Update of the BIPM comparison BIPM.RI(II)-K1.Lu-177 of activity measurements of the radionuclide ^{177}Lu to include the 2023 result of the CMI (Czechia)

Romain Coulon[1], **Carine Michotte**[1], **Sammy Courte**[1], **Manuel Nonis**[1], **Vincent Gressier**[1], **Jana Sochorová**[2], **Pavel Auerbach**[2]

[1] Bureau International des Poids et Mesures, Pavillon de Breteuil, F-92312 Sèvres Cedex, France.

[2] Czech Metrology Institute (CMI), Brno 638 00, Czechia.

E-mail: `cmichotte@bipm.org`

Abstract Since 2000, 8 laboratories have submitted 8 samples of ^{177}Lu 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.Lu-177. Recently, the CMI (Czechia) participated in the comparison. The degrees of equivalence between each equivalent activity measured in the SIR or linked to the SIR from the CCRI(II)-K2.Lu-177 comparison have been calculated and the results are given in the form of a table. A graphical representation is also given.

1. Introduction

The 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 BIPM that is then filled with 3.6 g of the radioactive solution. Each NMI completes a submission form that details the standardization method used to determine the absolute 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 all given in [1].

From its inception until 31 December 2023, the SIR has been used to measure 1054 ampoules to give 807 independent results for 72 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 continuous 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.Lu-177 key comparison. The results of earlier participations in this key comparison were published previously [3–5].

Successful participation in this comparison by a laboratory may provide evidential support for Calibration and Measurement Capability (CMC) claims for ^{177}Lu measured using the laboratory's method(s) used in the comparison or methods calibrated by those used for the comparison. This comparison may also be used to support CMC claims for those radionuclides measured in the laboratory using the same method and having a degree of difficulty at or below that of the radionuclide measured in this comparison as indicated in the current Measurement Methods Matrix (MMM) [6].

2. Participants

Laboratory details are given in Table 1, with the earlier submissions being taken from [3–5]. The dates of measurement in the SIR given in Table 1 are used in the KCDB and all references in this report.

Table 1: Details of the participants in the BIPM.RI(II)-K1.Lu-177.

NMI or laboratory	**Previous acronyms or other institutes**	**Full name**	**Country**	**Regional Metrology Organization (RMO)**	**Date of SIR measurement** yyyy-mm-dd
CMI	UVVVR, CMI-IIR	Czech Metrology Institute	Czechia	EURAMET	2023-09-12
IFIN-HH	-	Institutul National de Cercetare - Dezvoltare pentru Fizica si Inginerie Nucleara "Horia Hulubei"	Romania	EURAMET	2013-07-11
IRA	IER	Institut de Radiophysique	Switzerland	EURAMET	2022-06-23
JRC	IRMM, CBNM	EC-JRC Institute for Reference Materials and Measurements	European Union	EURAMET	2009-04-30
LNE-LNHB	LMRI, LPRI, BNM-LNHB	Université Paris-Saclay, CEA, List, Laboratoire National Henri Becquerel	France	EURAMET	2014-06-25
NIST	NBS	National Institute of Standards and Technology	United States	SIM	2000-02-15
NPL	-	National Physical Laboratory	United Kingdom	EURAMET	2009-04-28
PTB	-	Physikalisch-Technische Bundesanstalt	Germany	EURAMET	2000-01-26

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.

A brief description of the standardization methods used by the laboratories, the activities submitted, the relative standard uncertainties and the half life used by the participants are given in Table 2. The uncertainty budget for the new submission is given in Appendix D attached to this report; previous uncertainty budgets are given in the earlier reports [3–5]. The list of acronyms used to summarize the methods is given in Appendix E.

The half life used by the BIPM is 6.647(4) days as published in BIPM Monographie 5 vol. 2 [7].

Table 2: Standardization methods of the participants for ^{177}Lu.

