

LAB 26

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Calibration of radionuclide sources -

Activity, particle or photon emission rate, air kerma rate



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Changes since last edition

Section 2.6.1b) reference to BIPM CIPM MRA document and appendix reference to ILAC-P10:07/2020 updated.

1. Introduction

- 1.1 Laboratories that have been assessed by UKAS as meeting the requirements of ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories may be granted UKAS Accreditation. Several guidance publications on the application of these requirements, providing extra information, detail and limitations, are listed in the LAB series of documents listed in UKAS Publications.
- 1.2 This publication (LAB 26) provides guidance on the application of specific requirements for laboratories carrying out the calibration of radioactive sources in respect of activity, activity per unit mass, α, β, γ, X-ray or conversion electron emission rate, neutron emission rate or exposure/kerma rate. By following this guidance, laboratories will be able to demonstrate at assessment that they meet the requirements. Alternative methods may be used provided they are shown to have an equivalent outcome.
- 1.3 The guidance applies to such specific types of source as may be supplied by the laboratory or the organisation of which the laboratory may be part, according to a list submitted to UKAS before accreditation. It also applies to those sources of the same type which are submitted to the Laboratory by a third party for the purposes of being similarly standardised. Any subsequent addition to the list of approved types of source would require specific approval. The specific types of source should conform to the following general categories:
 - (a) standardised solutions to be calibrated in terms of activity and activity per unit mass;
 - (b) solid sources to be calibrated in terms of activity;
 - (c) radium-226 sources to be calibrated in terms of radium element content;
 - (d) sources to be calibrated in terms of γ -ray, or X-ray emission rate;
 - sources to be calibrated in terms of α-particle, β-particle or conversion electron emission rate;
 - (f) sources to be calibrated in terms of neutron emission rate;
 - (g) photon-emitting sources to be calibrated in terms of air kerma rate.
- 1.4 The laboratory should provide written procedures for sample preparation and measurement for each source type and radionuclide.
- 1.5 The primary standardisation of activity should be based upon primary reference measurement procedures. (As examples, such procedures include the 4πβ-γ coincidence method, Triple to Double Coincidence Ratio (TDCR), Defined Solid Angle counting (DSA)). For those reference measurement procedures which qualify, reference should be made to those procedures which are recognised by the BIPM Consultative Committee for Ionising Radiations, Section II (Measurement of radionuclides), as exemplified by inclusion in the determination of the Key Comparison Reference Value (KCRV) for any particular radionuclide.

2. Standardisation of radionuclide solutions

2.1 Materials

2.1.1 All materials intended for incorporation into radionuclide solutions should be ordered against written specifications and be subject to quality control examination and verification. All equipment used in source preparation shall be of suitable material and quality for the intended purpose. Where sources are supplied by a third party, that third party should be asked to provide as much information as possible in order to ensure that, where possible, the same level of quality control can be exercised.

2.2 Dispensing of solutions

- 2.2.1 The following requirements apply to the dispensing of radionuclide solutions:
 - (a) all containers should be of an appropriate type and specification;
 - (b) all containers and other glassware should be cleaned by an appropriate procedure;
 - (c) carrier solutions should be prepared in advance, filtered to remove suspended solids and stored in appropriate containers;
 - (d) sufficient aliquots of the radionuclidic solution in calibration containers should be prepared to enable independent checks on long term stability and all dilutions (see paragraph 2.4.1);
 - (e) equipment used for gravimetric or volumetric measurements should be calibrated in a manner traceable to national standards; weighings are subject to buoyancy corrections, as appropriate.

2.3 Preparation of solid sources

- 2.3.1 Where solid sources are required for the primary standardisation, the following criteria apply:
 - (a) sources should be prepared by dispensing gravimetrically solutions of appropriate dilution onto substrates which are appropriate for the intended measurement procedure, followed by evaporation to dryness;
 - (b) sufficient sources should be prepared from each dilution to demonstrate consistency within the required specification;
 - (c) when volatile material is involved due precautions should be taken to avoid loss of activity by evaporation and measurements shall be made to verify that there has been no loss of activity during the drying process.

