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Comparison reports

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# CCRI(II)-K2.Tc-99

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## Activity of the radionuclide $^{99}\text{Tc}$ per unit mass of aqueous solution

**KEY COMPARISON**

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## **Results of the CCRI(II)-K2.Tc-99 key comparison for the activity of the radionuclide <sup>99</sup>Tc per unit mass of aqueous solution**

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## **Abstract**

This is the final report for the CCRI(II)-K2.Tc-99 key comparison of the activity of the radionuclide <sup>99</sup>Tc per unit mass of aqueous solution which was organised in 2012 by the National Physical Laboratory of the United Kingdom. The Key Comparison Reference Value (KCRV) of 56.45 kBq g<sup>-1</sup> with a combined standard uncertainty of 0.13 kBq g<sup>-1</sup> was determined from the power-moderated mean of the results of 13 laboratories, with results from a further three laboratories being rejected by the accepted statistical criteria.

## **Keywords**

Radioactivity, primary measurement, activity per unit mass, <sup>99</sup>Tc radionuclide, environmental radioactivity, liquid scintillation counting

## **Introduction**

Technetium-99 is a long-lived beta emitting fission product which occurs in radioactive waste and in the environment. The need for a comparison was identified following the observation by several laboratories that the model-based liquid scintillation techniques were highly sensitive to the selection of beta emission spectrum used in the efficiency computations. A comparison of  $^{99}\text{Tc}$  (ground state) activity per unit mass of solution (sometimes termed “activity concentration” or “massic activity”) was agreed during the 21st meeting of the Consultative Committee for Ionising Radiation Section II (CCRI(II)) in 2011. The National Physical Laboratory (NPL) in the UK was the pilot laboratory for this exercise.

Participants were recommended to use the nuclear decay data from the Decay Data Evaluation Project (DDEP) published in BIPM Monographie 5 [1], which had been updated in 2010 to include an evaluation of the shape factor of the main ground state to ground state beta emission [2]. The recommended half-life was taken from the same data source converted into days by the piloting laboratory and was  $7.72(4) \times 10^7$  d. The reference date was set to 1 March 2012, 12 h UTC.

All the participants reported their results to BIPM directly using the Excel-based reporting forms. These have been interpreted manually to produce the tables and charts in this report.

Successful participation in this comparison by a laboratory may provide evidential support for Calibration and Measurement Capability (CMC) claims for  $^{99}\text{Tc}$  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) [3].

## **Participants**

The comparison had 16 participants which are listed in table A1.

## **Sample preparation and distribution**

The comparison material consisted of  $^{99}\text{Tc}$  as ammonium pertechnetate in  $0.1 \text{ mol dm}^{-3}$  ammonium hydroxide solution. The activity per unit mass was nominally  $50 \text{ kBq g}^{-1}$  to  $100 \text{ kBq g}^{-1}$ . Aliquots of nominally 3 g of solution were gravimetrically dispensed using a plastic pycnometer to a series of 30 flame-sealed glass 5 ml NBS ampoules in December 2011. Prior to use each ampoule had been washed with Crawley's solution (60 % v/v deionised water, 35 % nitric acid, 3 % hydrofluoric acid and 2 % teepol) using method III described on page 23 of BIPM Monographie 1 [4]. To check the homogeneity of the solution, an aliquot of nominally 0.1 g solution was dispensed to liquid scintillation vials containing 10 ml Ecoscint A between every five ampoules and bracketing the first and last ampoules. These vials were counted using a conventional two-photomultiplier system and the vials formed a consistent set with the relative standard deviation of the mean count rate being 0.26 %.

The samples were distributed to the participants in January 2012 with a reporting deadline of 31 May 2012. All participants received one ampoule each, with the exception of BIPM, CIEMAT and LNHB who each requested and were provided with additional ampoules.

### **Measurement methods**

The list of the participants and the methods used is provided in table A2. The majority of participants reported at least one result based on one of the free-parameter liquid scintillation counting techniques: either the CIEMAT/NIST technique (4P-BP-LS-00-00-CN, or CN for short) or the Triple- to Double-Coincidence Ratio (4P-BP-LS-00-00-TD, or TD for short) technique. Some participants elected to refer to these techniques as 4P-MX-LS-00-00-CN and 4P-MX-LS-00-00-TD respectively, depending on whether beta decay to the first excited state was considered. In summarising the data, these methods have been considered equivalent due to the probability of the decay to the first excited state ( $0.00145 \% \pm 0.00030 \%$ , DDEP) being far below any reported relative uncertainty. Both the CN and TD techniques are model-based and participants used a range of different codes to model the detection efficiencies. Critical model parameters typically include the ionisation quench factor (or selection of  $kB$  in the widely-used Birks' model [5]), linear energy transfer ("stopping power") and the shape of the beta spectrum, most notably in this case the effect of the beta-decay shape factor. Where reported, these data have

been tabulated in table A3. All participants used  $^3\text{H}$  as a tracer in the CN technique however it is of note that NMISA also used  $^{63}\text{Ni}$  as a tracer.

For a radionuclide exhibiting primarily ground state to ground state beta decay, the spectrum shape calculation is often one of the most significant factors in determining the activity by free parameter modelling techniques. The transition type for  $^{99}\text{Tc}$  is second forbidden non-unique ( $9/2^+ \rightarrow 5/2^+$ ). The majority of participants reported using either the shapefactor from the recommended data or another empirically-derived shapefactor in the form  $C(W) = q^2 + \lambda_2 p^2$  with  $\lambda_2$  assumed constant and in the range 0.522 to 0.54. In some cases (CIEMAT/NIST results from BIPM and ENEA) participants reported using an assumed value of  $\lambda_2$  of 1. Although the conventional conditions for the  $\xi$  approximation are met ( $2\xi/E_0 \approx 42$ ), for a decay of this type discrepancies might be expected [6] and a measured shape factor may yield more accurate activity determinations. At the time of this comparison, atomic electron exchange effects [7] were not generally taken into account, and not all modelling codes will have accounted for atomic screening effects [8]. These can be significant, as has been demonstrated for  $^{60}\text{Co}$  [9]. The beta spectrum of  $^{99}\text{Tc}$  has very recently been measured by Paulsen et al. [10].