NMI or laboratory	**Method used and the acronym**	**Activity** A_i**/MBq**	**Relative standard uncertainty /**10^{-2}		**Reference date**	**Half life /d**
			A	**B**	**yyyy-mm-dd**	
CMI	4π(PC)β-γ coincidence (4P-PC-BP-NA-GR-CO)	11 622.3	0.1	0.42	2023-09-08 10:00 UT	6.647(4) [7]
IFIN-HH	4π(PC)β-γ coincidence (4P-PC-BP-NA-GR-CO)	8155.72	0.55	0.7	2013-07-10 12:00 UT	[7]
IRA	$4\pi\beta$(PS)-$4\pi\gamma$ coincidence (4P-PS-BP-4P-NA-GR-CO)	20 924.5[a]	0.41	0.17	2022-06-15 12:00 UT	6.6463(15) [8]
JRC	CIEMAT/NIST (4P-LS-MX-00-00-CN) 4π(PC)β-γ coincidence (4P-PC-BP-NA-GR-CO)	2462[b]		1.64	2009-05-01 12:00 UT	6.647(4) [7]
LNE-LNHB	4π(PC)β-γ anti-coincidence (4P-PC-BP-NA-GR-AC)	5829.05	0.22	0.12	2014-06-23 12:00 UT	[7]
NIST	CIEMAT/NIST (4P-LS-MX-00-00-CN)	133 850	0.19	0.28	2000-02-01 17:00 UT	6.60(1)
NPL	4π(PC)β-γ coincidence (4P-PC-BP-NA-GR-CO) CIEMAT/NIST (4P-LS-MX-00-00-CN) 4π(LS)β-γ coincidence (4P-LS-BP-NA-GR-CO)	11 790[c]	0.03	0.3	2009-05-01 12:00 UT	6.647(4) [7]
PTB	CIEMAT/NIST (4P-LS-MX-00-00-CN)[d]	138 790[e]	0.2	0.4	2000-01-19 00:00 UT	6.646(5) [9]

... Continuation of Table 2.

NMI or laboratory	**Method used and the acronym**	**Activity A_i/MBq**	**Relative standard uncertainty /10^{-2}**		**Reference date**	**Half life /d**
			A	**B**	**yyyy-mm-dd**	
	4π(PC)β-γ coincidence (4P-PC-BP-NA-GR-CO)					

[a] The activity is the arithmetic mean of 12 efficiency extrapolated activities obtained with 4 plastic scintillation sources, from 2 dilutions, and 3 γ settings. The degrees of freedom of each of the twelve efficiency extrapolations ranged between 31 and 38.

[b] Partially weighted mean (power=1) of the rather discrepant results obtained with the two methods indicated.

[c] Corresponds to an activity concentration of 3286 kBq·g^{-1} evaluated as the arithmetic mean of the three results obtained with the three methods

[d] The result the weighted mean of the two methods.

[e] The result is the mean of the different methods.

Details regarding the solutions submitted are shown in Table 3, including any impurities, when present, as identified by the laboratories. When given, the standard uncertainties on the evaluations are shown.

Table 3: Details of each solution of ^{177}Lu submitted.

NMI or laboratory / SIR year	**Chemical composition**	**Solvent conc. /(mol dm^{-3})**	**Carrier conc. /(µg g^{-1})**	**Density /(g cm^{-3})**	**Relative activity of any impurity**[d]
CMI 2023	$LuCl_3$ in HCl	0.04	$LuCl_3$:1.25	1	none detected
IFIN-HH 2013	$LuCl_3$ in HCl	0.1	$LuCl_3$:2	1	^{177m}Lu: 12(6)x10^{-3} %
IRA 2022	nonactive Lu^{3+} ions in HCl	0.1	nonactive Lu^{3+} ions:20	1.000(5)	none detected
JRC 2009[a]	$LuCl_3$ in HCl	1	$LuCl_3$:20	1.037	^{177m}Lu: 40(10)x10^{-3} % 33.5(7)x10^{-3} % [e]
LNE-LNHB 2014	HCl	0.1	$LuCl_3$:10	1	^{177m}Lu: 10(1)x10^{-3} %
NIST 2000	Lu in HCl	0.01	-	1	^{177m}Lu: 27.1(5)x10^{-3} %[b]
NPL 2009[c]	$LuCl_3$ in HCl	1	$LuCl_3$:20	1	^{177m}Lu: 33(3)x10^{-3} %
PTB 2000	$LuCl_3$ in HCl	0.1	$LuCl_3$:20	0.999	^{177m}Lu: 6.9(6) x 10^{-3} %

[a] Same solution as for the CCRI(II)-K2 Lu-177 comparison but diluted by a factor of 4.955317

[b] Measured at the BIPM

[c] Same solution as for the CCRI(II)-K2 Lu-177 comparison

[d] The ratio of the activity of the impurity to the activity of ^{177}Lu at the reference date

[e] Weighted mean value from the CCRI(II)-K2.Lu-177 comparison, used for the evaluation of the KCRV and for the link of the K2 comparison

4. Results

All the submissions to the SIR since its inception in 1976 are maintained in a dedicated database based on CSV formatted files controlled by the Git version control system [10]. A machine-readable version of this report (XML document) is attached to this document [11]. The latest submission has added 1 ampoule for the activity measurements of ^{177}Lu giving rise to 8 ampoules in total.

