Technical Protocol for a EURAMET Key Comparison of ¹⁶⁶Ho (EURAMET Project #1435)

1. Introduction

This comparison is carried out in order to link 4 NMIs, i.e. CMI, ENEA-INMRI, CEA and NPL to the BIPM International Reference System (SIR) for the ¹⁶⁶Ho radionuclide.

2. Comparison details

2.1	Pilot laboratory:	CMI, Czech Republic
	Coordinator:	Jaroslav Solc, CMI
	Address:	Czech Metrology Institute
		Regional Branch Praha
		Radiova 1a
		102 00 Praha 10
		Czech Republic
	Tel:	+420 266 020 487
	E-mail:	jsolc@cmi.cz

2.2 Participants: see table 1

Table 1 : Participants of the comparison

NMI	Country	Contact person	Responsible person for activity measurements	E-mail contact person
СМІ	Czech Republic	Jaroslav Solc	Jaroslav Solc	jsolc@cmi.cz
ENEA- INMRI	Italy	Marco Capogni	Marco Capogni	marco.capogni@enea.it
CEA	France	Christophe Bobin	Christophe Bobin	christophe.bobin@cea.fr
NPL	United Kingdom	Andrew Fenwick	Andrew Fenwick	andrew.fenwick@npl.co.uk

2.3 Data of the ¹⁶⁶Ho master solution

Chemical composition of the solution: Approximate activity concentration:	15.9 mg/l Ho(NO₃)₃ in 1 M HNO₃ 25 MBq g⁻¹
Reference date:	29 th November 2017 11:00 UTC
Container:	flame-sealed SIR-type ampoule
Mass:	3.7 g
Recommended nuclear data:	Decay Data Evaluation Project [1], half-life: $T_{1/2}$ = 26.824 (30) h

2.4 Measurand

The measurand for this exercise is the activity per mass in the master solution and the evaluation of nuclear data for 166 Ho.

2.5 Schedule

The exercise started in the last week of November 2017 when a ¹⁶⁶Ho solution, with the chemical composition and approximate activity concentration reported above, was prepared at CMI. From the master solution the following sources were prepared:

- four glass ampoules, type BIPM SIR, ¹⁶⁶Ho filled-in in order to obtain a volume of 3.6 cm³
 liquid solution and ready-to-measure in the Ionization Chamber (IC) of each participating
 Institutes;
- one glass ampoule, type BIPM-SIR, filled-in in order to obtain a volume of 3.6 cm³ of liquid solution and sent to BIPM by CMI.

The set of four glass ampoules, type BIPM SIR, were measured at each participating NMI and at BIPM starting from November 29th 2018 over a period of two weeks. Each participant will provide a value of activity per mass for the received ampoule using a primary activity method ($4\pi\beta-\gamma$ coincidence counting, TDCR and/or CIEMAT/NIST method) or using the own IC calibrated for the ¹⁶⁶Ho through the primary activity methods.

CMI sent to the BIPM a glass SIR ampoule prepared by the same master solution above by 29th November 2017, with the characteristics described above, in order to link the results achieved in the comparison to the BIPM-SIR.

The schedule for reporting is proposed in table 2.

Action	Deadline
End of measurements in the own IC	December 15 th 2017
Data analysis of the IC measurements	February 28 th 2018
Report of Measurements	April 30 st 2018
Draft A sent to participants	May 15 th 2018
Draft A acceptance	May 31 th 2018
Draft B sent to participants	June 15 th 2018
Draft B acceptance	June 30 st 2018

Table 2 : Time schedule of the comparison

2.6 Costs and Funding

The costs associated with the shipping of the ¹⁶⁶Ho comparison solutions from CMI to the participating Institutes are borne by the participants. The costs for the shipping to BIPM are borne by CMI.

This project is carried out within the funded EMPIR joint research project MRTDosimetry.

2.7 Further information

To guarantee confidentiality, each institute will communicate its own results to the Executive Secretary of the CCRI(II), before the Report of Measurements deadline (Table 2), using the standard reporting form for the BIPM-SIR results [2] and describing the methods used for standardisation, the balance of uncertainty, any additional information useful for the comparison, and the final results achieved in own laboratory.

A result from a participant will not be considered complete without an associated uncertainty and will not be included in the comparison report unless it is accompanied by an uncertainty supported by a complete uncertainty budget.

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 the Joint Committee for Guides in Metrology (JCGM) [3]. In addition to the principal components of the uncertainty, common to both participants, each individual institute must add any other components they consider appropriate. Uncertainties are evaluated at a level of one standard deviation 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.

3. Preparation of the report on the comparison

According to the document "Measurement comparisons in the CIPM MRA" [4], the pilot laboratory, CMI, is responsible for the preparation of the Draft-A comparison report, as in the schedule above. For that purpose, immediately after the Report of Measurements deadline for reporting results (Table 2), but after a confirmed submission of the results of all participants to the Executive Secretary of the CCRI(II), the results shall be transmitted also to the pilot laboratory.

If, on examination of the complete set of contributions, the pilot laboratory finds results that appear to be anomalous, the pilot laboratory will invite the corresponding institute to check their result for transcription or arithmetic errors but without indication about the magnitude or sign of the apparent anomaly. If no numerical error is found, the result will stand.

Draft-A is considered as confidential to the participants and will include the results, uncertainties, methods, the analysis carried out, the conclusions reached and other details transmitted by the participants, identified by name. In particular, provisional degrees of equivalence for the for participating NMIs shall also be stated, using the link of CMI to the SIR and subject to the BIPM further update of the KCRV for this radionuclide.

The pilot laboratory will circulate the Draft A to all the participants for comments, with a reasonable deadline for reply. 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. If any controversial comments are received by the pilot laboratory, the discussion will continue until a consensus will be reached.

Note that once all participants have been informed of the results, individual results and uncertainties may be changed or removed or the complete comparison abandoned, only with the agreement of all participants and on the basis of some cause that renders the comparison or part of it invalid.

Due to the confidential character of the Draft A, copies will not be given to non-participants and graphs or other parts of the Draft A cannot be used in oral presentations 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 names of participants hidden. At this stage, a participant may publish experimental techniques or new developments as long as no information or comments are made about the comparison results.

Once the final version of Draft A is approved by the participants, the report is considered as Draft B and shall be sent to the CCRI Executive Secretary who will make a preliminary technical and editorial revision before circulation through the KCWG(II) and the CCRI(II), for comments within a reasonable period of time. At this stage, the results are not considered confidential and can be used to support CMCs and used for presentations and publications with the exception of the proposals for the reference value and degrees of equivalence.

The pilot lab shall take into account the comments received and revise the Draft B, obtaining the agreement of all the participants if necessary. The revised Draft B will be considered as Final Report and shall be sent to the CCRI Executive Secretary for verification purposes, upload into the KCDB and publication in the Metrologia Tech. Suppl. series.

This Technical Protocol was prepared by Dr Marco Capogni (ENEA) and Dr Jaroslav Solc (CMI) and approved by Dr Christophe Bobin (CEA) and Mr Andrew Fenwick (NPL).

References

- [1] http://www.nucleide.org/DDEP_WG/Nuclides/Ho-166_tables.pdf
- [2] https://www.bipm.org/utils/en/pdf/SIR-F-05.pdf
- [3] Evaluation of measurement data Guide to the expression of uncertainty in measurement (JCGM 100:2008) and supplements.
- [4] Measurement comparisons in the CIPM MRA, CIPM-MRA-D-05 (version 1.6). Available online: https://www.bipm.org/utils/common/documents/CIPM-MRA/CIPM-MRA-D-05.pdf