The  $kB$  values used in the calculation of efficiency are also tabulated in table A3. These should be interpreted with caution since they are multiplied by the linear energy transfer function in the Birks's equation, and it is not always meaningful to compare one without knowledge of the other [11]. For codes based on EFFY [12, 13], a  $kB$  of 0.0075-0.008  $\text{cm MeV}^{-1}$  was reported in CN measurements. For the TDCR code TDCR07c [14] a  $kB$  of 0.0075  $\text{cm MeV}^{-1}$  was exclusively used and for other TDCR codes use of a  $kB$  in the range 0.011-0.013  $\text{cm MeV}^{-1}$  was reported.

For liquid scintillation measurements the scintillation cocktail used can be significant when the radionuclide is in non-acidic media as is the case here. Count rate instability can be observed when counting radionuclides in basic solutions, possibly due to either micelle instability, precipitation or sorption or penetration into the vessel wall. Liquid scintillation sources containing basic solutions often also exhibit relatively high chemical quenching. These effects can be problematic in both the model-based methods and the coincidence-based methods.

It should be noted this comparison pre-dated the widespread implementation of micelle size effect corrections in the TD and CN methods, and as such participants did not generally report these in either methods or uncertainties. It has been suggested that any error introduced by neglecting these effects would likely be minimal in beta decay [15].

Several laboratories reported results based upon efficiency traced coincidence counting techniques (BARC, CIEMAT, IFIN-HH [16], NIST, NMISA, NRC and VNIIM). These techniques are not influenced greatly by input parameters, however the choice of tracer, the extrapolation model and the dead time correction method are significant. For measurements based on proportional counting, the choice of source mount and counting gas can also be important.

Where the extrapolation method and dead time correction method were reported, this data is tabulated in tables A4 and A5 for proportional counter and measurements based on liquid scintillation counting respectively. Participants specifying the use of a tracer radionuclide exclusively reported the use of  $^{60}\text{Co}$ . A comparison of the beta spectra of  $^{60}\text{Co}$  and  $^{99}\text{Tc}$  calculated with the BetaShape code [6, 17] is given in figure A1. Spectrum shape calculations are not required in coincidence-based techniques so reported sensitivities to the inclusion of exchange and screening corrections will have no impact on the reported results.

### **Radionuclidic impurities**

Eleven participants reported impurity measurements by high resolution gamma spectrometry. IFIN-HH and VNIIM reported detection limits determined by this method relative to technetium-99 of 0.01 % and 0.005 % respectively. In addition, CIEMAT and ENEA reported using the differential decay technique and NIST reported the use of alpha spectrometry. Three participants did not report impurity measurements. No radionuclidic impurities were identified by any of the participants.

### **Uncertainty Evaluations**

A complete breakdown of all the reported uncertainties can be found in table A6. In the “other uncertainties” section of the reporting form, uncertainties related to ionisation quench, counting time and photomultiplier tube (PMT) asymmetry were

commonly reported and have been tabulated separately. All other components reported under “other” have been summed in quadrature for conciseness.

### **Measurement results**

A complete tabulation of the results of all methods submitted is presented in table A7. The results submitted by the National Metrology Institutes (NMIs) for inclusion in the KCRV in table A7 are marked with an asterisk and are also plotted in figure A2. The values of various estimators for the activity per unit mass based on statistical analysis of the NMI results are presented in table A8.

A plot of all the submitted participant values (excluding means) sorted by method can be found in figure A3. For clarity, those results reported as “MX” and “BP” are reported together.

### **The Key Comparison Reference Value (KCRV)**

The key comparison reference value is based upon the power-moderated mean (PMM) given in table A8 and is 56.45 kBq g<sup>-1</sup> with a combined standard uncertainty of 0.13 kBq g<sup>-1</sup>. In the calculation of the KCRV, three values were excluded by the accepted statistical criteria of being more than 2.5 standard deviations away from the PMM. Due to the relatively high dispersion of the data in comparison with the reported uncertainties, the PMM is very close to the arithmetic mean of the reduced data set.

### **Degrees of equivalence**

The degree of equivalence between the result of a National Metrology Institute  $A_i$  and the KCRV (table A9 and figure A4) is a measure of the consistency of the individual measurement with the KCRV. This is expressed in terms of the deviation from the KCRV  $D_i$  and the expanded uncertainty of this deviation  $U_i$ , applying a coverage factor of  $k = 2$ :

$$D_i = A_i - KCRV \quad [1]$$

$$U_i = 2 u(D_i) \quad [2]$$

When the result of the institute  $i$  is included in the KCRV with a weight  $w_i$ , then:

$$u^2(D_i) = (1 - 2 w_i) u_i^2 + u^2(KCRV) \quad [3]$$

Where required, the degree of equivalence between any pair of National Metrology Institute measurements  $i$  and  $j$  is expressed in terms of their difference  $D_{i,j}$  and the expanded uncertainty of this difference  $U_{i,j}$ , again applying a coverage factor of  $k = 2$ . There is no longer a requirement to include these data in the report so long as the methodology required to derive them is described.

$$D_{i,j} = D_i - D_j = A_i - A_j \quad [4]$$

$$u^2(D_{i,j}) = u_i^2 + u_j^2 - 2 u(A_i, A_j) \quad [5]$$

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## Appendix

Table A1. List of participating organisations, principal contacts and other contributors. Organisational titles, affiliations and e-mail addresses are stated as correct at the time of the exercise.

Participant	Country	Primary contact	Other contributors
BARC	India	Leena Joseph ( <a href="mailto:leena@barc.gov.in">leena@barc.gov.in</a> )	D. B. Kulkarni Anuradha Ravindra
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Participant	Country	Primary contact	Other contributors
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VNIIM	Russia	S. V. Sepman ( <a href="mailto:ssv@vniim.ru">ssv@vniim.ru</a> )	I. A. Sokolova A. V. Zanevsky

Table A2. Measurement methods (part I) - all methods for which results were submitted by the participating laboratories.