The SIR equivalent activity, A_{ei}, for each ampoule received from each NMI, i, including both previous and new results, is given in Table 4. The relative standard uncertainties arising from the measurements in the SIR are also shown. This uncertainty is additional to that declared by the NMI ($u(A_i)$) for the activity measurement shown in Table 2. 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]. Table 4 also shows the comparison results selected for the KCRV as explained in section 4.1.

The CMI ampoule has been measured for four consecutive days in the SIR, yielding results that agree within one combined standard uncertainty.

Table 4: Results of SIR measurement of ^{177}Lu.

NMI or laboratory / SIR year	Mass m_i /g	A_i /MBq	^{226}**Ra source**	A_{ei} /MBq	**Relative uncert. from SIR /10^{-4}**	u_{ci} /MBq	A_{ei} **for KCRV /MBq**
CMI 2023	3.611 20(72)	11 622.3	1	559.4[a]	12	2.5	
IFIN-HH 2013	3.597 584	8155.72	1	550.3[a]	25	5.1	-
IRA 2022	3.569 06(20)	20 924.5	1	539.7[a]	16	2.5	-
JRC 2009	3.674 99	2462	1	566.3 565.2[e]	33 13	9.5[b] 9.3	 565.2(93)
LNE-LNHB 2014	3.65	5829.05	1	560.2[a]	16	1.7	-
NIST 2000	3.627 4	133 850	3	551.5	14[c]	2.0	-
NPL 2009	3.589 37	11 790	2	559.5	13[d]	1.8	559.5(18)
PTB 2000	3.675 5	138 790	3	559.0	11	2.7	559.0(27)

[a] It has been decided during the CCRI(II) meeting held in June 2023 to not update the KCRV, so this result is not included in the KCRV caculation.

[b] Dominated by the uncertainty in the impurity correction in the SIR measurement due to the large uncertainty on the ^{177m}Lu activity stated by the participant.

[c] Result not considered for the KCRV calculation, see details in [4]

[d] Result used to link to CCRI(II)-K2 comparison

[e] Result when the weighted mean ^{177m}Lu activity value from the CCRI(II)-K2 comparison is used in the impurity correction of the SIR measurement.

The CCRI(II)-K2.Lu-177 comparison was held in 2009 [12]. The results were linked to the BIPM.RI(II)-K1.Lu-177 comparison through the measurement in the SIR of at least one ampoule of the CCRI(II)-K2 comparison as explained in [4].

4.1. The key comparison reference value

In May 2013, the CCRI(II) decided to calculate the key comparison reference value (KCRV) by using the power-moderated weighted mean [13] 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 α smaller than two in the weighting factor. As proposed in [13], α is taken as $2 - 3/N$ where N is the number of results selected for the KCRV. Therefore, all SIR key comparison results can be selected for the KCRV with the following provisions:

(a) results for solutions standardized by only 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 other laboratory may use only 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.

In view that at least one participant found discrepant results depending on the method used without finding the reason, the CCRI(II) agreed in June 2023 not to update the KCRV and invited NMIs to carry out investigations on the standardization of Lu-177 and participate in the further SIR comparisons. The new CMI result enriches the comparison, but does not allow us to rule on the suspected outlier. Pending further participation, the KCRV is maintained identical to the value of 559.9(18) MBq published in 2014 [4]. This is in conformance with the SIRIC estimation of 560.1(31) MBq [14].

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). No recent submission has been identified as a pilot study so the most recent result of each NMI is normally eligible for inclusion on the KCDB platform of the CIPM MRA [2]. 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

$$D_i = A_{\mathrm{e}i} - \mathrm{KCRV} \tag{1}$$

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

$$U_i = 2u(D_i) \tag{2}$$

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

$$u^2(D_i) = (1 - 2w_i)u_i^2 + u^2(\mathrm{KCRV}) \tag{3}$$

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

$$u^2(D_i) = u_i^2 + u^2(\mathrm{KCRV}) \tag{4}$$

The introductory text in Appendix A is the one agreed by the CCRI(II) for all the K1 comparisons.

