

Update of the BIPM comparison BIPM.RI(II)-K1.Ho-166m of activity measurements of the radionuclide $^{166\text{m}}\text{Ho}$ to include the 2017 results of the NMISA (South Africa) and update of the KCRV

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Abstract

Since 1989, 6 national metrology institutes (NMI) have submitted 7 samples of known activity of $^{166\text{m}}\text{Ho}$ to the International Reference System (SIR) for activity comparison at the Bureau International des Poids et Mesures (BIPM), with comparison identifier BIPM.RI(II)-K1.Ho-166m. The values of the activity submitted were between about 70 kBq and 470 kBq. The key comparison reference value (KCRV) has been re-evaluated using the power-moderated weighted mean to include the PTB and NMISA results dated 2013 and 2017, respectively. There are now five valid results in the KCDB for the BIPM.RI(II)-K1.Ho-166m comparison and the degrees of equivalence between the equivalent activity measured in the SIR and the KCRV have been calculated. The degrees of equivalence for linked regional key comparisons (APMP and EURAMET) were updated.

1. Introduction

The Système International de Référence (SIR) for activity measurements of γ -ray-emitting radionuclides was established in 1976. Each national metrology institute (NMI) may request a standard ampoule from the Bureau International des Poids et Mesures (BIPM) that is then filled with 3.6 g of the radioactive solution. For radioactive gases, a different standard ampoule is used. Each NMI completes a submission form that details the standardization method used to determine the activity of the radionuclide and the full uncertainty budget for the evaluation. The ampoules are sent to the BIPM where they are compared with standard sources of ^{226}Ra using pressurized ionization chambers. Details of the SIR method, experimental set-up and the determination of the equivalent activity, A_e , are given in [1].

From its inception until 31 December 2018, the SIR has been used to measure 1008 ampoules to give 763 independent results for 70 different radionuclides. The SIR makes it possible for national laboratories to check the reliability of their activity measurements at any time. This is achieved by the determination of the equivalent

activity of the radionuclide and by comparison of the result with the key comparison reference value determined from the results of primary standardizations. These comparisons are described as BIPM ongoing comparisons and the results form the basis of the BIPM key comparison database (KCDB) of the Comité International des Poids et Mesures Mutual Recognition Arrangement (CIPM MRA) [2]. The comparison described in this report is known as the BIPM.RI(II)-K1.Ho-166m key comparison. The results of earlier participations in this key comparison were published previously [3, 4, 5].

2. Participants

The National Metrology Institute of South Africa (NMISA) has submitted ampoules in 2017 for inclusion in this comparison. The laboratory details are given in Table 1, with the earlier submissions being taken from [5]. In cases where the laboratory has changed its name since the original submission, both the earlier and the current acronyms are given, as it is the latter that are used in the KCDB. The date of measurement in the SIR given in Table 1 is used in the KCDB and all references in this report.

Table 1. Details of participants in the BIPM.RI(II)-K1.Ho-166m comparison

NMI	Original acronym	Full name	Country	Regional metrology organization	Date of measurement at the BIPM YYYY-MM-DD
LNE-LNHB	BNM-LNHB	Laboratoire national de métrologie et d'essais- Laboratoire national Henri Becquerel	France	EURAMET	1989-11-17
NMIJ	ETL	National Metrology Institute of Japan	Japan	APMP	1999-03-01
IRA	–	Institut de Radiophysique Appliquée	Switzerland	EURAMET	2006-12-22
NPL	–	National Physical Laboratory	United Kingdom	EURAMET	2009-03-02
PTB	–	Physikalisch-Technische Bundesanstalt	Germany	EURAMET	2013-04-03
NMISA	–	National Metrology Institute of South Africa	South Africa	AFRIMETS	2017-09-15

3. NMI standardization methods

Each NMI that submits ampoules to the SIR has measured the activity either by a primary standardization method or by using a secondary method, for example a calibrated ionization chamber. In the latter case, the traceability of the calibration needs to be clearly identified to ensure that appropriate correlations are taken into account.

