
indicative value
information value
informative value
value of a quantity or property of a reference material, which is provided for information only

Note 1 to entry: An indicative value cannot be used as a reference in a traceability chain

[SOURCE: ISO Guide 30]

3.6
calibrant
reference material used for calibration of equipment or a measurement procedure

[SOURCE: ISO Guide 30]

2.7
quality control material
reference material used for quality control of a measurement

[SOURCE: ISO Guide 30]

4 Symbols

α	risk of error of the first type (type I error)
β	risk of error of the second type (type II error)
χ^2	Chi-squared
d	measurement bias
k	coverage factor
s_{w}	standard deviation computed from repeat observations
σ_{wo}	required within-laboratory standard deviation
$u()$	standard uncertainty of the parameter in parenthesis
$U()$	expanded uncertainty of the parameter in parenthesis
u_{CRM}	standard uncertainty associated with property value of the CRM
u_{meas}	standard uncertainty associated with value obtained by measuring the CRM
u_{prep}	uncertainty associated with the value obtained from preparation of a calibrant
x_{CRM}	value of a specified property of the CRM
x_{meas}	value obtained by measuring the CRM
x_{prep}	value obtained from preparation of a calibrant
$\bar{x}$	average of repeat observations

5 Conventions

In this Guide, the following conventions are used.

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5.1 A measurand is specified in such a way that there exists a unique, but unknowable, 'true value'.

5.2 All statistical methods used in this Guide are based on the following assumptions.

a) The certified value is the best estimate of the true value of the property of the CRM.

b) All variation, be it associated with the material (i.e. homogeneity) or the measurement process, is random and follows a normal probability distribution. The values of probabilities stated in this Guide assume normality. The probability may be different if there is deviation from normality.

5.3 The concept of "certified reference material" (CRM) as used in this Guide also includes RMs with property values that are accompanied by the statements of metrological traceability or measurement uncertainty. These property values are assumed to be obtained through characterization as described in ISO Guides 34[1] and 35[2].

5.4 Where the term RM is used in this Guide, it means that any RM can be used for the purpose described. The use of a CRM is an option, but usually not the most economical one. In practice, in most cases an RM will be used that comes without property values, uncertainties and a traceability statement.

5.5 Values, given as "indicative", "informative", "for information" or otherwise identified as not being covered by the statements of metrological traceability or measurement uncertainty, are considered to be unfit for use in metrological applications requiring a value assigned to the measurand, such as, calibration, or the assignment of values to other materials. These values are however useful to verify whether an RM is suitable for precision control, or other applications that do not require a property value.

5.6 Throughout this Guide, the law of propagation of uncertainty is used. Other methods of propagating uncertainties can be applied as well, and in some cases such alternative methods are required by the circumstances of the application. Further guidance on these matters is given in the GUM and its supplements.

6 RMs and their role in measurement

6.1 Common applications of RMs

6.1.1 RMs, and CRMs in particular, are widely used for the following purposes:

— Calibration of equipment or a measurement procedure (Clause 10);

— Establishing metrological traceability (Clauses 9, 10 and 11);

— Method validation (Clauses 8 and 9);

— Assigning values to other materials (Clause 11);

— Quality control of a measurement or measurement procedure (Clause 8 and 9);

— Maintaining conventional scales (Clause 12).

Figure 1 gives an outline of a measurement, including sampling and sample preparation. The possible role(s) of CRMs are indicated.

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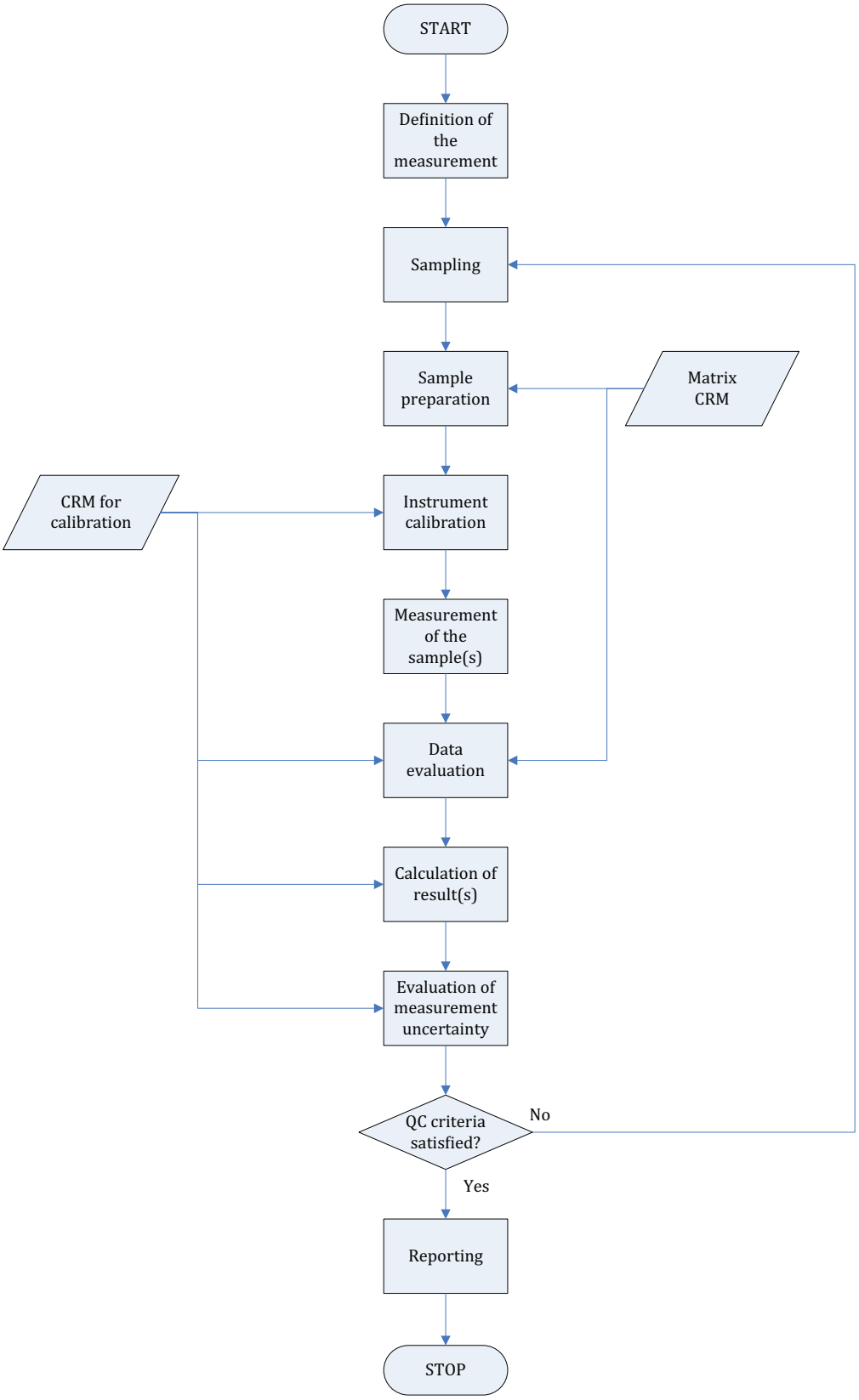

Figure 1 — Schematic outline of a measurement and the role of CRMs therein

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