International comparison of activity measurements of a solution of ^{238}Pu

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An international comparison of activity measurements of a solution of ^{238}Pu was organised by the BIPM under the auspices of the Comité Consultatif pour les Rayonnements Ionisants (CCRI(II)) in 2001. The importance of ^{238}Pu in environmental studies was the main reason for choosing this radionuclide. However, this exercise is also being used to widen the scope of the comparisons organised by the BIPM and to extend the SIR (Système International de Référence pour la Mesure d'Activité d'Émetteurs γ) to α-emitting radionuclides.

A reporting form (see Appendix 1) agreed by the Key Comparison Working Group (KCWG) members was issued by the BIPM and sent in December 2000 to the participating laboratories. Originally it was proposed to start the comparison in January 2001 with a deadline of 01 April 2001. For reasons principally related to customs problems and consequent difficulties for some laboratories to receive the ampoules, the deadline was postponed by a few months until 17 September 2001. Submissions up to 09 October 2001 were therefore accepted.

The solution of ^{238}Pu was supplied, prepared and dispensed by the NPL. The solution was dispensed to NBS-type ampoules that were supplied by the BIPM. As no attempt was made to measure the ampoules in the SIR chambers, the ampoules were dispatched directly to the twelve participants by the NPL in the first semester of 2001. Each participant listed in Table 1 received a flame-sealed ampoule, containing about 3.0 g to 3.2 g of the solution. The ^{238}Pu nominal activity concentration was 400 kBq/g of $PuCl_4$ in an aqueous solution of 1 M HCl. The solution contained high purity ^{238}Pu; some traces of ^{241}Pu (0.0121 % of the total activity), ^{240}Pu (0.0004 %) and ^{239}Pu (0.0027 %) were quoted by the NPL.

In order to harmonise the measurements, the same reference date 2001-04-01, 0h UTC was used for all reported measurements, the preliminary activity concentration measurements before opening of the ampoule, the impurity checks and the final activity concentration. All participants agreed to use the half-life value $T_{1/2}$ = (3203.0×10^2 d; $u = 1.1 \times 10^2$ d). The decay scheme of ^{238}Pu from Lagoutine et al.[1] was simplified following G. Triscone, and is shown in Fig. 1.

The twelve participating laboratories and the names of the persons who carried out the measurements are listed in Table 1.

Table 2 provides the list of the methods used by the laboratories. The method acronyms and codes that are used in Tables 3 and 4 and in Fig. 2 are also given. An overview of the codes is given in Appendix 2. For clarity the methods have been grouped into four categories characterized by their detection devices: liquid-scintillation counters, proportional counters pressurized or working at atmospheric pressure, defined solid angle counters and CsI(Tl) sandwich spectrometer.

Table 3 summarises the relative standard uncertainty components (1σ) as stated by the laboratories for the different methods applied. The uncertainties range from 0.12 % to 0.61 %, with most of the estimates around 0.30 %. There is no obvious link between the method used and the uncertainty stated.

The final results are presented in Table 4 and shown in Figure 2. Most of the results are enclosed in a band of about 0.5 % (+ 0.65 % and – 0.45 % respectively) at either side of the mean value of 360.40(0.29) kBq/g.
The value assessed by the KRISS is slightly lower when compared with the other results but remains acceptable considering the quoted uncertainties. The first result sent by the OMH (355.6(2.2) kBq/g) was identified as being discrepant with the other values and the OMH asked for the possibility to carry out further measurements after the deadline. These additional measurements gave a value of 356.3(1.1) kBq/g in agreement with the first result obtained by this laboratory.

The degrees of equivalence are shown in Figure 3.

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[1] Nuclear and Atomic Decay Data, CD version : 1-98 – 19/12/98, BNM – CEA/DTA/DAMRI/LPRI

