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Asia Pacific Laboratory Accreditation Cooperation

GUIDELINES FOR ACCEPTABILITY OF CHEMICAL REFERENCE MATERIALS AND COMMERCIAL CHEMICALS FOR CALIBRATION OF EQUIPMENT USED IN CHEMICAL TESTING

PURPOSE

This document provides guidelines on determining the acceptability of chemical reference materials and commercial chemicals used for calibration of instruments in chemical testing.

AUTHORSHIP

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1. INTRODUCTION

1.1 This document is applicable to chemical tests requiring the use of reference materials (RMs) for the purpose of calibration and provides guidance on determining the acceptability of certified reference materials (CRMs). Both ISO/IEC 17025 and ILAC-P10 stress that the CRMs used to establish metrological traceability are to be supplied by competent producers. It is thus essential for laboratories to obtain their CRMs used for the calibration of equipment from competent suppliers so that metrological traceability could be assured.

1.2 According to clause 5.6.2.2.1 of ISO/IEC 17025:2005, for testing laboratories, the requirements given in clause 5.6.2.1 apply for all measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from calibration contributes little to the total uncertainty of the test results.

1.3 Clause 5.6.2.1.1 of ISO/IEC 17025:2005 requires a laboratory to establish traceability of its own measurement standards and measurement instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. Where traceability of measurements to SI units is not possible and/or not relevant, traceability to, for example, CRMs, agreed methods and/or consensus standards are required (clause 5.6.2.2.2). CRMs provided by a competent supplier are to be used to give a reliable physical or chemical characterisation of a material (clause 5.6.2.1.2). ISO/IEC 17025:2005 further requires that RMs where possible, be traceable to SI units of measurement, or to CRMs. Internal reference materials need to be checked as far as technically and economically practicable (clause 5.6.3.2). VIM, International Vocabulary of Metrology - Basic and General Concepts and Associated Terms, 3rd edition (published by ISO as ISO/IEC Guide 99: 2007), defines metrological traceability as the 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.

1.4 VIM defines a RM as material, sufficiently homogeneous and stable with reference to specified properties, that has been established to be fit for its intended use in measurement or in examination of nominal properties. RMs with or without assigned quantity values can be used for measurement precision control whereas only reference materials with assigned quantity values can be used for calibration or measurement trueness control. Some RMs have assigned quantity values that are metrologically traceable to a measurement unit outside a system of units. Such materials include vaccines to which International Units (IU) have been assigned by the World Health Organisation. In a given measurement, a given RM can only be used for either calibration or quality assurance. The specifications of a RM should include its material traceability, indicating its origin and processing.

1.5   VIM defines a CRM as a reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties, and traceabilities using valid procedures. It explains that documentation is given in the form of a "certificate" (see ISO Guide 31:2000). Procedures for the production and certification of CRMs are given, e.g. in ISO Guide 34 and ISO Guide 35. Specified quantity values of CRMs require metrological traceability with associated measurement uncertainty.

1.6   ILAC-P10 ILAC Policy on Traceability of Measurement Results requires that laboratories accredited by ILAC Member Bodies shall be able to demonstrate that calibration of critical equipment, and hence the measurement results generated by that equipment, relevant to their scopes of accreditation, are traceable to the International System of Units (SI units). Where such traceability is not technically possible or reasonable, the laboratory, the client and other interested parties may agree to the use of certified reference materials provided by a competent supplier or of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned. ILAC does not, however, further elaborate how measurement traceability should be established for testing laboratories using reference materials. This document is intended to give some clarification to testing laboratories in this regard.

1.7   According to ISO/IEC 17025:2005 (NOTE under clause 5.6.2.2.1), the extent to which the requirements of metrological traceability (as given in clause 5.6.2.1) should be followed depends on the relative contribution of the calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements should be strictly followed. Therefore, when determining whether CRMs produced by National Measurement Institutes (NMIs) or Designated Institutes (DIs) and accredited RMPs are needed, laboratories should estimate the contribution of calibration uncertainty to the total uncertainty.

