International comparison of activity measurements of a solution of tritiated water <u>CCRI(II)-K2.H-3</u>

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Abstract:

An international comparison of activity measurements of a solution of tritiated water, CCRI(II)-K2.H-3, was organised in 2009 by the BIPM. Samples of this pure β emitter (provided by the LNE-LNHB) were measured using different techniques, but mostly with the TDCR method or methods relying on it. One result was identified as an outlier. Applying the PMM formalism a KCRV of 37.10(18) Bq/mg at 00:00 UTC on 31st May 2009 was obtained; degrees of equivalence for each participant have also been evaluated.

1. Introduction

An international comparison of activity measurements of a solution of tritiated water was organised under the auspices of the Comité Consultatif des Rayonnements Ionisants CCRI(II) in 2009, comparison CCRI(II)-K2.H-3. Samples were kindly made available to the participants by the LNE-LNHB. Tritium is a pure β emitter with the decay scheme shown in Figure 1. Nineteen laboratories participated in the exercise.

2. Relevant information about the comparison

The list of the participating institutions with information on the people who carried out the measurements is shown in Table 1. The tritiated water was prepared and dispatched to the participants by the LNE-LNHB. Table 2 gives some information about the main characteristics of the distributed ampoules. The laboratories who carried out appropriate tests reported no adsorption. No gamma-emitting impurities were detected in the samples (according to the results of measurements by ANSTO, NPL, PTB and RC). Table 3 provides the list of the methods used, together with the laboratories who applied these methods. Acronyms of the methods are also given, according to the CCRI(II) rules.

3. Results and evaluation of the KCRV and the Degrees of Equivalence

Nineteen laboratories took part in the exercise and reported a total of 22 independent results. Table 4 presents for each participant a detailed uncertainty budget as provided in their reporting forms. The reference time and date used was 00:00 UTC on 31^{st} May 2009, and a half life of 4496.862(9.131) d was used [1]. Most of the reported uncertainties lie in the range between 0.5 % and 1.0 %.

A summary of the results is given in Table 5. Table 6 shows the final results using only one result per participating laboratory, as required by the CIPM MRA rules. The figures displayed are taking into account the wishes of the laboratories, which are collated in footnotes.

Following a recommendation of the KCWG(II), the Power-Moderated weighted Mean formalism (PMM) [2] has to be used to evaluate the Key Comparison Reference Value (KCRV). It should be emphasized that the results obtained using ³H external standards were not included in the calculation of the KCRV. In addition, the MKEH value was identified as an outlier by the PMM procedure and was also excluded from the calculation of the KCRV. The KCRV calculated using the PMM method is thus 37.10(0.18) Bq/mg. An additional uncertainty of 0.539 Bq/mg was introduced in the evaluation of the KCRV applying the PMM with the empirical parameter $\alpha = 2-3/N$ that amounted to 1.77. The PMM is also used to evaluate degrees of equivalence for all participants in the comparison; these are shown in Table 7 and Figure 3.

4. Conclusion

The comparison of tritiated water was completed successfully. One outlier was identified but the rest of the laboratories obtained results in agreement with the key comparison reference value of 37.10(0.18) Bq/mg.

5. Acknowledgements

The authors are indebted to LNE-LNHB for supplying the samples of tritiated water, and to all the participants in the exercise. Thanks are also due to Steven Judge, BIPM, for editing the report.

References

[1] Monographie BIPM-5 (2006) *Table de Radionucléides*, BIPM, Pavillon de Breteuil, F-92310 Sèvres, pp. 1-4, Evaluation by V.P. Chechev.

[2] Pommé, S. & Keightley, J.D. 'Determination of a reference value and its uncertainty through a powermoderated mean' *Metrologia* 52 (2015) S200-S212

Decay scheme of tritium



Figure 1 – Decay scheme of ³H, taken from Monographie BIPM-5 (2006) *Table de Radionucléides*, BIPM, Pavillon de Breteuil, F-92310 Sèvres, pp. 1-4, Evaluation by V.P. Chechev.

