

Update of the ongoing comparison BIPM.RI(II)-K1. Ga-67 to include the activity measurements of the LNE-LNHB, France

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Abstract

Since 1978, eight national metrology institutes (NMIs) and one other laboratory have submitted fifteen samples of known activity of ^{67}Ga to the International Reference System (SIR) for activity comparison at the Bureau International des Poids et Mesures in the ongoing key comparison, BIPM.RI(II)-K1.Ga-67. The activities ranged from about 3 MBq to 600 MBq. The recent submission from the LNE-LNHB, France, replaces their earlier result in the key comparison database for this comparison. The key comparison reference value (KCRV) has been re-calculated and the degrees of equivalence between each equivalent activity measured in the SIR are given in the form of a matrix. A graphical presentation is also given.

1. Introduction

The SIR for activity measurements of γ -ray-emitting radionuclides was established in 1976. Each NMI may request a standard ampoule from the BIPM that is then filled (3.6 g) with the radionuclide in liquid form or a different standard ampoule for radioactive gases.. 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 2005, the SIR has measured 891 ampoules to give 651 independent results for 63 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 Mutual Recognition Arrangement (MRA) [2]. The comparison described in this report is known as the BIPM.RI(II)-K1.Ga-67 key comparison and the latest result updates those presented in [3, 4].

2. Participants

Eight NMIs and one other laboratory have submitted fifteen ampoules for the comparison of ^{67}Ga activity measurements since 1978. One other laboratory participated but ultimately withdrew its result. The laboratory details are given in Table 1. In cases where the laboratory has changed its name since the original submission, both the earlier and the current acronyms are given, as it is the latter that are used in the KCDB.

Table 1. Details of the participants in the BIPM.RI(II)-K1.Ga-67

Original acronym	NMI	Full name	Country	Regional metrology organization	Date of measurement at the BIPM
NBS	NIST	National Institute of Standards and Technology	United States	SIM	1978-03-21 1998-04-27 1999-04-28
UVVVR	CMI-IIR	Český Metrologický Institut/Czech Metrological Institute, Inspectorate for Ionizing Radiation	Czech Republic	EUROMET	1981-04-24
LMRI	LNE-LNHB	Laboratoire national de métrologie et d'essais - Laboratoire national Henri Becquerel	France	EUROMET	1981-11-10 2005-10-20
–	NPL	National Physical Laboratory	United Kingdom	EUROMET	1982-04-30
NIRH	–	National Institute of Radiation Hygiene	Denmark	EUROMET	1983-05-05
NAC*	CSIR-NML	National Metrology Laboratory	South Africa	SADCMET	1986-10-28
–	OMH	Országos Mérésügyi Hivatal	Hungary	EUROMET	1995-11-30
–	NMIJ	National Metrology Institute of Japan	Japan	APMP	2001-11-26 2002-05-17

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Table 1 continued. Details of the participants in the BIPM.RI(II)-K1.Ga-67

Original acronym	NMI	Full name	Country	Regional metrology organization	Date of measurement at the BIPM
–	CIEMAT	Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas	Spain	EUROMET	2003-03-19

* another laboratory in the country

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. The uncertainty budgets for comparison results post 1998 are given in [3]. The uncertainty budgets for the latest submissions of the LNE-LNHB and of the CIEMAT are in Appendix 1. The list of acronyms used to summarize the measurement methods is given in Appendix 2.

The half-lives given in Table 2 are the values (and standard uncertainties) as used by the participants. The half-life used previously by the BIPM was 3.261 (3) d [5] and this was changed to 3.2613 (5) d, following the recent evaluation published in *BIPM Monographie 5* [6]. All the results in Table 4 have been updated accordingly. The consequence of this has been non-significant changes of the equivalent activities and a reduction in many of the SIR uncertainties.

