DOI 10.1007/s11018-021-01983-5
Measurement Techniques, Vol. 64, No. 8, November, 2021

METROLOGICAL TRACEABILITY OF COOMET REFERENCE MATERIALS. PART. 1. INTERNATIONAL PRACTICE IN ESTABLISHING TRACEABILITY OF REFERENCE MATERIAL CERTIFIED VALUES

S. V. Medvedevskikh,[1] E. P. Sobina,[2] O. N. Kremleva,[2] M. Yu. Medvedevskikh,[2] A. V. Sobina,[2] and N. S. Taraeva[2]

UDC 543.08

Many countries are currently focused on expanding the range of national means for measurement unit transfer characterized by established metrological traceability within international organizations. As well as demonstrating the high level of measurement capabilities provided by organizations and countries, such activities facilitate a solution to the global problem of ensuring the comparability of measurement results obtained for different objects at various times in diverse parts of the world. The present article highlights issues involved in establishing the metrological traceability of physicochemical measurement results. Curent international practice in establishing traceability of reference material certified values is described. In addition, the authors provide a brief overview of internationally recognized concepts related to the metrological traceability of reference materials.

Keywords: *metrological traceability, certified reference materials, measurement standards, primary reference measurement procedures, interlaboratory comparisons.*

Introduction. Global recognition of the role played by a particular country in terms of creating new technologies and developing innovative scientific directions is validated by the national metrological traceability of measurement results. In other words, the importance of measurement integrity and level of confidence necessary to support socio-economic activities in a given country under contemporary social conditions is recognized at the highest level of the metrological community.

Issues related to establishing the metrological traceability of measurement results are addressed by many international organizations including the International Bureau of Weights and Measures (BIPM), the International Organization of Legal Metrology (OIML), the International Organization for Standardization (ISO), the International Laboratory Accreditation Organization (ILAC), the Euro-Asian Cooperation of National Metrological Institutions (COOMET), etc.

The main approaches to demonstrating the metrological traceability of measurement results are regulated by international documents recognized in different countries by the metrological community:

ISO/IEC 17025:2017, *General Requirement for the Competence of Testing and Calibration Laboratories*;

ISO 17034:2016, *General Requirements for the Competence of Reference Material Producers*;

ISO Guide 30:2015, *Reference Materials – Selected Terms and Definitions*;

ISO Guide 31:2015, *Reference Materials – Contents of Certificates, Labels and Accompanying Documentation*;

ISO Guide 33:2015, *Reference Materials – Good Practice in Using Reference Materials*;

ISO Guide 35:2017, *Reference Materials – Guidance for Characterization and Assessment of Homogeneity and Stability*;

ILAC P10:07/2020, *ILAC Policy on Metrological Traceability of Measurement Results*;

ISO 17511:2020, *In Vitro Diagnostic Medical Devices – Requirements for Establishing Metrological Traceability of Values Assigned to Calibrators, Trueness Control Materials and Human Samples*;

[1] Mendeleyev Institute for Metrology (VNIIM), St. Petersburg, Russia; e-mail: s.v.medvedevskih@vniim.ru.
[2] UNIIM – Affiliated Branch of Mendeleev Institute for Metrology, Ekaterinburg, Russia; e-mail: kremleva@uniim.ru.

Translated from Izmeritel'naya Tekhnika, No. 8, pp. 25–29, August, 2021. Original article submitted May 24, 2021. Accepted June 10, 2021.

Issues related to demonstrating the metrological traceability of measurement results in various industrial sectors are widely covered in the literature [1–9].

In general, the only way for testing and calibration laboratories to demonstrate the metrological traceability of measurement results consists in the use of reference materials (RMs), whose certified values must be traceable to a valid comparison basis, i.e., a reference point, a fixed point, or a measurement standard. Researchers and technical experts working in the field of metrology express interest in developing new and improving existing measurement procedures based on metrological principles. Currently, metrological traceability can only be achieved by globally ensuring measurement unit transfer from measuring instruments to the International System of Units (SI), internationally recognized artifacts, or primary reference measurement procedures. The most accessible and portable transfer media are certified reference materials (CRMs). A significant expansion of development and production activities in the CRM industry in Russia and abroad is based on the use of CRMs to ensure metrological traceability to the SI. However, the rapidly growing market for CRMs results in a need to ensure their metrological traceability.

