Key comparison BIPM.RI(I)-K5 of the air-kerma standards of the LMRI-CIEMAT, Spain and the BIPM in ^{137}Cs gamma radiation

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Abstract

A first comparison of the standards for air kerma of the Laboratorio de Metrología de Radiaciones Ionizantes - Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (LMRI-CIEMAT), Spain, and of the Bureau International des Poids et Mesures (BIPM) was carried out in the ^{137}Cs radiation beam using the radiation protection facility of the International Atomic Energy Agency (IAEA) in Seibersdorf, Austria, in September 2023[a]. The comparison was done using the CIEMAT and the BIPM primary standards and a transfer instrument belonging to the CIEMAT. The direct comparison result, evaluated as a ratio of the CIEMAT and the BIPM standards for air kerma, is 0.9992 with a combined standard uncertainty of 2.2 parts in 10^3. The result for the indirect comparison is in agreement with the direct result at the level of 2.2 parts in 10^3. The results are analysed and presented in terms of degrees of equivalence, suitable for entry in the BIPM key comparison database, to update the 2014 APMP comparison result.

1. Introduction

A first comparison of the standards for air kerma of the Laboratorio de Metrología de Radiaciones Ionizantes - Centro de Investigaciones Energéticas Medioambientales y Tecnológicas (LMRI-CIEMAT), Spain, and of the Bureau International des Poids et Mesures (BIPM) was carried out in September 2023 in the ^{137}Cs radiation beam at the International Atomic Energy Agency (IAEA) in Seibersdorf, Austria. The comparison result is published in the BIPM key comparison database (KCDB 2023) under the reference BIPM.RI(I)-K5. The comparison was carried out after the implementation of the recommendations of ICRU Report 90 (ICRU 2016) at both laboratories.

The comparison was undertaken using one of the primary standards of the CIEMAT (direct comparison) and a spherical ionization chamber as a transfer instrument (indirect comparison).

[a] Since 2018, the BIPM no longer maintains reference radiation protection beams for ^{137}Cs and ^{60}Co. To continue the services of calibration and comparison (BIPM.RI(I)-K5) in ^{137}Cs beams, in 2022 the BIPM has installed the primary standard together with a BIPM measurement and data acquisition system at the IAEA laboratories in Seibersdorf, Austria.

2. Details of the standards

The air-kerma standards of the CIEMAT for ^{137}Cs are graphite-walled cavity ionization chambers constructed by the Laboratoire National de Métrologie et d'Essais – Laboratoire National Henri Becquerel (LNE-LNHB), France, referenced as CS-001 and SP-001. The chambers were characterized by the CIEMAT (Cornejo Díaz 2022). For the present comparison, only measurements using the CS-001 standard were made. The main characteristics of the primary standards are given in Table 1. Details of the transfer chamber used for the indirect comparison are also included in Table 1.

The BIPM standard is a parallel-plate graphite-walled cavity ionization chamber with a volume of about 6.8 cm^3 (Boutillon and Niatel 1973, Kessler *et al.* 2009, Burns and Kessler 2018).

Table 1. Characteristics of the CIEMAT standards for air kerma and the transfer chamber

Parameters	Primary standards		Transfer chamber
	CS-001	SP-001	PTW 32005-00047
Shape	Cylindrical with hemispherical ends	Spherical	Spherical
Outer height / mm	39	-	-
Outer diameter / mm	28	28	44.22
Inner height / mm	33	-	-
Inner diameter / mm	22	22	38.22
Wall material	Graphite (99.998 % purity)		Polyoxymethylene, graphite coated
Wall density / g cm^{-3}	1.8281		0.453
Wall thickness / mm	3	3	3.24
Electrode material	graphite		PMMA, graphite coated
Electrode diameter / mm	3	3	4.2
Electrode height / mm	23	12	29.7
Volume / cm^3	9.6126	5.5104	27.9 (nominal)
Applied voltage to outer electrode	400 V; both polarities		400 V (positive polarity)