Participant	Methods used (acronym)
BARC	4P-BP-PC-GR-NA-CT, 4P-BP-LS-GR-NA-CT (x2), 4P-BP-LS-00-00-CN
BIPM	4P-BP-LS-00-00-CN (x3)
CIEMAT	4P-BP-LS-00-00-CN, 4P-BP-PC-GR-NA-CT
CNEA	4P-BP-LS-00-00-TD
ENEA	4P-MX-LS-00-00-CN, 4P-MX-LS-00-00-TD (x2)
IFIN-HH	4P-BP-LS-00-00-TD, 4P-BP-PC-GR-NA-CT
JRC	4P-BP-LS-00-00-CN, 4P-BP-LS-00-00-TD
LNE-LNHB	4P-BP-LS-00-00-TD (x2)
NIST	4P-BP-LS-GR-NA-AT, 4P-BP-LS-00-00-CN, 4P-BP-LS-00-00-TD
NMIJ	4P-BP-LS-00-00-CN
NMISA	4P-BP-LS-00-00-TD, 4P-BP-LS-00-00-CN, 4P-BP-LS-GR-NA-CT
NPL	4P-BP-LS-00-00-CN, 4P-BP-LS-00-00-TD
NRC	4P-BP-PP-GR-NA-AT
POLATOM	4P-BP-LS-00-00-TD
PTB	4P-MX-LS-00-00-CN, 4P-MX-LS-00-00-TD
VNIIM	4P-BP-PC-00-00-HE, 4P-BP-PC-00-00-HE

Table A3. Measurement methods (part I) – parameters of free parameter liquid scintillation based techniques. Where information is not relevant, this is marked as “not applicable” (n/a). Where information could not be found in the submitted forms, this is marked as “not specified” (n/s).

Laboratory	Tracer	Scintillation cocktail(s)	Number of PMTs	Max. Eff. <sup>99</sup> Tc	Model code	Ionisation quench parameter $kB$ / cm MeV <sup>-1</sup>	Beta decay shapefactor
BARC	<sup>3</sup> H	Ultima Gold	2	0.9049	CN2003	0.0075	Not specified
BIPM (CN1)	<sup>3</sup> H	BioFluor+	2	0.97	EFFY5	0.0075	$q^2 + p^2$
BIPM (CN2)	<sup>3</sup> H	Hionic Fluor	2	0.962	EFFY5	0.0075	$q^2 + p^2$
BIPM (CN3)	<sup>3</sup> H	Ultima Gold	2	0.97	EFFY5	0.0075	$q^2 + p^2$
CIEMAT	<sup>3</sup> H	Hisafe III	2	0.964	EFFY	0.0075	$q^2 + 0.529 p^2$
CNEA	n/a	Ultima Gold AB	3	0.9653	TDCR11	0.011-0.013	$q^2 + 0.54 p^2$
ENEA (CN)	<sup>3</sup> H	Ultima Gold	2	0.9541	CN2004	0.0075	$q^2 + p^2$
ENEA (TD)	n/a	Ultima Gold	3	0.9724	TDCR07c	0.0075	$q^2 + 0.54 p^2$
JRC (TD)	n/a	Ultima Gold/Instafluor plus	3	0.955	TDCRB-02p	0.0115	$q^2 + 0.54 p^2$
JRC (CN)	<sup>3</sup> H	Ultima Gold/Instafluor plus	2	0.963	CN2005	0.0075	$q^2 + 0.54 p^2$
LNE-LNHB	n/a	Ultima Gold/Ultima Gold AB/Hionic Fluor	3	0.97	TDCR07c	0.007-0.015	$q^2 + 0.54 p^2$

Laboratory	Tracer	Scintillation cocktail(s)	Number of PMTs	Max. Eff. <sup>99</sup> Tc	Model code	Ionisation quench parameter $kB$ / cm MeV <sup>-1</sup>	Beta decay shapefactor
NIST (CN)	<sup>3</sup> H	Hionic Fluor/Hisafe III	2	0.955	TRACER	0.013 ± 0.001	$q^2 + (0.54 \pm 0.02) p^2$
NIST (TD)	n/a	Hionic Fluor	3	0.94	Local	0.012 ± 0.001	$q^2 + (0.54 \pm 0.02) p^2$
NMISA (CN)	<sup>3</sup> H/ <sup>63</sup> Ni	Quicksafe A + Aliquat-336	2	0.949	Local/EFFY2	0.008 ± 0.001	$q^2 + 0.522 p^2$
NMISA (TD)	n/a	Quicksafe A + Aliquat-336	3	0.896	Local/EFFY2	0.008 ± 0.001	$q^2 + 0.522 p^2$
NPL (CN)	<sup>3</sup> H	Hisafe III	2	0.959	Local/BETA	0.0075	$q^2 + 0.529 p^2$
NPL (TD)	n/a	Hisafe III	3	0.97	TDCRb02	0.012	n/s
POLATOM	n/a	Ultima Gold	3	0.9547	TDCRB-03	0.012	$q^2 + 0.54 p^2$
PTB (CN)	<sup>3</sup> H	Ultima Gold	2	0.958	Local/EFFY4	0.0075	$q^2 + 0.54 p^2$
PTB (TD)	n/a	Ultima Gold	3	0.977	Local/EFFY4	0.0075	$q^2 + 0.54 p^2$

Table A4. Measurement methods (part II) – parameters of proportional-counter based techniques.

Laboratory	Tracer	Source type	Wetting agents	Counting gas	Max. efficiency	Dead time / $\mu$ s	Dead time characteristics	Extrapolation method
BARC	$^{60}\text{Co}$	VYNS	Teflon	LPG	0.86	$5.1 \pm 0.2$	Non-extending	Voltage reduction
CIEMAT	$^{60}\text{Co}$	VYNS	Not specified	P10	0.85	$10.00 \pm 0.01$	Non-extending	Not specified
IFIN-HH	$^{60}\text{Co}$	VYNS	Not specified	Methane	0.83	$10.0 \pm 0.5$	Non-extending	Not specified
NRC	$^{60}\text{Co}$	VYNS	Ludox, Catanac	P10	0.92	$5.33 \pm 0.01$	Extending, live timed	Threshold variation
VNIIM	None	Cellulose nitrate	Insulin	P10	0.98	$1.2 \pm 0.1$	Non-extending	Not specified
VNIIM	Not specified	Cellulose nitrate	Insulin	P10	0.90	$1.2 \pm 0.1$	Non-extending	Not specified

LPG: Liquified petroleum gas

P10: 90 % argon, 10 % methane

Table A5. Measurement methods (part III) – parameters of LSC-based coincidence techniques. [1] Ultima Gold and Hionic Fluor are trademarks of PerkinElmer Inc. [2] Quicksafe is a trademark of Zinsser Analytic GmbH.

