Proposal and protocol for a CCRI Supplementary Comparison of ^{57}Co

1. Introduction

In 2004, the International Atomic Energy Agency (IAEA) initiated a Coordinated Research Project (CRP), entitled "Harmonization of Quality Practices for Nuclear Medicine Radioactivity Measurements". One of goals of the project is to develop the necessary methodologies for laboratories to act as secondary radioactivity measurement laboratories in their respective countries as part of the IAEA/World Health Organization (WHO) Secondary Standards Dosimetry Laboratory network, which currently provides calibrations and testing of dosimetry quantities (i.e., absorbed dose).

Because it is envisioned that each member of the network will provide radioactivity calibrations and standards to end users in their respective country, it is necessary for each laboratory to establish measurement traceability to international standards in order to ensure consistency in the results between all laboratories in all countries. One of the primary ways to establish so-called equivalence for the measurement of a particular radionuclide is through direct comparisons of aliquots of the same master solution.

For National Metrology Institutes (NMIs), which are laboratories designated by their respective countries as being responsible for maintaining and disseminating physical standards, equivalence to other NMIs is normally established through participation in Key Comparisons conducted under the aegis of the CCRI(II) of the CIPM. Participation in such exercises is limited, however, to only those institutes that are signatories to the Metre Convention and of the CIPM MRA. For the majority of Member States of the IAEA, these conditions exclude them from the comparisons.

As a follow-up to a recently conducted CCRI Supplementary Comparison of ^{131}I (CCRI(II)-S6.I-131) that was conducted under the above-mentioned CRP, the IAEA is proposing to conduct a comparison of ^{57}Co. The rationale for choosing this particular radionuclide is that comparing the participating laboratories' performance for measurement of a low-energy photon-emitter such as ^{57}Co was necessary to ensure that the participants were in a reasonable position to conduct the next phase of the CRP, which is to organize national comparisons of ^{99}Tc^{m}. Additionally, it provides an opportunity for the participants that are not NMIs to establish traceability to their NMI counterparts for this radionuclide. It is also anticipated that the laboratories that are able to do so (BARC, LNMRI/IRD, CMI-IIR, IFIN-HH, NIST) will prepare ampoules for submission to the SIR as part of the ongoing comparison BIPM.RI(II)-K1.Co-57. It is understood, however, that any such submission is separate from the proposed Supplementary Comparison and can only be made by appropriately accredited laboratories.

2. Comparison Protocol

Pilot Institute: IAEA, with assistance from NIST

List of Participants:
LNMRI/IRD (BRA)
CENTIS (CUB)
CMI-IIR (CZE)
BARC (IND)
Nuclear Science & Technology Research Institute (IRA)
Korean Food and Drug Administration (ROK)
IFIN-HH (ROM)

Department of Nuclear Medicine, University of Ankara (TUR)
NIST (USA)

Comparison Nuclide Solution: ^{57}Co chloride containing nominally 100 MBq at time of shipment to be dispatched by QSA Global GmbH (or other appropriate source supplier); all aliquots are to be dispensed from the same master solution.

Container: Shipping vial chosen by source supplier. Empty vials will be sent to participants prior to shipment of radioactive material to allow for ionization chamber correction factors to be determined.

Mass: approximately 5 g; mass to be provided by source supplier to within 0.1 mg

Chemistry: in carrier containing 1 mol·L^{-1} HCl and approx. 5 μg/g of solution nonactive $CoCl_2$; actual composition to be provided by source supplier

Recommended Nuclear Data: BIPM Monographie BIPM-5, Vol 1, pp 83-89.

2.1 Schedule

Distribution: It is proposed that the solutions will be prepared and distributed to participants in February, 2008.

Based on the assumption that the sources will be distributed in mid-February, the following schedule for reporting is proposed:

Reporting deadline: 14 April 2008

Draft A sent to participants: 16 May 2008

Draft A acceptance deadline: 16 June 2008

Draft B sent to participants: 21 July 2008

Draft B acceptance deadline: 18 August 2008

2.2 Measurand: **The measurand for this exercise is activity per unit mass.**

2.3 The starting date for this programme is 10 December, 2007.

2.4 IAEA shall be responsible for maintaining up-to-date key comparison status reports and shall transmit them to the executive secretary of CCRI(II).