Details regarding the solutions submitted are shown in Table 2, including any impurities, when present, as identified by the laboratories. When given, the standard uncertainties on the evaluations are shown.

Table 2. Details of each solution of ^{166m}Ho submitted

NMI / SIR year	Chemical composition	Solvent conc. / (mol dm^{-3})	Carrier: conc. / ($\mu\text{g g}^{-1}$)	Density / (g cm^{-3})	Relative activity of any impurity / $10^{-2} \dagger$
LNE-LNHB 1989	HoCl_3 in HCl	1	–	0.998	^{134}Cs : 1.19(5) ^{160}Tb : 0.18(1) ^{154}Eu : 0.21(5) ^{152}Eu : 0.32(2)
NMIJ 1999 [#]	HoCl_3 in HCl	0.1	HoCl_3 : 7700	1.01	^{152}Eu : 0.15(5) ^{154}Eu : 0.60(10)
IRA 2006	HoCl_3 in HCl	0.15	HoCl_3 : 2300	–	– ††
NPL 2009	HoCl_3 in HCl	1	HoCl_3 : 100	1	^{152}Eu : 0.122(12) ^{154}Eu : 0.239(24)
PTB 2013 ^{##}	HoCl_3 in HCl	1	Unknown* : ~ 3900	~1.02	– [9]
NMISA 2017	HoCl_3 in HCl	0.02	HoCl_3 : 1610	1.00	^{152}Eu : 0.09(2) ^{154}Eu : 0.19(4)

[†] the ratio of the activity of the impurity to the activity of ^{166m}Ho at the reference date

^{††} trace impurities of $^{152}, ^{154}, ^{155}\text{Eu}$ had been removed by ion-exchange chromatography

[#] the same solution as used in the APMP comparison (1999)

^{##} dilution of the solution used in the EURAMET comparison (2013)

* mainly rare earth salts

The NMISA used a $4\pi\beta\text{-}\gamma$ coincidence method with four different energy windows in the gamma channel ((1) all energies greater than ~620 keV; (2) ~620 keV to 870 keV; (3) over the 184 keV peak; (4) all energies greater than ~ 74 keV). A brief description of the standardization methods, the activities submitted, the relative standard uncertainties ($k = 1$) and the half life used by the participants is given in Table 3. The

corresponding uncertainty budgets for the present submissions are given in Appendix 1 attached to this report. Previous uncertainty budgets are given in the earlier K1 report [3, 4, 5]. The list of acronyms used to summarize the methods is given in Appendix 2.

Table 3. Standardization methods used by the participants in the ^{166m}Ho comparison

NMI	Method used and acronym (see Appendix 2)	Half-life / a	Activity A_i / kBq	Reference date YYYY-MM-DD	Relative standard uncertainty / 10^{-2} by method of evaluation	
					A	B
LNE-LNHB	$4\pi\gamma(\text{NaI}(\text{Tl}))$ 4P-NA-GR-00-00-HE	1200(200) [6]	110.7 110.9 [#]	1989-10-21 12 h UTC	0.13	0.13
NMIJ	$4\pi\beta\text{-}\gamma$ coincidence 4P-PC-BP-NA-GR-CO	1200	471	1999-03-01 0 h UTC	0.31	0.15
IRA	$4\pi\beta\text{-}\gamma$ coincidence 4P-PC-BP-NA-GR-CO	1200(180) [7]	161.4	2006-02-01 0 h UTC	0.10	0.19
NPL	$4\pi\beta(\text{PPC})\text{-}\gamma$ coincidence* 4P-PP-BP-NA-GR-CO $4\pi\beta(\text{LS})\text{-}\gamma$ coincidence 4P-LS-BP-NA-GR-CO	1200(200) [6]	69.06	2009-02-15 12 h UTC	0.07	0.25
			68.91		0.07	0.25
PTB	$4\pi(\text{LS})$ CIEMAT/ NIST 4P-LS-MX-00-00-CN $4\pi(\text{LS})$ TDCR 4P-LS-MX-00-00-TD	1132.6(3.9) [8]	134.49**	2013-01-01 0 h UTC	0.06***	0.21***
NMISA	$4\pi(\text{LS})\beta\text{-}\gamma$ coincidence 4P-LS-BP-NA-GR-CO	1200(180) [7]	95.285	2017-06-21 10 h UTC	0.03	0.37

