

Activity measurements of the radionuclide ^{109}Cd
for the PTB, Germany and the NIST, USA
in the ongoing comparison BIPM.RI(II)-K1.Cd-109

G. Ratel*, C. Michotte*, H. Janßen**, K. Kossert**, L. Lucas[#] and L. Karam[#]
*BIPM, **PTB(Germany), [#]NIST (USA)

Abstract

In 2004, the Physikalisch-Technische Bundesanstalt (PTB, Germany) and the National Institute of Standards and Technology (NIST, USA) each submitted one sample of known activity of ^{109}Cd to the International Reference System (SIR). The PTB result replaces their previous measurement of 1994 and the NIST result updates their 1986 CCRI(II) comparison result. The values of the activity submitted were about 15 MBq and 42 MBq. The new key comparison results have replaced the earlier values in the matrix of degrees of equivalence that now contains six results, identifier BIPM.RI(II)-K1.Cd-109, to which the remaining eleven results from the CCRI(II)-K2.Cd-109 held in 1986 are still linked.

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 (3.6 g) with the radionuclide in liquid (or gaseous) form. The 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 2004, the SIR has measured 872 ampoules to give 634 independent results for 62 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 realizations. These comparisons are described as BIPM ongoing comparisons and the results form the basis of the BIPM key comparison database (KCDB) that was set up under the Mutual Recognition Arrangement (MRA) [2]. The comparison described in this report is known as the BIPM.RI(II)-K1.Cd-109 key comparison.

In addition, an international comparison was held in 1986 for this radionuclide, CCRI(II)-K2.Cd-109 [3] and this comparison has been given the status of having provisional equivalence in the KCDB. Although eighteen laboratories took part in this

comparison, five of them have since submitted ampoules to the SIR. A further ten NMIs and one international laboratory are eligible to be linked to the BIPM key comparison through this CCRI(II) comparison [4].

2. Participants

The PTB and the NIST have each submitted one ampoule in 2004 for this comparison which complements the 20 previous submissions to the SIR from seven NMIs and two other laboratories for the comparison of ^{109}Cd activity measurements since 1978. The PTB result replaces their 1994 SIR measurement while the NIST submission is their first measurement of this radionuclide in the SIR, replacing their 1986 CCRI(II) comparison result in the KCDB. The laboratory details are given in Table 1 for the two current participants while the details of the previous participants are given in [4].

Table 1. Details of the recent participation in the comparison BIPM.RI(II)-K1.Cd-109

NMI	Full name	Country	Regional metrology organization	Date of measurement at the BIPM
PTB	Physikalisch-Technische Bundesanstalt	Germany	EUROMET	1994-11-24 2004-09-24
NIST	National Institute of Standards and Technology	United States	SIM	2004-11-22

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 any correlations are taken into account.

A brief description of the standardization method for the laboratory, the activities submitted and the relative standard uncertainties ($k = 1$) are given in Table 2. The uncertainty budgets are given in Appendix 1.

The half-life now used by the BIPM is 461.4 (12) d as published in BIPM Monographie 5 [5].

Table 2. Standardization methods of the recent participants for ^{109}Cd

NMI	Method used and acronym (see Appendix 2)	Half-life / d	Activity / kBq	Reference date YY-MM-DD	Relative standard uncertainty $\times 100$ by method of evaluation	
					A	B
PTB	Pressurized IC ^a 4P-IC-GR-00-00-00	–	7 405	94-10-01 0 h UTC	0.05	0.50
	Pressurized IC 4P-IC-GR-00-00-00 calibrated in March 2005 by a combination of 4π (PPC)ce counting, 4P-PP-CE-00-00-HE LS counting and 4P-LS-CE-00-00-00 HPGe spectrometry UA-GH-GR-00-00-00	[5]	14 962	04-01-01 0 h UTC	0.06	0.29
NIST	Pressurized IC 4P-IC-GR-00-00-00 calibrated in Oct. 1986 ^b by a combination of 4π LS counting and 4P-LS-CE-00-00-00 4π NaI(Tl) counting 4P-NA-GR-00-00-00	462 (1)	42 410	04-11-15 12 h UTC	0.04	0.51

^a calibrated by a combination of 4P-PP-CE-00-00-HE and UA-GH-GR-00-00-00 for the nuclide considered

^b calibration traceable to the CCRI(II)-K2 NIST comparison result in 1986.