2.5 Each participating institution is responsible for its own costs associated with the measurements, as well as those for transportation and customs and any damage that may occur within its country. The costs associated with organization of the comparison, preparing, calibrating, and shipping the ^{57}Co comparison solutions will be borne by IAEA.

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

2.7 Participants must provide a list and evaluation of the principal components of the uncertainty budget based on the Guide to the Expression of Uncertainty in Measurement, published by ISO. In addition to the principal components of the uncertainty, common to all of the participants, individual institutes must add any other components they consider

appropriate. Uncertainties are evaluated at a level of one standard uncertainty and information must be given on the number of effective degrees of freedom, required for a proper estimate of the level of confidence, where this is appropriate.

2.8 Transport of the ^{57}Co vials to the participants will be arranged by IAEA and QSA Global (or other source supplier of the IAEA's choosing) using their normal radioactive shipment arrangements. Immediately after receipt, the participating institute shall check for any damage to the samples and report this to IAEA.

2.9 If delays occur, IAEA shall inform the participants and revise the schedule, if necessary.

2.10 Other details of the protocol can be found in Annex 1.

3. Preparation of the report on the comparison

NIST and the IAEA will be responsible for the preparation of a report on the comparison. The report passes through a number of stages before publication, and these are referred to here as drafts A and B.

3.1 During the comparison, as the results are received by IAEA/NIST, they are kept confidential until all the participants have completed their measurements and all the results have been received, or until the deadline for receipt of results has passed.

3.2 A result from a participant is not considered complete without an associated uncertainty, and is not included in the draft report unless it is accompanied by an uncertainty supported by a complete uncertainty budget. Uncertainties are drawn up following the guidance given in the technical protocol.

3.3 If, on examination of the complete set of results, IAEA/NIST finds results that appear to be anomalous, the corresponding institutes are 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.

3.4 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.

3.5 Draft A of the 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.

3.6 If any controversial or contradictory comments are received by IAEA/NIST, they will be circulated to all participants and discussion continues until a consensus is reached.

3.7 Draft A is considered as 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 frame of the comparison, as long as no information or comments are made about the comparison results.)

3.8 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 travelling standard (the radioactive solution in this case) or some other phenomenon that renders the comparison or part of it invalid.

3.9 An institute that considers its result unrepresentative of its standards may request a subsequent separate bilateral comparison with one of the other participants. This should take place as soon as possible after the completion of the comparison in progress. The subsequent bilateral comparison is considered as a new and distinct comparison.

3.10 On receipt of final comments from participants, the second draft, draft B, is prepared by the IAEA/NIST incorporating the agreed comments on the draft A.

3.11 The draft B is circulated through the participants. Once agreed, draft B is not considered confidential and may be the subject of a publication, with the exception of the Appendix containing proposals for the reference value and degrees of equivalence.

3.12 The agreed Draft B will be sent to the CCRI(II) for review and approval.

3.13 In the event that there is disagreement concerning the results or the interpretation of the results of the comparison, and the disagreement cannot be resolved by the participants, by the key comparison working group or by the Consultative Committee, the matter is referred to the CIPM for a decision.

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ANNEX 1

REVISED PROTOCOL FOR PERFORMING RADIOACTIVITY MEASUREMENT COMPARISONS BETWEEN CRP PARTICIPANTS

1. Prior to comparison
   a. IAEA to propose comparison as CCRI(II) Supplementary Comparison to BIPM at next meeting of the Key Comparisons Working Group of the BIPM.
   b. IAEA to confirm schedule for comparison after selection of source supplier and notify participants of planned shipping date and reporting due date at least two weeks prior to shipment of source.
   c. IAEA to notify participants of vial type and solution composition once source supplier has been chosen and order placed.
   d. In case of deviation from agreed start date, IAEA to confirm availability of participants for proposed alternative date.
   e. Participants to obtain appropriate licences for shipping/receipt through contact with source supplier and provide appropriate shipping address and contact information (IAEA to facilitate communication between participants and supplier).
   f. Participants to make arrangements for clearance of source through customs and provide appropriate paperwork to source supplier in a timely fashion.
   g. Participants to make up carrier solution with composition identical to that used by source producer, if needed. Information will be provided to participants by IAEA.
   h. IAEA to provide half-life data (with uncertainty), reference date, and reporting forms prior to shipment of sources. Data will be taken from BIPM Monographie 5.