In the present article, a comparative analysis of various international practices in establishing metrological traceability of RMs is presented.

Different interpretations of the term "metrological traceability." Definitions of metrological traceability can be found in several basic international documents: ISO 17034:2016, ISO Guide 30:2015, ILAC P10:07/2020, ISO 17511:2020, and JCGM 200:2008, *International Vocabulary of Metrology – Basic and General Concepts and Associated Terms* (VIM). According to VIM, "metrological traceability – 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*."

In common with the ISO Committee on Reference Materials (ISO/REMCO), ISO generally adheres to the term presented in JCGM 200:2008 in terms of practice and developed documents. ISO 17511:2020, which comprises one of the international standards on laboratory and clinical medicine, also uses the term "metrological traceability" as defined in JCGM 200:2008.

In legal terms, Russian regulatory documents provide a slightly different interpretation of "metrological traceability." For example, the Federal Law of 06.26.2008 No. 102-FZ, *On Uniformity of Measurements* (as amended on 07.21.2014 No. 254-FZ), states that "traceability constitutes a property of standards for measurement units, measuring instruments, or measurement results, consisting in the documented establishment of their relation to a *state primary standard* or a foreign *national primary measurement standard* for the *corresponding measurement unit* via comparison of measurement standards, as well as verification and calibration of measuring instruments." In terms of measurement unit correspondence, it follows that the legal metrology of the Russian Federation adopts a stricter interpretation of the establishment of metrological traceability, emphasizing the hierarchical chain of SI calibrations to the primary measurement standard for this particular measurement unit.

The following sections present an analysis of several internationally recognized practices in ensuring the metrological traceability of RMs (within the BIPM, ISO, and ILAC).

Provision of metrological traceability according to BIPM policy. National metrology institutes are tasked with the creation of unified internationally recognized measurement standards. Therefore, the establishment of metrological traceability to SI units is primarily based on international key comparisons of measurement standards, which serve as the basis of declared recognition of the calibration and measurement capabilities (CMCs) of national metrology institutes (NMIs) within the BIPM according to the CIPM Mutual Recognition Arrangement (CIPM MRA documents, BIPM, https://www.bipm.org/en/cipm-mra/cipm-mra-documents). The CIPM MRA provides a technical basis for the mutual recognition of national measurement standards and calibration certificates issued by NMIs.

In international chemistry, the technical basis for reference measurement procedures (as opposed to measurement standards) are generally constituted by those implemented at corresponding NMIs; the BIPM Consultative Committee for Amount of Substance mainly considers rational methods, less often empirical, giving preference to primary measurement methods. Otherwise, CMCs are recognized in the field of chemistry under the CIPM MRA in the same way as for classical measurements.

Data on all recognized CMCs for different types of measurements are presented in the KCDB 2.0 (BIPM Key Comparison Database, http://kcdb.bipm.org), which provides information on CIPM MRA participants, key and supplementary comparisons, as well as peer-reviewed CMCs.

When measuring the amount of a substance, metrological traceability is usually established through the use of CRMs that constitute the NMI calibration service. BIPM recognizes and takes into account measurement results traceable to the SI, which is accomplished by using primary measurement methods. Such methods do not require pre-calibration involving measurement units that characterize the content of components in liquid and solid substances and materials to enable the highest accuracy in applying NMI CRMs (referred to as the primary reference materials of NMIs). The characterization of RMs from the KCDB 2.0 as means for demonstrating approved measurement capabilities should not be based on the use of commercial (freely marketed) reference materials produced by (non-)accredited manufacturers other than NMIs. The BIPM does not generally address issues related to the metrological traceability of empirical methods (e.g., measurement of moisture content in solids via thermogravimetric analysis); moreover, reference values may not be obtained using several measurement methods implemented at one or different laboratories.