Table 1 - List of participants

ANSTO	Australian Nuclear Science and Technology Organisation, Lucas Heights, Australia (Li Mo and Lindsey Bignell)
BARC	Bhabha Atomic Research Centre, Trombay, Mumbai, India (Leena Joseph, R. Anuradha and D.B. Kulkarni)
BIPM	Bureau International des Poids et Mesures, Sèvres, France (S. Courte and G. Ratel, source preparation: M. Nonis and G. Ratel, measurements)
ČMI-IIR	Czech Metrology Institute – Inspectorate for Ionizing Radiations, Prague, Czech Republic
CNEA	(P. Auerbach and J. Sochorová) Comisión Nacional de Energía Atómica, Laboratorio de Metrología de Radioisótopos, Buenos Aires, Argentina
IFIN	(Dario Rodrigues and Pablo Arenillas) National Institute of Research and Development for Physics and Nuclear Engineering - "Horia Hulubei" - IFIN-HH, Bucharest, Romania
IRMM	(Anamaria Cristina Wätjen, Andrei Antohe, Maria Sahagia) Institute for Reference Materials and Measurements, Geel, Belgium
KRISS	(T. Altzitzoglou) Korea Research Institute of Standards and Science, Yuseong, Daejon, Republic of Korea
LNE-LNHB	(K.B. Lee, Tae Soon Park, Jong Man Lee and Sang-Han Lee) Laboratoire National d'Essais – Laboratoire National Henri Becquerel, Gif-sur-Yvette, France
MKEH	(P. Cassette, C. Bobin and E. Verdeau) Magyar Kereskedelmi Engedélyezési Hivatal (Hungarian Trade Licensing Office) Budapest, Hungary
NIM	(K. Rózsa, L. Szücs and A. Zsinka) National Institute of Metrology, Beijing, Peoples Republic of China (Juncheng Liang and Jiacheng Liu, source preparation:
NMISA	Yang Yuandi, Juncheng Liang and Jiacheng Liu, measurements) National Metrology Institute of South Africa, Cape Town, South Africa (B.R.S. Simpson, M.I. van Staden, J. Lubbe and W.M. van Wyngaardt)
NPL	National Physical Laboratory, Teddington, United Kingdom (Hilary Phillips)
РТВ	(Inaly Finings) Physikalisch-Technische Bundesanstalt, Braunschweig, Germany (Ole Nähle and Karsten Kossert, TDCR; Karsten Kossert, CIEMAT/NIST;
RC	Oliver Ott, γ-spectrometry for impurity check) Institute of Atomic Energy, Radioisotope Centre POLATOM, Otwock-Świerk, Poland (R. Broda, T. Dziel and A. Muklanowicz)

Note: Since the comparison was carried out, the names and acronyms of some participating organizations have changed. These are: RC (now POLATOM) and IRMM (now JRC).

Laboratory	Ampoule number from	Adsorption	Impurity
	the mother solution		
	H3-LNHB-11-07		
ANSTO	N° 1	No adsorption found ¹⁾	No γ impurity ⁸⁾
BARC			
BIPM	N° 23		
ČMI-IIR			
CNEA	N° 5		
IFIN	N° 6		
IRMM	N° 8	2)	
KRISS			
LNE-LNHB	N° 22 & N° 23		
MKEH	N° 3	3)	
NIM		No adsorption found ⁴⁾	
NMISA	N° 15	No adsorption found ⁵⁾	
NPL	N° 19	-	No γ impurity ⁹⁾
РТВ	N° 20	6)	No γ impurity ¹⁰⁾
RC	N° 21	No significant adsorption found ⁷⁾	No γ impurity ¹¹

Table 2 – Characteristics of the distributed solutions

¹⁾ after measure of the original ampoule in the TDCR system after twice rinsing with Ultima GoldTMLLT.

²⁾ 110 Bq after first rinsing, 3 Bq after the second rinsing and 0 Bq after the third rinsing. Only included in the uncertainty budget.

 $^{^{3)}}$ a mass of 5.0044 g of solution was found in the ampoule.

⁴⁾ after three rinsings. A mass of 4.88896 g of solution was found in the ampoule.

 $^{^{5)}}$ after three rinsings. A mass of 5.02195 g of solution was found in the ampoule.