Laboratory	Tracer	Scintillant cocktail	PMTs	Max. efficiency	Dead time / $\mu\text{s}$	Dead time characteristics	Extrapolation method
BARC	$^{60}\text{Co}$	Ultima Gold [1]	1	0.902	$4.60 \pm 0.23$	Non-extending	Voltage reduction
BARC	$^{60}\text{Co}$	Ultima Gold [1]	1	0.902	$4.60 \pm 0.23$	Non-extending	Quench addition
NIST	$^{60}\text{Co}$	Hionic fluor [1], Aliquat-336	1	0.93	30-60 (anti-coincidence)	Extending	Threshold variation
NMISA	$^{60}\text{Co}$	Quicksafe A [2], Aliquat-336	2	0.88	$1.13-1.19 \pm 0.10$	Non-extending	Threshold variation





Table A6 (continued)

Quantity Q	BIPM 4P-BP-LS-00-00-CN (3)			CIEMAT 4P-BP-LS-00-00-CN			CIEMAT 4P-BP-PC-GR-NA-CT		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	0.07	0.07	A	0.1	0.1	A	*	0.43	A
Weighing	0.023	0.023	B	0.04	0.04	B	*	0.17	B
Background	4.47	0.02	A	0.04	0.04	A	*	0.78	A
Dead/live time	-	-	-	0.1	0.1	B	*	0.00017	B
Resolving time	-	-	-	-	-	-	*	0.13	B
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	negligible	*	*	*	0.02	*	-	-	-
Quenching	*	0.25	B	0.25	0.08	B	-	-	-
Tracer	0.51	0.25	B	0.4	0.04	B	*	0.38	B
Extrapolation/Interpolation	-	-	-	*	0.36	*	-	-	-
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	0.52	negligible	B	0	0	B	-	-	-
Impurities	-	-	-	0	0	B	-	-	-
Adsorption	-	-	-	0.05	0.05	B	-	-	-
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $kB$ )	-	-	-	*	0.1	B	-	-	-
Counting time	-	-	-	*	0.1	B	-	-	-
PMT asymmetry	-	-	-	-	-	-	-	-	-
Other	-	-	-	-	-	-	*	0.018	A

Table A6 (continued)

Quantity Q	CNEA 4P-BP-LS-00-00-TD			ENEA 4P-MX-LS-00-00-CN			ENEA 4P-MX-LS-00-00-TD (1)		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	0.03	0.03	A	0.250	*	A	0.250	*	A
Weighing	0.1	0.1	B	0.05	*	A	0.05	*	A
Background	0.03	0.01	A	0.004	*	A	0.004	*	A
Dead/live time	0.02	0.02	B	0.1	*	B	0.1	*	B
Resolving time	-	-	-	-	-	-	-	-	-
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	5	0.07	B	0.05	*	B	0.05	*	B
Quenching	-	-	-	0.3	*	A	-	-	-
Tracer	-	-	-	0.03	*	B	-	-	-
Extrapolation/Interpolation	-	-	-	-	-	-	-	-	-
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	0.5	< 0.01	B	0.01	*	B	0.01	*	B
Impurities	-	-	-	0.07	*	A/B	0.07	*	A/B
Adsorption	-	-	-	0.02	*	B	0.02	*	B
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $kB$ )	18	0.08	B	0.2	*	B	0.2	*	B
Counting time	-	-	-	0.01	*	*	-	-	-
PMT asymmetry	-	-	-	0.1	*	-	0.1	*	B
Other	-	-	-	0.11	*	B	*	*	B

Table A6 (continued)

Quantity Q	ENEA 4P-MX-LS-00-00-TD (2)			IFIN 4P-BP-LS-00-00-TD			IFIN-HH 4P-BP-PC-GR-NA-CT		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	0.250	*	A	0.132	0.132	A	0.75	0.75	A
Weighing	0.05	*	A	0.1	0.1	B	0.1	0.15	B
Background	0.004	*	A	*	0.005	B	50 and 15	0.4	B
Dead/live time	0.1	*	B	0.83	0.15	B	5	0.1	B
Resolving time	-	-	-	-	-	-	0.5	0.008	B
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	0.05	*	B	0.48	0.01	B	-	-	-
Quenching	-	-	-	-	-	-	-	-	-
Tracer	-	-	-	-	-	-	0.27	0.27	B
Extrapolation/Interpolation	-	-	-	-	-	-	0.36	0.36	B
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	0.01	*	B	-	-	-	-	-	-
Impurities	0.07	*	A/B	0.01	0.01	B	0.01	0.01	B
Adsorption	0.02	*	B	0.1	0.1	B	0.1	0.1	B
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $kB$ )	0.2	*	B	0.07	0.07	B	-	-	-
Counting time	-	-	-	-	-	-	-	-	-
PMT asymmetry	0.2	*	B	-	-	-	-	-	-
Other	0.14	*	B	0.30	0.30	B	-	-	-

Table A6 (continued)

