Activity measurements of the radionuclide ^{51}Cr for the NMIJ, Japan and the NPL, UK in the ongoing key comparison BIPM.RI(II)-K1.Cr-51 and the linking of the activity measurement of the VNIIM, Russian Federation in the APMP.RI(II)-K2.Cr-51 regional key comparison.

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Abstract

In 2004, the NMIJ, Japan and the NPL, UK submitted two new samples of known activity of ^{51}Cr to the International Reference System (SIR) for activity comparison at the Bureau International des Poids et Mesures, with comparison identifier BIPM.RI(II)-K1.Cr-51. The activities were about 3.8 MBq and 7.9 MBq. The measurements update earlier submissions. The degrees of equivalence have been calculated between each of the equivalent activity results for the nine national metrology institutes measured in the SIR and the key comparison reference value (KCRV). At the same time as their submission to the SIR, the NMIJ sent an ampoule to the VNIIM to participate in the APMP.RI(II)-K2.Cr-51 comparison. The results of the VNIIM as described in this report are consequently linked to the BIPM comparison through the NMIJ. All the results are given in the form of a matrix with a graphical presentation.

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 up 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) of the CIPM Mutual Recognition Arrangement (MRA) [2]. The comparison described in this report is known as the BIPM.RI(II)-K1.Cr-51 key comparison and updates the previous results published in [3].

In addition, the NMIJ piloted the APMP.RI(II)-K2.Cr-51 comparison with the VNIIM. The details are included in this report so that the results of the VNIIM can be linked to those of the other NMIs.

2. Participants

The NMIJ and NPL, six other NMIs and three other laboratories have submitted a total of twenty-eight ampoules for the comparison of ^{51}Cr activity measurements since 1977 [3]. The details of the present and past submissions of the current participants are given in Table 1a while those of the additional participant in the APMP.RI(II)-K2.Cr-51 comparison are given in Table 1b.

Table 1a. Details of the participants in the BIPM.RI(II)-K1.Cr-51

Original acronym	NMI	Full name	Country	Regional metrology organization	Date of measurement at the BIPM
–	NPL	National Physical Laboratory	United Kingdom	EUROMET	1980-12-01 2004-05-28
ETL	NMIJ	National Metrology Institute of Japan	Japan	APMP	1993-11-24 2004-03-15

Table 1b. Details of the participants in the 2004 APMP.RI(II)-K2.Cr-51

NMI	Full name	Country	Regional metrology organization
NMIJ	National Metrology Institute of Japan	Japan	APMP
VNIIM	D.I. Medeleyev Institute for Metrology	Russian Federation	COOMET

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 methods for each laboratory, the activities submitted and the relative standard uncertainties ($k = 1$) are given in Table 2, while those of the VNIIM for the APMP comparison are given in Table 4b. The uncertainty budgets are given in Appendix 1 attached to this report.

Table 2. Standardization methods of the participants for ^{51}Cr

NMI	Method used and acronym (see Appendix 2)	Half-life / d	Activity A_i / kBq	Reference date YY-MM-DD	Relative standard uncertainty × 100 by method of evaluation	
					A	B
NPL	4πβ-γ coincidence 4P-PC-BP-NA-GR-CO	–	15 533	80-12-05 0 h UTC	0.24	0.33
		27.7009 (20)	3 803	04-05-25 12 h UTC	0.10	0.66
NMIJ	4π(e,x)-γ coincidence 4P-PC-MX-NA-GR-CO	–	5 938	93-11-01 0 h UTC	0.04	0.30
		27.702 (3) [7]	7 896	04-03-01 0 h UTC	0.18	0.21

The half-life used by the BIPM is 27.70 (4) days [4], which is in agreement with the value recommended by the IAEA, 27.706 (7) d [5] and the more recent value 27.703 (3) d in [6]. As the SIR measurements are generally performed within one month following the reference date, the use of the more recent value would not change the results significantly. The half-life used in the APMP comparison was 27.702 (3) d [7] which is in agreement with the value used in the SIR within the uncertainties.

Details regarding the solution submitted are shown in Table 3, including any impurities, when present, as identified by the laboratories. When given, the standard uncertainties on the evaluations are shown. The BIPM has developed a standard method for evaluating the activity of impurities using a calibrated Ge(Li) spectrometer [8]. The CCRI(II) agreed in 1999 [9] that this method should be followed according to the protocol described in [10] when an NMI makes such a request or when there appear to be discrepancies. No impurity was found in the NMIJ solution and this solution was also used in the APMP comparison with the VNIIM. The VNIIM used a hyperpure gamma spectrometer to measure that there was no impurity, at a level of 3×10^{-4}.

