Protocol for the CCRI(II)-K2.Cd-109 key comparison

1. Introduction

The main application of ^{109}Cd is the calibration of high-resolution gamma spectrometers. These spectrometers are used in the nuclear industry, and also for checking the radioactivity content of foodstuffs and the different types of environmental matrices – as a consequence, all nuclear sites and independent environmental monitoring laboratories have a set of such instruments. These spectrometers are normally calibrated [1, 2] using a mixed–radionuclide solution that is available from national measurement institutes or from commercial suppliers; ^{109}Cd is an essential component of the mix as it provides a low-energy calibration point at 88 keV. In addition, the Measurement Method Matrix of the Consultative Committee for Ionizing Radiation, Section II (CCRI(II)) shows that measurements of ^{109}Cd may support CMCs for many other radionuclides.

The previous comparison of a solution of ^{109}Cd was organized by the CCRI(II) and was carried out in 1986 (see here). Primary standardizations of ^{109}Cd are challenging due to the delayed state in the daughter nuclide ^{109}Ag. Nevertheless, the results from the comparison in 1986 showed reasonably good agreement between the participants.

The results from the 1986 comparison are no longer valid to support CMCs in the CIPM MRA [3]. Consequently, the CCRI(II) decided to repeat this comparison and the BIPM volunteered to be the pilot laboratory, with the support of the LNE-LNHB.

2. Comparison Protocol

Pilot Laboratory:

- Bureau International des Poids et Mesures (C. Michotte)
- Laboratoire National de Métrologie et d'Essais -Laboratoire National Henri Becquerel, France (C. Fréchou)

The Pilot laboratories are encouraged to consult the Key Comparison Working Group of Section II of the Consultative Committee on Ionizing Radiation [KCWG(II)] for assistance in designing, coordinating, analyzing, and reporting on this comparison and its results.

List of participants (22):

BARC, BEV, BIPM, BFKH, CIEMAT, CMI-IIR, ENEA-INMRI, IFIN-HH, ININ, KRISS, LNE-LNHB, LNMRI/IRD, NIM, NIST, NMIJ, NMISA, NPL, NRC, PTB, POLATOM, SMU, NUKEN/TENMAK

2.1 Comparison nuclide solution

The ^{109}Cd mother solution will be diluted and distributed in glass ampoules by the LNE-LNHB. Homogeneity between the ampoules will be verified by ionization chamber, 4πNaI(Tl) or HPGe spectrometry measurements before despatching to participants by the BIPM.

Container: Flame sealed ampoules : LMRI-type (1.7 cm diam x 8 cm height)

Solution mass: **2 g**

Activity concentration: at least **250 kBq/g**

Chemistry: 0.1 mol.L^{-1} HCl with Cd carrier concentration of 20 μg/g

One ampoule with a stronger activity (about 50 MBq) will be measured in the SIR at the BIPM for linking the present key comparison to BIPM.RI(II)-K1.Cd-109.

It is part of the exercise for the participant to identify and measure the activity of any possible impurity. However, no significant impurity is expected in the solution that will be distributed.

2.2 Measurand

The measurand for this exercise is the activity concentration (kBq g^{-1}) of ^{109}Cd, at the **reference date of 1st September 2021 12:00 UT**.

2.3 Nuclear Data

Nuclear data from **Monographie BIPM-5, Vol 8, pp 129-134 (2016)** must be used, in particular the half-life of 461.9(4) days.

2.4 Schedule

Shipment preparation: Deadline for submission of the registration form with all appropriate shipping, customs, and special handling information is **5 March 2021.**

Despatch: Despatch of the ampoules is planned for **July 2021**.

Reporting deadline: 31 January 2022

Draft A distributed: 31 May 2022

The aim is to publish the Final Report by end of 2022.

2.5 LNE-LNHB role

The LNE-LNHB will be responsible for preparing the samples, checking the homogeneity of the ampoules. The LNE-LNHB will endorse the costs related to these operations.

2.6 BIPM role

The BIPM will be responsible for buying the ^{109}Cd solution, checking the homogeneity of the ampoules produced by the LNE-LNHB and despatching* them to the participants (door-to-door delivery, if possible), including the related costs.

The BIPM will inform the participants if there is a delay in the organization of the comparison and will keep the key comparison status on the KCDB up to date.

The BIPM will prepare the reporting form for the comparison results.

2.7 Participant role

Participants should send any specific instruction for them to receive the package. Immediately after receipt of the sample, the participant shall check for any damage or contamination of the sample and report this to the pilot laboratories.

Each participant is responsible for its own costs associated with the measurements.

All results, method of standardisation, associated uncertainties, and any additional requested information shall be transmitted to the BIPM using the reporting forms to be provided.

