

International comparison of extrapolation chamber measurements of the absorbed dose rate in tissue for beta radiation

EUROMET project No. 739

BIPM KCDB: EUROMET.RI(I)-S2

Final Report

- R. Behrens, K. Helmstädter, P. Ambrosi,
Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100,
38116 Braunschweig, Germany
- J.-M. Bordy, C. Lecante,
Laboratoire National de métrologie et d'Essais, Laboratoire National Henri
Becquerel (LNE-LNHB), CEA/SACLAY, FR - 91191 Gif sur Yvette Cedex, France
- M. Bovi,
Istituto Nazionale di Metrologia delle Radiazioni Ionizzanti (ENEA-INMRI) CR
Casaccia (ENEA), Via Anguillarese 301. I-00060 SM Galeria (Roma), Italy
- I. Jokelainen,
Radiation and Nuclear Safety Authority (STUK), P.O. Box 14,
FI - 00881 Helsinki, Finland
- C. G. Soares,
National Institute of Standards & Technology (NIST), 100 Bureau Dr., Stop 8460
Gaithersburg, MD 20899-8460 U.S.A.
- P. R. B. Saull,
National Research Council of Canada (NRC), Ionizing Radiation Standards, 1200
Montreal Road, M-35, Ottawa, Canada, K1A 0R6
- I. A. Kharitonov, S. Fedina,
All-Russian Institute for Metrology (VNIIM), Ionizing Radiation Laboratory, 19
Moscovsky prosp., St. Petersburg, 198005, Russia
- T. Kurosawa, M. Kato,
National Metrology Institute of Japan (NMIJ), National Institute of Advanced
Industrial Science and Technology (AIST), 1-1-1 Umezono, Tsukuba, Ibaraki
305-8568, Japan

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ABSTRACT

For quality assurance in the realization and transfer by the national standard laboratories of the unit of the absorbed dose rate at 0.07 mm tissue depth for beta radiation, comparison measurements among the primary standard facilities are needed. Although some bilateral comparisons have taken place for this quantity, the CCRI decided in May 2003 that a EUROMET supplementary comparison would be appropriate if inclusive of primary standards laboratories of other regional metrology organizations. The operation and results of such a comparison are reported here. A flat ionization chamber and measurement system was used as the transfer instrument. A comparison was made of the calibration coefficients of this transfer instrument in various beta-particle laboratory reference fields (Pm-147, Kr-85, Tl-204, and Sr-90/Y-90) measured by each of the eight participants from France, Italy, Finland, Germany, Russian Federation, USA, Canada and Japan. The PTB was the pilot laboratory and the comparison ran from January 2004 until April 2007 under EUROMET project No. 739 and EUROMET.RI(I)-S2. The results for most of the participants are consistent with the stated uncertainties although an extreme deviation is apparent for Pm-147 beta radiation for one participant.

1 Introduction

In October 1999, national metrology laboratories worldwide signed the CIPM Mutual Recognition Arrangement (MRA: 'Arrangement on the mutual recognition of the equivalence of national standards and of calibration certificates issued by national metrology institutes') with the aim of establishing a basis for the mutual recognition of calibrations. In this context, the BIPM has published on its web pages a list of the Calibration and Measurement Capabilities (CMC-lists) of the institutes which have signed the CIPM MRA. Calibration services can, however, only be entered if a quality management system (for example one according to ISO standard 17025) has been established. Quality assurance and confidence in the capabilities of other laboratories is ensured if a laboratory has successfully taken part in a comparison in which the degree of equivalence with other national metrology institutes or calibration laboratories has been determined.

In recent years, a great change has taken place in the field of radiation protection dosimetry: the concept of radiation protection quantities, developed by ICRU between 1985 and 1993, has been adopted by the European Union and anchored in Council Directive 96/29/Euratom. For beta radiation, the personal dose equivalent $H_p(0.07)$ and the directional dose equivalent $H'(0.07)$ have been introduced as the operational quantities for individual and area monitoring. With the transposition of this directive into the national law of the EU member states, the national standard laboratories must be able to realize and disseminate this unit.

For quality assurance of the realization and transfer of the unit of the absorbed dose rate in 0.07 mm tissue depth for beta radiation, comparison measurements among the primary standard facilities are needed. This may be accomplished by the use of transfer ionization chambers. These devices are the basis for the measurement of the two operational quantities $H'(0.07)$ and $H_p(0.07)$ in the field of radiation protection.