⁶⁾ a mass of 5.00363 g of solution was found in the ampoule.

⁷⁾ 164 Bq remaining in the original ampoule after two rinsings.

 $^{^{8)}}$ detection by γ spectrometry with HPGe detector.

 $^{^{9)}}$ detection by γ spectrometry with HPGe detector.

 $^{^{10)}}$ by γ spectrometry.

¹¹⁾ detection by γ spectrometry with HPGe detector, with a detection limit < 0.1 %.

Table 3 – List of the methods use
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Method acronym Number of times used	Description of the method	Laboratories using this method
CIEMAT/NIST method 4P-LS-BP-00-00-CN (1 x)	4π liquid-scintillation counting using the CIEMAT/NIST method with ³ H as an external standard,	NIM
CIEMAT/NIST method 4P-LS-MX-00-00-CN (1 x)	4π liquid-scintillation counting using the CIEMAT/NIST method with ⁵⁴ Mn as an external standard	РТВ
TDCR method 4P-LS-BP-00-00-TD (13 x)	4π liquid-scintillation counting using the Triple-to-Double-Coincidence- Ratio method	ANSTO, BIPM, ČMI-IIR, CNEA, IFIN, IRMM, KRISS, LNE-LNHB, NIM, NMISA, NPL, PTB, RC
TDCR method 4P-LS-BP-00-00-TD (1 x)	4π liquid-scintillation counting using the Triple-to-Double-Coincidence- Ratio method and a digital MAC3 ¹²⁾	LNE-LNHB
TDCR method 4P-LS-BP-GH-GR-TT (1 x)	4π liquid-scintillation counting using the Triple-to-Double-Coincidence- Ratio method coupled to the Compton efficiency tracing using ²⁴¹ Am	LNE-LNHB
4π(LS)β efficiency tracing 4P-LS-MX-00-00-ET (3 x)	$4\pi\beta$ liquid-scintillation secondary standardization against an ³ H standard	BARC, IRMM, NIM
4π(LS)β–γ efficiency tracing 4P-LS-MX-00-00-ET (1 x)	$4\pi\beta-\gamma$ efficiency tracing using the liquid-scintillation technique using ⁵⁴ Mn as a tracer	МКЕН
4π(PC) β 4P-PC-BP-00-00-IG	Differential proportional counting	LNE-LNHB, NPL

¹²⁾ See <u>http://www.nucleide.org/ICRM_LSCWG/icrmtdcr.htm#MAC3</u>, accessed on 13th April 2018

Uncertainty			Labor	atory		
components			Meth	nod		
		ANSTO		BARC		
	4:	$\pi(LS)\beta$ TDCR	<u>_</u>	$4\pi(LS)$	secondary sta	ndardization
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.29	А		0.5 ¹³⁾	А	
weighing	0.30	В		0.06	В	
background	0.10	В		14)	А	
half-life	0.10	В		0.01	В	
kB	0.59	А				
standard deviation between liquid scintillators	0.15	А				
tracer				0.6	В	
extrapolation of efficiency curve				1.35	А	
quench indicator parameter				0.25	А	
combined uncertainty (as quadratic sum of all uncertainty components)	0.75			1.58		

Table 4 – Components of the standard uncertainties in % of the activity concentration

¹³⁾ including background¹⁴⁾ included in the counting statistics

Uncertainty	Laboratory						
components							
	1.						
	42	t(LS)p IDCR	Con aitirritar		$4\pi(LS)\beta$ IDCR		
	Relative value / (%)	evaluation type	factor	Relative value / (%)	evaluation type	factor	
counting statistics	< 0.24	А		0.25			
weighing	< 0.015	В		0.01			
dead time				0.1			
background	15)	А		0.05			
adsorption				0.2			
counting time	negligible ¹⁶⁾						
impurities	not determined						
input parameters and statistical model				0.6			
half-life	0.1817)	В	0.0092				
extrapolation of efficiency curve with respect to <i>kB</i>	< 0.35	В					
dispersion of individual source measurements	1.41	В					
combined uncertainty (as quadratic sum of all uncertainty components)	1.48			0.68			

¹⁵⁾ included in the counting statistics
¹⁶⁾ quartz precision better than 10⁻³
¹⁷⁾ for the 60 d of measurements (measurement to reference date uncertainty).