Quantity Q	JRC 4P-BP-LS-00-00-CN			JRC 4P-BP-LS-00-00-TD			LNHB 4P-BP-LS-00-00-TD (1)		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	0.18	0.18	A	0.2	0.2	A	0.05	0.05	A
Weighing	0.12	0.12	B	0.12	0.12	B	$7 \cdot 10^{-4}$	0.07	B
Background	6.2	0.006	A	1.8	0.01	A	0.05	0.05	A
Dead/live time	0.1	0.1	B	0.1	0.1	B	0.005	0.005	B
Resolving time	-	-	-	-	-	-	$1 \cdot 10^{-4}$	0.1	B
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	0.003	0.003	B
Decay data	0.4	0.4	B	0.4	0.4	B	0.5	0.02	B
Quenching	1	0.001	B	-	-	-	-	-	-
Tracer	0.7	0.16	B	-	-	-	-	-	-
Extrapolation/Interpolation	0.3	0.07	B	-	-	-	-	-	-
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	$7.30 \cdot 10^{-7}$	$7.30 \cdot 10^{-7}$	B	$7.30 \cdot 10^{-7}$	$7.30 \cdot 10^{-7}$	B	0.52	$3 \cdot 10^{-6}$	B
Impurities	-	-	-	-	-	-	0.01	0.01	B
Adsorption	0.005	0.005	A	0.005	0.005	A	0	0.03	*
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $k_B$ )	-	-	-	-	-	-	25	0.13	A
Counting time	-	-	-	-	-	-	-	-	-
PMT asymmetry	-	-	-	-	-	-	-	-	-
Other	0.1	0.1	B	0.2	0.2	B	0.14	0.14	B

Table A6 (continued)

Quantity Q	LNHB 4P-BP-LS-00-00-TD (2)			NIST 4P-BP-LS-GR-NA-AT			NIST 4P-BP-LS-00-00-CN		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	*	0.1	A	*	0.13	A	*	0.08	A
Weighing	*	0.1	B	*	0.1	B	*	0.1	B
Background	*	0.05	A	-	-	-	*	-	B
Dead/live time	*	0.01	B	*	0.1	B	*	0.06	B
Resolving time	-	-	-	-	-	-	-	-	-
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	*	-	-	-	-	-	*	0.3	B
Quenching	-	-	-	-	-	-	-	-	-
Tracer	-	-	-	-	-	-	0.16	$5 \cdot 10^{-4}$	B
Extrapolation/Interpolation	-	-	-	*	0.20	B	-	-	-
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	*	0.05	B	*	0.003	B	0.52	0	B
Impurities	-	-	-	*	$5 \cdot 10^{-6}$	B	*	0.002	B
Adsorption	-	-	-	-	-	-	-	-	-
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $kB$ )	*	0.2	B	-	-	-	-	-	-
Counting time	-	-	-	-	-	-	-	-	-
PMT asymmetry	*	0.05	B	-	-	-	-	-	-
Other	-	0.3	B	-	-	-	-	-	-

Table A6 (continued)

Quantity Q	NIST 4P-BP-LS-00-00-TD			NMIJ 4P-BP-LS-00-00-CN			NMISA 4P-BP-LS-00-00-TD		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	*	0.01	A	0.1	0.1	A	0.025	0.025	A
Weighing	*	0.05	B	0.05	0.05	B	0.125	0.125	B
Background	*	$3 \cdot 10^{-5}$	A	0.02	0.02	B	2.3	0.01	A
Dead/live time	-	-	-	-	-	-	10	0.05	B
Resolving time	-	-	-	-	-	-	-	-	-
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	0.48	0.07	A	-	-	-	3.4	0.03	B
Quenching	-	-	-	-	-	-	12.5	0.1	B
Tracer	-	-	-	0.8	0.8	B	-	-	-
Extrapolation/Interpolation	-	-	-	-	-	-	-	-	-
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	*	$9.40 \cdot 10^{-8}$	A	0.07	0.07	B	0.52	0	B
Impurities	-	-	-	-	-	-	-	-	-
Adsorption	-	-	-	-	-	-	15.5	0.0071	B
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $kB$ )	*	0.06	B	-	-	-	-	-	-
Counting time	-	-	-	-	-	-	-	-	-
PMT asymmetry	-	-	-	-	-	-	-	-	-
Other	-	-	-	-	-	-	-	0.33	B

Table A6 (continued)

Quantity Q	NMISA 4P-BP-LS-00-00-CN			NMISA 4P-BP-LS-GR-NA-CT			NPL 4P-BP-LS-00-00-CN		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	0.07	0.07	A	0.11	0.11	A	0.0297	0.0297	A
Weighing	0.16	0.16	B	0.15	0.15	B	0.014	0.006	B
Background	1.3	0.01	A	5.15	0.02	A	10.9	0.0012	A
Dead/live time	10	0.05	B	10	0.003	B	0.15	0.087	B
Resolving time	-	-	-	4.15	0.011	B	-	-	-
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	3.4	0.13	B	-	-	-	0.87	0.87	B
Quenching	12.5	0.16	B	-	-	-	-	-	B
Tracer	0.66	0.15	B	0.3	0.3	B	1.2	0.0741	B
Extrapolation/Interpolation	-	-	-	0.1	0.1	B	0.0358	0.0358	A
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	0.52	0	B	0.52	0	B	<0.001	<0.001	B
Impurities	-	-	-	-	-	-	-	-	-
Adsorption	15.5	0.0071	B	15.5	0.0071	B	*	*	B
Self-absorption	-	-	-	-	-	-	-	-	-
Ionisation quench ( $kB$ )	-	-	-	-	-	-	-	-	-
Counting time	-	-	-	-	-	-	-	-	-
PMT asymmetry	-	-	-	-	-	-	-	-	-
Other	-	0.28	B	-	0.16	B	-	0.16	B

Table A6 (continued)

Quantity Q	NPL 4P-BP-LS-00-00-TD			NRC 4P-BP-PP-GR-NA-AT			POLATOM 4P-BP-LS-00-00-TD		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	0.07	0.07	A	*	0.2	A	0.020	0.020	A
Weighing	0.02	0.011	B	*	0.02	B	0.005	0.054	B
Background	0.06	0.06	A	*	0.01	B	-	-	-
Dead/live time	0.02	0.02	B	*	0.001	B	-	-	-
Resolving time	-	-	-	-	-	-	-	-	-
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	0.03	0.03	B	-	-	-	-	-	-
Decay data	0.6	0.6	B	-	-	-	3.704	0.018	B
Quenching	n/a	n/a	B	-	-	-	-	-	-
Tracer	-	-	-	*	0.1	B	-	-	-
Extrapolation/Interpolation	0.95	0.95	B	*	1.3	B	0.209	0.209	B
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	<0.001	*	B	-	-	-	0.518	1.07e-7	B
Impurities	-	-	-	-	-	-	0.1	0.1	B
Adsorption	*	*	B	-	-	-	0.01	0.01	B
Self-absorption	<0.001	<0.001	B	-	-	-	-	-	-
Ionisation quench ( <i>kB</i> )	-	-	-	-	-	-	0.150	0.150	B
Counting time	-	-	-	-	-	-	-	-	-
PMT asymmetry	-	-	-	-	-	-	-	-	-
Other	-	0.14	B	-	-	-	0.200	0.2	B