Up to now, no comparisons for radiation protection quantities using beta radiation have been performed within the scope of the EUROMET or the CCRI. Some bilateral comparisons have taken place between the LPRI (LNE-LNHB) and PTB (1996), PTB and VNIIM (1999/2001), NIST and PTB (2001), and the LPRI (LNE-LNHB) and VNIIM (2001). Section I of CCRI decided at its meeting in May 2003 not to propose a worldwide key comparison of this kind under the auspices of the CCRI, but to support a EUROMET comparison, which is presented here.

A flat ionization chamber was used as the transfer instrument. Together with this chamber, a complete electronic measurement system was circulated. The aim of this comparison was to compare the calibration coefficients of the transfer instrument obtained by each participant.

Eight laboratories registered for the comparison: In 2003: LNE-LNHB (FR), ENEA-INMRI (IT), STUK (FI), PTB (DE), VNIIM (RU), NIST (US), NRC (CA); and in 2006: NMIJ (JP). The contact persons and the addresses of the partners are listed in Annex D. They each calibrated the comparison device according to their quality system and their beta-particle reference fields (one or more of Pm-147, Kr-85, Tl-204, and Sr-90/Y-90).

The comparison was coordinated by the PTB as pilot laboratory, and PTB also evaluated the results. The circulation of the transfer chamber and of the electronic

system was performed in a star pattern. Each participant returned the comparison equipment (chamber and measurement device) to the pilot laboratory for testing after the measurements were finished (see Table 1). The circulation scheme is shown in Figure 1.

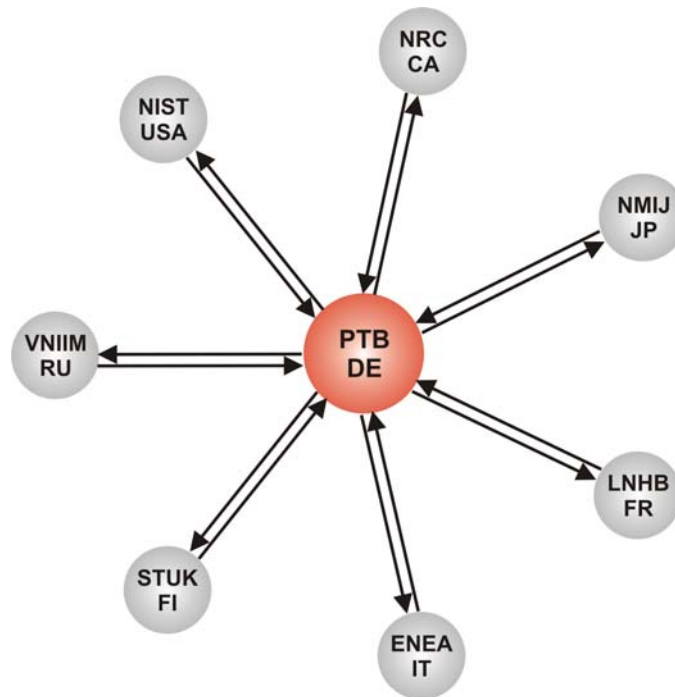


Figure 1: Circulation scheme of the comparison

The following time schedule, see Table 1, was agreed upon by the 7 partners. The start of the comparison was January 2004.

Table 1: Time schedule for the comparison

Participant	Calibration measurements at the participant's	Repeat measurements at PTB / DE	Transfer to the next participant or return to PTB	Report of the results from the participant
PTB / DE	01-03 / 2004		04 / 2004	
STUK / FI	04-05 / 2004		05 / 2004	06 / 2004
PTB / DE		06-07 / 2004	08 / 2004	
LNE-LNHB / FR	09 / 2004		09 / 2004	10 / 2004
PTB / DE		10 / 2004	10 / 2004	
NIST / US	11 - 12 / 2004		01 / 2005	02 / 2005
PTB / DE		02 / 2005	03 / 2005	
ENEA-INMRI / IT	03 - 04 / 2005		04 / 2005	07 / 2005
PTB / DE		04 / 2005	05 / 2005	
NRC / Canada	06 - 07 / 2005		07 / 2005	09 / 2005
PTB / DE		08 / 2005	09 / 2005	
VNIIM / RU	09 - 12 / 2005		12 / 2005	01 / 2006
PTB / DE		02-03 / 2006		
PTB / DE		02 / 2007	02 / 2007	
NMIJ / JP	03 / 2007		03 / 2007	04 / 2007
PTB / DE		04 / 2007		