[#] two ampoules submitted

* method selected by the NPL for the KCDB

** result obtained as the weighted mean of the results obtained with the methods 4P-LS-MX-00-00-CN and 4P-LS-MX-00-00-TD

*** relative uncertainty of TDCR method adopted by the PTB

4. Results

The SIR equivalent activity, A_{ei} , is given in Table 4 for each NMI, i . The relative standard uncertainties arising from the measurements in the SIR are also shown. This uncertainty is additional to that declared by the NMI for the activity measurement shown in Table 3. Although submitted activities are compared with a given source of

^{226}Ra , all the SIR results are normalized to the radium source number 5 [1]. The $^{166\text{m}}\text{Ho}$ half life used by the BIPM is 1200(200) years [6].

The SIR corrections for impurity are generally small, with a maximum value of 1.016 for LNE-LNHB (1989). This impurity correction for the LNE-LNHB (1989) ampoule has been re-evaluated using recent data for the impurities, giving a result about 0.2 % lower than in the previously published reports [3, 4, 5].

The LNE-LNHB ampoules were measured again in the SIR after 13 years, at which time the impurity correction is ten times lower because of the decay, and the results agree within one combined standard uncertainty.

The NMII ampoule has been re-measured at the BIPM after periods of up to 4 years later producing the same comparison result.

Table 4. Results of SIR measurements of $^{166\text{m}}\text{Ho}$

NMI / SIR year	Mass of solution m_i / g	No. of Ra source used	SIR $A_{e,i}$ / kBq	Relative uncertainty from SIR / 10^{-4}	Combined standard uncertainty u_i / kBq
LNE-LNHB 1989	3.667 74	1	9976	19	26
	3.674 32	1	9998 ^{*,a}	15	24
NMIJ 1999	3.612 12	2	10 051	11	36
IRA 2006	3.536 2(1)	1	9 913	14	26
NPL 2009	3.598 29	1	9 932 ^c	15	30
			9 909		
PTB 2013	3.61719	1	9 912	17.7	28
NMISA 2017	3.571 39	1	9 973	14	40

^{*} results updated as described in the text

^a the mean of the two A_e values is used in the KCDB with an averaged uncertainty, as attributed to an individual entry [11].

^c result from the method 4P-PP-BP-NA-GR-CO selected by the NPL for the KCDB.

No recent submission has been identified as a pilot study so the most recent result of each NMI is normally eligible for inclusion in Appendix B of the MRA.

Two regional comparisons for this radionuclide, APMP.RI(II)-K2.Ho-166m and EURAMET.RI(II)-K2.Ho-166m were held in 1999 and 2013, respectively. More details about these comparisons and links to the BIPM.RI(II)-K1.Ho-166m comparison can be found in [3, 10] for APMP and in [5, 9] for EURAMET.

4.1 The key comparison reference value

In May 2013 the CCRI(II) decided to calculate the key comparison reference value (KCRV) using the power-moderated weighted mean [12] rather than an unweighted mean, as had been the policy. This type of weighted mean is similar to a Mandel-Paule mean in that the NMIs' uncertainties may be increased until the reduced chi-squared value is one. In addition, it allows for a power smaller than two in the weighting factor. Therefore, all SIR key comparison results can be selected for the KCRV with the following constraints:

- a) only results for solutions standardized by primary techniques are accepted, with the exception of radioactive gas standards (for which results from transfer instrument measurements that are directly traceable to a primary measurement in the laboratory may be included);
- b) each NMI or DI may only use one result (normally the most recent result or the mean if more than one ampoule is submitted);
- c) results more than 20 years old are included in the calculation of the KCRV (but are not included in data shown in the KCDB or in the plots in this report as they have expired);
- d) possible outliers can be identified on a mathematical basis and excluded from the KCRV using the normalized error test with a test value of 2.5 and using the modified uncertainties;
- e) results can also be excluded for technical reasons; and
- f) the CCRI(II) is always the final arbiter regarding excluding any data from the calculation of the KCRV.