Uncertainty	Laboratory					
components	CNEA			IFIN		
	4:	$\pi(LS)\beta$ TDCR		$4\pi(LS)\beta$ TDCR		
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.10	А		0.63	А	
weighing	0.08	В		0.1	В	
background double coincidence				0.01	В	0.01
background triple coincidence				0.08	В	0.04
kB				0.6 ¹⁸⁾		
input parameters and statistical model	0.90	В				
decay-scheme parameters	0.02	В				
measurement variability	0.10					
combined uncertainty (as quadratic sum of all uncertainty components)	0.91			0.88		

¹⁸⁾ difference between activities calculated using two successive kB values

Uncertainty	Laboratory					
components		IRMM	wieth		IRMM	
	47	τ(LS)β TDCR		$4\pi(\text{LS})\beta$ efficiency tracing		
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.3	А	1	0.1	А	1
weighing	0.2	А	1	0.2	А	1
dead time	0.2	А	1	0.1	А	1
background	0.1	А	1	0.05	А	1
counting time	0.01	А	1	0.05	А	1
adsorption	0.05	В	1	0.05	В	1
impurities	0.05	В	1	0.05	В	1
tracer				0.7	А	1
Interpolation from calibration curve				0.5	А	1
input parameters and statistical model	0.9	В	1			
half-life	0.01	В	1	0.01	В	1
photomultiplier asymmetry	0.1	А	1			
combined uncertainty (as quadratic sum of all uncertainty components)	1.0			0.9		

Uncertainty	Laboratory					
components	KRISS			LNE-LNHB		
	4	$\pi(LS)\beta$ TDCR		Differential proportional counter		
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.17 ¹⁹⁾	А	1	0.05	А	
weighing	0.05 ²⁰⁾	В	1			
background				0.3	А	
volume				0.5	В	
input parameters and statistical model	0.50 ²¹⁾	В	1			
quenching	0.32 ²²⁾	В	1			
half-life				0.001	В	
<i>kB</i> ionization quenching factor	1.78 ²³⁾	В	1			
photomultiplier asymmetry	0.14 ²⁴⁾	В	1			
extrapolation of efficiency curve threshold				0.1 ²⁵⁾	В	
correction due to the non-ionising particles				0.07	В	
combined uncertainty (as quadratic sum of all uncertainty components)	1.89			0.6		

¹⁹⁾ standard deviation of the counting of 9 samples

²⁰⁾ 10 mg mass sampling uncertainty due to the systematic effects of the micro-balance used

²¹⁾ dependence on the Poisson and Pólya statistics and on the end point energy of the ³H β -spectrum

²²⁾ dependence on the liquid scintillators used (Ultima Gold® and Hionic Fluor®)

²³⁾ standard uncertainty for the ³H efficiency difference for kB = 0.005 through kB = 0.018

²⁴⁾ difference between the TDCR models with unequal or equal photomultiplier gains

²⁵⁾ correction due to the low-level threshold

Uncertainty	Laboratory					
	LNE-LNHB $4\pi(LS)\beta$ TDCR CET (Compton Efficiency Tracing)			LNE-LNHB $4\pi(LS)\beta$ TDCR		
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.2	А	1	0.16	А	
weighing	0.1	В	1	0.01	В	
dead time	0.01	В	1	< 0.01	В	
background	0.03	А	1	0.03	А	
pile-up	0.02	В	1	0.02	В	
counting time	0.0001	В	1	0.0001	В	
input parameters and statistical model	0.3	В	1			
Quenching (kB factor)	0.13	В	1	0.64	В	
half-life	0.01	В	1	0.01	В	
influence of the scintillator	0.1	В	1			
counter effect				0.31 ²⁶⁾	В	
combined uncertainty (as quadratic sum of all uncertainty components)	0.41			0.73		

 $^{^{\}rm 26)}\ensuremath{\,\rm variability}\xspace$ between 2 different TDCR counters