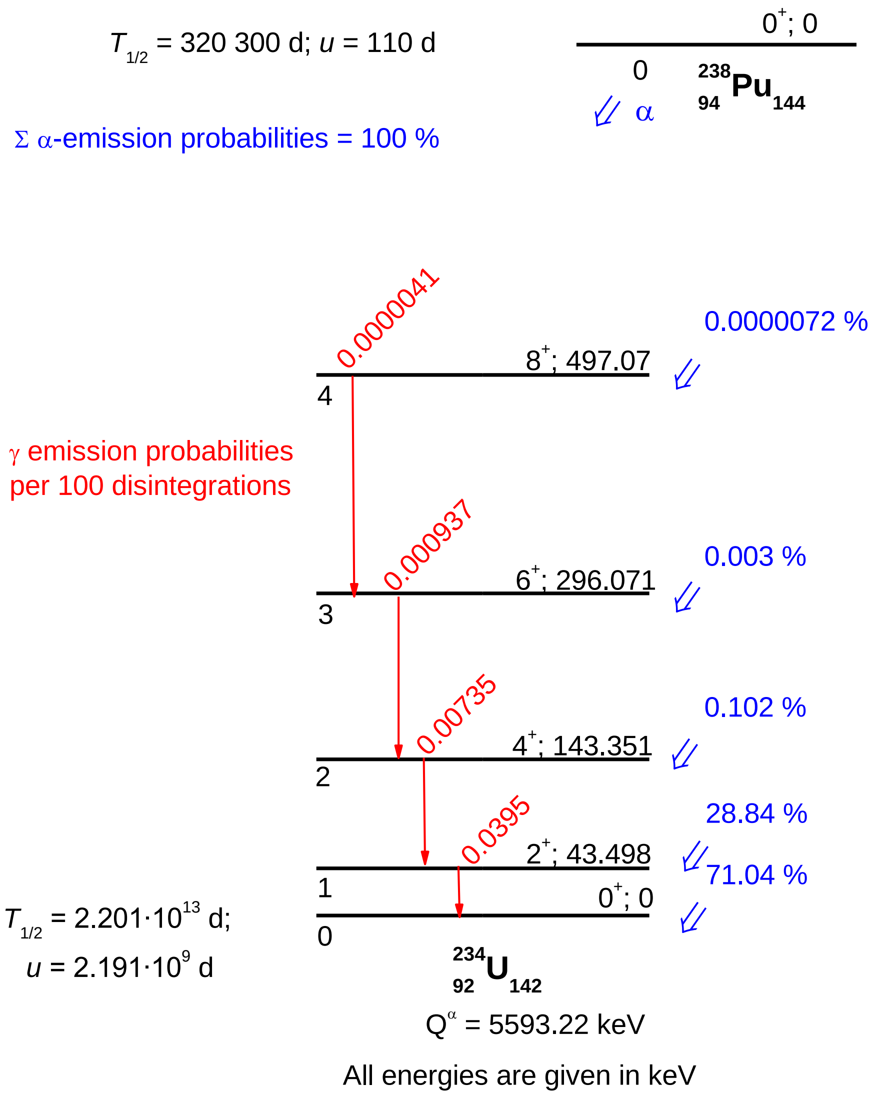

Fig. 1 - Simplified decay scheme of ${}^{238}_{94}\mathrm{Pu}_{144}$.

Table 1 - List of participants

BIPM	Bureau International des Poids et Mesures, Sèvres, France (C. Colas, C. Michotte and G. Ratel)
BNM-LNHB	Bureau National de Métrologie - Laboratoire National Henri Becquerel, Saclay, France (Ph. Cassette, F. Dayras, J. de Sanoit, C. Collin and B. Leprince)
CIEMAT	Centro de Investigaciones Energéticas Medioambientales y Technológicas, Madrid, Spain (M. Teresa Crespo and E. Garcia-Toraño)
CNEA	Comision Nacional de Energia Atomica, Laboratorio de Metrología de Radioisótopos, Buenos Aires, Argentina (J. Aguiar, P. Arenillas and M. Lobbe)
IRA	Institut Universitaire de Radiophysique Appliquée, Lausanne, Suisse (G. Triscone and J.-Ch. Gostely)
IRMM	Institute for Reference Materials and Measurements, Geel, Belgium (S. Pommé, T. Altzitzoglou, L. Johansson, G. Sibbens and B. Denecke, measurements; T. Altzitzoglou, G. Sibbens, S. Pommé, L. Johansson, source preparation; T. Altzitzoglou, adsorption tests; T. Altzitzoglou, G. Sibbens, impurity tests)
KRISS	Korea Research Institute of Standards and Science, Taejon, Korea (Jong Man Lee, Pil Jae Oh and Tae Soon Park)
NPL	National Physical Laboratory, Teddington, United Kingdom (A. Stroak, D. Woods, A. Woodman and A. Pearce)
OMH	Országos Mérésügyi Hivatal, Budapest, Hungary (L. Szücsi, Gy. Hegyi and K. Rózsa)
PTB	Physikalisch-Technische Bundesanstalt, Braunschweig, Germany (E. Günther, H. Janßen and R. Klein)
RC	Radioisotope Center “POLATOM”, Świerk, Poland (R. Broda and T. Terlikowska)
VNIIM	D.I. Mendeleyev Institute for Metrology, St. Petersburg, Russia (T.E. Sazonova, M.A. Rasko and V. Zanevsky)

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

Table 2 – List of the methods used

Method acronym	Description of the method	Laboratories using this method
4π(LS)α 4P-LS-AP-00-00-00	4πα counting with a liquid-scintillation spectrometer	BIPM, BNM-LNHB, CIEMAT, CNEA, IRA, IRMM, RC, PTB
4π(LS)α-γ tracer technique 4P-LS-AP-GR-CT-00	4πα-γ coincidence tracer technique method using the liquid-scintillation technique and ^{241}Am as a tracer	NPL
4π(LS)α TDCR method 4P-LS-TD-AP-00-00	4πα detection using the liquid-scintillation technique and the Triple-to-Double Coincidence Ratio method	RC
4π(PC)α 4P-PC-AP-00-00-00	4πα counting using a proportional counter at atmospheric pressure	BIPM, OMH, PTB
4π(PPC)α 4P-PP-AP-00-00-00	4πα counting using a pressurized proportional counter	IRMM
4π(PC)α-γ tracer technique 4P-PC-AP-GR-CT-00	4πα-γ coincidence tracer technique method using a proportional counter at atmospheric pressure for α-detection, a NaI(Tl) scintillator for γ-ray detection and ^{241}Am as a tracer	NPL
4π(PPC)α-γ tracer technique 4P-PP-AP-GR-CT-00	4πα-γ coincidence tracer technique method using a pressurized proportional counter for α-detection, a NaI(Tl) scintillator for γ-ray detection and ^{241}Am as a tracer	NPL
4π(PC)α-Lx coincidence 4P-PC-AP-XR-CO-00	4πα-Lx coincidence counting using a proportional counter at a pressure slightly above the atmospheric pressure for α-detection and a NaI(Tl) scintillator for x-ray detection	VNIIM
α(DSA) SA-AP-00-00-00-00	α counting under defined solid angle	BNM-LNHB, VNIIM
α(DLSA) SA-PS-AP-00-00-00	α counting under defined low solid angle	CNEA, IRMM, PTB
α-x(DSA) coincidence SA-AP-XR-CT-00-00	α-x coincidence counting under defined solid angle	KRISS
2π(GIC)α 2P-GC-AP-00-00-00	α counting using a grid ionization chamber with a 2π geometry	CIEMAT
4πCsI(Tl)α 4P-CS-AP-00-00-00	α counting with a 4π-CsI(Tl) sandwich-spectrometer	IRMM

