TECHNICAL REPORT

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First edition

# Reference materials — Establishing and expressing metrological traceability of quantity values assigned to reference materials

*Matériaux de référence — Etablissement et expression de la traçabilité métrologique de valeurs assignées à des matériaux de référence*



PROOF/ÉPREUVE

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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).

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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

ISO/TR 16476 was prepared by the ISO Committee on Reference Materials (ISO/REMCO).



 

# Introduction

Reference materials (RM), in particular when certified (CRM), are a major tool for assuring the quality and reliability of results obtained in measurement and testing. CRM property values, in particular used for assessing the trueness of a measurement procedure as implemented in a laboratory, also establish traceability of the measurement result. Which reference the property values assigned to (C)RM should be traceable to, and how this traceability should be established, demonstrated, and reported on certificates is, therefore, a question of primary importance, mainly for RM producers. However, users of (C)RMs should also know what the endpoint of their traceability chain is, in particular for all purposes of cross-border acceptance of measurement results.

It was therefore considered necessary to conduct a study into existing principles for, and requirements to, the traceability of (C)RM, in particular with a specific view to the current definition of metrological traceability given by the Vocabulary of International Metrology (VIM), edition 3, 2007.

# Reference materials — Establishing and expressing metrological traceability of quantity values assigned to reference materials

## 1 Scope

This Technical Report investigates, discusses, and specifies further, the general principles of establishing traceability of measurement results laid down in the Joint BIPM, OIML, ILAC and ISO Declaration on Metrological Traceability [1], in particular for values assigned to (certified) reference materials. The document covers the following topics:

a) a study into existing principles for, and requirements to, the traceability of the value assigned to the property of a (C)RM, with a specific view to the current definition of metrological traceability given by the 2007 edition of the VIM (published also as JCGM 200:2008[2] and ISO/IEC Guide 99:2007[21]);

b) the development of a sensible, widely applicable approach to the understanding of the traceability of a value assigned to (C)RM property;

c) recommendations on how traceability should be established, demonstrated, and reported on certificates and other documents accompanying (C)RM.

The developed approach is exemplified for measurement procedures not covered earlier by other guidance documents on the topic.



## 2 The VIM definition of metrological traceability

The recent edition of the VIM[2],[21] defines *metrological traceability* (term 2.41) as

| property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty | |
|---|---|
| NOTE 1 | For this definition, a 'reference' can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard. |
| NOTE 2 | Metrological traceability requires an established calibration hierarchy. |
| NOTE 3 | Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed. |
| NOTE 4 | For measurements with more than one input quantity in the measurement model, each of the input quantity values should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network. The effort involved in establishing metrological traceability for each input quantity value should be commensurate with its relative contribution to the measurement result. |
| NOTE 5 | Metrological traceability of a measurement result does not ensure that the measurement uncertainty is adequate for a given purpose or that there is an absence of mistakes. |

| | |
|---|---|
| NOTE 6 | A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and, if necessary, correct the quantity value and measurement uncertainty attributed to one of the measurement standards. |
| NOTE 7 | The ILAC considers the elements for confirming metrological traceability to be an unbroken metrological traceability chain to an international measurement standard or a national measurement standard, a documented measurement uncertainty, a documented measurement procedure, accredited technical competence, metrological traceability to the SI, and calibration intervals (see ILAC P-10:2002). |
| NOTE 8 | The abbreviated term "traceability" is sometimes used to mean 'metrological traceability' as well as other concepts, such as 'sample traceability' or 'document traceability' or 'instrument traceability' or 'material traceability', where the history ("trace") of an item is meant. Therefore, the full term of "metrological traceability" is preferred if there is any risk of confusion. |

NOTE 7 makes clear that the measurement method/procedure is a part of the traceability statement, but insufficient if taken alone. This implies that a measurement result or the assigned value of a (C)RM can be traceable to a method or a series of methods used, but not to the method alone, although such statements can still be found on CRM certificates. Without any doubt, the measurement procedure used will mostly be reflected in the definition of the measurand, but additionally, the value assigned to the measurand should be made traceable to stated references given the procedure applied, thus, the latter cannot be the endpoint of the traceability chain for the assigned value. Recent presentations on the topic (e.g. Reference [3]) support this viewpoint.