The main advantage of the BIPM approach consists in providing a valid demonstration of metrological traceability to internationally recognized measurement standards.

However, when using this approach, it takes a long time to create national primary measurement standards and demonstrate recognition of CMCs declared by NMIs. Thus, while the respective countries may be significantly advanced in terms of their technological development, NMIs do not always have enough time to support the global economy with the necessary CRM types.

Provision of metrological traceability according to the ISO/REMCO policy. In view of the difficulties experienced by NMIs in creating an extensive range of CRMs, as well as the variety of objects and components to be determined, ISO/REMCO offers superior capabilities than those offered by BIPM for demonstrating metrological traceability of commercially produced (including matrix) RMs.

ISO/IEC 17025:2017, ISO 17034:2016, ISO Guide 30:2015, ISO Guide 31:2015, ISO Guide 33:2015, and ISO Guide 35:2017 provide metrological traceability requirements designed for a wide range of international (including accredited) reference material producers (RMPs). Furthermore, according to ISO/TR 16476:2016, *Reference Materials – Establishing and Expressing Metrological Traceability of Quantity Values Assigned to Reference Materials*, "horizontal traceability" is permitted. With such traceability, one of the following options is assumed:

measurements performed at a separate laboratory using a separate (primary) method;

measurements conducted using two or more independent standard methods at the same laboratory;

measurement performed by a laboratory network using one or more methods of demonstrated accuracy;

measurements performed using a particular method estimating only the values of properties.

Thus, ISO/TR 16476:2016 permits measurements performed by a laboratory network, as well as the use of empirical methods, which must be specified in RM certificates.

As compared to the BIMP policy, the ISO/REMCO approach offers a more flexible, cost-effective, and reliable demonstration of metrological traceability for RMs. The main disadvantage of this approach is that, even in RM certification cases where all the results obtained from the network of laboratories can be separately traced, the comparison extent and basis of the traceability chain of the combined result remain entirely unclear. This problem is greatly exacerbated if results from individual laboratories are traceable to different comparison bases or are obtained via different paths. The disadvantages also include the "unclear" traceability of commercial RMs.

Provision of metrological traceability according to the ILAC policy. The main document specifying ILAC metrological traceability requirements is the international ILAC P10:07/2020 document. According to ILAC policy, metrological traceability requires a continuous chain of calibrations traceable to recognized measurement standards or CRMs having established uncertainty values.

A common misconception exists in the metrology community that traceability can be provided in relation to an organization (e.g., traceability to a certain NMI), contributing to a persistent misunderstanding of the traceability procedure (its "nature"). The present authors argue that metrological traceability has to do with the reference values of measurement standards and results rather than the organization providing them.

In [1], Mike Sargent analyzes the wording of metrological traceability statements in certificates for the CRMs of pure substances, solutions, as well as matrix CRMs, of different producers. According to the analysis results [1], it is recommended to include references to NMI CRMs to which the certified value of a particular reference material is traceable when drawing up RM certificates, without the need to additionally clarify the traceability paths of the indicated NMI CRMs.

The ILAC policy on metrological traceability (ILAC P10:07/2020) to be provided by producers of reference materials (RMPs) via CRMs states that the certified values assigned to CRMs establish valid metrological traceability in the following cases:

"4) CRMs are produced by NMIs using a service that is included in the BIPM KCDB.

or

5) CRMs are produced by an accredited RMP under its scope of accreditation and the Accreditation Body is covered by the ILAC Arrangement or by Regional Arrangements recognized by ILAC.

or

6) The certified values assigned to CRMs are covered by entries in the Joint Committee for Traceability in Laboratory Medicine (JCTLM) database.

Recognizing that the accreditation of RMPs is still developing and CRMs may not be available from accredited RMPs, where CRMs are produced by non-accredited RMPs, Accredited Organizations shall demonstrate that CRMs have been provided by a competent RMP and that they are suitable for their intended use.