The data set used for the evaluation of the KCRVs is known as the "KCRV file" and is a reduced data set from the "SIR master-file". Although the KCRV may be modified when other NMIs participate, on the advice of the Key Comparison Working Group of the CCRI(II), such modifications are made only by the CCRI(II) during one of its biennial meetings or by consensus through electronic means (i.e., e-mail) as discussed at the CCRI(II) meeting in 2013.

Consequently, the KCRV for $^{166\text{m}}\text{Ho}$ has been calculated to be 9959(22) kBq with the power $\alpha = 1.5$ on the basis of the SIR results from the LNE-LNHB, NMIJ, IRA, NPL, PTB and NMISA. This can be compared with the previous KCRV value of 9978(33) kBq published in 2009 [4] and the value of 10 040(76) kBq obtained using the SIRIC efficiency curve of the SIR [13].

4.2 Degrees of equivalence

Every participant in a comparison is entitled to have one result included in the KCDB as long as the NMI is a signatory or designated institute listed in the CIPM MRA, and the result is valid (i.e., not older than 20 years). Normally, the most recent result is the one included. An NMI may withdraw its result only if all other participants agree.

The degree of equivalence of a given measurement standard is the degree to which this standard is consistent with the KCRV [2]. The degree of equivalence is expressed quantitatively in terms of the deviation from the key comparison reference value and

the expanded uncertainty of this deviation ($k = 2$). The degree of equivalence between any pair of national measurement standards is expressed in terms of their difference and the expanded uncertainty of this difference and is independent of the choice of key comparison reference value.

4.2.1 Comparison of a given NMI result with the KCRV

The degree of equivalence of the result of a particular NMI, i , with the key comparison reference value is expressed as the difference D_i between the values

$$D_i = A_{e_i} - \text{KCRV} \quad (1)$$

and the expanded uncertainty ($k = 2$) of this difference, U_i , known as the equivalence uncertainty; hence

$$U_i = 2u(D_i). \quad (2)$$

When the result of the NMI i is included in the KCRV with a weight w_i , then

$$u^2(D_i) = (1-2w_i) u_i^2 + u^2(\text{KCRV}). \quad (3)$$

However, when the result of the NMI i is not included in the KCRV, then

$$u^2(D_i) = u_i^2 + u^2(\text{KCRV}). \quad (4)$$

4.2.2 Comparison between pairs of NMI results

The degree of equivalence between the results of any pair of NMIs, i and j , is expressed as the difference D_{ij} in the values

$$D_{ij} = D_i - D_j = A_{e_i} - A_{e_j} \quad (5)$$

and the expanded uncertainty ($k = 2$) of this difference, $U_{ij} = 2u(D_{ij})$, where

$$u_{D_{ij}}^2 = u_i^2 + u_j^2 - 2u(A_{e_i}, A_{e_j}) \quad (6)$$

where any obvious correlations between the NMIs (such as a traceable calibration, or correlations normally coming from the SIR or from the linking factor in the case of linked comparison) are subtracted using the covariance $u(A_{e_i}, A_{e_j})$ (see [14] for more detail). However, the CCRI decided in 2011 that these “pair-wise degrees of equivalence” no longer need to be published as long as the methodology is explained.