3. Determination of the air kerma

For a cavity chamber with measuring volume *V*, the air-kerma rate is determined by the relation

$$\dot{K} = \frac{I}{\rho_{\mathrm{air}} V} \frac{W}{e} \frac{1}{1-\bar{g}} \left(\frac{\mu_{\mathrm{en}}}{\rho}\right)_{\mathrm{a,c}} \bar{s}_{\mathrm{c,a}} \prod k_i \qquad (1)$$

where

ρ_{air} is the density of air under reference conditions,

I is the ionization current under the same conditions,

W is the average energy spent by an electron of charge e to produce an ion pair in dry air,

$\bar{g}$ is the fraction of electron energy lost by radiative processes in air,

$(\mu_{\mathrm{en}}/\rho)_{\mathrm{a,c}}$ is the ratio of the mean mass energy-absorption coefficients of air and graphite,

$\bar{s}_{\mathrm{c,a}}$ is the ratio of the mean mass electronic stopping powers for electrons in graphite and air,

$\prod k_i$ is the product of the correction factors to be applied to the standard.

Physical data and correction factors

The values used for the physical constants, the correction factors, the volume of the primary standards entering in equation (1), and the associated uncertainties for the ^{137}Cs radiation beam at the IAEA are given in Table 2.

Table 2. Physical constants and correction factors with their relative standard uncertainties of the BIPM and CIEMAT standards for the ^{137}Cs radiation beam at the IAEA

		BIPM	CH 6.3		CIEMAT	CS-001	
		values	uncertainty [1]		values	uncertainty [1]	
			100 $u_{i\mathrm{A}}$	100 $u_{i\mathrm{B}}$		100 $u_{i\mathrm{A}}$	100 $u_{i\mathrm{B}}$
Physical Constants							
ρ_{air}	dry air density [2] / kg m^{-3}	1.2930	–	0.01	1.2930	–	0.01
$s_{\mathrm{c,a}}$	ratio of mass stopping powers	1.0023	–	0.12 [3]	1.0034	–	0.13 [3]
W/e	mean energy per charge / J C^{-1}	33.97	–		33.97	–	
$(\mu_{\mathrm{en}}/\rho)_{\mathrm{a,c}}$	ratio of mass energy-absorption coefficients	0.9990	0.01	0.04	0.9994	–	0.10
g_{a}	fraction of energy lost in radiative processes	0.0012	–	0.02	0.0015	–	
Correction factors:							
k_{s}	recombination losses	1.0013	0.01	0.02	1.0009	0.01	0.02
k_{h}	humidity	0.9970	–	0.03	0.9970	–	0.03
k_{st}	stem scattering	0.9998	0.01	–	0.9986	0.03	–
k_{wall}	wall attenuation and scattering	1.0002	0.01	– [4]	1.0233	0.01	0.10 [5]
k_{an}	axial non-uniformity	1.0018	–	0.04	1.0000	0.05	0.03
k_{rn}	radial non-uniformity	1.0009	0.01	0.03	1.0004	0.01	0.03
k_{or}	orientation	0.9997	0.01	0.01	–	–	–
k_{SA}	Spencer-Attix theory	–	–	–	1.0000		0.10
Measurement of I / V							
V	chamber volume / cm^3	6.8313	–	0.08	9.6126	–	0.10 [6]
k_{vol}	volume correction	1.0004	–				
I	ionization current / pA	–	0.02	0.02	–	0.02	0.02
	reproducibility		0.03	–		0.01	–
Relative standard uncertainty							
quadratic summation			0.04	0.16		0.06	0.25
combined uncertainty			**0.17**			**0.25** [7]	
Removing correlations			0.04	0.10		0.06	0.18

(1) Expressed as one standard deviation
$u_{i\mathrm{A}}$ represents the type A relative standard uncertainty estimated by statistical methods,
$u_{i\mathrm{B}}$ represents the type B relative standard uncertainty estimated by other means

(2) At 101 325 Pa and 273.15 K

(3) Combined uncertainty for the product of $s_{\mathrm{c,a}}$ and W/e (Burns and Kessler 2018; Cornejo Díaz 2022). At the BIPM it was adopted in January 2019