When metrological traceability to the SI is not technically possible, it is the responsibility of the Accredited Organization to:

7a) Choose a way to satisfy metrological traceability requirements by using certified values of certified reference materials provided by a competent producer

or

7b) Document the results of a suitable comparison to reference measurement procedures, specified methods, or consensus standards that are clearly described and accepted as providing measurement results fit for their intended use. Evidence of this comparison shall be assessed by the Accreditation Body.

Note 4. When metrological traceability to solely SI units is not appropriate or applicable to the application, a clearly defined measurand should be selected. Establishing metrological traceability therefore includes both the proof of identity of the property measured and the comparison of the results to an appropriate stated reference. The comparison is established by ensuring the measurement procedures are properly validated and/or verified, that measuring equipment is appropriately calibrated, and that conditions of measurement (such as environmental conditions) are under sufficient control to provide a reliable result.

Note 5. Surplus test materials are often available from proficiency testing (PT) providers. It should be checked whether the PT provider can provide additional stability information to demonstrate the ongoing stability of the property value and matrix of the test material. If this cannot be provided, these test materials should not be considered as an alternative way to ensure the validity of results."

Thus, guided by the BIPM principles, ILAC takes into account the problem of providing testing and calibration laboratories only with CRMs produced by NMIs. Therefore, it is permitted to use the CRMs of other RMPs provided their metrological traceability can be demonstrated. In addition, ILAC supports approaches to demonstrating metrological traceability recommended by ISO/REMCO documents without clearly defining reference points to which metrological traceability is established, which leads to an inconclusive assessment by the accreditation body when evaluating the metrological traceability of RMs.

Comparison of different approaches to establishing metrological traceability. The performed analysis of three metrological traceability criteria applied by the BIPM, ISO/REMCO, and ILAC indicates conceptual differences in establishing metrological traceability despite the common methodological approach. The differences mainly arise when it is impossible to trace measurement results back to SI units and/or when empirical measurement methods or interlaboratory experiments are used in RM characterization.

Conclusion. The performed analysis of international practice in establishing traceability for RM certified values reveals different definitions of the term "metrological traceability" found in international and national documents. A brief

comparative review of the main internationally recognized concepts underlying the demonstration of metrological traceability for RMs describes the advantages and disadvantages of the BIPM, ISO, and ILAC policies.

REFERENCES

1. M. Sargent, *Accredit. Qual. Assur.*, **25**, 367–372 (2020), https://doi.org/10.1007/s00769-020-01450-8.
2. M. do Ceu Ferreira, A. Matos, and R. P. Leal, *Accredit. Qual. Assur.*, **20**, 457–464 (2015), https://doi.org/10.1007/s00769-015-1149-9.
3. R. J. C. Brown and H. Andres, *Accredit. Qual. Assur.*, **25**, 161–166 (2020), https://doi.org/10.1007/s00769-020-01424-w.
4. O. Velychko and T. Gordiyenko, *Standards, Methods and Solutions of Metrology*, L. Cocco (ed.), IntechOpen (2019), https://doi.org/10.5772/intechopen.84853.
5. T. Kyriacos and M. Sappho, "Metrological requirements for accredited laboratories," *17th Int. Congr. of Metrology*, Paris, France, Sept. 21–24, 2015, EDP Sciences, Paris (2015), https://doi.org/10.1051/metrology/201502016.
6. E. Bulska, *Metrology in Chemistry. Lecture Notes in Chemistry* (2018), Book 101, pp. 35–51, https://doi.org/10.1007/978-3-319-99206-8_4.
7. W. G. Miller, N. Greenberg, J. Budd, and V. Delatour, *Clin. Chim. Acta*, **514**, 84–89 (2021), https://doi.org/10.1016/j.cca.2020.12.021.
8. V. I. Paneva, "Role of interlaboratory comparisons in assuring the quality of analytical measurements used in conformity assessment," *Stand. Obraztsy*, No. 3, 7–14 (2009).
9. V. G. Kutyaikin, P. A. Gorbachev, E. Yu. Geiger, and K. K. Savrovskii, "Metrological traceability of test results," *Kompetentnost*, No. 7, 30–36 (2020), https://doi.org/10.24411/1993-8780-2020-10705.