Table 3 - Uncertainty components* of the activity concentration (in %)

Laboratory	BIPM		BNM-LNHB		CIEMAT	
Method	4π(LS)α	4π(PC)α[2]	4π(LS)α	α(DSA)	2π(GIC)α	4π(LS)α
Components due to:						
counting statistics	~ 0.07	0.12 [3]	0.04	0.03	0.11	0.04
weighing	0.28	#	0.1	0.1	0.1[5]	0.08 [3]
dead time		#	< 0.001	0.001		0.01
background	~ 0.02	negligible	< 0.01	0.001		
pile-up			< 0.001		0.01	0.01
timing			< 0.01			0.01
adsorption					0.01	0.09
impurities						0.01
tracer						
input parameters and statistical model						
quenching						[6]
interpolation from calibration curve						
decay-scheme parameters						
half life ($T_{1/2} = 320.3 \times 10^2$ d; $u = 1.1 \times 10^5$ d)	< 0.0001	0.001	0.0002	0.0001		
self absorption				< 0.0001	0.3	
extrapolation of efficiency curve		#				
spread of the measurements	0.15					
wall effect			0.05			
geometry factor				0.056		
tail extrapolation and backscattering					0.2	
differences between counters and series of measurements						0.2
uncertainty of method		0.3 [4]				
combined uncertainty (as quadratic sum of all uncertainty components)	0.33	0.32	0.12	0.12	0.39	0.24

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* The uncertainty components are to be considered as approximations of the corresponding standard deviations (see also *Metrologia*, 1981, **17**, 73 and *Guide to expression of uncertainty in measurement*, ISO, corrected and reprinted 1995).

[2] with efficiency extrapolation using the VYNS film addition technique

[3] includes items marked by #

[4] estimated from the bias observed when measuring ^{241}Am sources of known activities with the same method

[5] including dilution

[6] no visible effect on alpha counting

Table 3 - Uncertainty components of the activity concentration (in %) (continued)

Laboratory	CNEA		IRA	IRMM	
Method	4π(LS)α	α(DSLA)	4π(LS)α	4π (PPC)α	4π(LS)α
Components due to:					
counting statistics	0.25		0.05 [8)]	0.1	0.06
weighing	0.01		0.06 [9)]	0.05	0.08
dead time	0.01			0.02	0.1 [15)]
background	< 0.01		0.001 [10)]	0.01	0.005
pile-up					
timing			0.04 [11)]		0.05
adsorption	0.08			0.008	0.008 [16)]
impurities				0.007	0.008 [17)]
tracer					
input parameters and statistical model	0.48 [7)]				
quenching			0.1 [12)]		
interpolation from calibration curve					
decay-scheme parameters					0.25 [18)]
half life ($T_{1/2} = 320.3 \times 10^2$ d; $u = 1.1 \times 10^5$ d)	< 0.01		0.001 [13)]	0.001	0.001
self absorption	0.9			[14)]	
degradation of the cocktail quality with time			0.3		
foil absorption				0.2	
low energy tail extrapolation				0.1	
variation amongst sources				0.25	
combined uncertainty (as quadratic sum of all uncertainty components)	0.58		0.4	0.35	0.29

[7)] solid angle

[8)] standard deviation of the experimental data

[9)] $\frac{\Delta m}{\bar{m}}$; $\Delta m = 22$ µg and $\bar{m}$ is the mean distributed mass of the four sources used

[10)] propagation of the background uncertainty on the result for the source having the lowest activity

[11)] $\frac{\Delta t}{\bar{t}}$; $\Delta t = 0.01$ min and $\bar{t}$ is the mean measuring time of the four sources used

[12)] estimated from the content of the ten first channels divided by the total counting for the source of lowest activity

[13)] propagation of the uncertainty of the half life on the activity concentration

[14)] included in foil absorption

[15)] automatically corrected for by the instrument

[16)] 50 % of the adsorption

[17)] 50 % of the impurities

[18)] assumption of a 100 % efficiency

Table 3 - Uncertainty components of the activity concentration (in %) (continued)