(4) The uncertainties for k_{wall} and k_{an} are included in the determination of the effective volume (Burns *et al* 2007)

(5) Includes the uncertainty corresponding to the angular dependence of the chamber response

(6) Includes the uncertainty assessed from the difference between the air kerma rates determined using the CS-001 and SP-001 standards

(7) The combined uncertainty at the CIEMAT is 3.2 parts in 10^3

The correction factors and the associated uncertainties for the CIEMAT primary standard at the CIEMAT are described in Cornejo Díaz (2022). The combined standard uncertainty at the CIEMAT is 3.2 parts in 10^3. The increase results mainly from the CIEMAT measuring system and the characteristics of the CIEMAT beam.

Reference values

The BIPM reference air-kerma rate $\dot{K}_{\mathrm{BIPM}}$ is taken as the mean of three measurements made during the period of the comparison. The $\dot{K}_{\mathrm{BIPM}}$ value refers to an evacuated path length between source and standard corrected to the reference date of 2023-01-01, 0 h UTC. The half-life of ^{137}Cs was taken as 10 976 days (u = 30 days) (Bé *et al.* 2006). The correction for air attenuation between source and standard uses the ambient air density at the time of the measurement and the air attenuation coefficient 0.010 m^{-1} for ^{137}Cs.

At the CIEMAT, the $\dot{K}_{\mathrm{CIEMAT}}$ value is taken as the mean of the values determined using both primary standards. Measurements have been carried out quarterly since 2019. By convention it is given at the reference date of 2019-01-01 0 h UTC using the half-life value 10964 days (u = 8 days) for the source decay correction (http://www.lnhb.fr/nuclear-data/nuclear-data-table/).

Reference conditions and beam characteristics

The characteristics of the IAEA and CIEMAT beams are given in Table 3. The distance from source to reference plane is 1 m and 2 m at the IAEA and the CIEMAT, respectively. The values for the nominal air-kerma rate are corrected to the date of 2023-01-01.

Table 3. Characteristics of the ^{137}Cs beams at the CIEMAT and the IAEA

^{137}Cs beam	Source dimensions / mm		Nominal $\dot{K}$/ µGy s^{-1}	Field diameter / mm
	diameter	length		
CIEMAT source	32.0	54.7	139.5	260 at 2 m
IAEA source	14.3	19.9	17.5	230 at 1 m

4. Experimental method

The BIPM and the CIEMAT experimental method for measurements is described in the following paragraphs.

Positioning

At each laboratory the chambers were positioned with the stem perpendicular to the beam direction and with the appropriate marking on the stem facing the source.

Applied voltage and polarity

A collecting voltage of 400 V (both polarities) and 400 V (positive polarity) was applied to the outer electrode of the CIEMAT standard and the transfer chamber, respectively, at least 40 min before any measurements were made.

Charge and leakage measurements

The charge Q collected by the CIEMAT chambers was measured by the BIPM at the IAEA using a Keithley electrometer, model 6517B. The source is exposed during the entire measurement series and the charge is collected for the appropriate, electronically controlled, time interval. A pre-irradiation was made for at least 40 min before any measurements.

Leakage current was measured before and after each series of measurements. The measured leakage value for the standard was around 1 fA, 2 parts in 10^4 in relative terms, of the ionization current. The standard was measured on two separate occasions, the chamber being repositioned.

The ionization current measured using the transfer chamber was corrected for the leakage current; in relative value it was less than 2 parts in 10^4.

At the LMRI-CIEMAT, the charge was measured using a SCANDITRONIX WELLHOFER-IBA model DOSE 1 Reference Class electrometer. The chambers are exposed to the beam during the entire measurement sequence and the charge is collected for the preselected, electronically controlled, time interval. The chambers were placed at the reference point at least 2 hours before the beginning of measurements, with the polarizing voltage applied at least 30 min before the measurements start and with a pre-irradiation of at least 15 min. The leakage current was measured before and after each series of measurements. The leakage correction for the standards was less than 2 parts in 10^4 and less than 1 part in 10^4 for the transfer chamber.