As expressed in NOTE 2, the definition as given above is governed by the (assumed) existence of a straightforward, single-route top-down reference standard hierarchy. Reference [4], as a guidance to implement the above VIM definition in chemistry, almost always assumes the existence of higher-order reference materials, conveniently certified at the highest level by using allegedly primary methods. This Technical Report does not go into further details of these situations since References [4] to [6] provide sufficient guidance. Considerations and guidance on traceability hierarchies together with graphs visualizing and illustrating traceability chains, including branched ones, can also be found in Reference [7].

The described philosophy works fine for all levels which still have a "higher-order" level above, or fields where primary methods exist and can readily be used for a valid and reliable value assignment to the measurand. However, at some point, the top with no "higher-order" is reached. It may also be stated that for a huge amount of certified reference materials at this level, no primary method is available for assigning values to the measurands. This causes the general problem of traceability for property values of (C)RMs allocated at prominent places in the hierarchy. More critical points will be discussed under Clause 3.

NOTE An annotation document is being developed. Its aim is to give further explanations to the VIM definitions; it will also provide advice regarding the application of these definitions.

# 3 Challenges arising from the definition of metrological traceability

## 3.1 Conventions

For the purposes of this Technical Report, the following conventions apply.

— "Traceability of an RM"

is in common and daily use, it is understood throughout as the traceability of the quantity value assigned to a (certified) reference material.

— "(Analytical) method"

is used in the sense of defining the instrumental implementation of the (most often physical) principle of obtaining, from an appropriately pre-processed and/or transformed object under investigation, a signal (subject to further processing) reflecting the sought-after property. Such implementations are, for example, ToF-IDMS, GC-FID, LC-MS/MS, HPLC-DAD, FT-IR, etc.

— "Measurement protocol"

is used to refer to measurement procedures prescribed or standardized to an extent that the value(s) assigned to the material becomes senseless without direct reference to these prescriptions, i.e. where not only the *conditions* under which measurements have to be taken but *also form, structure, shape, size* and/or *composition* of the specimen are prescribed.

— "RM document", sometimes also called "property value sheet" or "product information sheet" (see ISO Guide 31)

is used as an analogue to, and distinction from, the term "certificate" as defined in ISO Guide 31. Certificates refer to CRM, while an RM document provides the necessary information on the properties of a (non-certified) RM.

— "Matrix (C)RM"

an RM made out of natural-born substance(s) or synthetically re-constituted ingredients, characterised for composition.

— "Property (C)RM"

an RM characterized for a property other than the content of main components and/or impurities as e.g. tensile strength or Charpy impact for an alloyed steel.

NOTE This Technical Report refers to the requirements of ISO Guide 34, in force at the time of publication of this Technical Report. For traceability issues, the future ISO 17034 will also follow the principles of the named ISO Guide 34.



### 3.2 (C)RM as the carrier of traceable values

In the context of (C)RM production, a basic problem of the definition of metrological traceability (see Clause 2) is that it refers to traceability of a result of a measurement. (C)RMs are normally considered as artefacts providing traceability of a measurement result.

NOTE An RM which comes without a (measured) value attributed to its properties, e.g. in cases where the material has an intended purpose not requiring such attributed value, does not experience the need of establishing traceability.

The value and uncertainty carried by an (C)RM are, in virtually almost all cases, combinations of results of various measurements. These results may refer to the different steps of RM production, namely homogeneity and stability estimation, and to measurements taken in the characterization step based upon independent implementations of the same measurement procedure, or implementations of different independent measurement procedures.

Even in cases when all of the single results obtained in an RM certification are traceable, it remains unclear to which extent and endpoint of the traceability chain a combined result is traceable. This problem increases considerably if the results to be combined are traceable to different endpoints, or at least via different pathways all having different lengths and reliabilities.