Table A6 (continued)

Quantity Q	PTB 4P-MX-LS-00-00-CN			PTB 4P-MX-LS-00-00-TD			VNIIM 4P-BP-PC-00-00-HE		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	*	0.03	*	*	0.01	*	*	0.029	B
Weighing	*	0.06	*	*	0.06	*	*	0.056	A
Background	*	0.03	*	*	0.03	*	*	0.058	A
Dead/live time	*	0.10	*	*	0.03	*	*	0.017	B
Resolving time	-	-	-	-	-	-	-	-	-
Gandy effect	-	-	-	-	-	-	-	-	-
Pile-up	-	-	-	-	-	-	-	-	-
Decay data	*	0.40	*	*	0.18	*	-	-	-
Quenching	*	0.03	*	*	0.01	*	-	-	-
Tracer	*	0.06	*	-	-	-	-	-	-
Extrapolation/Interpolation	-	-	-	-	-	-	-	-	-
Calibration factor	-	-	-	-	-	-	-	-	-
Half-life	*	< 0.01	*	*	<0.01	*	*	0	B
Impurities	*	0.03	*	*	0.03	*	-	-	-
Adsorption	*	0.05	*	*	0.05	*	-	-	-
Self-absorption		-	-	-	-		*	0.539	A
Ionisation quench ( $kB$ )	*	0.08	*	*	0.14	*	-	-	-
Counting time	*	< 0.01	*	*	< 0.01	*	-	-	-
PMT asymmetry	-	-	-	*	0.02	*	-	-	-
Other	-	-	-	-	-	-	*	0.172	A

Table A6 (continued)

Quantity Q	VNIIM 4P-PC-BP-GR-NA-CT		
	$u(Q)/Q$ / %	$u(A)/A$ / %	Type (A/B)
Counting statistics	*	0.055	B
Weighing	*	0.348	A
Background	*	0.262	A
Dead/live time	*	0.023	B
Resolving time	*	0.007	A
Gandy effect	-	-	-
Pile-up	-	-	-
Decay data	-	-	-
Quenching	-	-	-
Tracer	-	-	-
Extrapolation/Interpolation	*	0.281	A
Calibration factor	-	-	-
Half-life	*	0	B
Impurities	-	-	-
Adsorption	-	-	-
Self-absorption	-	-	-
Ionisation quench ( $k_B$ )	-	-	-
Counting time	-	-	-
PMT asymmetry	-	-	-
Other	-	-	-

Table A7. Compiled reported laboratory results with their combined standard uncertainties. Rows marked with an asterisk (\*) are those values selected by the laboratory for inclusion in the KCRV. Where the participant reported a mean result for inclusion in the KCRV, these are also tabulated, but means have not been calculated or tabulated unless reported by the participant.

Laboratory	Method acronym	Result / kBq g <sup>-1</sup>	Uncertainty / kBq g <sup>-1</sup>
BARC	4P-BP-PC-NA-GR-CT	55.23	0.52
BARC	4P-BP-LS-NA-GR-CT	55.26	0.43
BARC	4P-BP-LS-NA-GR-CT	55.72	0.47
BARC	4P-BP-LS-00-00-CN	55.31	0.32
BARC *	Arithmetic mean	55.38	0.22
BIPM	4P-BP-LS-00-00-CN	56.2	0.2
BIPM	4P-BP-LS-00-00-CN	56.08	0.2
BIPM *	4P-BP-LS-00-00-CN	56.09	0.2
CIEMAT	4P-BP-LS-00-00-CN	55.7	0.24
CIEMAT	4P-BP-PC-NA-GR-CT	55.8	0.5
CIEMAT *	Weighted mean	55.72	0.22
CNEA *	4P-BP-LS-00-00-TD	56.58	0.10
ENEA	4P-MX-LS-00-00-CN	56.7	0.28
ENEA	4P-MX-LS-00-00-TD	56.43	0.16
ENEA	Arithmetic mean	56.57	0.16
IFIN-HH *	4P-BP-LS-00-00-TD	56.43	0.23
IFIN-HH	4P-BP-PC-NA-GR-CT	56.04	0.55
JRC	4P-MX-LS-00-00-CN	56.6	0.3
JRC	4P-MX-LS-00-00-TD	56.6	0.3
JRC *	Arithmetic mean	56.6	0.3
LNE-LNHB *	4P-BP-LS-00-00-TD	56.46	0.14
LNE-LNHB	4P-BP-LS-00-00-TD	56.52	0.22
NIST *	4P-BP-LS-NA-GR-AT	56.55	0.15
NIST	4P-BP-LS-00-00-CN	56.49	0.19
NIST	4P-BP-LS-00-00-TD	56.4	0.06
NMIJ	4P-BP-LS-00-00-CN	59.4	0.5
NMISA *	4P-BP-LS-00-00-TD	57.03	0.22

Laboratory	Method acronym	Result / kBq g <sup>-1</sup>	Uncertainty / kBq g <sup>-1</sup>
NMISA	4P-BP-LS-00-00-CN	57.11	0.24
NMISA	4P-BP-LS-BA-GR-CT	56.79	0.23
NPL *	4P-BP-LS-00-00-CN	56.7	0.5
NPL	4P-BP-LS-00-00-TD	56.5	0.64
NRC *	4P-BP-PP-NA-GR-AT	59.00	0.77
POLATOM *	4P-BP-LS-00-00-TD	57.14	0.20
PTB	4P-MX-LS-00-00-CN	56.68	0.25
PTB	4P-MX-LS-00-00-TD	56.60	0.15
PTB *	Weighted mean	56.62	0.13
VNIIM	4P-BP-PC-NA-GR-CT	51.745	0.296
VNIIM *	4P-BP-PC-00-00-HE	52.995	0.276

Table A8. Results of statistical analysis of results marked for inclusion in the KCRV. The power moderated mean calculated using the technique described in rejected the values submitted by NMIJ, NRC and VNIIM. The weighted mean with Limitation of Relative Statistical Weights and outlier rejection by the Chauvenet's criterion, calculated using the LWEIGHT tool [18], rejected values submitted by BARC, CIEMAT, NMIJ, NRC and VNIIM.