Uncertainty	Laboratory Mathod					
		I NF-I NHR	Mich	МКЕН		
	$4\pi(LS)\beta$ TD	CR coupled w	ith a dioital	4π (I S)B efficiency tracing with ⁵⁴ Mn		
	MAC3 unit			as a tracer		
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.1	В	1	0.16	А	1
weighing	0.05	В	1	0.01	В	1
dead time	0.0127)	В	1			
background	0.05	А	1	0.003	В	1
input parameters and statistical model	0.1	В	1			
quenching (kB factor)	1.1	В	1			
half-life	0.01	В	1	0.19	В	1
accidental coincidences	0.1 ²⁸⁾	В	1			
counter effect	0.31 ²⁹⁾	В	1			
activity concentration of the MKEH standard				2.31 ³⁰⁾	В	1
reproducibility of the TriCarb 2910TR spectrometer ³¹⁾				0.73	В	
combined uncertainty (as quadratic sum of all uncertainty components)	1.1			2.4		

²⁷⁾ live-time

²⁸⁾ photomultipliers self-coincidences
²⁹⁾ variability observed between 2 different TDCR counters
³⁰⁾ uncertainty for the standardization of ⁵⁴Mn given on next page
³¹⁾ due to geometry, vial dimensions, etc.

Uncertainty components		Laboratory Method			
	MKEH 4π (NaI(Tl)) γ coincidence counting standardization of the ⁵⁴ Mn solution used as external standard				
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor		
counting statistics	1.5	А	1		
weighing	0.01	В	1		
dead time	0.006	В	1		
background	0.01	В	1		
counting time	0.006	В	1		
tracer	0.031	В	1		
decay-scheme parameters	0.005	В	1		
half-life	< 0.001	В	1		
extrapolation of efficiency curve	1.75	В	1		
coincidence resolving time	0.017	В	1		
delay mismatch	0.05	В	1		
combined uncertainty (as quadratic sum of all uncertainty components)	2.31				

Uncertainty components	Laboratory Method					
	NIM			NIM		
	$4\pi(LS)\beta$ CIEMAT/NIST			4π (LS) β TDCR		
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.10	А	1	0.10	А	1
weighing	0.05	В	1	0.05	В	1
dead time	0.01	В	1	< 0.01 ³²⁾	В	1
background				0.2	А	1
tracer	0.20	В	1			
input parameters and statistical model	0.86	В	1	0.65 ³³⁾	В	1
quenching	0.02	В	1			
decay-scheme parameters	0.10	В	1			
half-life	< 0.01	В	1	< 0.01	В	1
extrapolation of efficiency curve with respect to <i>kB</i>						
dispersion of individual source measurements						
combined uncertainty (as quadratic sum of all uncertainty components)	0.90			0.69		

³²⁾ live-time clock
³³⁾ including TDCR and *kB* values

Uncertainty components	Laboratory Method					
components	NMISA			NPL		
	$4\pi(LS)\beta$ TDCR		$4\pi(LS)\beta$ TDCR			
	Relative value / (%)	Uncertainty evaluation type	Relative sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.10	А	0.5112	0.08		
weighing	0.05	В	1.0000	0.03		
dead time	0.05	В	0.0050	0.02		
background	0.10	А	0.0352	0.02		
pile-up				0.03		
counting time	0.001	В	1.0000	0.001		
adsorption	0.05	В	0.5000	0.001		
impurities				0.1		
input parameters and statistical model	0.47	В	0.0423	0.94		
half-life	0.01	В	0.0492	0.001		
afterpulsing	0.5	В	0.0087			
combined uncertainty (as quadratic sum of all uncertainty components)	0.71			0.95		

Uncertainty components	Laboratory Method					
	NPL			PTB		
	Differential proportional counter		$4\pi(LS)\beta$ CIEMAT/NIST			
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.52			0.48	А	
weighing				0.02	В	
dead time	0.1			0.10	В	
background	0.5			0.05	А	
counting time	< 0.001			0.01	В	
adsorption				0.05	В	
impurities	< 0.001			0.20 ³⁴⁾	В	
dilution	1.56					
conversion to gas	0.6					
half-life	0.04			< 0.01	В	
extrapolation	0.4 ³⁵⁾					
input parameters and statistical model				0.70	В	
interpolation from calibration curve				36)		
decay-scheme parameters				37)		
combined uncertainty (as quadratic sum of all uncertainty components)	1.87					