Laboratory	IRMM		KRISS	NPL	
Method	α(DLSA)	4πCsI(Tl)α	α-x (DSA) coincidence	4π(PC)α-γ tracer technique	4π(LS)α-γ tracer technique
Components due to:					
counting statistics	0.05	0.1	0.45 21)	0.04	0.092
weighing	0.05	0.05	0.03	0.03	0.017
dead time	0.01	0.02	0.03 22)	0.09	α 0.116; γ 0.002
background	0.05	(0.05) 19)	0.1 23)	< 0.01	0.282
pile-up	0.0	0.0		< 0.01	
timing	0.0005	0.0005	0.001 24)	< 0.01	0.041
adsorption	0.008	0.008		0.1	
impurities	0.007	0.007		0.05	0.050
tracer				0.16	0.175
input parameters and statistical model		0.0			
quenching					
interpolation from calibration curve					
decay-scheme parameters	0.0	0.0		0.11	
half life ($T_{1/2}$ = 320.3 × 10^2 d; u = 1.1 × 10^5 d)	0.001	0.001	0.002 25)	< 0.01	0.003; tracer 0.003
self absorption		20)			
extrapolation of efficiency curve			0.23 26)	0.13	0.272
foil absorption		0.25			
low energy tail extrapolation	0.005	0.03			
variation amongst sources	0.25	0.25			
detector efficiency	0.01				
scattering at diaphragm & wall	0.01				
solid angle					
evaporation during source preparation	0.09-0.02 0.1	0.03			
choice of fit to efficiency function					0.016
uncertainty in fit					0.028 27)
combined uncertainty (as quadratic sum of all uncertainty components)	0.30	0.36	0.52	0.28	0.29

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19) included in counting statistics

20) included in foil absorption

21) standard deviation of the means obtained for 8 sources

22) calculated from measured uncertainty

23) estimated from background data set

24) estimated from time distribution

25) calculated from the given data

26) evaluated from residuals of the linear extrapolation

27) effect of using Cox-Isham formula instead of Campion formula for dead-time correction

Table 3 - Uncertainty components of the activity concentration (in %) (continued)

Laboratory	NPL	OMH	PTB		
Method	4π(PPC)α-γ tracer technique	4π(PC)α 28)	4π(LS)α	α(DLSA)	4π(PC)α
Components due to:					
counting statistics	0.17	0.085	0.05	0.15	0.1
weighing	0.016	0.005	0.08 30)	0.15 36)	0.15
dead time	0.046	0.005	0.07 31)	0.01 37)	0.01
background	0.049	< 0.001	< 0.03	< 0.01	0.01
pile-up			< 0.03	0.03 38)	
timing	0.086	0.005	0.05	< 0.01	< 0.01 40)
adsorption			0.07 32)		41)
impurities		0.02	0.05		39)
tracer	0.18		< 0.03 33)		0.2 42)
input parameters and statistical model			< 0.03 34)	0.25 39)	
quenching			< 0.03 32)		
interpolation from calibration curve			< 0.03 32)		
decay-scheme parameters					43)
half life ($T_{1/2}$ = 320.3 × 10^2 d; u = 1.1 × 10^5 d)	29)	< 0.001	< 0.001 35)	< 0.001	< 0.001 41)
self absorption		0.60			
extrapolation of efficiency curve	0.13				
scattering at diaphragm & wall					
tracer mixing ratio	0.049				
energy transfer from the α and the scintillator			0.07		
scattering of α in the spectrometer				0.1	
combined uncertainty (as quadratic sum of all uncertainty components)	0.28	0.61	0.18	0.35	0.27

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28) self absorption was estimated from a ^{241}Am standardization carried out using 4πα-γ coincidence counting under the same conditions as those used for the pure α measurements

29) negligible contribution

30) weights of about 40 mg

31) count rates approximately 1700 s^{-1}

32) sample spread 0.07 %

33) efficiency 100 %, no influence

34) no influence

35) for a time difference of 12 days between the date of reference and the date of measurements

36) for the set of sources

37) count rates lower than 20 s^{-1}

38) live-time correction of the multichannel analyser

39) geometry factors

40) time of measurement

41) not investigated

42) from efficiency measurements

43) negligible

Table 3 - Uncertainty components of the activity concentration (in %) (continued)

Laboratory	RC		VNIIM	
Method	4π(LS)α TDCR method	4π(LS)α	4π(PC)α-Lx coincidence	α(DSA)
Components due to:				
counting statistics	0.2	0.2	0.02	0.03
weighing	0.19	0.19	0.05	0.05
dead time				
background	0.03	0.02	0.15	0.05
pile-up				
timing	0.03			
adsorption	0.01	0.001		
impurities				
tracer				
input parameters and statistical model	0.1	0.02		
quenching				
interpolation from calibration curve				
decay-scheme parameters	0.14	0.14		
half life ($T_{1/2}$ = 320.3 × 10^2 d; u = 1.1 × 10^5 d)	0.001	0.001	0.001	0.001
dilution			0.03	0.03
resolving time			0.003	
G- factor				0.07
discrimination level				0.1
combined uncertainty (as quadratic sum of all uncertainty components)	0.33	0.31	0.16	0.15

Table 4 – Final results

Laboratory Method	Activity concentration ($\mathrm{kBq g^{-1}}$)	Combined uncertainty ($\mathrm{kBq g^{-1}}$)	Relative uncertainty / %
BIPM			
4π(LS)α	359.1*	1.2	0.33
4π(PC)α	361.1	1.2	0.32
BNM-LNHB			
4π(LS)α	361.77*	0.40	0.11
α(DSA)	362.05	0.43	0.12
CIEMAT			
2π(GIC)α	359.4	1.4	0.39
4π(LS)α	361.7*	0.9	0.25
CNEA			
4π(LS)α	358.80*	0.97	0.27
α(DLSA)	359.4	1.7	0.48
IRA			
4π(LS)α	360.9*	1.5	0.4
IRMM			
4π(PPC)α	360.2[1]	1.1	0.3
4π(LS)α	360.4	1.1	0.35
α(DLSA)	360.3	1.1	0.3
4πCsI(Tl)α	359.6	1.3	0.35
KRISS			
α-x (DSA) coincidence	358.1*	1.9	0.52
NPL			
4π(PC)α-γ tracer technique	362.7*	1.0	0.28
4π(LS)α-γ tracer technique	361.2	1.7	0.46
4π(PPC)α-γ tracer technique	362.0	1.1	0.28
OMH			
4π(PC)α	355.6*	2.2	0.61
PTB			
4π(LS)α	360.8[2]	0.7	0.18
α(DLSA)	360.7	1.3	0.35
4π(PC)α	360.8	1.0	0.27
RC			
4π(LS)α TDCR method	360.3	1.2	0.33
4π(LS)α	360.3*	1.1	0.31
VNIIM			
4π(PC)α-Lx coincidence	361.5	0.6	0.16
α(DSA)	361.3*	0.5	0.15