Table 2. Standardization methods of the participants for ^{67}Ga

NMI	Method used and acronym (see Appendix 2)	Half-life	Activity A_i / kBq	Reference date YY-MM-DD	Relative standard uncertainty $\times 100$ by method of evaluation	
					A	B
NIST	Pressurized IC* 4P-IC-GR-00-00-00	–	565 200	78-03-15 19 h UT	0.01	1.49
		3.2614 (6) d	94 860	98-04-27 12 h UT	0.03	0.27
			53 990	99-04-28 12 h UT	0.01	0.30

continued overleaf

Table 2 continued . Standardization methods of the participants for ⁶⁷Ga

NMI	Method used and acronym (see Appendix 2)	Half-life	Activity A _i / kBq	Reference date YY-MM-DD	Relative standard uncertainty × 100 by method of evaluation	
					A	B
CMI-IIR	4π(e,x)-γ coincidence 4P-PP-MX-NA-GR-CO	78.26 h	22 750	81-04-08 12 h UT	0.05	0.87
LNE-LNHB	4π(e _A ,x)-γ anti-coincidence 4P-PP-MX-NA-GR-AC	–	4 772 [†] 4 771	81-11-13 12 h UT	0.02	0.38
	4πLS-γ anti-coincidence 4P-LS-MX-NA-GR-AC	3.2613 (5) d [6]	3 269 [†] 3 253	05-10-18 12 h UT	0.25	0.08
NPL	Pressurized IC** 4P-IC-GR-00-00-00	–	30 090	82-04-28 0 h UT	0.04	1.22
NIRH	Pressurized IC 4P-IC-GR-00-00-00	–	118 290	83-05-04 12 h UT	0.04	0.60
CSIR-NML	4π(LS)(e,x)-γ coincidence [7] 4P-LS-MX-NA-GR-CO	78.26 h	222 770	86-10-24 10 h UT	0.15	0.17
OMH	4π(e _A ,x)-γ anti-coincidence 4P-PP-MX-NA-GR-AC	3.26154 (54) d [8]	6 817	95-12-01 0 h UT	0.06	0.51
NMIJ	Pressurized IC*** 4P-IC-GR-00-00-00	3.2612 d	21 850	01-11-30 12 h UT	0.06	0.41
			36 650	02-05-15 12 h UT	0.06	0.36
CIEMAT	4πβ(PPC)-γ coincidence 4P-PP-AE-NA-GR-CO	3.259 (10) d	7 916	03-03-12 10 h UT	0.70	0.52

[†] two ampoules submitted

* calibrated by 4π(e_A,x)-γ coincidence (4P-PP-MX-NA-GR-CO) for ⁶⁷Ga in 1977

** calibrated by 4π(e_A,x)-γ coincidence (4P-PC-MX-NA-GR-CO) for ⁶⁷Ga

*** calibrated by 4π(e,x)-γ anti-coincidence (4P-PP-MX-NA-GR-AC) in August 2001 for ⁶⁷Ga.

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 a standard method for evaluating the activity of impurities using a calibrated Ge(Li) spectrometer [9]. The CCRI(II) agreed in 1999 [10] that this method should be followed according to the protocol described in [11] when an NMI makes such a request or when there appear to be discrepancies. No impurity was identified in the 2005 LNE-LNHB solution.

The same primary standardization was used to calibrate the ionization chamber used for the NIST submissions of 1978, 1998 and 1999. However, the latter two samples have smaller uncertainties due to better source characterizations and so the 1998 value, being closer in time to the 1977 calibration than the 1999 result, has been used for the KCRV (see section 4.1).

In view of the uncertainty over the Ga-citric acid solution of the 2001 NMIJ ampoule, the result of 2002 has been used in the KCRV rather than the result from 2001 although the two results are consistent within the uncertainties [3].