Table 5 shows the matrix of all the degrees of equivalence as they will appear in the KCDB. It should be noted that for consistency within the KCDB, a simplified level of nomenclature is used with A_{e_i} replaced by x_i . The introductory text is that agreed for the comparison. The graph of the results in Table 5, corresponding to the degrees of equivalence with respect to the KCRV (identified as x_R in the KCDB), is shown in Figure 1. This graphical representation indicates in part the degree of equivalence between the NMIs but obviously does not take into account the correlations between the different NMIs. It should be noted that the final data in this paper, while correct at the time of publication, will become out-of-date as NMIs make new comparisons. The formal results under the CIPM MRA [2] are those available in the KCDB.

Conclusion

The BIPM ongoing key comparison for $^{166\text{m}}\text{Ho}$, BIPM.RI(II)-K1.Ho-166m currently comprises five valid results. The key comparison reference value has been re-evaluated using the power-moderated weighted mean to include the PTB and NMISA results. The SIR results have been analysed with respect to the updated KCRV determined for this radionuclide, providing degrees of equivalence for these five participants.

The results of six other NMIs that took part in the APMP.RI(II)-K2.Ho-166m comparison and of three other NMIs and the IRMM (now called JRC) that took part in the EURAMET.RI(II)-K2.Ho-166m comparison were previously linked to the BIPM on-going key comparison. These linked results are included in the matrix of degrees of equivalence and are shown on the graph. This means that the table of degrees of equivalence includes fifteen results in total.

The degrees of equivalence have been approved by the CCRI(II) and are published in the BIPM key comparison database. Further results may be added when other NMIs contribute $^{166\text{m}}\text{Ho}$ activity measurements to the ongoing K1 comparison or take part in other linked comparisons.

References

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Table 5. Table of degrees of equivalence and introductory text for ^{166m}Ho

Key comparison BIPM.RI(II)-K1.Ho-166m

MEASURAND : Equivalent activity of ^{166m}Ho

Key comparison reference value: the SIR reference value for this radionuclide is $x_R = 9959$ kBq with a standard uncertainty, $u_R = 22$ kBq (see Section 4.1 of the Final Report). The value x_i is the equivalent activity for laboratory i .

The degree of equivalence of each laboratory with respect to the reference value is given by a pair of terms:

$D_i = (x_i - x_R)$ and U_i , its expanded uncertainty ($k = 2$), both expressed in MBq, and $U_i = 2((1 - 2w_i)u_i^2 + u_R^2)^{1/2}$ when each laboratory has contributed to the calculation of x_R .

Linking APMP.RI(II)-K2.Ho-166m (1999) to BIPM.RI(II)-K1.Ho-166m

The value x_i is the equivalent activity for laboratory i participant in APMP.RI(II)-K2.Ho-166m having been normalized using the value of the linking laboratory, NMIJ (see 2003 Final report).

The degree of equivalence of laboratory i participant in APMP.RI(II)-K2.Ho-166m with respect to the key comparison reference value is given by a pair of terms: $D_i = (x_i - x_R)$ and U_i , its expanded uncertainty ($k = 2$), both expressed in MBq. The approximation $U_i = 2(u_i^2 + u_R^2)^{1/2}$ is used in the following table.

Linking EURAMET.RI(II)-K2.Ho-166m (2013) to BIPM.RI(II)-K1.Ho-166m

The value x_i is the equivalent activity for laboratory i participant in EURAMET.RI(II)-K2.Ho-166m having been normalized using the value of the linking laboratory, PTB (see 2015 Final report).

The degree of equivalence of laboratory i participant in EURAMET.RI(II)-K2.Ho-166m with respect to the key comparison reference value is given by a pair of terms: $D_i = (x_i - x_R)$ and U_i , its expanded uncertainty ($k = 2$), both expressed in MBq. The approximation $U_i = 2(u_i^2 + u_R^2)^{1/2}$ is used in the following table.

These statements make it possible to extend the BIPM.RI(II)-K1.Ho-166m matrices of equivalence to the other participants in the APMP.RI(II)-K2.Ho-166m and EURAMET.RI(II)-K2.Ho-166m.