Notes

* Value used in estimating the KCRV and the degrees of equivalence

[1] Value to be used was given by IRMM as the mean of the four results: 360.1(10) kBq/g

[2] Value to be used was given by PTB as 360.8(10) kBq/g

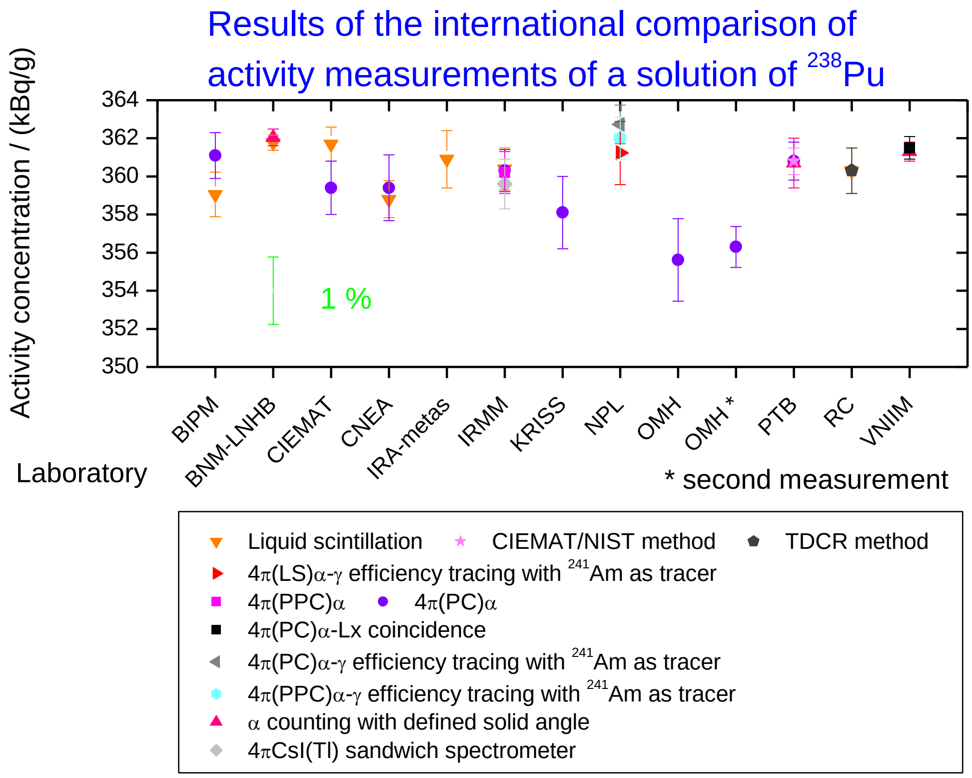

Figure 2 – Final results of the ^{238}Pu international comparison of activity concentration.

Notes

Further details of the methods used are given in Table 2. The result using the CIEMAT/NIST method by PTB has been reclassified as 4π(LS)α.

Lab *i*	**D_i**	**U_i**
	/ kBq.g^{-1}	
BIPM	**-1.5**	2.4
BNM-LNHB	**1.3**	1.2
CIEMAT	**1.2**	1.9
CNEA	**-1.7**	2.0
IRA	**0.4**	3.0
IRMM	**-0.3**	2.2
KRISS	**-2.4**	3.7
NPL	**2.2**	2.1
OMH	**-4.9**	4.3
PTB	**0.3**	1.6
RC	**-0.2**	2.2
VNIIM	**0.8**	1.3