Method	Result / kBq g <sup>-1</sup>	Uncertainty / kBq g <sup>-1</sup>
Arithmetic mean of all values and standard deviation of mean	56.58	0.35
Weighted mean with limitation of relative statistical weights (LRSW)	56.42	0.20 (external) 0.05 (internal)
Weighted mean with LRSW and outlier rejection by Chauvenet's criterion	56.59	0.07 (external) 0.05 (internal)
Power-moderated mean (PMM) and uncertainty, $\alpha = 1.8$	56.45	0.13
Median and estimated standard deviation based on median absolute deviation	56.58	0.44

Table A9. Degrees of equivalence between National Metrology Institute results and the KCRV, expressed in terms of the deviation  $D_i$  and the expanded uncertainty of the deviation  $U_i$ .

$i$	Laboratory $i$	$D_i$ / kBq g <sup>-1</sup>	$U_i$ / kBq g <sup>-1</sup>
1	BARC	-1.07	0.48
2	BIPM	-0.36	0.45
3	CIEMAT	-0.73	0.48
4	CNEA	0.13	0.32
5	ENEA	0.12	0.39
6	IFIN-HH	-0.02	0.50
7	JRC	0.15	0.62
8	LNE-LNHB	0.01	0.37
9	NIST	0.10	0.38
10	NMIJ	2.95	1.03
11	NMISA	0.58	0.48
12	NPL	0.25	0.99
13	NRC	2.55	1.56
14	POLATOM	0.69	0.45
15	PTB	0.17	0.36
16	VNIIM	-3.45	0.60

Figure A1. Comparison of the beta-decay spectrum of  $^{99}\text{Tc}$  with that of the  $^{60}\text{Co}$  transition to the 3<sup>rd</sup> excited state of  $^{60}\text{Ni}$ . The  $^{99}\text{Tc}$  spectrum in red is that calculated using the shapefactor from DDEP of  $C(W) = q^2 + \lambda_2 p^2$  where  $\lambda_2 = 0.529 \pm 0.018$  and  $q$  and  $p$  are respectively the neutrino and electron linear momentum. For comparison, the spectrum obtained by using the  $\xi$  approximation with a calculated, energy-dependent  $\lambda_2$  is also shown. All spectra were calculated using the BetaShape code, version 1.0.

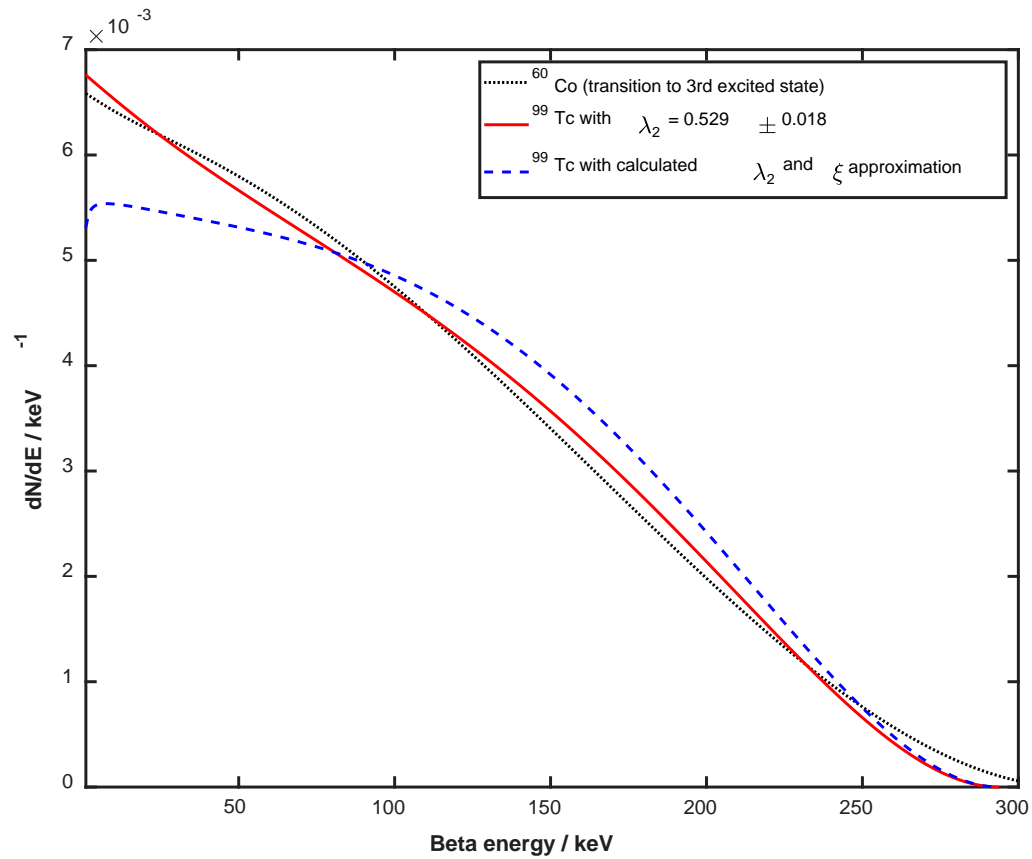


Figure A2. Results submitted by the participants for inclusion in the KCRV. The uncertainty bars represent one standard uncertainty. The solid line represents the PMM and the dashed lines represent the boundaries of the confidence interval at  $k = 2.5$ .