Table 2: Measurement programme in the reference fields used by the participants

Participant	Reference fields of the radionuclides		
	¹⁴⁷ Pm	⁸⁵ Kr	⁹⁰ Sr/ ⁹⁰ Y
PTB	yes (0°, 45°, 60°)	yes (0°, 45°, 60°)	yes (0°, 45°, 60°)
STUK	no	no	yes (0°)
LNE-LNHB	yes (0°)	yes (0°)	yes (0°)
NIST	yes (0°, 45°, 60°)	yes (0°, 45°, 60°)	yes (0°, 45°, 60°)
ENEA-INMRI	yes (0°, 45°, 60°)	yes (0°, 45°, 60°)	yes (0°, 45°, 60°)
NRC	yes (0°)	yes (0°)	yes (0°)
NMIJ	yes (0°)	yes (0°)	yes (0°)
VNIIM	yes (0°)	yes (0°) ²⁰⁴ Tl	yes (0°)

The calibration coefficient N_n is defined as the quotient of the conventional true value of the quantity, $\dot{D}_t(0.07; \alpha)$, on the date of and for the ambient air conditions during the calibration measurements, and the indicated value of the ionization current, I_c , at the point of test and only corrected by the measurement software to the current at reference conditions in the collecting volume. It is expressed as

$$N_n = \frac{\dot{D}_t(0.07; \alpha)}{I_c},$$

where N_n is the calibration coefficient of the transfer chamber in mGy/(h A), measured by the partner n;

$\dot{D}_t(0.07; \alpha)$ is the conventional true dose rate at a depth of 0.07 mm in a tissue-equivalent slab phantom in mGy/h for the angle of incidence α , measured by means of an extrapolation chamber (primary standard) and corrected to the ambient air conditions during the calibration measurements;

I_c is the ionization current I in A, measured in the flat chamber at the positive chamber voltage at the ambient air conditions during the calibration measurements, corrected by the measurement software to the current at reference conditions in the collecting volume.

The ISO standards ISO 6980-2:2004 [1], ISO 6980-1:2006 [2], and ISO 6980-3:2006 [3] were used as a guide for the calculation of the conventional true dose rate, $\dot{D}_t(0.07; \alpha)$.

The values of the half-lives that have been used are those of the ISO 6980-2:2004 [1] and the PTB-report PTB-Ra-16/5 (2000) [4]:

$$\begin{aligned} &(958.2 \pm 8) \text{ days for Pm-147} \\ &(1381 \pm 8) \text{ days for Tl-204} \\ &(3915 \pm 3) \text{ days for Kr-85} \\ &(10523 \pm 35) \text{ days for Sr-90/Y-90} \end{aligned}$$

The comparison reference value x_R was determined for each beta-particle reference field (Pm-147, Kr-85 or Tl-204, and Sr-90/Y-90) as the weighted mean of the calibration coefficients N_n of the participants taking the correlations between the participants into account, see Annex A. These reference values x_R were used to determine the degree of equivalence for each participating laboratory.

4 Measurement system for the international comparison

4.1 Overview

The calibration measurements for the comparison were carried out using the transfer chamber and the complete electronic measurement device, sent together in a transport box. The transport box contained two further boxes: a black case containing the transfer chamber, and a blue box containing the complete electronic measurement system, see Figure 2.



Figure 2: The two transport boxes: the black one containing the transfer chamber, the blue one the electronic measurement system

4.2 Transfer chamber

As transfer instrument, a flat ionization chamber was used, see Figure 3. The chamber consists of a measurement part with a backscatter plate and a rod, fastened to each other. The main material of the chamber is polyethylene terephthalate (PET).

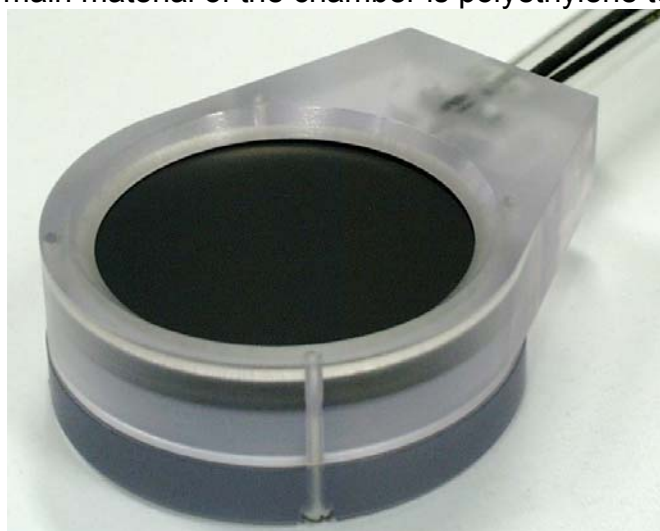


Figure 3: Transfer ionization chamber; view of the chamber's front face

The chamber has the geometry of a circular slab. A sketch of the flat ionization chamber is shown in Figure 4. The outside dimensions are 90 mm in diameter, with a total thickness of 40 mm. The active volume is about 8 cm³. In front of the active volume, an entrance window, consisting of a polycarbonate foil (7.24 mg/cm² tissue equivalence), is fixed. During calibrations, the front face of the chamber had to be irradiated completely.