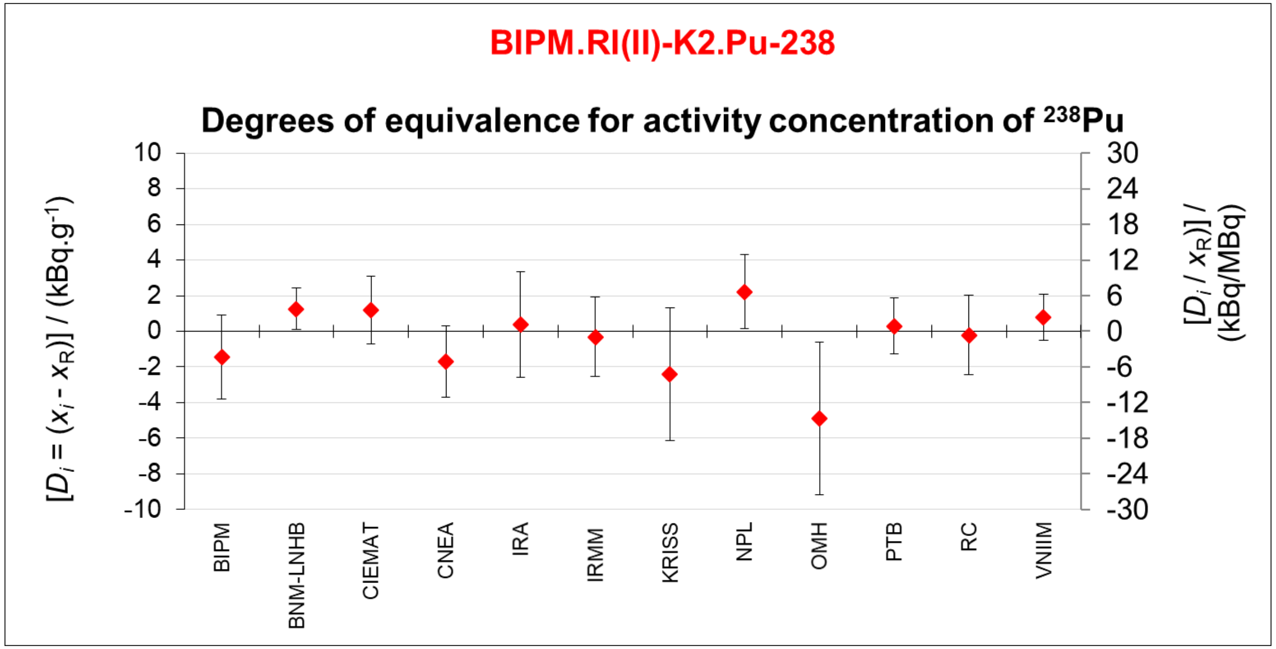

Figure 3 - Degrees of equivalence for activity concentration for ^{238}Pu

Notes:

1) The measurand is the activity concentration of ^{238}Pu
2) The Key Comparison Reference Value is the power moderated weighted mean of the results (x_R = 360.51 kBq.g^{-1} with a standard uncertainty u_R = 0.48 kBq.g^{-1})
3) The value x_i is the activity concentration for laboratory *i*.
4) 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 $U_i = 2((1 - 2w_i)u_i^2 + u_R^2)^{½}$, where w_i is the weight of laboratory *i* contributing to the calculation of x_R.
5) The right-hand axis shows approximate values only.
6) Since the comparison was carried out, the names and acronyms of some participating organizations have changed. These are: BNM-LNHB (now LNE-LNHB), IRMM (now JRC), OMH(now MKEH) and RC (now POLATOM).

Appendix 1 – Agreed reporting form for the ^{238}Pu international comparison

BUREAU INTERNATIONAL DES POIDS ET MESURES

Comparison of activity measurements of a solution of ^{238}Pu
(January 2001)

Participating laboratory :

$T_{½}$ = (320.3 × 10^2 d; *u* = 1.1 × 10^2 d)*

Ampoule number _ _ _ _ _ _

Mass of solution, according to distributing laboratory _ _ _ _ _ g

Name(s) of the person(s) who carried out the measurements :

Date :

Please send the filled-in form and any additional information to the BIPM
not later than ***April 1st, 2001.***

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* Ulrich Schötzig und Heinrich Schrader, Halbwertszeiten und Photonen-Emissionswahrscheinlichkeiten von häufig verwendeten Radionukliden, PTB-Bericht PTB-Ra-16/5, Braunschweig, September 1998.

A. Preliminary measurements

A.1. Method used for preliminary measurements

- calibrated ionization chamber YES ☐ NO ☐
- well crystal YES ☐ NO ☐
- other method YES ☐ NO ☐

A.2. Results obtained

Radioactivity concentration, in kBq g^{-1} (2001-04-01, 00 h UTC)

- before opening the original ampoule _ _ _ _ _

date of this measurement _ _ _ _ _

- after transfer to another ampoule _ _ _ _ _

date of this measurement _ _ _ _ _

Total mass of solution found in the ampoule_ _ _ _ _ g.

A.3. Adsorption tests

Please take into account the adsorption tests in the evaluation of the final results.

A.3.1. *Adsorption tests carried out with liquid-scintillation counting*

Please keep in mind the following :

- No rinsings are necessary,
- Use a water immiscible cocktail to measure the residual activity.

Activity remaining in the "empty" original ampoule _ _ _ _ _ Bq.

Date of this test _ _ _ _ _ _ _ _ _ _ _ _ .

Please explain the measuring procedure used :

A.3.2. *Adsorption tests carried out with proportional counting*

Please rinse the ampoule with an aggressive solution to remove most of the activity and prepare solid source(s) to measure this residual activity.

Activity remaining in the "empty" original ampoule _ _ _ _ _ Bq.

Date of this test _ _ _ _ _ _ _ _ _ _ _ .

A.4. Impurity checks*

Method of measurement _.

Nuclide _ _ _ _ _ _ _ _ _ _ _ _,

Impurity to ^{238}Pu activity ratio _ _ _ _ _ _ _ _ _ and its uncertainty _ _ _ _ _ _ _ _ _
at reference date (2001-04-01, 00 h UTC).

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* Pease give this information for each impurity found.