Table 3. Details of the solution of ^{67}Ga submitted

NMI	Chemical composition	Solvent conc. / (mol dm ⁻³)	Carrier: conc. / (μg g ⁻¹)	Density / (g cm ⁻³)	Relative activity of impurity [†]
NIST	^{67}Ga in HCl	2	–	1.032	–
	GaCl ₃ in HCl	2.1	GaCl ₃ : 1950	1.036 (2)	–
			GaCl ₃ : 805	1.035	–
CMI-IIR	GaCl ₃ in HCl	1	GaCl ₃ : 50	–	< 0.1 %
LNE-LNHB	^{67}Ga citrate* and NaCl in HCl	0.1	NaCl: 180	1.006	^{57}Co : $5.1 (1.0) \times 10^{-4}$ % ^{60}Co : $7.2 (1.5) \times 10^{-4}$ %
	GaCl ₃ in HCl	0.1	GaCl ₃ : 48	1.000	–
NPL	^{67}Ga in HCl	0.1	–	1.0015	–
NIRH	^{67}Ga citrate* in NaCl solution	–	–	–	–
CSIR-NML	Na citrate* in HCl	1.0	Na ₃ C ₆ O ₇ H ₅ ·2H ₂ O: 2600 Ga: 100	1.0143	–
OMH	^{67}Ga citrate* in NaCl solution	–	NaCl: 8000	–	–
NMIJ	Ga citrate* and NaCl in HCl	0.1	GaC ₆ O ₇ H ₅ : 200 NaCl: 9000	1.02	–
	GaCl ₃ in HCl	0.1	GaCl ₃ : 100	1.002	–
CIEMAT	Ga citrate* in HCl	0.1	Na ₃ C ₆ O ₇ H ₅ ·2H ₂ O : 230	1	–

[†] the ratio of the activity of the impurity to the activity of ^{67}Ga at the reference date

* gallium citrate: GaC₆O₇H₅ ; sodium citrate: Na₃C₆O₇H₅·2H₂O.

4. Results

All the submissions to the SIR since its inception in 1976 are maintained in a database master-file. The activity measurements for ^{67}Ga arise from twelve ampoules and the SIR equivalent activity for each ampoule, A_{ei} , is given in Table 4 for each NMI, i . The dates of measurement in the SIR are given in Table 1.

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 1 to 8 half-lives later (periods of up to one month) produced results in agreement within one SIR standard uncertainty for the CMI-IIR, LNE-LNHB (1981 and 2005), NPL, NIRH, CSIR-NML, OMH, NIST (1998 and 1999), NMIJ (2001 and 2002) and for the CIEMAT [3, 4]. These measurements confirm the validity of the half-life value used.

The latest LNE-LNHB result agrees with their 1981 result within two combined standard uncertainties, while both results are slightly lower than most other SIR results. This deviation may be linked to the ^{67}Zn meta-stable state (9 μs) populated by the ^{67}Ga decay, which makes the standardization of this radionuclide by the laboratories using the coincidence method rather challenging. As the LNE-LNHB uses anti-coincidence counting with extended dead-times, no correction related to the meta-stable state is necessary [12].

No recent submission has been identified as a pilot study so the most recent result of each NMI is normally eligible for Appendix B of the MRA. However, the NIRH is not the designated laboratory of Denmark and therefore their result is not included in the KCDB.

No international or regional comparison for this radionuclide has been held to date so no linking data are identified.

4.1 The key comparison reference value

The key comparison reference value is derived from the unweighted mean of all the results submitted to the SIR with the following provisions:

- a) only primary standardized solutions are accepted, with the exception of radioactive gas standards, for which results from transfer instrument measurements that are directly traceable to a primary measurement in the laboratory may be included;
- b) each NMI or other laboratory has only one result (normally the most recent result or the mean if more than one ampoule is submitted);
- c) any outliers are identified using a reduced chi-squared test and, if necessary, excluded from the KCRV using the normalized error test with a test value of four;
- d) exclusions must be approved by the CCRI(II).