Details regarding the solutions submitted are shown in Table 3, including any impurities, when present, as identified by the laboratories.

Table 3. Details of the solutions of ^{109}Cd submitted

NMI	Chemical composition	Solvent conc. / (mol dm^{-3})	Carrier: conc. /($\mu\text{g g}^{-1}$)	Density /(g cm^{-3})	Relative activity of any impurities [†]
PTB	CdCl_2 in HCl	0.1	CdCl_2 : 45	1.00	–
	CdCl in HCl	0.1	CdCl : 28	1.00	–
NIST	CdCl_2 in HCl	0.5	CdCl_2 : 500	1.007 (1)	–

[†] the ratio of the activity of the impurity to the activity of ^{109}Cd at the reference date

Recently the BIPM has developed a standard method for evaluating the activity of impurities using a calibrated Ge(Li) spectrometer [6]. The CCRI(II) agreed in 1999 [7] that this method should be followed according to the protocol described in [8] when an NMI makes such a request or when there appear to be discrepancies. The absence of impurity in the PTB (2004) solution was confirmed at the BIPM.

4. Results

All the submissions to the SIR since its inception in 1976 are maintained in a database known as the "mother-file". The previous activity measurements for ^{109}Cd arise from twenty ampoules and the SIR equivalent activity, A_{ei} , for each ampoule is given in [4] for each NMI, i . The SIR equivalent activity for the PTB and the NIST ampoules are given in Table 4a. The dates of measurement in the SIR are given in Table 1 and are used in the KCDB and all references in this report. The relative standard uncertainty arising from the measurements in the SIR is also shown. This uncertainty is additional to that declared by the NMI for the activity measurement shown in Table 2. Although activities submitted are compared with a given source of ^{226}Ra , all the SIR results are normalized to the radium source number 5 [1].

Table 4a. Results of SIR measurements for recent participants in the ^{109}Cd comparison

NMI	Mass of solution / g	Activity submitted / kBq	N° of Ra source used	SIR A_e / MBq	Relative uncertainty from SIR	Total uncertainty $u_{c,i}$ / MBq
PTB	3.698 9	7 405	1	8153	37×10^{-4}	51
	3.640 9 (9)	14 962	1	8170	29×10^{-4}	34
NIST	3.613 9 (2)	42 410	1	8168 [†]	16×10^{-4}	44

[†] supersedes the result of the 1986 CCRI(II) comparison in the KCDB.

Measurements repeated at the BIPM after a period of about 8 months give results in agreement within one standard SIR uncertainty for the NIST and about two SIR standard uncertainties for the PTB (2004).

The present result of the PTB agrees with their previous result to within one standard uncertainty of the SIR.

The 1986 result for the NIST that was linked through the international comparison is copied in Table 4b from [4]. This result and the present SIR equivalent activity value reported in Table 4a are both traceable to the same 1986 $4\pi\text{LS}$ and $4\pi\text{NaI(Tl)}$ measurements, giving a correlation coefficient of 0.36. Taking this correlation into account, the 1986 and 2004 NIST equivalent activities agree within 2 standard uncertainties.

Table 4b. NIST result of 1986 CCRI(II) comparison of ^{109}Cd linked to the SIR

NMI	Activity * concentration $(A/m)_i$ / (kBq g ⁻¹)	Relative standard uncertainty u_i × 10 ²	Linked SIR A_{ei} / MBq	$u(A_{ei})$ / MBq
NIST	5972 [†]	0.55	8107	47

* reference date 1986-03-01 at 0h UTC

[†] weighted mean of $^{109}\text{Cd}/^{109}\text{Pd}$ measurement and $4\pi\text{LS}$ counting combined with $4\pi\text{NaI(Tl)}$ counting.