Table 3. Details of the solution of ^{51}Cr submitted

NMI	Chemical composition	Solvent conc. / (mol dm^{-3})	Carrier: conc. /(µg g^{-1})	Density /(g cm^{-3})	Relative activity of impurity[†]
NPL	$CrCl_3$ in HCl	0.1	Cr : 30	1.001	–
	Na_2CrO_4 in H_2O	–	Na_2CrO_4 : 10	1	–
NMIJ	$CrCl_2$ in HCl	0.1	$CrCl_2$: 50	1.00	–
			$CrCl_2$: 100*	1.002	–

[†] the ratio of the activity of the impurity to the activity of ^{51}Cr at the reference date

* same solution used in the APMP comparison.

4. Results

All the submissions to the SIR since its inception in 1976 are maintained in a database known as the "mother-file". The activity measurements for ^{51}Cr arise from twenty-eight ampoules. The SIR equivalent activities, A_{ei}, for the previous and new NMIJ and NPL ampoules are given in Table 4a. The dates of measurement in the SIR are given in Table 1a. The relative standard uncertainties arising from the measurements in the SIR are 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].

Measurements repeated at the BIPM after a period of one month produced a comparison result in agreement within two SIR combined standard uncertainties for the NMIJ (2004).

The new results for the NPL and the NMIJ agree with their previous SIR result within one and two standard uncertainties, respectively.

Table 4a. Recent results of SIR measurements of ^{51}Cr

NMI	Mass of solution m_i / g	Activity submitted A_i / kBq	N° of Ra source used	SIR A_e / MBq	Relative uncertainty from SIR	Combined uncertainty $u_{c,i}$ / MBq
NPL	3.390 1	15 533	2	488.3	9 × 10^{-4}	2.0
	3.537 36	3 803	1	487.0	19 × 10^{-4}	3.4
NMIJ	3.654 09	5 938	1	484.7	15 × 10^{-4}	1.6
	3.602 52	7 896	1	487.1*	18 × 10^{-4}	1.6

* result used to link the APMP comparison result for the VNIIM

The results $(A/m)_i$ for the APMP comparison are given in Table 4b. The VNIIM result is linked to the SIR through the measurement in the SIR of an ampoule of the same solution standardized by the NMIJ. The link is made using a normalization ratio deduced from the line indicated in Table 4a:

$$A_{\mathrm{e}i} = (A/m)_i \times (A_{\mathrm{e,NMIJ}} / (A/m)_{\mathrm{NMIJ}}) = (A/m)_i \times 0.2222 \qquad (1)$$

The details of the links are given in Table 4b. The uncertainties for the regional comparison linked to the SIR are comprised of the original uncertainties together with the uncertainty in the link, 18×10^{-4}, given by the uncertainty of the SIR measurement of the NMIJ ampoule.

Table 4b. Results of the 2004 APMP regional comparison of ^{51}Cr activity and links to the SIR

NMI	Measurement method and acronym (see Appendix 2)	Activity * concentration measured $(A/m)_i$ / (kBq g^{-1})	Evaluation by category of relative standard uncertainty × 100		Equivalent SIR activity $A_{\mathrm{e}i}$ / MBq	Combined standard uncertainty $u_{\mathrm{c}\,i}$ / MBq
			A	B		
NMIJ**	4π(e,x)-γ coincidence 4P-PC-MX-NA-GR-CO	2 191.8	0.18	0.21	487.1$	1.6
VNIIM	4π(PC)AX-γ coincidence 4P-PC-MX-NA-GR-CO	2 199.9	0.06	0.22	488.8	1.4
	KX -γ coincidence UA-NA-XR-NA-GR-CO	2 194.4	0.07	0.09	487.6	1.0

*referenced to 2004-03-01 0 h UT

** linking laboratory

$ SIR result from Table 4a.

The VNIIM has requested that the arithmetic mean of their two measurement results of the same NMIJ solution be used as their final result with a combined uncertainty of 0.16 % [11]. Hence the final result linked to the SIR for the VNIIM is 488.2 (1.2) MBq.

4.1 The key comparison reference value

The KCRV for ^{51}Cr has been identified in [3] as 487.4 (5) MBq using the results from the ANSTO (1978), AECL, NPL, IRMM, CMI-IIR (1982), ASMW, OMH (1989), NMIJ (1993), BNM-LNHB (1994), PTB (1998) and the NIST (1981). Although the KCRV may be modified by the CCRI(II) whenever an NMI participates, such modifications are normally made during one of the CCRI(II)'s biennial meetings. It is interesting to note that if the two new primary determinations replaced their earlier ones in the evaluation of the KCRV, this would change its value to 487.5 (5) MBq.