Table 4. Results of SIR measurements of ⁶⁷Ga

NMI	Mass of solution m_i / g	Activity submitted A_i / kBq	N° of Ra source used	SIR A_e / kBq	Relative uncertainty from SIR	Combined uncertainty $u_{c,i}$ / kBq
NIST	3.689 38	565 200	5	115 600	8×10^{-4}	1700
	3.746 00	94 860	5	116 090	8×10^{-4}	330
	3.711 19	53 990	4	116 230	8×10^{-4}	360
CMI-IIR	3.457 40	22 750	1	118 800	19×10^{-4}	1100
LNE-LNHB	3.612 5	4 772	3	114 616	10×10^{-4}	450
	3.611 9	4 771		114 597	9×10^{-4}	450
	3.557 1	3 269	2	113 955 [†]	11×10^{-4}	320
	3.539 5	3 253		113 695		320
NPL	3.682 1	30 090	4	116 000	9×10^{-4}	1400
NIRH	3.573 6	118 290	5	115 640	8×10^{-4}	710
CSIR-NML	3.605 65	222 770	5	116 430	8×10^{-4}	280
OMH	3.639 4	6 817	3	115 210	9×10^{-4}	600
NMIJ	3.603 96	21 850	4	114 670	8×10^{-4}	480
	3.607 28	36 650	4	115 210	8×10^{-4}	430
CIEMAT	3.663	7 916	1	117 960	13×10^{-4}	1040

[†] the mean of the two A_e values is used with an averaged uncertainty, as attributed to an individual entry [13].

The reduced data set used for the evaluation of the KCRVs is known as the KCRV file and is the reduced data set from the SIR master-file. Although the KCRV may be modified when other NMIs participate, on the advice of the Key Comparison Working Group of the CCRI(II) as is the case for the LNE-LNHB new result, such modifications are only made by the CCRI(II) whose members also agreed to include the result of the CIEMAT in the KCRV during the biennial meeting in 2005.

Consequently, the KCRV for ⁶⁷Ga has changed from 116 040 (520) kBq to the new value of 116 190 (560) kBq using the results from the CMI-IIR, NPL, CSIR-NML, OMH, NIST (1998), NMIJ (2002), CIEMAT and the LNE-LNHB (2005).

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. An 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

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

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 [14].

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 $u(A_{e_i}, A_{e_j})$, 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_{e_i} 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 where the black

squares indicate results obtained prior to 1985. 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.

It should be noted that the equivalent activity of value 115 200 (2 000) kBq obtained independently using the SIR efficiency curve [15] for ^{67}Ga has a 1.7 % relative uncertainty arising from the large uncertainties in the gamma emission intensities [6]. In addition, there are some doubts about the decay scheme [16] and this makes it difficult to discriminate in favour of any particular grouping of SIR results shown in Figure 1.

Conclusion

The BIPM ongoing key comparison for ^{67}Ga , BIPM.RI(II)-K1.Ga-67 currently comprises eight results including the latest result from the LNE-LNHB. All the results 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. Other results may be added as and when other NMIs contribute ^{67}Ga activity measurements to this comparison.