2.4 Radionuclide purity

2.4.1 Radionuclide purity should be assessed by appropriate means and verified.

2.5 Quality checks on accuracy of dilutions and stability

2.5.1 Where possible, calibration containers of γ-ray emitting solutions of sufficient activity should be checked by measurement in a transfer instrument that has been calibrated in a manner traceable to national standards. The preferred transfer instrument, because of its long-term stability, is a well type, sealed high pressure ionization chamber. Alternative instruments such as gamma spectrometers or liquid scintillation counters may be employed but additional quality control measures may be necessary because of their susceptibility to short-term variations in response.

2.6 Traceability of standardisation measurements

- 2.6.1 Primary Measurements made by the National Measurement Institute (NMI)
 - (a) Whichever primary reference measurement procedure is used, the competence of the NMI for that method and for the individual radionuclide concerned must have been validated via a satisfactory demonstration of international equivalence using the BIPM programme of Key Comparisons within the prescribed period for revalidation (typically 10 years). This system allows competence for the radionuclide concerned using the particular measurement procedure to be demonstrated by a direct comparison for the radionuclide concerned or by satisfactory performance with an equivalent radionuclide using the CCRI(II) traffic light system.
 - (b) The establishment of equivalence is prescribed by the Mutual Recognition Arrangement (MRA) of the International Committee for Weights and Measures. *Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes,* International Committee for Weights and Measures, 1999 (Technical Supplement revised 2003) - see <u>Documents on the CIPM MRA - BIPM.</u>
 - (c) The uncertainty on the value of activity should not be stated as being less than the corresponding degree of equivalence with the BIPM Key Comparison Reference Value.
- 2.6.2 Measurements made by non-NMIs

Where the laboratory is not the National Measurement Institute, measurements made by the laboratory should be traceable to national standards. Traceability should be established and maintained through the regular exchange and measurement of a representative range of radionuclide samples with the National Metrology Institute.

3. Solution sources

- 3.1 Solutions (including gas standards) of β - γ emitting radionuclides may be calibrated by either of the following methods:
 - (a) a primary reference measurement procedure as recognised by CCRI(II) (see 1.5) absolute standardisation by the $4\pi\beta$ - γ coincidence method or an agreed variant thereof;
 - (b) a secondary standardisation procedure by means of a transfer instrument that has itself been calibrated in a manner traceable to national standards. The preferred transfer instrument, because of its long-term stability, is a well-type, sealed high pressure ionization chamber. Alternative instruments such as gamma spectrometers or liquid scintillation counters may be employed but additional quality control measures may be necessary because of their susceptibility to short term variations in response.

4. Solid sources

- 4.1 Photon (γ- and X-ray) emitting sources should be calibrated for activity by means of an ionisation chamber, or other suitable detector, that has itself been calibrated using standard sources of appropriate geometry. These standard sources shall either be prepared from previously standardised solutions, or subsequently standardised by dissolution and measurement in a manner traceable to national standards.
- 4.2 Solid sources prepared from standardised solutions should, in the absence of a more direct method of calibration, be provided with a calibration derived from the known mass or volume of solution dispensed.

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5. Radium content: calibration of ²²⁶Ra sealed sources

5.1 Radium sources comprising encapsulated radium salt should be calibrated in terms of radium element content by comparison with suitable sources which have themselves been calibrated in a manner traceable to the 1934 international radium standards of Hönigschmid.

6. α-particle, β-particle or conversion electron emission rate: calibration of sources

- 6.1 Small area sources with thick backings should be calibrated by $2\pi\alpha$ or $2\pi\beta$ counting, using a detector with a response determined in a manner traceable to national standards.
- 6.2 Large area α-particle or β-particle emitting sources should be calibrated by means of a large area proportional counter or other appropriate detector which has itself been calibrated using appropriate reference sources whose emission rate calibration is traceable to national standards.