Note: The Bureau International des Poids et Mésures (BIPM) http://www.bipm.org/en/cipm-mra/documents/ states that the Commité International des Poids et Mésures (CIPM) MRA introduced the concept of designated institutes (DIs) as responsible for certain national standards and associated services that are not covered by the actvities of the "traditional" NMI. In general, governments, an appropriate authority, or in some cases the NMI itself when it is authorized to do so, have identified and appointed specialist institutes to deal with metrological responsibilities in many new areas.

1.8   A summary of the guidelines is given in the flowchart in Annex I.

2.   GENERAL

2.1   The onus is on laboratories to demonstrate that

(a)   full records are kept of the identity, lot number and source of each reference material used;

(b) the documentation of certified values includes details of the mode of establishment of metrological traceability of the assigned values by the producers and/or laboratories, uncertainties of the assigned values, instructions for storage and use, homogeneity and stability data, and expiration dates;

(c) the matrices of the reference materials used match those of the laboratory's test samples or the effect of any non-matching of matrices is determined and accounted for; and

(d) uncertainties of the assigned values and of the verifications of RMs are appropriate for the test methods concerned, and that their contributions, where significant, are included in the estimation of the total measurement uncertainties of the test results.

2.2 When a standard test method specifies the use of a particular RM for calibration, the laboratory is deemed to have satisfied the metrological traceability of ISO/IEC 17025 when the specified RM is used. In such a case, the test reports should clearly state the standard test method used. Similarly, when a regulation mandates the use of a particular RM, the laboratory is deemed to have satisfied the metrological traceability requirement when the specified RM is used.

2.3 Laboratories should take particular note that reference material producers that are certified to ISO 9001 or other standards containing management system requirements only but not specific requirements for reference material producers are not considered as competent suppliers because the technical competence of the reference material producer may not have been determined. RMs and CRMs produced by these producers need to be verified before use (see Section 5). Similarly, RMs and CRMs supplied by manufacturers of equipment need to be verified before use unless the manufacturers meet the requirements given in Section 4.

3.   **USE OF REFERENCE MATERIALS PRODUCED BY NMIs OR DIs**

3.1 CRMs supplied by an appropriate National Metrology Institute (NMI) or Designated Institute (DI) are considered acceptable as providing metrological traceability when the certificates of the CRMs include information on the method of establishing metrological traceability to SI units, where relevant, or to a reference, and on the uncertainties of the certified values.

Note: ILAC P-10 states that "ILAC considers an "appropriate" national metrology institute to be one that participates regularly and successfully in relevant international interlaboratory comparisons performed by BIPM and/or by regional metrology bodies." In this regard, for chemical reference materials, there are key comparisons organized by the CIPM Consultative Committee for Amount of Substance (CCQM).

3.2 Laboratories using CRMs obtained from NMIs or DIs should check the statement of metrological traceability. Laboratories should take note that measurement results should not be regarded as traceable to a particular NMI or DI. Their measurement results should be regarded as traceable to the SI units, or other references, through the use of CRMs produced by a particular NMI or DI.

3.3 Owing to the wide scope and variety of components, concentrations and matrices, it is impossible for these institutes to encompass the whole field of analytical chemistry. Reference materials produced by producers other than NMIs or DIs may, therefore, have to be used in many cases.

4. USE OF REFERENCE MATERIALS PRODUCED BY ACCREDITED PRODUCERS

4.1 Some accreditation bodies are now providing an accreditation service to reference material producers (RMPs). In September 2005, the International Laboratory Accreditation Cooperation (ILAC) passed a resolution that accreditation of RMPs to ISO Guide 34 in combination with ISO/IEC 17025:2005 be included under the ILAC Mutual Recognition Arrangement (MRA). Currently ILAC is working on establishing the MRA. In the Asia Pacific region, based on the accreditation criteria stipulated by ILAC, the Asia Pacific Laboratory Accreditation Cooperation (APLAC) MRA for reference material producer accreditation was established in December 2007.

4.2 RMPs accredited by signatories to the APLAC MRA for Reference Material Producers have been assessed and found to meet the accreditation requirements of ISO Guide 34 and ISO/IEC 17025 in combination. Accredited RMPs can, therefore, be considered as competent, and the use of CRMs produced by them is considered to meet the metrological traceability requirement of ISO/IEC 17025. Laboratories using these CRMs are not required to further verify the competence of the accredited RMPs or to verify the CRMs produced by them.