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

$$D_{ij} = D_i - D_j = A_{\mathrm{e}i} - A_{\mathrm{e}j} \tag{5}$$

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

$$u^2(D_{ij}) = u_i^2 + u_j^2 - 2u(A_{\mathrm{e}i}, A_{\mathrm{e}j}) \tag{6}$$

where any obvious correlations between the NMIs (such as a traceable calibration, correlations normally coming from the SIR, or from the linking factor in the case of linked comparison) are subtracted using the covariance $u(A_{\mathrm{e}i}, A_{\mathrm{e}j})$ (see [15] 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.

Appendix B 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_{ei} 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 C1. This graphical representation indicates in part the degree of equivalence between the NMIs but obviously does not take into account the correlations between 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.

5. Conclusion

The BIPM continuous key comparison for ^{177}Lu, BIPM.RI(II)-K1.Lu-177, currently comprises 6 valid results. The SIR results, together with the previously published CCRI(II)-K2.Lu-177 results, have been analyzed with respect to the KCRV, providing degrees of equivalence for 13 national metrology institutes. Other results may be added when other NMIs contribute ^{177}Lu activity measurements to this comparison or take part in other linked comparisons.

6. References

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Appendix A. Introductory text for ^{177}Lu degrees of equivalence

Key comparison BIPM.RI(II)-K1.Lu-177

MEASURAND: Equivalent activity of ^{177}Lu

Key comparison reference value: the SIR reference value x_{R} for this radionuclide is 559.9 MBq , with a standard uncertainty,u_{R} equal to 1.8 MBq (see Section 4.1 of the Final Report). The value x_i is taken as the equivalent activityfor a laboratory i.

The degree of equivalence of each laboratory with respect to the reference value is given by a pair of terms: $D_i = (x_i - x_{\mathrm{R}})$ and U_i, its expanded uncertainty ($k = 2$), both expressed in MBq, and $U_i = 2((1 - 2w_i)u_i^2 + u_{\mathrm{R}}^2)^{1/2}$, where w_i is the weight of laboratory i contributing to the calculation of x_{R}.

Appendix B. Table of degrees of equivalence for BIPM.RI(II)-K1.Lu-177

Table B1: The table of degrees of equivalence for BIPM.RI(II)-K1.Lu-177

NMI i	D_i **/MBq**	U_i **/MBq**
JRC	5	17
NPL	-0.4	3.5
IFIN-HH	-10	11
LNE-LNHB	0.2	5.0
IRA	-20.2	6.2
CMI	-0.5	6.2

Table B2: The table of degrees of equivalence for the CCRI(II)-K2.Lu-177(2009) comparison

NMI i	D_i **/MBq**	U_i **/MBq**
ANSTO	-4.1	4.7
ENEA-INMRI	4.2	8.5
LNMRI-IRD	-2.1	7.8
NIST	-0.5	5.3
NMISA	0.7	4.8
POLATOM	-1.7	7.2
PTB	-1.3	4.4

Appendix C. Graph of degrees of equivalence with the KCRV for ^{177}Lu (as it appears in Appendix B of the MRA)