Participants must provide a list and evaluation of the uncertainty components using the reporting form provided by the BIPM. Participants must add any other components they consider appropriate. Uncertainties are evaluated at a level of one standard uncertainty following the Guide to the Expression of Uncertainty in Measurement, published by the JCGM (JCGM 100:2008, http://www.bipm.org/en/publications/guides/#gum.html).

If a participant uses several measurement methods and reports several results, the single result to be used for equivalence should be indicated.

3. Preparation of the report on the comparison

The BIPM is responsible for the preparation of the report on the comparison. The report passes through a number of stages before publication, and these are referred to as drafts A and B [3].

A result from a participant is not considered complete without an associated uncertainty; a result is not included in the draft report unless it is accompanied by an uncertainty

* the package should be Exempt from class 7 (no UN number)

supported by a complete uncertainty budget. Uncertainties are drawn up following the guidance given in the technical protocol.

If, on examination of the complete set of results, the BIPM finds results that appear to be anomalous, those participants will be invited to check their results for numerical errors but without being informed as to the magnitude or sign of the apparent anomaly. If no numerical error is found the result stands and the complete set of results is sent to all participants.

The first draft, draft A, is prepared as soon as all the results have been received from and, if necessary, confirmed by the participants. It includes the results, uncertainties, standardization methods and experimental details transmitted by the participants, identified by name.

The draft A report is confidential to the participants. Copies are not given to non-participants, and graphs or other parts of the draft are not used in oral presentations at an external conference without the specific agreement of all the participants[†]. The results may be the subject of an internal report if they are shown in relative terms and the name of participants hidden. At this stage, a participant may publish experimental techniques of special interest or new developments of a measurement method made in the context of the comparison, as long as no information or comments are made about the comparison results.

The draft A report is sent as soon as possible after completion of the comparison to all the participants for comment, with a reasonable deadline for replies. The date at which this draft is sent to the participants is taken to be the end date for the comparison and is subsequently referred to as such.

A participant that considers its result unrepresentative of its standards may submit another solution to the SIR or organize a bilateral comparison with another participant in the CCRI(II) comparison. The subsequent comparison is considered as a new and distinct comparison.

On receipt of final comments from participants, the second draft, draft B, is prepared incorporating the agreed comments on the draft A, and a preliminary comparison reference value with preliminary degrees of equivalence.

A draft B report is not considered confidential and *may* be the subject of publications or presentations at conferences, with the exception of the preliminary comparison reference value and degrees of equivalence. At this stage, the results can be used to support CMCs [4].

The BIPM will circulate the draft B report to all participants for comments and corrections. Any comments and corrections received will be addressed by the BIPM in correspondence with the participants. If any controversial or contradictory comments are received by BIPM, they will be circulated to all participants and discussion continues until a consensus is reached.

The agreed draft B report will be forwarded to the CCRI Executive Secretary who will circulate the report to the members of the CCRI Section II and the KCWG(II) for review,

[†] Note that once all participants have been informed of the results, individual values and uncertainties may be changed or removed, or the complete comparison abandoned, only with the agreement of all participants and on the basis of a clear failure of the comparison artefact or some other phenomenon that renders the comparison or part of it invalid.

allowing a period of 6 weeks. Following the review period, the CCRI Executive Secretary will collate any responses and return the report to the person responsible at the BIPM for any further actions needed. Once these actions have been completed, the draft B report will be sent to the Chair of CCRI Section II for approval. Once approved, the CCRI Executive Secretary will arrange publication on the KCDB.

As the comparison will be linked to the SIR, the KCRV (in terms of SIR Equivalent Activity) will be based on previous results of the SIR as well as the result of the linking laboratory for the present CCRI(II) comparison. The evaluation method of the KCRV is the power moderated weighted mean [5]. The final KCRV and the degrees of equivalence will be determined by the BIPM in a separate report which will be circulated to the key comparison working group KCWG(II) and the CCRI(II) for approval.

In the event that there is disagreement concerning the results or the interpretation of the results of a key comparison, and the disagreement cannot be resolved by the participants, by the KCWG(II) or by the CCRI(II), the matter is referred to the CCRI and then the CIPM for decision.

4. References

[1] Gilmore G. and Hemingway J., Practical gamma-ray spectrometry, John Wiley & Sons, 1995.

[2] ISO20042 Measurement of radioactivity -- Gamma-ray emitting radionuclides -- Generic test method using gamma-ray spectrometry. To be published in 2019.

[3] CIPM MRA: *Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes*, International Committee for Weights and Measures, 1999, 45 pp. http://www.bipm.org/en/cipm-mra/.

[4] Measurement comparisons in the CIPM MRA: Guidelines for organizing, participating and reporting, Jan. 2021, 26 pp. https://www.bipm.org/utils/common/documents/CIPM-MRA/CIPM-MRA-G-11.pdf.

[5] Pommé S. and Keightley J., Determination of a reference value and its uncertainty through a power-moderated mean, *Metrologia*, 2015, **52(3),** S200.