B. Source preparation

B.1. Methods used for source preparation :

Possible remarks about drying, precipitation, foils used (gold-coated or not, number, etc.), type of balance used…

B.2. Solutions, sources

B.2.1. *For beta counting and photon counting (if relevant)*

Diluent:

dilution number

	1	2 3	...
- dilution factor	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _
possible remarks	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _
- number of sources prepared	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _
- dispensed mass of solution (approx.)	_ _ _ _ _	_ _ _ _ _	_ _ _ _ _

B.2.2. *For liquid-scintillation counting*

Diluent _ _ _ _ _

Dilution factor _ _ _ _ _

Scintillator used to prepare the sources _ _ _ _ _

Volume of scintillator used _ _ _ _ _ cm^3

Chemicals used to stabilize the solution _ _ _ _ _

Substances used as quenching agent _ _ _ _ _

Type of vials used _ _ _ _ _

C. Procedures used for the activity measurements

Method of measurement used

(e.g. 2π(PC)α counting, 4π(PC)α counting, absolute counting with defined solid angle, α-x coincidence counting with defined solid angle, liquid-scintillation counting or other)

Please list the values for all the decay-scheme parameters (branching ratios, transition intensities, internal conversion coefficients, etc.) relevant to your measurements.

In case you used more than one method, please assemble the relevant information on separate sheets.

D. Detectors, counting equipment

D.1. Alpha counting (channel 1)

D.1.1. *Proportional counter* _ _ _ _ _

Solid angle _ _ _ _ _ sr

Wall material _ _ _ _ _   Height of each half _ _ _ _ _ mm

Anode

    Nature _ _ _ _ _

    Wire diameter _ _ _ _ _ mm   Wire length _ _ _ _ _ mm

    Distance from source _ _ _ _ _ mm

    Voltage applied _ _ _ _ _ kV

Gas

    Nature _ _ _ _ _   Pressure

    (above atmospheric pressure) _ _ _ _ _ MPa

Remarks

D.1.2. *Liquid-scintillation equipment*

D.1.2.1. *CIEMAT/NIST method*

D.1.2.1.1. *Characterization of the liquid-scintillation counter (LSC)*

Type of the counter _ _ _ _ _
Age _ _ _ _ _
Quench parameter _ _ _ _ _
Nuclide used for the determination of the quench parameter _ _ _ _ _
Efficiency obtained with an unquenched standard of ^{3}H _ _ _ _ _
Background (unquenched standard in toluene scintillator,
0 keV to 2000 keV or more) _ _ _ _ _
Options used (e.g. low-level counting) _ _ _ _ _

D.1.2.1.2. *Characterization of the tracer* (e. g. ^{3}H)

Standard used and its origin _ _ _ _ _
Uncertainty on the standard _ _ _ _ _
Date of preparation of the tracer samples _ _ _ _ _
Chemical composition of the tracer samples _ _ _ _ _

D.1.2.2. *TDCR method*

D.1.2.2.1. *Characteristics of the experimental equipment*

Type of phototubes _ _ _ _ _
Operating temperature _ _ _ _ _

Discrimination level _ _ _ _ _
Coincidence resolving time _ _ _ _ _

Type of dead time extending ☐ non-extending ☐
Minimum dead-time length _ _ _ _ _ µs
Efficiency variation method:
- defocusing _ _ _ _ _
- grey filters _ _ _ _ _
- chemical quenching _ _ _ _ _
- other ones (please describe) _ _ _ _ _

External standard (^{3}H or other) used
for the determination of the figure of merit _ _ _ _ _

D.1.2.3. *Other method(s)*

D.2. Photon counting (channel 2)

D.2.1. *Scintillator detector*

Crystal material _ _ _ _ _ _ _ _ _ Solid angle _ _ _ _ _ sr
Number of crystals _ _ _ _ _ _ _ _ _ Well type YES ☐ NO ☐
Crystal diameter _ _ _ _ _ mm Crystal height _ _ _ _ _ mm
Well diameter _ _ _ _ _ mm Well depth _ _ _ _ _ mm
Window material _ _ _ _ _ _ _ _ _ Window thickness _ _ _ _ _ mm
Distance between photon counter and source _ _ _ _ _ mm
Resolution at _ _ _ _ _ keV, FWHM[*] _ _ _ _ _ %, _ _ _ _ _ keV

Please add a typical pulse-height spectrum.

D.2.2. *Semiconductor detector*

Nature _ _ _ _ _ _ _ _ _ Solid angle _ _ _ _ _ sr
Type _ _ _ _ _ _ _ _ _ Coaxial ☐ Planar ☐
Number of detectors _ _ _ _ _ _ _ _ _ Well type YES ☐ NO ☐
Diameter _ _ _ _ _ mm Volume _ _ _ _ _ cm^3
Window material _ _ _ _ _ Window thickness _ _ _ _ _ mm
Distance between photon counter and source _ _ _ _ _ mm
Resolution at _ _ _ _ _ keV, FWHM*_ _ _ _ _ %, _ _ _ _ _ keV

Please add a typical pulse-height spectrum and an efficiency curve.

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[*] full width at half maximum

Radionuclides used for an efficiency determination (if relevant)

	E_γ (keV)	P_γ (%)
– –	– – – – –	– – – – –
– –	– – – – –	– – – – –
– –	– – – – –	– – – – –
– –	– – – – –	– – – – –
– –	– – – – –	– – – – –
– –	– – – – –	– – – – –

D.2.3. *Other detectors used (e.g. ionization chamber)*

D.3. Parameters of counting equipment

(Give a brief description and/or a block diagram of the experimental arrangement.)