4.3 When the required CRMs are available from both NMIs or DIs and accredited RMPs, laboratories may choose the most appropriate source that meets their need. Factors to be considered include matching of matrices, concentration levels of the analytes, measurement uncertainties of assigned values as well as costs, and the need to have the test results traceable through a particular NMI or DI.

5. USE OF REFERENCE MATERIALS FROM PRODUCERS OTHER THAN NMIs OR DIs AND ACCREDITED RMPs

5.1 RMs and CRMs supplied by producers other than NMIs or DIs and accredited RMPs need to be verified. The purpose of verification is to ascertain and demonstrate that the assigned values are reliable and that the materials are sufficiently homogenous and stable for use as RMs and CRMs. The extent of verification depends on the information supplied by the producers, as well as on the nature of the RMs and CRMs, and the certified properties.

5.2 Laboratories should use two independent sources of the reference materials, when available. Laboratories should, as far as practicable, verify the two separately sourced reference materials against each other to ascertain that the values agree with each other within specified limits that are suitable for the application.
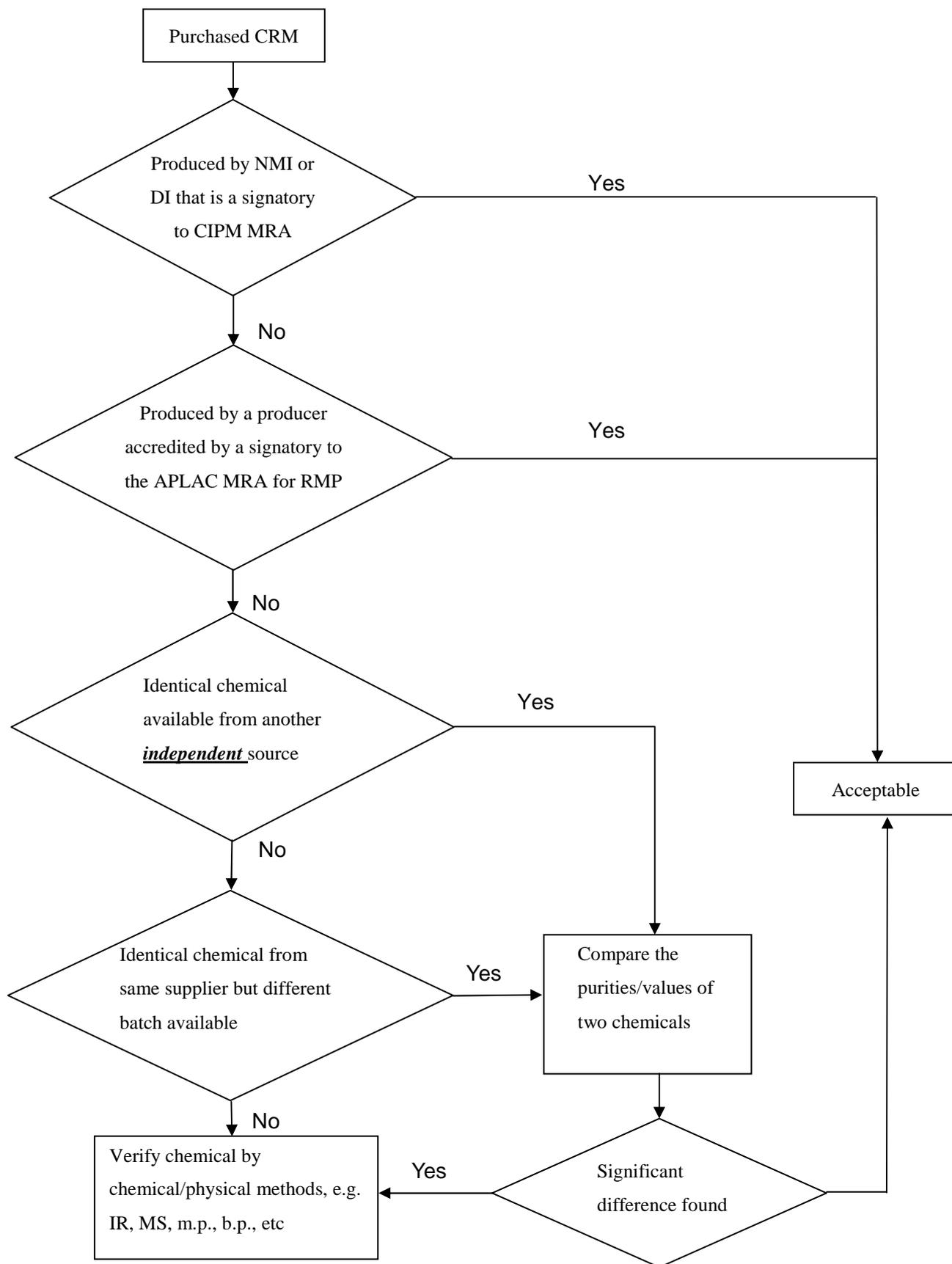
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5.3   If another independent source is not available, a less preferred method is to use a reference material from the same supplier but from a different batch.