 $^{^{34)}}$ contains also contribution due to the purity of ^{54}Mn $^{35\,)}$ losses below threshold

³⁶⁾ included in tracer uncertainty
³⁷⁾ combined with input parameters

Uncertainty components	Laboratory Method					
	PTB		RC			
	$4\pi(LS)\beta$ TDCR		$4\pi(LS)\beta$ TDCR			
	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor	Relative value / (%)	Uncertainty evaluation type	Sensitivity factor
counting statistics	0.20	А		0.05		
weighing	0.02	В		0.02		
dead time	0.03	В				
background	0.05	А		0.04		
counting time	0.01	В		0.001		
adsorption	0.05	В		0.07		
impurities	0.05 ³⁸⁾	В				
input parameters and statistical model	0.90 ³⁹⁾	В		0.4		
decay-scheme parameters	40)			0.001		
half-life	< 0.01	В		0.003		
measurement of TDCR and asymmetry correction	0.08	В				
combined uncertainty (as quadratic sum of all uncertainty components)	0.93			0.41		

³⁸⁾ no impurity detected ³⁹⁾ including uncertainty of the model for Q(E) and kB⁴⁰⁾ combined with input parameters

Table 5 – Results

Laboratory	Activity	Combined standard	Relative standard
Method	$(kBa g^{-1})$	$(kBa g^{-1})$	/ %
	(KDq g)		/ /0
ANSTO			
$4\pi(LS)\beta$ TDCR	37.46 ⁴¹⁾	0.28	0.75
BARC			
$4\pi(LS)\beta$ efficiency tracing	36.46	0.58	1.6
(with ${}^{3}H$ as a tracer)			
BIPM			
$4\pi(LS)\beta$ TDCR	35.66 ⁴²⁾	0.53	1.5
ČMI-IIR			
$4\pi(LS)\beta$ TDCR	36.40	0.25	0.68
CNFA			
$4\pi(LS)\beta$ TDCR	37.65	0.34	0.91
IEIN			
$4\pi(LS)\beta$ TDCR	36.49	0.32	0.88
IRMM $4\pi(LS)\beta$ TDCR	37 55	0 38	1.0
4π (LS) β efficiency tracing	37.7	0.3	0.9
(with 3 H as a tracer)			
KRISS			
$4\pi(LS)\beta$ TDCR	37.60	0.71	1.9
I NE-I NHB			
Differential proportional counting	36.65	0.22	0.60
4π (LS) β TDCR with CET	36.40	0.15	0.41
$4\pi(LS)\beta$ TDCR	36.75	0.27	0.73
$4\pi(LS)\beta$ TDCR with a digital MAC3	36.60	0.41	1.1

⁴¹⁾ arithmetic mean of the individual results
⁴²⁾ arithmetic mean of the individual results for five different sources

Laboratory	Activity	Combined standard	Relative standard
	concentration	uncertainty	uncertainty
Method	$(kBq g^{-1})$	$(kBq g^{-1})$	/ %
МКЕН			
4π (LS) β efficiency tracing (with ⁵⁴ Mn as a tracer)	31.15	0.76	2.5
NIM			
4π (LS) β CIEMAT/NIST	37.30	0.33	0.90
$4\pi(LS)\beta$ TDCR	37.03	0.26	0.70
NMISA			
$4\pi(LS)\beta$ TDCR	37.56	0.27	0.71
NPL			
Differential proportional counting	37.29 ⁴³⁾	0.70	1.9
$4\pi(LS)\beta$ TDCR	37.51	0.36	0.95
РТВ			
4π (LS) β CIEMAT/NIST	36 69	0.37	1.0
$4\pi(LS)\beta$ TDCR	36.69	0.34	0.93
PC .			
4π (LS)β TDCR	37.90	0.16	0.41

Table 5 – Results (continued)

⁴³⁾ mean of eight measurements

International comparison of activity measurements of a solution of ³H All results (22)



Figure 2 – Individual results of the international comparison of activity concentration of a solution of 3 H (22 individual results).