Ambient conditions

During a series of measurements, the air temperature is measured for each current measurement and was stable to better than 0.2 °C at the IAEA. At the CIEMAT, the air temperature was stable to better than 0.2 °C during each series of measurements. At both laboratories, the current measured using the standards is normalized to 273.15 K and 101.325 kPa. The current measured using the transfer instrument is normalized to the reference conditions of 293.15 K and 101.325 kPa.

Relative humidity is controlled and it was between 45 % and 55 % at the IAEA and between 39 % and 60 % at the CIEMAT.

5. Results of the comparison

Direct comparison

The CIEMAT primary standard was set-up and measured in the IAEA ^{137}Cs beam on two separate occasions. The results were reproducible to better than 1 part in 10^4. The values of the ionization currents measured by the BIPM using the CIEMAT standard are given in Table 4.

Table 4. The experimental results using the CIEMAT standard in the IAEA beam

CIEMAT standard	I_+ and I_- / pA		I_{mean} / pA
CS-001	6.2262	-6.2343	6.2302
	6.2284	-6.2307	6.2295
Mean current			6.2299

The result of the comparison R_K is expressed in the form

$$R_K = \dot{K}_{\text{CIEMAT}} / \dot{K}_{\text{BIPM}} \tag{2}$$

where $\dot{K}_{\text{CIEMAT}}$ and $\dot{K}_{\text{BIPM}}$ are the air-kerma rate values measured by the BIPM using the CIEMAT CS-001 and the BIPM CH6.3 primary standards, respectively; the results are presented in Table 5.

Table 5. Final result of the CIEMAT/BIPM comparison of standards for ^{137}Cs air kerma

CIEMAT standard	$\dot{K}_{\text{CIEMAT}}$ / µGy s^{-1}	$\dot{K}_{\text{BIPM}}$ / µGy s^{-1}	R_K	u_c
CS-001	17.445	17.460	0.9992	0.0022

The combined standard uncertainty u_c for the comparison result R_K is presented in Table 6.

The ratio of the air-kerma rate values determined by the BIPM using the CIEMAT and the BIPM standards is 0.9992 with a combined standard uncertainty, u_c, of 0.0022. Some of the uncertainties in $\dot{K}$ that appear in both the BIPM and the CIEMAT determinations (such as air density, W/e, μ_{en}/ρ, $\bar{g}$, $\bar{s}_{c,a}$ and k_h) cancel when evaluating the uncertainty of R_K.

Table 6. Uncertainties associated with the comparison result

Relative standard uncertainty	100 u_{iA}	100 u_{iB}
$\dot{K}_{\text{CIEMAT}}/\dot{K}_{\text{BIPM}}$	0.08	0.21$^{(1)}$
Relative standard uncertainty of R_K	u_c = 0.0022	

(1) Takes account of correlation in type B uncertainties.

Indirect comparison

The transfer chamber was set-up and measured by the BIPM in the IAEA ^{137}Cs beam on three separate occasions. The comparison result is evaluated as the ratio of the calibration coefficients $N_{K,\text{lab}}$ determined at each laboratory. The calibration coefficient is given by

$$N_{K,\text{lab}} = \dot{K}_{\text{lab}}/I_{\text{lab}} \tag{3}$$

where $\dot{K}_{\text{lab}}$ is the air-kerma rate at each lab and I_{lab} is the ionization current of a transfer chamber measured by the CIEMAT at the CIEMAT or by the BIPM at the IAEA. Table 7 lists the relevant values of N_K at the stated reference conditions (293.15 K and 101.325 kPa) and the final results of the indirect comparison. The uncertainties associated with the calibration of the transfer chamber at each laboratory and with the indirect comparison are presented in Table 8 and Table 9, respectively.