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 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

$$D_i = A_{ei} - \mathrm{KCRV} \qquad (1)$$

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

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

taking correlations into account as appropriate [12].

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

$$D_{ij} = D_i - D_j = A_{ei} - A_{ej} \qquad (3)$$

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

$$u_{D_{ij}}{}^2 = u_i{}^2 + u_j{}^2 - 2u(A_{e,i}, A_{e,j}) \qquad (4)$$

and any obvious correlations between the NMIs (such as a traceable calibration) are subtracted 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 will appear in Appendix B of the KCDB. It should be noted that for consistency within the KCDB, a simplified level of nomenclature is used with A_{ei} replaced by x_i. The introductory text is that agreed for the BIPM and linked APMP comparisons. 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 where the black squares indicate results obtained prior to 1983. The graphical representation 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 ^{51}Cr, BIPM.RI(II)-K1.Cr-51 currently comprises nine results. These have been analysed with respect to the KCRV determined for this radionuclide, and with respect to each other. The new results for the NMIJ and the NPL agree with the KCRV within a standard uncertainty.

The result of the VNIIM that took part in the APMP.RI(II)-K2.Cr-51 comparison in 2004 has been linked to the BIPM ongoing comparison through an ampoule of the comparison solution standardized by the NMIJ and measured in the SIR.

The matrix of degrees of equivalence has been approved by the CCRI(II) and is published in the BIPM key comparison database. Other results may be added as and when other NMIs contribute ^{51}Cr activity measurements to this or a linked comparison.

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Table 5. Table of degrees of equivalence and introductory text for ^{51}Cr

Key comparison BIPM.RI(II)-K1.Cr-51

MEASURAND : **Equivalent activity of ^{51}Cr**

Key comparison reference value: the SIR reference value x_R for this radionuclide is 487.4 MBq,
with a standard uncertainty of 0.5 MBq (see Section 4.1 of the Report),
the value x_i is taken as 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
$U_i = 2((1 - 2/n)u_i^2 + (1/n^2)\Sigma u_i^2)^{1/2}$ 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 numbers:
$D_{ij} = D_i - D_j = (x_i - x_j)$ and U_{ij}, its expanded uncertainty ($k = 2$), both expressed in MBq.
The approximation $U_{ij}^2 \sim 2^2(u_i^2 + u_j^2)$ is used in the following table.

Linking APMP.RI(II)-K2.Cr-51 (2004) to BIPM.RI(II)-K1.Cr-51

The value x_i is the equivalent activity for laboratory *i* participant in APMP.RI(II)-K2.Cr-51
having been normalized to the value of the NMIJ as the linking laboratory (see Final report).

The degree of equivalence of laboratory *i* participant in APMP.RI(II)-K2.Cr-51 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*, 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
$U_{ij} = 2(u_i^2 + u_j^2 - 2fu_l^2)^{1/2}$ is used where *l* is the NMIJ and *f* is the correlation coefficient.

These statements make it possible to extend the BIPM.RI(II)-K1.Cr-51 matrices of equivalence
to the participant in the APMP.RI(II)-K2.Cr-51 comparison