4.1 The key comparison reference value

The key comparison reference value for ^{109}Cd given in [4] is 8136 (14) MBq. It is interesting to note that if both the recent submissions contributed to the KCRV, replacing earlier values where relevant, the revised value for the KCRV would be 8141 (13) MBq. The KCRV may indeed be modified when there are new submissions to the SIR but such modifications are only made by the CCRI(II), normally during one of its biennial meetings, the next of which is scheduled for 2007.

4.2 Degrees of equivalence

Every NMI that has submitted ampoules to the SIR is entitled to have one result included in Appendix B of the KCDB as long as the NMI is a signatory or designated institute listed in the MRA. Normally, the most recent result is the one included. Any NMI may withdraw its result only if all the participants agree. The results used are taken from [4] except for the two new results reported here.

The degree of equivalence of a given measurement standard is the degree to which this standard is consistent with the key comparison reference value [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 with the KCRV*

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

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

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

$$U_i = 2u_{D_i}, \quad (2)$$

taking correlations into account as appropriate [10].

4.2.2 *Comparison of any two NMIs with each other*

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

$$D_{ij} = D_i - D_j = A_{e_i} - A_{e_j} \quad (3)$$

and the expanded uncertainty of this difference U_{ij} where

$$u_{D_{ij}}^2 = u_i^2 + u_j^2 - 2u(A_{e_i}, A_{e_j}) \quad (4)$$

where any obvious correlations between the NMIs (such as a traceable calibration) are subtracted using the covariance $u(A_{ei}, A_{ej})$, as are normally those correlations coming from the SIR.

The uncertainties of the differences between the values assigned by individual NMIs and the key comparison reference value (KCRV) are not necessarily the same uncertainties that enter into the calculation of the uncertainties in the degrees of equivalence between a pair of participants. Consequently, the uncertainties in the table of degrees of equivalence cannot be generated from the column in the table that gives the uncertainty of each participant with respect to the KCRV. However, the effects of correlations have been treated in a simplified way as the degree of confidence in the uncertainties themselves does not warrant a more rigorous approach.

Table 5 shows the matrix of all the degrees of equivalence as they appear in Appendix B of the KCDB. The core of the matrix is based on six values from the SIR. The additional matrix cells show the eleven remaining results from the 1986 international (CCRI(II)) comparison linked to those of the SIR. 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 first column of results in Table 5, corresponding to the degrees of equivalence with respect to the KCRV (identified as x_R in the KCDB), is shown in Figure 1. The graph indicates in part the degree of equivalence between the NMIs but does not take into account the correlations between the different NMIs. However, the matrix of degrees of equivalence shown in yellow in Table 5 does take the known correlations into account.

Conclusion

The BIPM ongoing key comparison for ^{109}Cd , BIPM.RI(II)-K1.Cd-109 currently comprises six results. These have been analysed with respect to the KCRV determined for this radionuclide, and with respect to each other. The matrix of degrees of equivalence has been approved by the CCRI(II) and is published in the BIPM key comparison database.

The results of eleven other NMIs that took part in the CCRI(II)-K2.Cd-109 comparison in 1986 are linked to the BIPM ongoing key comparison through two ampoules of the comparison standardized at the BIPM and measured in the SIR. These linked results are included in the matrix of degrees of equivalence approved by the CCRI(II).

Other results may be added as and when NMIs contribute ^{109}Cd activity measurements to this comparison or take part in other linked comparisons.

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Table 5. Table of degrees of equivalence and introductory text for ^{109}Cd

Key comparison BIPM.RI(II)-K1.Cd-109

MEASURAND : Equivalent activity of ^{109}Cd

Key comparison reference value: the SIR reference value for this radionuclide is $x_R = 8136 \text{ MBq}$ with a standard uncertainty, $u_R = 14 \text{ MBq}$ (see Section 4.1 of the Final Report).
The value x_i is the equivalent activity for laboratory i .