D.3.1. *Channel 1 (alphas)*

a) Discrimination level _ _ _ _ _ keV
(or window)

b) Dead times and their uncertainties (standard deviation)

Dead time τ_1 = _ _ _ _ μs; *u* = _ _ _ _ μs

Type of dead time extending ☐ non-extending ☐
Method used for measurement _

Live time clock Yes ☐ No ☐
Pulser technique Yes ☐ No ☐
Loss free counting Yes ☐ No ☐

c) Pile-up rejector Yes ☐ No ☐

D.3.2. *Channel 2 (photons) (if relevant)*

a) Discrimination level _ _ _ _ _ keV
(or window)

b) Dead times and their uncertainties (standard deviation)

Dead time τ_2 = _ _ _ _ μs; *u* = _ _ _ _ μs

Type of dead time extending ☐ non-extending ☐
Method used for measurement _
_ _

D.3.3. *Coincidence unit (if relevant)*

Coincidence resolving time τ_R = _ _ _ _ µs; u = _ _ _ _ µs

Method used for measurement _

_ _

D.3.4. *Other modules used*

(for LSC see section D.1.2.)

E. Relevant data, corrections and uncertainties

E.1. Date of measurement

(Mean date on which your measurements were performed)

E.2. Measuring data

E.2.1. *Channel 1 (alphas)*

Dead time _ _ _ _ _ μs Number of sources measured _ _ _ _ _
Background rate _ _ _ _ _ s^{-1} Typical count rate _ _ _ _ _ s^{-1}
Typical time for one measurement _ _ _ _ _ s
Discrimination threshold or window _ _ _ _ _ keV

E.2.2. *Channel 2 (photons) (if relevant)*

Dead time _ _ _ _ _ μs Number of sources measured _ _ _ _ _
Background rate _ _ _ _ _ s^{-1} Typical count rate _ _ _ _ _ s^{-1}
Typical time for one measurement _ _ _ _ _ s
Discrimination threshold or window _ _ _ _ _ keV

E.2.3. *Extrapolation of efficiency function (coincidence method)*

Maximum achieved efficiency _ _ _ _ _ %
Method used for varying the efficiency

Number of degrees of freedom _ _ _ _ _
Please add a figure, if possible.

E.2.4. *Liquid-scintillation parameters*

Numerical codes used _ _ _ _ _
kB value _ _ _ _ _ unit _ _ _ _ _

Formula used to calculate the ionization quenching correction factor Q(E) _ _ _ _ _

Are M, N, ... captures taken into account? _ _ _ _ _
Are M, N, ... x-ray and Auger electrons taken into account? _ _ _ _ _

Model used to evaluate the interaction probability of the photons with the scintillator _ _ _ _ _
Values used for cross section of interaction _ _ _ _ _

E.2.5. *Calculated data for the liquid-scintillation method*

Total efficiency for ^{238}Pu _ _ _ _ _

E.2.6. *Corrections applied*

E.2.7. *Uncertainty components**, in % of the activity concentration, due to

		Remarks
counting statistics	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
weighing	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
dead time	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
background	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
pile-up	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
timing	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
adsorption	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
impurities	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
tracer	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
input parameters and statistical model	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
quenching	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
interpolation from calibration curve	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
decay-scheme parameters	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
half life (= 320.3×10^2 d; $u = 1.1 \times 10^2$ d)	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
self absorption	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
extrapolation of efficency curve (coincidence method)	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
other effects (if relevant) (explain)	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
combined uncertainty (as quadratic sum of all uncertainty components)	_ _ _ _ _	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _

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* The uncertainty components are to be considered as approximations of the corresponding standard deviations (see also *Metrologia*, 1981, **17**, 73) and *Guide to expression of uncertainty in measurement*, ISO, corrected and reprinted, 1995).

F. Combination of individual results

(obtained from the individual dilutions, source preparation, etc.)

How have the individual results been used for arriving at the final result (statistical weights, coverage factor, etc.) ?

G. Final result

The radioactivity concentration of the ^{238}Pu solution on the reference date*

(2001-04-01, 00 h UTC) is

_ _ _ _ _ _ _ _ _ kBq g^{-1} ,

and the combined uncertainty is

_ _ _ _ _ _ _ _ _ kBq g^{-1}, _ _ _ _ %.

Remarks

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* To adjust your result to the reference date, please use the half-life value given on page 1.

Appendix 2 - KCWG proposal for acronyms used to identify different measurement methods

Geometry	**acronym**	**Detector**	**acronym**
4π	4P	proportional counter	PC
defined solid angle	SA	pressurized proportional counter	PP
2π	2P	liquid scintillation counting	LS
		NaI(Tl)	NA
		Ge(HP)	GH
		Ge-Li	GL
		Si-Li	SL
		CsI	CS
		ionisation chamber	IC
		bolometer	BO
		calorimeter	CA
		PIPS detector	PS
		Grid ionisation chamber	GC

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
bremsstrahlung	BS	coincidence	CO
gamma ray	GR	anti-coincidence	AC
x - rays	XR	coincidence counting with efficiency tracing	CT
alpha - particle	AP	anti-coincidence counting with efficiency tracing	AT
mixture of various radiation e.g. x and gamma	MX	triple-to-double coincidence ratio counting	TD
		selective sampling	SA

Examples	
method	**acronym**
4π(PC)β–γ-coincidence counting	4P-PC-BP-NA-GR-CO
4π(PPC)β–γ-coincidence counting efficiency. tracing	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-00-00-00
calibrated IC	4P-IC-GR-00-00-00
internal gas counting	4P-PC-BP-00-00-IG