Table 5 continued

Lab *j* ⇨

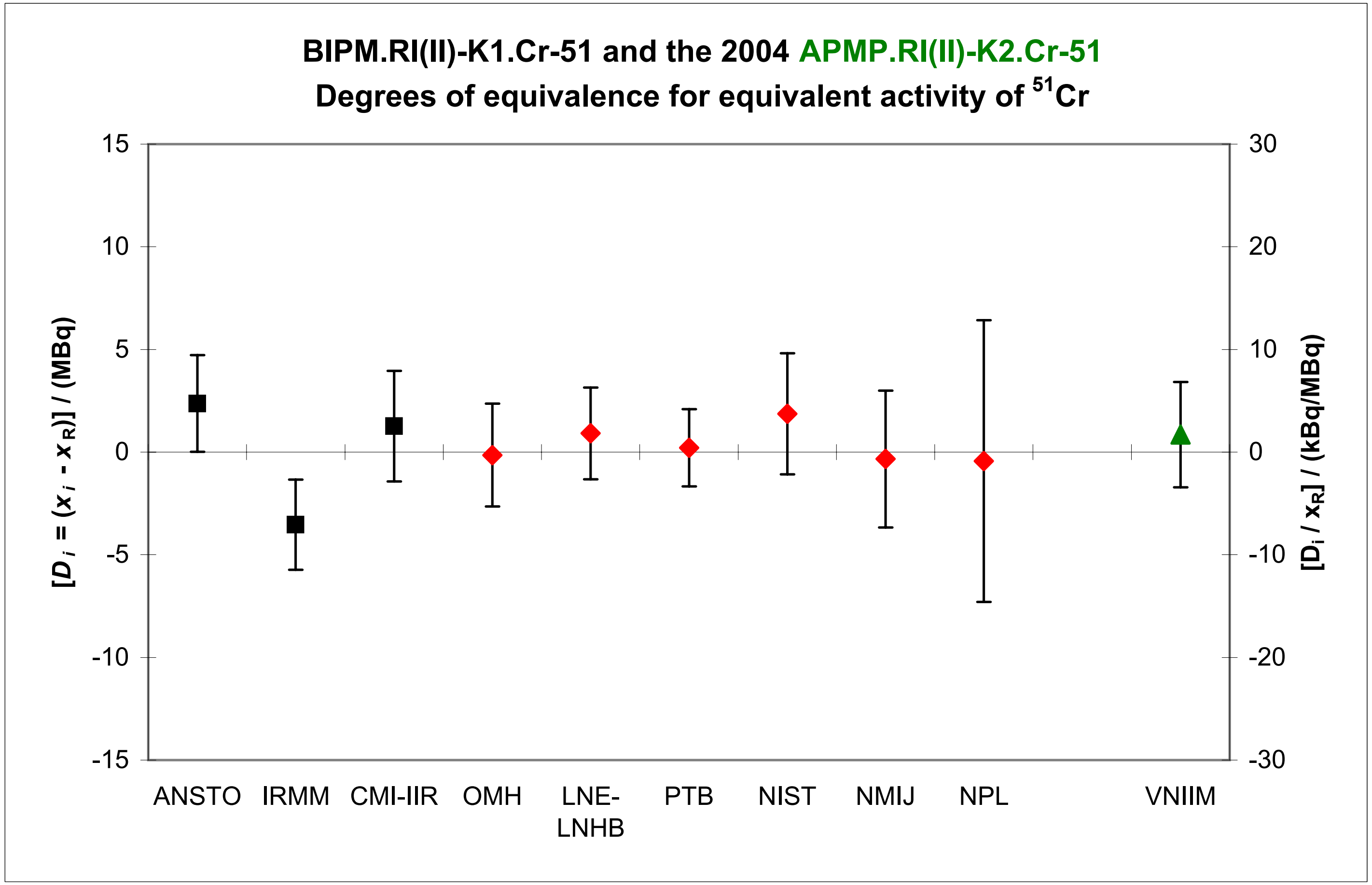
Lab *i* ⇩

Lab i	D_i	U_i	ANSTO		IRMM		CMI-IIR		OMH		LNE-LNHB	
	/ MBq		D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}
ANSTO	**2.4**	2.4			**5.9**	3.3	**1.1**	3.7	**2.5**	3.5	**1.4**	3.3
IRMM	**-3.5**	2.2	**-5.9**	3.3			**-4.8**	3.6	**-3.4**	3.4	**-4.5**	3.1
CMI-IIR	**1.3**	2.7	**-1.1**	3.7	**4.8**	3.6			**1.4**	3.8	**0.3**	3.6
OMH	**-0.1**	2.5	**-2.5**	3.5	**3.4**	3.4	**-1.4**	3.8			**-1.1**	3.4
LNE-LNHB	**0.9**	2.2	**-1.4**	3.3	**4.5**	3.1	**-0.3**	3.6	**1.1**	3.4		
PTB	**0.2**	1.9	**-2.2**	3.0	**3.7**	2.9	**-1.1**	3.3	**0.4**	3.2	**-0.7**	2.9
NIST	**1.9**	2.9	**-0.5**	3.7	**5.4**	3.6	**0.6**	4.0	**2.0**	3.8	**0.9**	3.6
NMIJ	**-0.3**	3.3	**-2.7**	4.0	**3.2**	3.9	**-1.6**	4.3	**-0.2**	4.1	**-1.3**	3.9
NPL	**-0.4**	6.9	**-2.8**	7.2	**3.1**	7.1	**-1.7**	7.4	**-0.3**	7.3	**-1.4**	7.2
VNIIM	**0.9**	2.6	**-1.5**	3.4	**4.4**	3.3	**-0.4**	3.7	**1.0**	3.5	**-0.1**	3.3