Table 5 continued

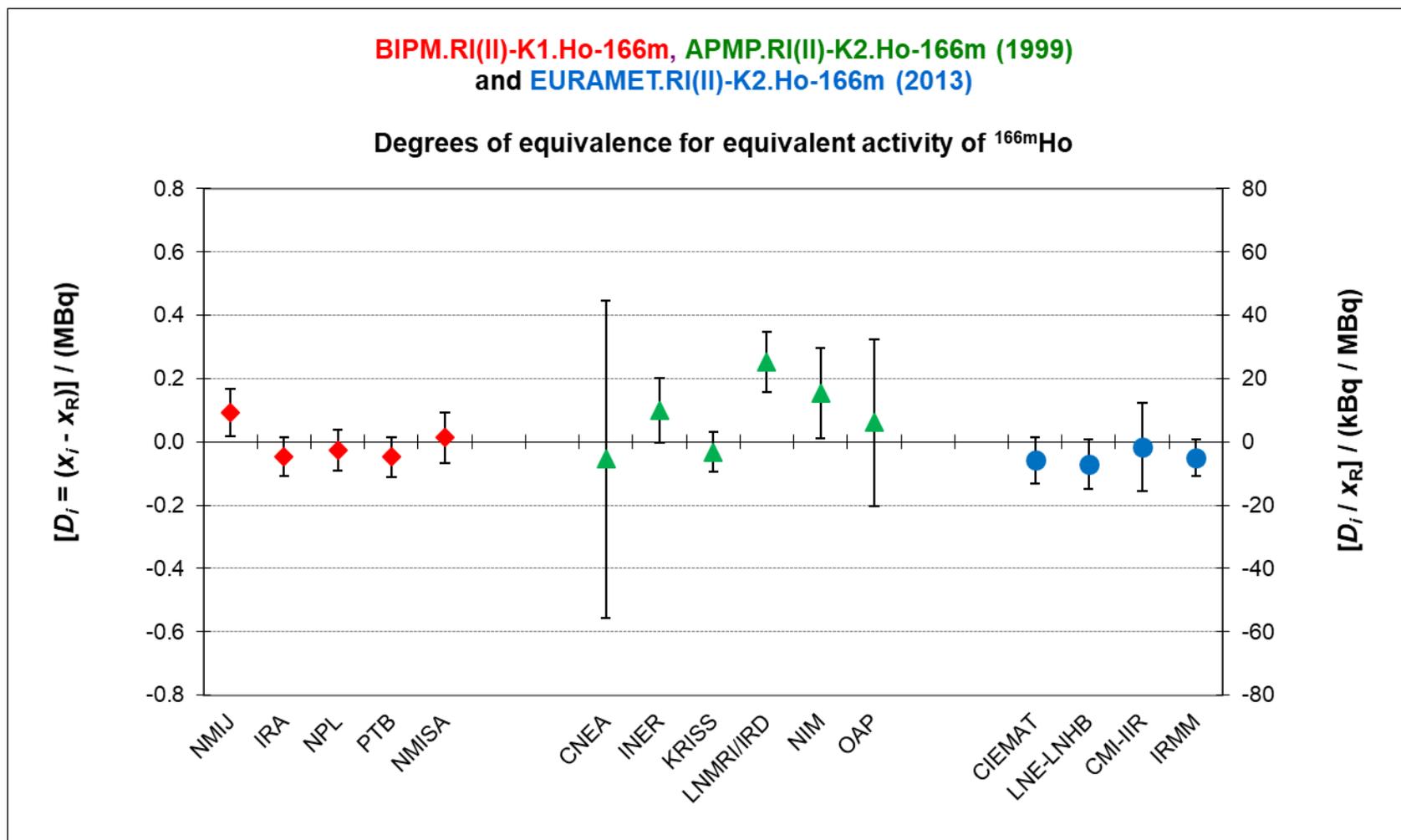
Lab *i*

	D_i	U_i
	/ MBq	
NMIJ	0.09	0.07
IRA	-0.05	0.06
NPL	-0.03	0.07
PTB	-0.05	0.07
NMISA	0.01	0.08

CNEA	-0.05	0.50
INER	0.10	0.10
KRISS	-0.03	0.06
LNMRI/IRD	0.25	0.10
NIM	0.15	0.14
OAP	0.06	0.26

CIEMAT	-0.06	0.07
LNE-LNHB	-0.07	0.08
CMI-IIR	-0.02	0.14
IRMM	-0.05	0.06

Figure 1. Graph of degrees of equivalence with the KCRV for ^{166m}Ho
 (as it appears in Appendix B of the MRA)