2. When sources are being prepared/shipped
   a. Masses of dispensed solution from the same stock solution to be determined to within 0.1 mg.
   b. Source supplier to provide participants with nominal activity values and actual masses (to within 0.1 mg) of dispensed solution when sources are shipped.
   c. IAEA will notify participants at least two weeks in advance of shipment.

3. Receiving source at SSDL
   a. Check the integrity of the source, confirm that source matches paperwork, including source ID.
   b. Notify IAEA immediately if discrepancy/compromised source discovered.

4. Determination of activity concentration
   a. Activity concentration can be determined by primary calibration if those capabilities are available in the participant’s laboratory. A determination of activity concentration is also possible using pre-calibrated reference instruments (i.e., ionization chamber calibrated against primary method for the specific radionuclide in standard geometry, or alternatively, a calibrated gamma-ray detector).

b. Secondary standards laboratories may determine activity concentration using radionuclide calibrators if they have a calibration factor for their instrument for the specific radionuclide in the geometry in which the incoming source is shipped using procedure in Appendix 1.
c. If no calibration factor for the specific radionuclide in the incoming shipping container is available, the procedure in Appendix 2 must be followed.

5. Additional optional measurements
   a. Impurities
   b. Adsorption

6. Calculation of uncertainties

Uncertainties are to be evaluated in accordance with the ISO *Guide to the Expression of Uncertainties in Measurement*. The reporting forms, which will be provided when the sources are shipped, will provide guidance on identifying and assessing the magnitude of common uncertainty components.

7. Reporting results

The result should be determined, following the method in Appendix 1 or Appendix 2 as appropriate, and reported as activity/mass (kBq/g) at the reference date, together with the combined standard uncertainty (kBq/g).

Appendix I. Determination of activity concentration when calibration factor is available for specific radionuclide in specific container

1. Zero correction (if appropriate)
2. Background correction or measurement
3. Apply calibration factor appropriate for radionuclide in specific measurement geometry (e.g., container type and solution volume)
4. Measure check source, if appropriate (e.g., Ra-226 or Cs-137)
5. Perform a minimum 10 measurement repetitions, removing source from chamber between measurements. Record time of each measurement (middle of measurement if integrating over time period)
6. Take mean of measured activity values; calculate standard deviation
7. Check /re-measure zero; determine mean value/calculate difference (if any) from first measurement; calculate standard deviation
8. Check /re-measure background; determine mean background value/calculate difference (if any) from first measurement; calculate standard deviation
9. If necessary, subtract mean background from mean activity reading
10. Make any corrections for impurities (if determined)
11. Make any additional corrections for geometry, if not already included
12. Divide corrected activity value by solution mass to obtain activity concentration
13. Decay correct the determined value of activity to the reference time.

Appendix 2. Determination of activity concentration when calibration factor is known for container other than shipping container

1. The container, for which the calibration factor is known, will be called in the following the “standard container”.

2. Transfer the solution from the shipping container to the standard container
    a. Weigh empty standard container.
    b. Estimate amount of solution required to be transferred into standard geometry (volume and container)
    c. Transfer the amount of solution from “b“ into the standard container
    d. Weigh the standard container with the transferred solution
    e. Calculate mass of transferred solution by difference of values obtained in “d“ and “a“.
    f. Calculate and transfer the amount of carrier solution (use distilled water if no proper carrier solution is available) needed to bring solution in the standard container to desired volume (if necessary).
3. Determination of activity concentration
    a. Zero correction (if appropriate)
    b. Background correction or measurement
    c. Apply calibration factor appropriate for radionuclide in specific measurement geometry (e.g., container type and solution volume)
    d. Measure check source, if appropriate (e.g., Ra-226 or Cs-137)
    e. Perform a minimum 10 measurement repetitions, removing source from chamber between measurements. Record time of each measurement (middle of measurement if integrating over time period)
    f. Take mean of measured activity values; calculate standard deviation
    g. Check /re-measure zero; determine mean value/calculate difference (if any) from first measurement; calculate standard deviation
    h. Check /re-measure background; determine mean background value/calculate difference (if any) from first measurement; calculate standard deviation
    i. If necessary, subtract mean background from mean activity reading
    j. Make any corrections for impurities (if determined)
    k. Make any additional corrections for geometry, if not already included
    l. To determine activity concentration divide the corrected activity value by the amount of solution transferred from the shipping container (see 2e)
    m. Decay correct the determined value of activity to the reference time.