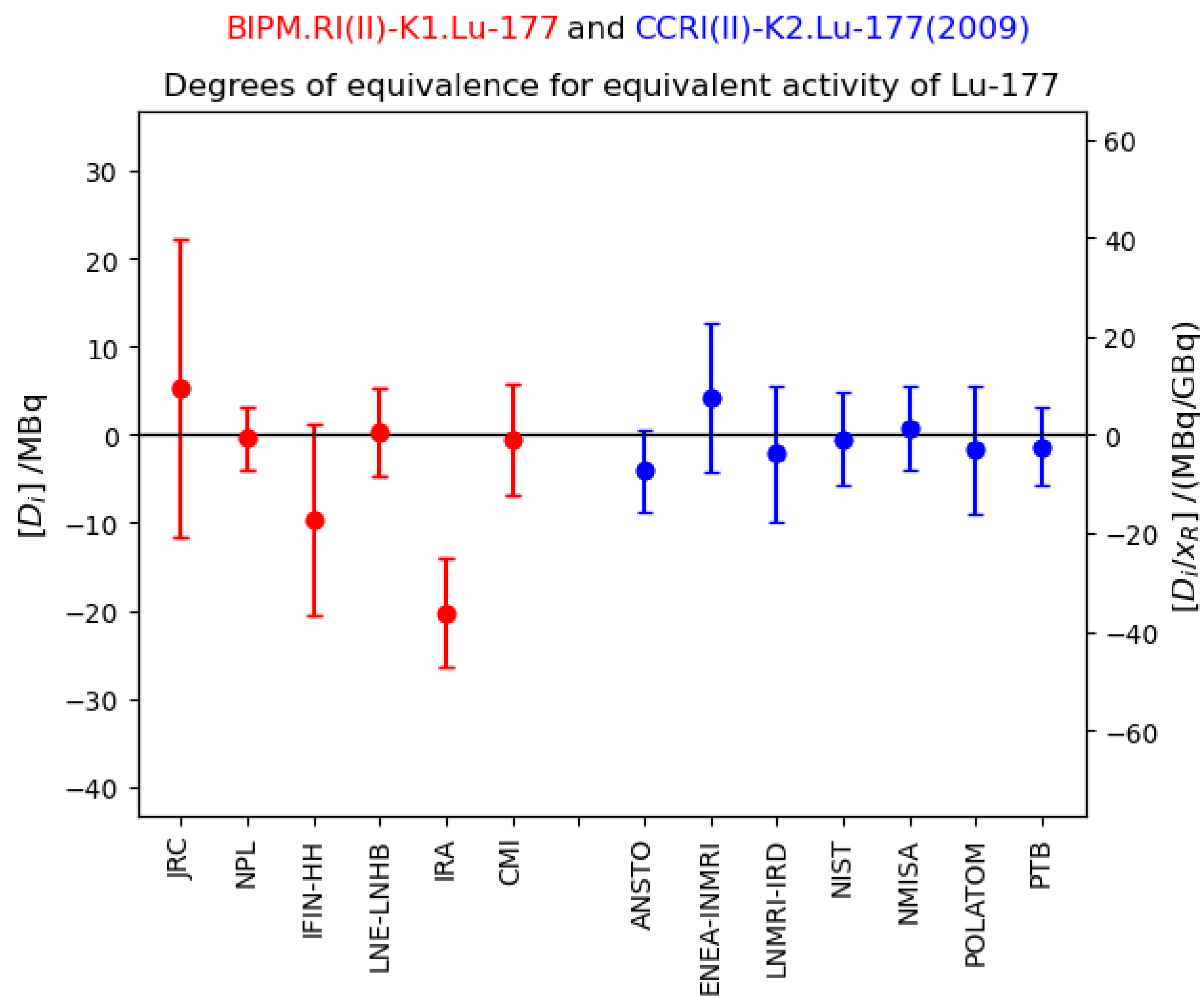

Figure C1. Degrees of equivalence for equivalent activity of ^{177}Lu.

Appendix D. Uncertainty budgets for the activity of ^{177}Lu submitted to the SIR

Uncertainty budget from CMI

SIR/SIRTI reporting form - radioactive solution page 3a

BIPM.RI(II)-K1 or BIPM.RI(II)-K4

Measurement method	4π (PPC) β-γ coincidence	
ACRONYM	4P-PC-BP-NA-GR-CO	**Comments:**
Activity concentration at reference date / kBq g^{-1}	3218.4000	
Relative standard uncertainty / 10^{-2}	0.42	
Date of measurement at the NMI (YYYY-MM-DD)	2023-09-08	

For relative methods:

Primary methods or standards used for calibration	
Date of calibration	
Date of primary measurement	

Uncertainty budget

Uncertainty component	Relative uncertainty / 10^{-2}	Evaluation type (A or B)	Comment
Counting statistics	0.100	A	
Background	0.100	B	
Weighing	0.010	B	
Dilution	0.050	B	
Dead time	0.010	B	
Resolving time	0.020	B	
Pile-up, afterpulse			
Adsorption			
Impurities	0.030	B	
Decay correction			
Decay data			
Extra-/Inter-polation of efficiency curve	0.400	B	
Quenching, kB value			
Tracer			
Reproducibility			
Combined standard uncertainty	**0.420**		

Appendix E. 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**
4 π	4P	proportional counter	PC
defined solid angle	SA	press. Prop. Counter	PP
2 π	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
		CeBr3	CB

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	anticoincidence	AC
gamma rays	GR	coincidence counting with efficiency tracing	CT
x-rays	XR	anticoincidence counting with efficiency tracing	AT
photons $(x + \gamma)$	PH	triple-to-double coincidence ratio counting	TD
photons + electrons	PE	selective sampling	SS
alpha particle	AP	high efficiency	HE
mixture of various radiation	MX	digital coincidence counting	DC

Examples of methods	**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
4πCsI-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