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

$D_i = (x_i - x_R)$ and U_i , its expanded uncertainty ($k = 2$), both expressed in MBq, and

$U_i = 2((1 - 2/n)u_i^2 + (1/n^2)\sum u_i^2)^{1/2}$ when each laboratory has contributed to the calculation of x_R , with n the number of laboratories.

The degree of equivalence between two laboratories is given by a pair of terms:

$D_{ij} = D_i - D_j = (x_i - x_j)$ and U_{ij} , its expanded uncertainty ($k = 2$), both expressed in MBq.

The approximation $U_{ij} \sim 2(u_i^2 + u_j^2)^{1/2}$ is used in the following table.

Linking CCRI(II)-K2.Cd-109 (1986) to BIPM.RI(II)-K1.Cd-109

The value x_i is the equivalent activity for laboratory i participant in CCRI(II)-K2.Cd-109 having been normalized to the value of the BIPM as the linking laboratory (see Final report)

The degree of equivalence of laboratory i participant in CCRI(II)-K2.Cd-109 with respect to the key comparison reference value is given by a pair of terms: $D_i = (x_i - x_R)$ and U_i , its expanded uncertainty ($k = 2$), both expressed in MBq.

The approximation $U_i = 2(u_i^2 + u_R^2)^{1/2}$ is used in the following table.

The degree of equivalence between two laboratories i and j , one participant in BIPM.RI(II)-K1.Cd-109 and one in CCRI(II)-K2.Cd-109, or both participant in CCRI(II)-K2.Cd-109, is given by a pair of terms: $D_{ij} = D_i - D_j$ and U_{ij} , its expanded uncertainty ($k = 2$), both expressed in MBq, where the approximation $U_{ij} = 2(u_i^2 + u_j^2 - 2fu_i^2)^{1/2}$ is used with l being the linking laboratory when both laboratories are linked, and f is the correlation coefficient.