EXAMPLE Karl Fischer titration and the oven (drying) method have different endpoints of their traceability chains. It might be sensible to include the method in the definition of the measurand which solves the problem of the traceability endpoint.

However, for the seemingly clear specification "water in a matrix", the above mentioned problem arises. A sensible traceability statement for the value combined from results of both methods might be based upon the more direct oven method (see also 5.3.2).

## 3.3 Implicit traceability to the unit of the measurement scale

The measurement procedure is a convention, it most often also includes transformation(s) of the measurand. This holds for most areas in chemistry, biology, or life sciences. Different conventions (i.e. different measurement procedures) for the same measurand may lead to different results, i.e. they turn out to be incompatible. This is reflected in NOTE 5 to the definition saying that traceability is a necessary condition for *comparability* of measurement results, but insufficient for their *compatibility*.

Generally speaking, the measurement procedure has a non-negligible influence on the value assigned. The principal approach is that traceability can only be established "given the specified measurement procedure used". The specification of this procedure in a written standard, an SOP, etc., is a nominal prescription. Any implementation in a specific place, by a specific operator using specific equipment will cause inevitable deviations from the prescription, no matter whether these are negligibly small or introduce a real contribution to the total uncertainty. The deviations should be assessed in specific investigations virtually considered as calibrations against the nominal prescription.

EXAMPLE ISO 148-3 describes the production of samples for Charpy impact tests. ISO 148-3 provides nominal values for the size of the sample and the location and shape of the V notch. Despite the fact that the instrument measuring the size (e.g. a calliper) should duly be calibrated and introduces a measurement uncertainty, the machining tool also has a certain variability. Both should be considered.

NOTE In chemical analysis, a ruggedness test as part of a thorough method validation assesses most of the deviations occurring from implementation of a method under real, and varying within specified limits, laboratory conditions.

All of these calibrations will normally not introduce real corrections to the measured value but contribute to the overall uncertainty which makes the approach compatible with the VIM definition.

It is commonly accepted that the combination of metrological traceability and proper measurement uncertainty is the only way how measurement results can legitimately be compared.[8] Moreover, measurement uncertainty estimation of the calibration steps is a mandatory prerequisite for the establishment of traceability. The concept of calibration against a nominal requirement closes the gap in cases where the routes to measurement scales (SI and others) are considered "indirect". Demonstration of compliance with nominal requirements has to be carried out using measuring instruments which are, for the measurand they tackle, traceable to the corresponding unit of the scale (callipers, balances, volumetric flasks, etc.). This concept is formalised as approach B under Clause 4.

The significance and influence of indirect pathways to measurement scales is also recognized in the Joint Declaration on Metrological Traceability[1] stating that "*In general, ... references are the International System of Units (SI), but where such traceability is not yet feasible, measurement results should be traceable to other internationally agreed references...*"

It might seem viable to attribute all the peculiarities of the measurement method or procedure to the definition of the measurand as proposed in Reference [9]. In general, one should remember that the VIM defines the measurand as the "quantity intended to be measured", not as the procedures necessary for accomplishing the intention. At the same time, the faults that might happen when the quantity is measured are not considered.

Two other points have to be considered.

— Firstly, the approach of Reference [9] will work only with one single method of determination which then (according to the requirements of ISO Guide 34) should be a primary method, a restriction limiting the applicability of the approach to special cases.

— Secondly, it will limit the field of application of the material or its commutability and, thus, considerably reduce its technical and commercial value.

A sensible and balanced distribution of method impact on the measurement result between the definition of the measurand and the traceability chain(s) to units of scale is therefore crucial (see also 5.4).



 

### 3.4 Traceability networks

NOTE 4 in the definition of *metrological traceability* (Clause 2) suggests that for measurements with more than one input quantity in the measurement model (a situation which is daily practice in chemical analysis and virtually all fields of testing), each of the input quantity values should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network. The effort involved in establishing metrological traceability for each input quantity value should be commensurate with its relative contribution to the measurement result.