7. X-ray or γ-ray emission rate: calibration of sources

7.1 Solid or solution sources of well-defined geometry should be calibrated using a suitable detector that has itself been calibrated in a manner traceable to national standards.

8. Neutron emission rate: calibration of sources

- 8.1 The neutron emission rate from sealed radionuclide neutron sources should be determined by one of the following methods:
 - (a) directly, using a manganese sulphate bath;
 - (b) indirectly, by comparison with an appropriate source whose emission rate has been determined with traceability to national standards.
- 8.2 Where the manganese bath technique is used to determine the absolute emission rate, the detectors shall be calibrated using ⁵⁶Mn that has been measured with traceability to national standards. The appropriate corrections must be applied for loss of neutrons by absorption by materials other than the manganese.
- 8.3 Indirect measurements of emission rate of a source should be made using a suitable moderatordetector arrangement with the source either placed inside the moderator or mounted externally to it.
- 8.4 Where necessary, due allowance should be made for any effect arising from anisotropy of neutron output.

9. Air kerma rate: calibration of sources

- 9.1 The calibration of photon-emitting sources in terms of air kerma rate should be carried out directly, by measurement of the source output (air kerma rate) in a known geometry using a calibrated instrument.
- 9.2 Direct measurements of source output should be carried out in an adequately defined sourcedetector geometry. An ionisation chamber of the appropriate sensitivity should be used and

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the energy dependence of the chamber response should be known over the range of photon energies to be measured. The calibration of this chamber should be traceable to national standards. Scattering from the surroundings should be kept to a practical minimum and appropriate corrections should be applied.

10. General

- 10.1 For any calibrated ionisation chamber or similar system providing the basis of a calibration facility, the constancy of the system response should be monitored at appropriate intervals by means of a suitable radionuclide check source.
- 10.2 Since activity, emission and exposure rates are stated in units which include the reciprocal of time, the laboratory should demonstrate the traceability of its time measurement to a suitable reference standard.

APPENDIX - Definitions

As an informative guide to this document, some relevant definitions and paragraph numbers are given below and are extracted from JCGM 200:2012, International vocabulary of metrology – Basic and general concepts and associated terms (VIM) 3rd edition.

(Downloadable from https://www.bipm.org/en/committees/jc/jcgm/publications/ Vocabulary of Metrology (VIM))

2.40 Calibration hierarchy

Sequence of **calibrations** from a reference to the final **measuring system**, where the outcome of each calibration depends on the outcome of the previous calibration.

2.41 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 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-P10:07/2020).

2.8 Primary reference measurement procedure

Reference measurement procedure used to obtain a measurement result without relation to a measurement standard for a quantity of the same kind.

5.1 Measurement standard

Realization of the definition of a given **quantity**, with stated **quantity value** and associated **measurement uncertainty**, used as a reference.

5.2 International measurement standard

Measurement standard recognized by signatories to an international agreement and intended to serve worldwide.

5.3 National measurement standard

Measurement standard recognized by national authority to serve in a state or economy as the basis for assigning **quantity values** to other measurement standards for the **kind of quantity** concerned.

5.4 Primary measurement standard

Measurement standard established using a **primary reference measurement procedure**, or created as an artefact, chosen by convention.

5.5 Secondary measurement standard

Measurement standard established through calibration with respect to a primary measurement standard for a quantity of the same kind.

NOTE 1 Calibration may be obtained directly between a primary measurement standard and a secondary measurement standard or involve an intermediate **measuring system** calibrated by the primary measurement standard and assigning a **measurement result** to the secondary measurement standard.

NOTE 2 A measurement standard having its **quantity value** assigned by a ratio **primary reference measurement procedure** is a secondary measurement standard.

5.6 Reference measurement standard

Measurement standard designated for the **calibration** of other measurement standards for **quantities** of a given kind in a given organization or at a given location.

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