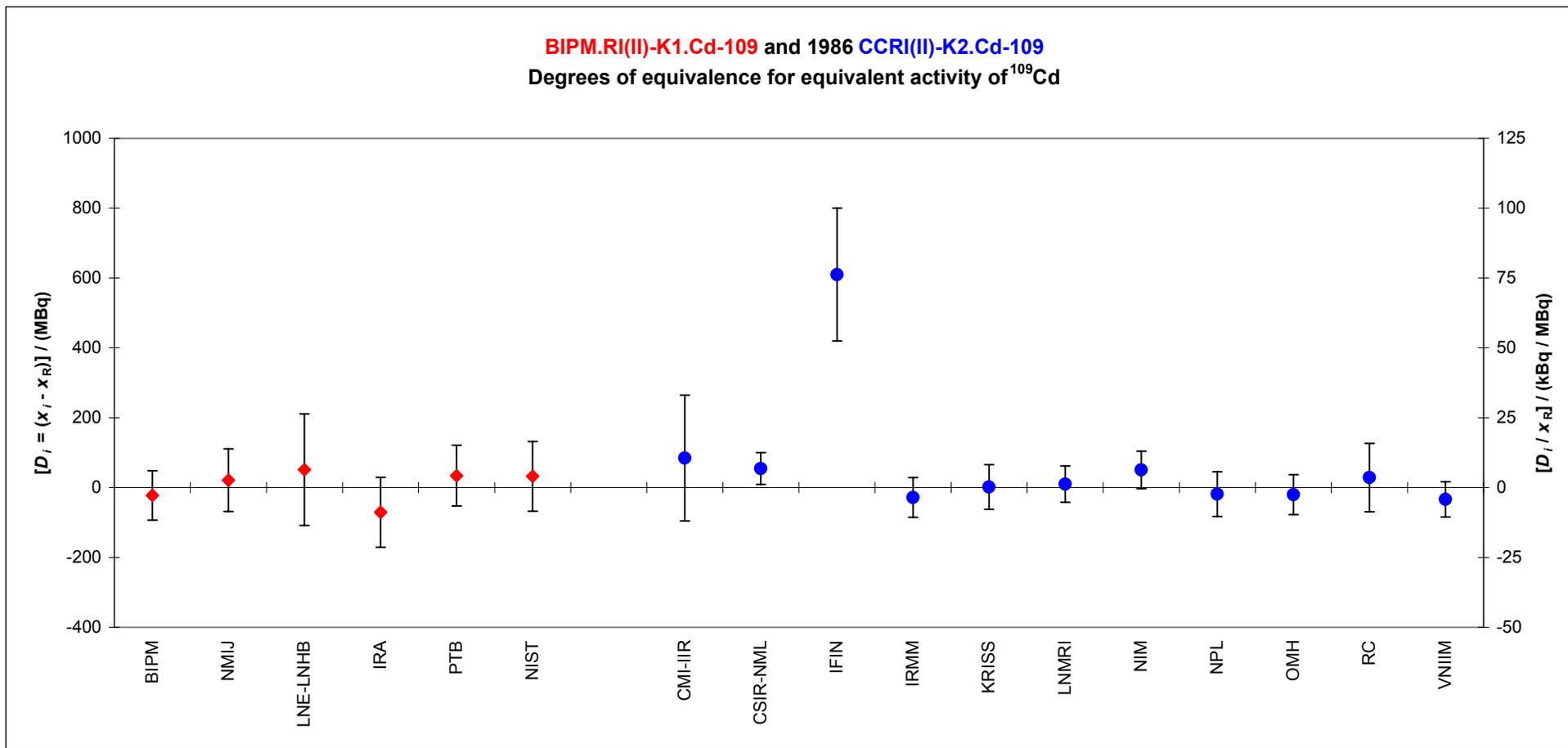
These statements make it possible to extend the BIPM.RI(II)-K1.Cd-109 matrices of equivalence to the other participants in CCRI(II)-K2.Cd-109.

Table 5 continued. Degrees of equivalence for ¹⁰⁹Cd

		Lab j →																		
Lab i ↓	D_i U_i		BIPM		NMIJ		LNE-LNHB		IRA		PTB		NIST		CMI-IIR		CSIR-NML		IFIN	
	/ MBq		D_{ij}	U_{ij}																
BIPM	-23	71																		
NMIJ	21	90	44	97																
LNE-LNHB	51	160	74	180	30	190			120	200										
IRA	-71	100	-48	110	-92	130	-120	200			-110	120								
PTB	34	87	57	86	13	110	-17	190	110	120										
NIST	32	100	55	100	11	120	-19	200	100	130	-2	110								
CMI-IIR	85	180	110	190	63	200	33	250	160	210	50	190	52	200						
CSIR-NML	55	45	77	51	33	90	3	180	130	110	20	77	22	95	-30	180				
IFIN	610	190	630	200	590	210	560	260	680	220	570	200	570	210	520	260	550	190		
IRMM	-28	57	-5	62	-49	96	-79	180	43	110	-62	84	-60	100	-110	190	-83	49	-640	190
KRISS	2	64	24	69	-20	100	-50	190	72	120	-33	89	-31	110	-83	190	-53	58	-610	200
LNMRI	10	52	33	57	-11	93	-41	180	81	110	-24	81	-22	98	-75	180	-45	43	-600	190
NIM	51	54	73	59	29	94	-1	180	120	110	16	82	18	99	-34	180	-4	45	-560	190
NPL	-19	64	4	69	-40	100	-70	190	52	120	-53	89	-51	110	-100	190	-73	58	-630	200
OMH	-20	57	3	62	-41	96	-71	180	51	110	-54	84	-52	100	-100	190	-75	49	-630	190
RC	29	98	52	100	8	120	-22	200	100	140	-5	120	-3	130	-56	200	-26	94	-580	210
VNIM	-34	50	-11	56	-55	92	-85	180	37	110	-68	80	-66	98	-120	180	-88	41	-640	190