N.B. Right-hand axis shows approximate values only

Appendix 1. Uncertainty budget for measurements of the activity of $^{166\text{m}}\text{Ho}$ submitted to the SIR

The NMISA has submitted a detailed uncertainty budget as follows:

Component	Relative standard uncertainties		Comment	Relative sensitivity factors
	$u_{rel,i} / 10^{-2}$	Method		
counting statistics	0.03	A	Standard deviation of the mean of 56 values	0.13
weighing (primary source prep.)	0.05	B		1
weighing (SIR ampoule prep.)	0.01	B		1
dead time	0.01	B	$\Delta\tau_D \pm 0.05 \mu\text{s}$	0.002
background	0.02	B	Background square root statistics applied	0.002
counting time	0.001	B	Calibration of timer	1
decay correction	0.0001	B		8×10^{-6}
efficiency extrapolation	0.3	B	Half range between extrapolation using 1 st order and 2 nd order polynomials, divided by $\sqrt{3}$	1
coincidence resolving time	0.01	B	$\Delta\tau_R \pm 0.01 \mu\text{s}$	0.005
afterpulse correction	0.04	B	Based on formula for $\Delta\theta$	0.0007
Adsorption in LS glass vial	0.05	B	Count rates after multiple rinsings relative to the count rate from the source measured beforehand	1
radionuclide impurities	0.2	B	HPGe measurements	1
Relative combined standard uncertainty, u_c	0.37			

Appendix 2. Acronyms used to identify different measurement methods

Each acronym has six components, geometry-detector (1)-radiation (1)-detector (2)-radiation (2)-mode. When a component is unknown, ?? is used and when it is not applicable 00 is used.

Geometry	acronym	Detector	acronym
4π	4P	proportional counter	PC
defined solid angle	SA	press. prop counter	PP
2π	2P	liquid scintillation counting	LS
undefined solid angle	UA	NaI(Tl)	NA
		Ge(HP)	GH
		Ge(Li)	GL
		Si(Li)	SL
		CsI(Tl)	CS
		ionization chamber	IC
		grid ionization chamber	GC
		Cerenkov detector	CD
		calorimeter	CA
		solid plastic scintillator	SP
		PIPS detector	PS
Radiation	acronym	Mode	acronym
positron	PO	efficiency tracing	ET
beta particle	BP	internal gas counting	IG
Auger electron	AE	CIEMAT/NIST	CN
conversion electron	CE	sum counting	SC
mixed electrons	ME	coincidence	CO
bremsstrahlung	BS	anti-coincidence	AC
gamma rays	GR	coincidence counting with efficiency tracing	CT
X - rays	XR	anti-coincidence counting with efficiency tracing	AT
photons ($x + \gamma$)	PH	triple-to-double coincidence ratio counting	TD
alpha - particle	AP	high efficiency	HE
mixture of various radiation	MX		

Examples

method	acronym
4π (PC) β - γ -coincidence counting	4P-PC-BP-NA-GR-CO
4π (PPC) β - γ -coincidence counting eff. trac.	4P-PP-MX-NA-GR-CT
defined solid angle α -particle counting with a PIPS detector	SA-PS-AP-00-00-00
4π (PPC)AX- γ (GeHP)-anticoincidence counting	4P-PP-MX-GH-GR-AC
4π CsI- β ,AX, γ counting	4P-CS-MX-00-00-HE
calibrated IC	4P-IC-GR-00-00-00
internal gas counting	4P-PC-BP-00-00-IG