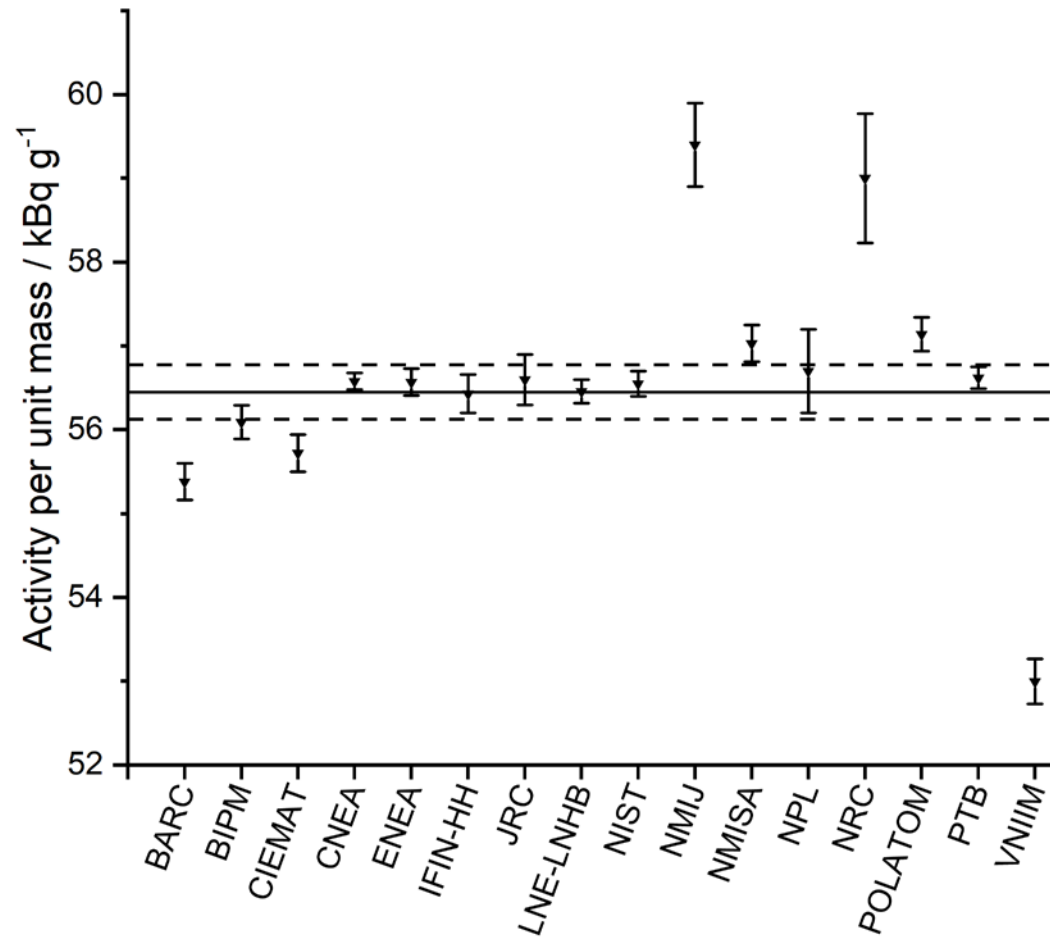


Figure A3. All submitted participant results arranged by method. Methods described by participants as “MX” and “BP” have been grouped together under “BP” for clarity. The uncertainty bars represent one standard uncertainty. The solid line represents the PMM and the dashed lines represent the boundaries of the confidence interval at  $k = 2.5$ .

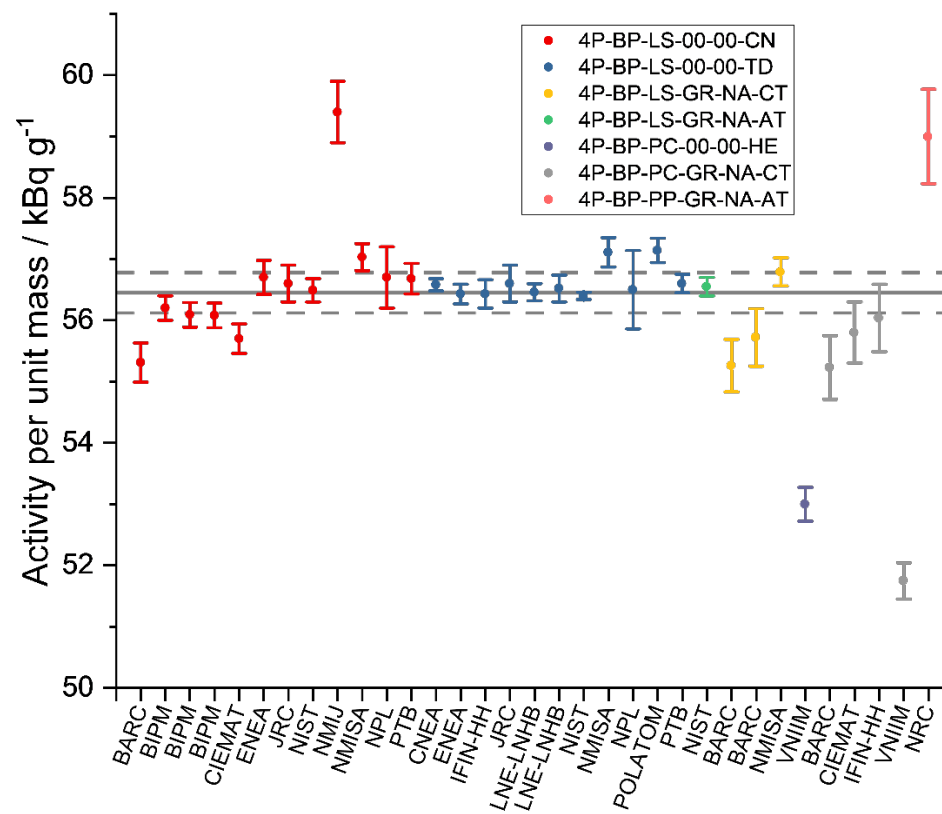


Figure A4. Degrees of equivalence  $D_i$  for all participating laboratories. The uncertainty bars represent  $U_i = 2 u(D_i)$ .