5.4   When neither 5.2 nor 5.3 are possible, the laboratory should consider using methods based on physical or chemical properties of the reference materials, such as melting point, boiling point, mass spectrum, infra-red spectrum, etc, to confirm their identity and purity.

5.5   Internal or secondary reference materials should also be subject to appropriate verifications and the provisions identified in the clauses of this section and in clause 2.1 apply.

6.   USE OF COMMERCIAL CHEMICALS

6.1   Laboratories sometimes use commercial chemicals to prepare "standard" mixtures for calibration of analytical instruments. These prepared "standard" mixtures are prepared by, for example, mixing known amounts of chemicals or dissolving the chemicals in a solvent. Laboratories also sometimes purchase and use "standard solutions" that are available commercially. These solutions are however often not certified and thus measurement traceability is not provided by these solutions.

6.2   Laboratories should determine the need to strictly follow the metrological traceability of ISO/IEC 17025 by estimating the contribution of calibration uncertainty to the total uncertainty (see clause 1.7). If the contribution is significant, then these standard solutions or other calibration mixtures such as calibration gas mixtures used for calibration of equipment are functioning as reference materials and the requirements given in Section 5 apply. In such cases, laboratories should have a defined system and procedure for verification of standard solutions or other calibration mixtures used for calibration, whether produced in-house or purchased directly from outside suppliers.

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Annex I

Flowchart for Acceptability of RM

Flowchart:

- Purchased CRM
  - → Produced by NMI or DI that is a signatory to CIPM MRA
    - Yes → Acceptable
    - No → Produced by a producer accredited by a signatory to the APLAC MRA for RMP
      - Yes → Acceptable
      - No → Identical chemical available from another *independent* source
        - Yes → Compare the purities/values of two chemicals
        - No → Identical chemical from same supplier but different batch available
          - Yes → Compare the purities/values of two chemicals
          - No → Verify chemical by chemical/physical methods, e.g. IR, MS, m.p., b.p., etc
      - Compare the purities/values of two chemicals → Significant difference found
        - Yes → Verify chemical by chemical/physical methods, e.g. IR, MS, m.p., b.p., etc
        - No → Acceptable

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Annex II
Bibliography

The following is a list of references that contain useful information on the use of RMs and CRMs.

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

2. ISO Guide 31: 2000 Reference Materials – Contents of certificates and labels

3. ISO Guide 32: 1997 Calibration in analytical chemistry using certified reference materials

4. ISO Guide 33: 2000 Uses of certified reference materials

5. ISO Guide 34: 2009 General requirements for the competence of reference material producers

6. ISO Guide 35: 2006 Reference materials – General and statistical principles for certification

7. ISO Guide 99:2007 International vocabulary of metrology - Basic and general concepts and associated terms (VIM)

8. ILAC-P10:2002 ILAC Policy on Traceability of Measurement Results

9. ILAC-G9: 2005 Guidelines for the selection and use of reference materials (www.ilac.org)

10. APLAC TC-008 2010 APLAC Guidelines on the approach to the assessment of reference material producers and the resulting scope of accreditation

11. Guidelines for the in-house production of reference materials, Laboratory of the Government Chemist, 1998 (www.vam.org.uk)

12. ISO The role of reference materials – Achieving quality in analytical chemistry 2000