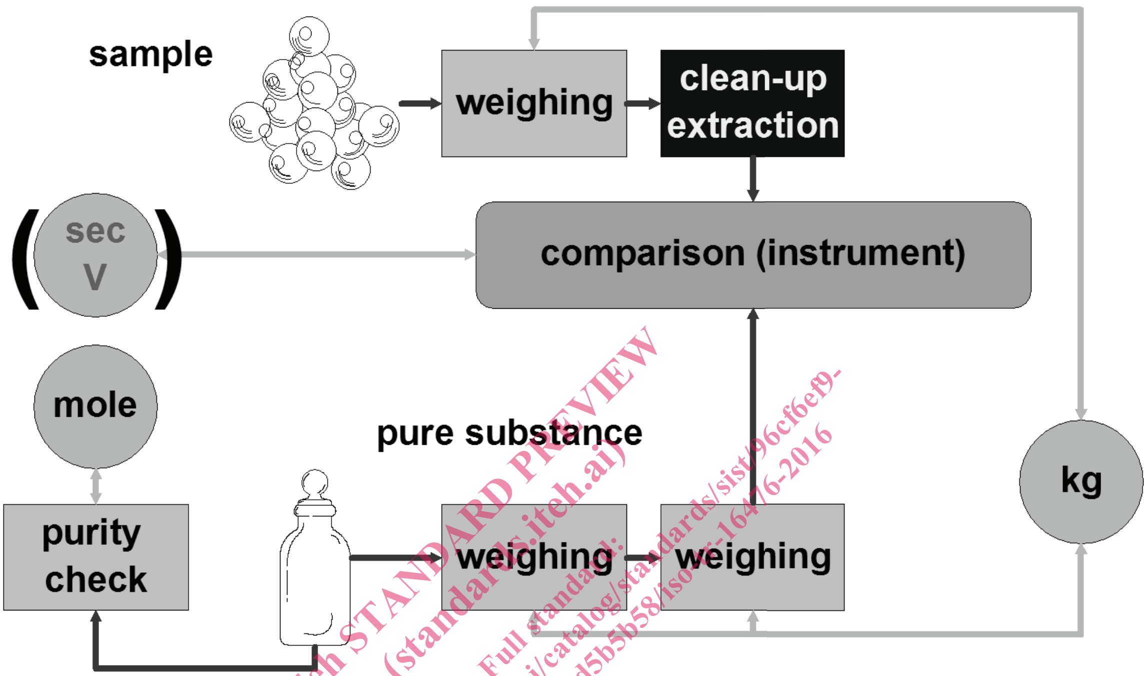


**Figure 1 — "Horizontal" traceability network relating the measured quantities in a measurement procedure model to a set of SI units**



It is assumed that this explicitly allows a "horizontal" networked strategy for establishing the traceability of a particular implementation of a measurement procedure as visualized in Figure 1. The concept formalized under approach B of Clause 4 is a consequence of NOTE 4 to the definition of metrological traceability and implements this "horizontal" strategy. It is the only feasible approach when no higher-order reference is available in the vertical direction.

### 3.5 Properties expressed in units of measurement scales other than the SI

Most of the scales other than the SI have nevertheless similarities with the latter, namely that they are realised by a single or a set of materialisations/artefacts with assigned values expressed predominantly in real numbers (e.g. pH scale). Furthermore, fractions or multiples of the basic unit exist. Some scales use ordinal numbers which express a cardinal "smaller-larger" relationship between the realizations of the points on the scale (e.g. Mohs hardness, see Reference [10]) rather than an explicit proportionality or a counting result. Here, specific problems with the resolution of the scale may arise. However, for establishment of traceability to these scales the same rules and recommendations may be applied as given in this Technical Report for traceability to the endpoint SI.

A prominent, widely used non-SI measurement scale is the series of natural numbers. It is the basic measurement scale in all areas of measurement where counting is involved, e.g. of specified objects (pollen in a certain amount of air, *E.coli* bacteria in a specified volume of a food product, etc.). The peculiarity of this scale is that no materialization of the unit exists to which traceability might be established by direct comparison (calibration).