Lab *i* ⇩

Lab i	D_i	U_i	PTB		NIST		NMIJ		NPL		VNIIM	
	/ MBq		D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}	D_{ij} / MBq	U_{ij}
ANSTO	**2.4**	2.4	**2.2**	3.0	**0.5**	3.7	**2.7**	4.0	**2.8**	7.2	**1.5**	3.4
IRMM	**-3.5**	2.2	**-3.7**	2.9	**-5.4**	3.6	**-3.2**	3.9	**-3.1**	7.1	**-4.4**	3.3
CMI-IIR	**1.3**	2.7	**1.1**	3.3	**-0.6**	4.0	**1.6**	4.3	**1.7**	7.4	**0.4**	3.7
OMH	**-0.1**	2.5	**-0.4**	3.2	**-2.0**	3.8	**0.2**	4.1	**0.3**	7.3	**-1.0**	3.5
LNE-LNHB	**0.9**	2.2	**0.7**	2.9	**-0.9**	3.6	**1.3**	3.9	**1.4**	7.2	**0.1**	3.3
PTB	**0.2**	1.9			**-1.7**	3.3	**0.5**	3.7	**0.6**	7.0	**-0.6**	3.0
NIST	**1.9**	2.9	**1.7**	3.3			**2.2**	4.3	**2.3**	7.4	**1.0**	3.7
NMIJ	**-0.3**	3.3	**-0.5**	3.7	**-2.2**	4.3			**0.1**	7.5	**-1.2**	3.1
NPL	**-0.4**	6.9	**-0.6**	7.0	**-2.3**	7.4	**-0.1**	7.5			**-1.3**	7.2
VNIIM	**0.9**	2.6	**0.6**	3.0	**-1.0**	3.7	**1.2**	3.1	**1.3**	7.2		

Figure 1. Graph of degrees of equivalence with the KCRV for ^{51}Cr (as it appears in Appendix B of the MRA)



N.B. The right-hand scale gives approximate relative values only

Appendix 1. Uncertainty budgets for the activity of ^{51}Cr

Uncertainty evaluation of the NMIJ (2004) submitted to the SIR

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
Contributions due to	**A**	**B**
Linear extrapolation, including weighing and counting type A uncertainties	18	–
Fixed dead time	–	1
Resolving time	–	3
Timing	–	2
Half-life	–	3
Weighing	–	5
Background	–	20
Quadratic summation	**18**	**21**
Total relative combined uncertainty u_c	**28**	

Uncertainty evaluation of the NPL (2004) submitted to the SIR

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
Contributions due to	**A**	**B**
Counting statistics	10	–
Dead-time	–	< 1
Pile-up	–	< 1
Counting time	–	1
Extrapolation	–	59
Choice of fit	–	24
Half-life	–	< 1
Dilution factor	–	3
Weighing	–	4
Background	–	10
Quadratic summation	**10**	**66**
Total relative combined uncertainty u_c	**67**	

Uncertainty evaluations of the VNIIM in the 2004 APMP comparison

for the 4P-PC-MX-NA-GR-CO method

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
Contributions due to	**A**	**B**
Counting statistics	6	–
Weighing	–	6
Dead-time	–	< 1
Resolving time	–	5
Extrapolation	–	17
Half-life	–	< 1
Background	–	11
Quadratic summation	**6**	**22**
Total relative combined uncertainty u_c	**23**	

for the UA-NA-XR-NA-GR-CO method

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
Contributions due to	**A**	**B**
Counting statistics	7	–
Dead-time	–	< 1
Resolving time	–	9
Half-life	–	< 1
Gamma-ray sensitivity of the KX detector	–	2
Weighing	–	1
Background	–	< 1
Quadratic summation	**7**	**9**
Total relative combined uncertainty u_c	**12**	

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	NaI(Tl)	NA
		Ge(HP)	GH
		Ge(Li)	GL
		Si(Li)	SL
		CsI(Tl)	CS
		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 + γ)	PH	triple-to-double coincidence ratio counting	TD
photons + electrons	PE	selective sampling	SS
alpha - particle	AP	high efficiency	HE
mixture of various radiations	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(Ge(HP))-anticoincidence counting	4P-PP-MX-GH-GR-AC
4π CsI-β,AX,γ counting	4P-CS-MX-00-00-HE
calibrated IC	4P-IC-GR-00-00-00
internal gas counting	4P-PC-BP-00-00-IG