Table 7. Results of the indirect comparison

Transfer chamber	$N_{K,\text{CIEMAT}}$/ Gy µC^{-1}			$N_{K,\text{BIPM}}$ / Gy µC^{-1}	R_K	u_c
	pre-BIPM	post-BIPM	overall mean			
PTW 32005	1.1181	1.1166	1.1173	1.1158	1.0014	0.0034

The calibration coefficients measured before and after the measurements at the IAEA give rise to the relative standard uncertainty s_{tr}, which represents the uncertainty in the N_K arising from the transfer chamber stability, included in Table 9.

Note that at the CIEMAT, the current measured using the PTW 32005 is normally corrected for losses due to ion recombination. As volume recombination is negligible at the present ^{137}Cs dose rates at this polarizing voltage, and the initial recombination loss will be the same in the two laboratories, no correction for recombination was applied to the measured current

for the present comparison; a relative uncertainty component of 1 part in 10^4 is included in Table 9.
The radial non-uniformity correction is estimated to be 1.0005 and 1.0008 at the IAEA and the CIEMAT, respectively; no radial non-uniformity correction was applied and a relative uncertainty component of 2 parts in 10^4 is included in Table 9.

Table 8. Uncertainties associated with the transfer chamber calibration

Transfer chamber	BIPM		CIEMAT	
Relative standard uncertainty	100 u_{iA}	100 u_{iB}	100 u_{iA}	100 u_{iB}
Air-kerma rate	0.04	0.16	0.09	0.31
Ionization current for the transfer chamber	0.01	0.02	0.01	0.12
Distance	0.01	–	–	0.06
Reproducibility	0.02	–	0.02	–
Temperature, pressure	–	–		0.04
$N_{K,\mathrm{lab}}$	0.05	0.17	0.09	0.34

The result of the indirect comparison taken from Table 7 is 1.0014 with a combined standard uncertainty, u_c, of 0.0034. This result is in agreement with the direct comparison at the level of 2.2 parts in 10^3, which is within the standard uncertainty of the calibration procedure. The result of the direct comparison is used to evaluate the degrees of equivalence for entry in the key comparison database (KCDB).

Table 9. Uncertainties associated with the indirect comparison

Indirect comparison result	100 u_{iA}	100 u_{iB}
$N_{K,\mathrm{CIEMAT}}$ / $N_{K,\mathrm{BIPM}}$	0.10	0.31(1)
Short-term stability s_{tr}	0.08	–
Ion recombination	–	0.01
Radial non-uniformity	–	0.02
$N_{K,\mathrm{CIEMAT}}$ / $N_{K,\mathrm{BIPM}}$	u_c = 0.0034	

(1) The combined standard uncertainty of the comparison result takes into account correlation in the type B uncertainties associated with the physical constants and the humidity correction

6. Degrees of equivalence

Comparison of a given NMI with the key comparison reference value

Following a decision of the CCRI, the BIPM determination of the dosimetric quantity, here K_{BIPM}, is taken as the key comparison reference value (KCRV) (Allisy *et al.* 2009). It follows that for each NMI i having a BIPM comparison result x_i with combined standard uncertainty u_i, the degree of equivalence with respect to the reference value is the relative difference $D_i = (K_i - K_{\mathrm{BIPM},i}) / K_{\mathrm{BIPM},i} = x_i - 1$ and its expanded uncertainty $U_i = 2\,u_i$.

The results for D_i and U_i are usually expressed in mGy/Gy. Table 10 gives the values for D_i and U_i for each NMI, *i*, taken from the KCDB of the CIPM MRA (1999) and this report. These data are presented graphically in Figure 1.

Table 10. Degrees of equivalence

For each laboratory *i*, the degree of equivalence with respect to the key comparison reference value is the difference D_i and its expanded uncertainty U_i. Tables formatted as they appear in the BIPM key comparison database

BIPM.RI(I)-K5

Lab *i*	D_i	U_i
	/ (mGy/Gy)	
GUM	-0.5	5.8
IST/ITN	1.3	4.2
LNE-LNHB	-1.6	5.2
MKEH	5.3	5.0
VNIIM	1.3	5.4
KRISS	-1.4	4.4
NIST	-1.0	7.0
NMIJ	-2.3	5.2
PTB	2.4	5.4
NIM	-3.3	4.2
ININ	4.8	4.0
VSL	-4.8	7.6
SMU	5.1	5.4
BEV	4.3	5.4
CIEMAT	-0.8	4.4