		Lab j →																
Lab i ↓	D_i U_i		IRMM		KRISS		LNMRI		NIM		NPL		OMH		RC		VNIM	
	/ MBq		D_{ij}	U_{ij}														
BIPM	-23	71	5	62	-24	69	-33	57	-73	59	-4	69	-3	62	-52	100	11	56
NMIJ	21	90	49	96	20	100	11	93	-29	94	40	100	41	96	-8	120	55	92
LNE-LNHB	51	160	79	180	50	190	41	180	1	180	70	190	71	180	22	200	85	180
IRA	-71	100	-43	110	-72	120	-81	110	-120	110	-52	120	-51	110	-100	140	-37	110
PTB	34	87	62	84	33	89	24	81	-16	82	53	89	54	84	5	120	68	80
NIST	32	100	60	100	31	110	22	98	-18	99	51	110	52	100	3	130	66	98
CMI-IIR	85	180	110	190	83	190	75	180	34	180	100	190	100	190	56	200	120	180
CSIR-NML	55	45	83	49	53	58	45	43	4	45	73	58	75	49	26	94	88	41
IFIN	610	190	640	190	610	200	600	190	560	190	630	200	630	190	580	210	640	190
IRMM	-28	57			-30	67	-38	56	-79	57	-10	67	-8	60	-57	100	5	54
KRISS	2	64	30	67			-8	63	-49	64	20	73	22	67	-27	100	35	61
LNMRI	10	52	38	56	8	63			-41	52	29	63	30	56	-19	97	43	48
NIM	51	54	79	57	49	64	41	52			69	64	71	57	22	98	84	50
NPL	-19	64	10	67	-20	73	-29	63	-69	64			1	67	-48	100	15	61
OMH	-20	57	8	60	-22	67	-30	56	-71	57	-1	67			-49	100	14	54
RC	29	98	57	100	27	100	19	97	-22	98	48	100	49	100			62	96
VNIM	-34	50	-5	54	-35	61	-43	48	-84	50	-15	61	-14	54	-62	96		

Figure 1. Graph of degrees of equivalence with the KCRV for ^{109}Cd
 (as it appears in Appendix B of the MRA)



N.B. The right-hand axis shows approximate relative values only

Appendix 1. Uncertainty budgets for the activity of ^{109}Cd submitted to the SIR**Uncertainty budget for the PTB (2004)**

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
	A	B
Contributions due to		
Statistics (ionization current measurement)	5	–
Linearity of current measurement	–	5
Radium reference source current	2.5	–
Geometry correction	–	5
Calibration factor for ^{109}Cd	–	27
Adsorption	–	< 0.1
Impurities (detection limit for ^{57}Co)	–	< 0.1
Decay correction for ^{109}Cd ($\Delta t = 140$ d)	–	7.8
Weighing	–	2.4
Background	–	1
Quadratic summation	5.6	29.1
Relative combined standard uncertainty, u_c	30	

Uncertainty budget for the NIST (2004)

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
	A	B
Contributions due to		
PIC A net response per gram of solution, measured relative to RRS3	3	–
PIC A net response per Bq of ^{109}Cd in solution, measured relative to RRS3	3	–
PIC A charge collection	–	5
Live time	–	5
Source positioning	–	5
Activity used to calibrate PIC A net response per Bq of ^{109}Cd in solution	–	50
Decay correction for ^{109}Cd	1	–
Decay correction for ^{226}Ra	0.4	–
Photon-emitting impurities	–	1
Gravimetric measurements	–	5
Quadratic summation	4	51
Relative combined standard uncertainty, u_c	51	

Appendix 2. Acronyms used to identify different measurement methods

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

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

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