Laboratory	Activity	Combined standard	Relative standard	Result used
	concentration	uncertainty	uncertainty	for the KCRV
	$(kBq g^{-1})$	$(kBq g^{-1})$	/ %	$(kBq g^{-1})$
ANSTO	37.46	0.28	0.75	37.46(28)
BARC	36.46	0.58	1.58	-
BIPM	35.66	0.53	1.48	35.66(53)
ČMI-IIR	36.40	0.25	0.68	36.40(25)
CNEA	37.65	0.34	0.91	37.65(34)
IFIN	36.49	0.32	0.88	36.49(32)
IRMM	37.6344)	0.34 ⁴⁹⁾	0.9	37.55(38) [TDCR]
KRISS	37.60	0.71	1.89	37.60(71)
LNE-LNHB	36.62 ⁴⁵⁾	0.22^{50}	0.6	36.62(22)
MKEH	31.15	0.76	2.45	_
NIM	37.0346)	0.26 ⁵¹⁾	0.70	37.03(26)
NMISA	37.56	0.27	0.71	37.56(27)
NPL	37.5147)	0.36 ⁵²⁾	0.95	37.51(36)
РТВ	36.6948)	0.26 ⁵³⁾	0.69	36.69(26)
RC	37.90	0.16	0.41	37.90(16)

Table 6 – Results: one value per participating institute

Notes: The result from BARC was omitted from the calculation of the KCRV as the method used a ³H tracer. The result from MKEH was identified as an outlier by the PMM method.

⁴⁴⁾ weighted mean of the values obtained for the two methods

⁴⁵⁾ final result taken as the mean of the value obtained by the gas counting method and the mean of the three liquid-scintillation determinations, i.e. 36.65(0.22) kBqg⁻¹ and 36.58(0.18) kBq g⁻¹ respectively

⁴⁶⁾ TDCR value

⁴⁷⁾ TDCR value

⁴⁸⁾ weighted mean of the two results

⁴⁹⁾ uncertainty of the most precise determination (efficiency tracing)

⁵⁰⁾ uncertainty taken as the largest uncertainty of the two results quoted in note ⁴⁵⁾

⁵¹⁾ TDCR value; originally given for k = 2

⁵²⁾ TDCR value

⁵³⁾ uncertainty taken as the largest individual uncertainty 0.37 kBqg⁻¹ (obtained for the CIEMAT/NIST method) divided by $\sqrt{2}$

Table 7.Table of degrees of equivalence and introductory text for ³H

Key comparison CCRI(II)-K2.H-3

MEASURAND : Activity concentration of ³H

Key comparison reference value: the power moderated weighted mean of the results $x_R = 37.10 \text{ kBq.g}^{-1}$ with a standard uncertainty, $u_R = 0.18 \text{ kBq.g}^{-1}$

The value *x_i* is the activity concentration 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 kBq.g⁻¹, and

 $U_i = 2((1 - 2w_i)u_i^2 + u_R^2)^{1/2}$, where w_i is the weight of laboratory *i* contributing to the calculation of x_R .

Lab <i>i</i>	Di	Ui	
	/ kBq.g⁻¹		
ANSTO	0.4	0.6	
BARC	-0.6	1.2	
BIPM	-1.4	1.1	
CMI-IIR	-0.7	0.6	
CNEA	0.6	0.7	
IFIN	-0.6	0.7	
IRMM	0.5	0.8	
KRISS	0.5	1.4	
LNE-LNHB	-0.5	0.5	
МКЕН	-5.9	1.6	
NIM	-0.1	0.6	
NMISA	0.5	0.6	
NPL	0.4	0.8	
РТВ	-0.4	0.6	
RC	0.8	0.5	



Figure 3: Graph of degrees of equivalence with the KCRV for ³H (as it appears in Appendix B of the MRA).

The KCRV and the degrees of equivalence have been evaluated with the Power-Moderated weighted Mean (PMM) [2], k = 1. The result from BARC was omitted from the calculation of the KCRV as the method used a ³H tracer. The result from MKEH was identified as an outlier by the PMM method.