Acknowledgements

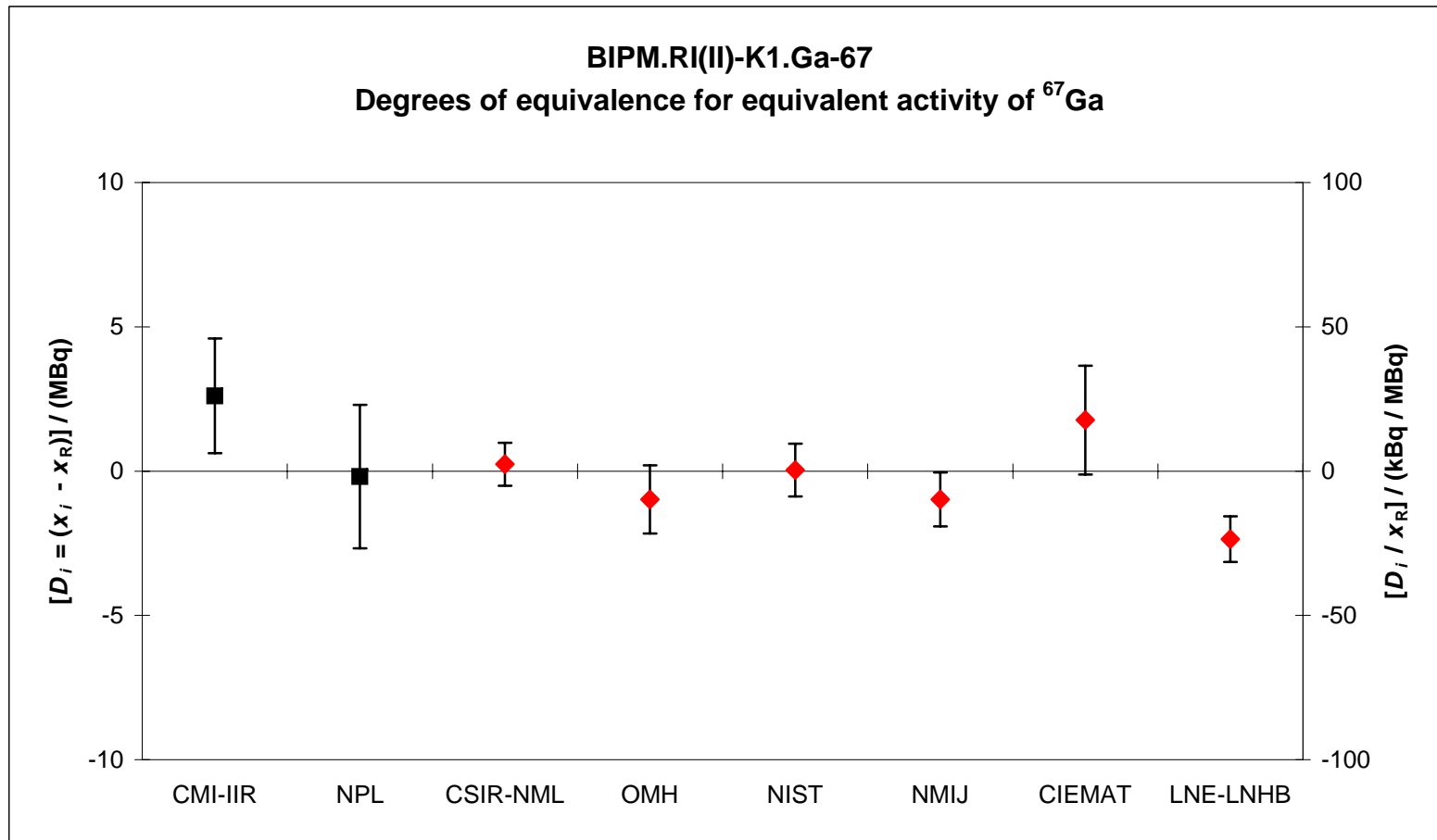
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Figure 1. Graph of degrees of equivalence with the KCRV for ^{67}Ga
(as it appears in Appendix B of the MRA)



N.B. Right-hand axis represents approximate values only

Appendix 1. Recent uncertainty budgets for ⁶⁷Ga activity measurements**Uncertainty budget for the LNE-LNHB submitted to the SIR in 2005**

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
	A	B
Contributions due to		
counting statistics	6	–
dead time (live time technique)	–	< 1
counting time	–	< 1
accidental coincidences in the LS channel	3	–
extrapolation of efficiency curve	23	–
weighing	–	5
background	6	–
half-life	–	6
Quadratic summation	25	8
Relative combined standard uncertainty, u_c	26	

CIEMAT uncertainty budget (ampoule BIPM-301) submitted to the SIR in 2003

Relative standard uncertainties	$u_i \times 10^4$ evaluated by method	
	A	B
Contributions due to		
counting	27	–
dead time	–	45
coincidence resolving time	–	22
Gandy effect	–	1
extrapolation of efficiency curve	62	–
weighing of source	–	10
background	19	–
half-life (3.259 (10) d)	–	10
Quadratic summation	70	52
Total relative combined uncertainty u_c	87	

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 ($x + \gamma$)	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 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