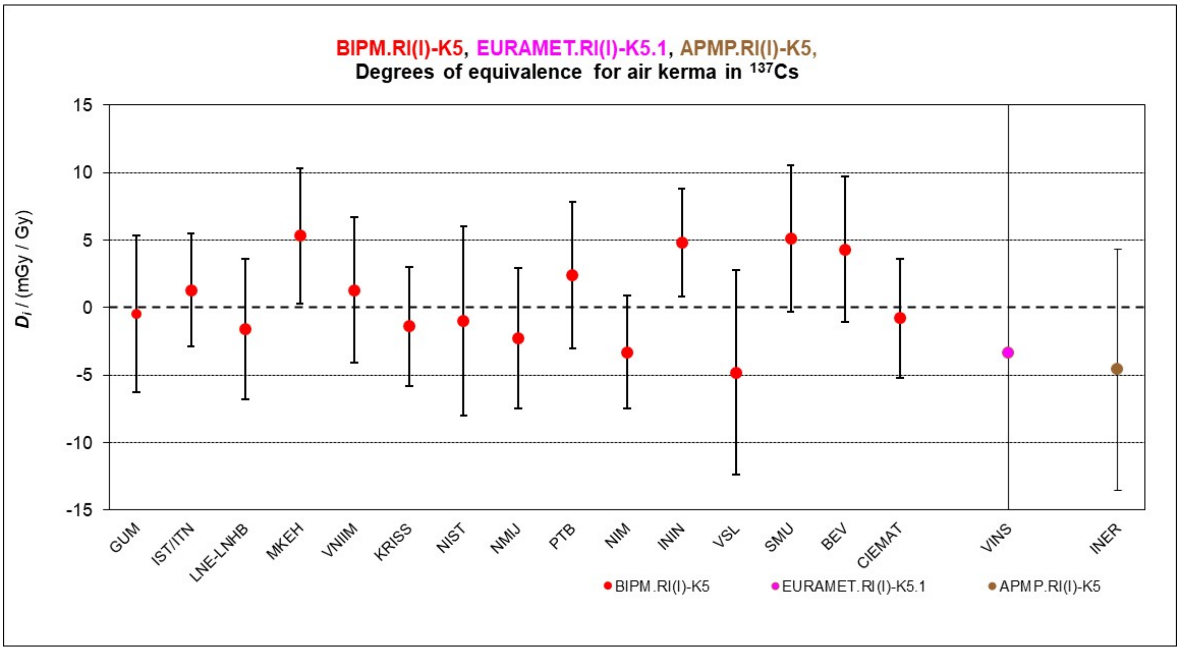
EURAMET.RI(I)-K5.1

Lab *i*	D_i	U_i
	/ (mGy/Gy)	
VINS	-3.3	20.2

APMP.RI(I)-K5

Lab *i*	D_i	U_i
	/ (mGy/Gy)	
INER	-4.6	8.9

Figure 1. Graph of degrees of equivalence with the KCRV



Note that the data presented in Table 10, while correct at the time of publication of the present report, become out-of-date as NMIs make new comparisons. In addition, revised validity rules for comparison data have been agreed by the CCRI(I) so that any results older than 15 years are no longer considered valid and have been removed from the KCDB. The formal results under the CIPM MRA are those available in the key comparison database.

7. Conclusion

The CIEMAT standard for air kerma in ^{137}Cs gamma radiation compared with the BIPM air-kerma standard gives a comparison result of 0.9992 (22). The indirect and direct comparison results are in agreement at the level of 2.2 parts in 10^3, which is within the standard uncertainty of the calibration procedure.

The CIEMAT is in agreement within the expanded uncertainty with all the NMIs having taken part in the BIPM.RI(I)-K5 ongoing key comparison for air-kerma standards in ^{137}Cs gamma-ray beams.

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