The electric field for separating the electrons and the ion charges is produced by two electrodes arranged in parallel. The HV electrode in front of the active volume was set to a potential of +100 V. The electrode at the back was set to zero potential.

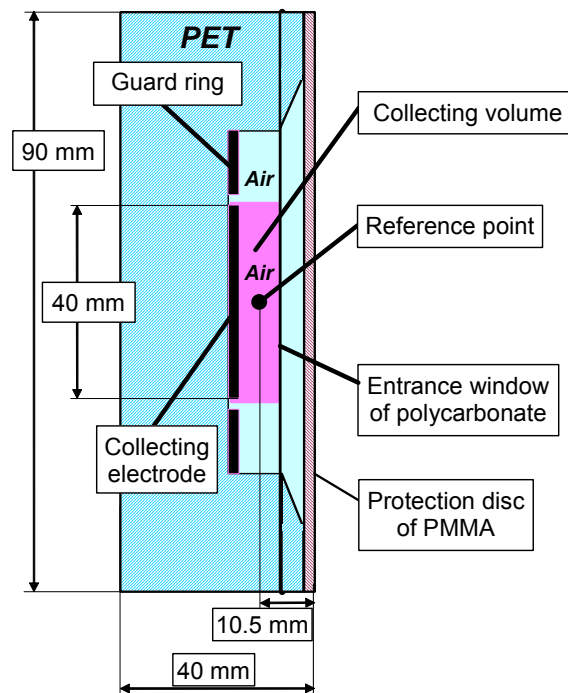


Figure 4: Sketch of the flat ionization chamber

The **reference point** of the chamber lies inside the active volume, 10.5 mm below the front face of the protection disc which is made of polymethyl methacrylate, PMMA (see Figure 4). The reference point of the chamber had to be positioned at the point of test, for which the conventional true value of $\dot{D}_t(0.07)$ in a tissue-equivalent slab phantom is known.

At a beam incidence of 0° , the radiation axis had to be normal with respect to the front face of the chamber. In addition, the radiation axis must pass through the reference point.

The chamber had to be calibrated at different angles of incidence. For the rotation of the chamber, the rotary axis had to pass through the reference point of the chamber.

The active volume of the chamber is rotationally symmetric with respect to an axis which is normal with respect to the front face of the chamber. This axis passes through the reference point of the chamber. However, small deviations both from symmetry (as, for example, small differences in the thickness of the entrance window and graphite layers of the chamber) and from the adjustment of the angle of incidence, α , can lead to different measurement results, depending on the beam incident angle $+\alpha$ or $-\alpha$. To reduce this influence, the calibration measurements were to be performed for the radiation qualities at the angles $+\alpha$ and $-\alpha$, i.e. with the beam incident on both sides of the normal on the chamber's front surface. The mean value from the two measurements was to be used for determining the calibration coefficients for the respective radiation quality. The chamber was deemed to be correctly adjusted to the beam axis if the calibration coefficients determined for Kr-85 at $+45^\circ$ and -45° agreed within $\pm 1.2\%$ with the mean value.

4.3 Electronic system

The calibration measurements were carried out using the complete electronic measurement device supplied together with the transfer chamber, see Figure 5. It consists of an electrometer, a high-voltage power supply unit, a time controlling device, a computer interface, a temperature, pressure and relative humidity sensor and, for automatic data recording, a laptop equipped with measurement software written by the pilot laboratory. The electrometer consists of an integrator module with an integrator amplifier that has an input bias current of less than 3 fA. Six charge measuring ranges from 20 pC to 2 μ C are available [5].

The current measured by the chamber under ambient conditions was corrected by the measurement software to the current for reference air conditions in the collecting volume (pressure: 101.3 kPa, temperature: 293.15 K).

The measurement result furnished by the software was the value of the mean corrected current of the chamber, adjusted for leakage, along with its standard uncertainty.

During the comparison, it was found that the correction regarding the relative humidity was not being carried out by the software. Thus, after the measurements, all the values were corrected to 65% relative humidity by the participants, in addition to the correction already made by the software.



Figure 5: Transfer chamber and electronic measurement device

The stability of the complete measurement system was checked by the pilot laboratory after each participant completed his measurements. The results are given in Annexes B and C.

5 Description of the beta-particle reference fields

Figure 6 shows a sketch of the irradiation arrangement of the transfer chamber for the calibration measurements.

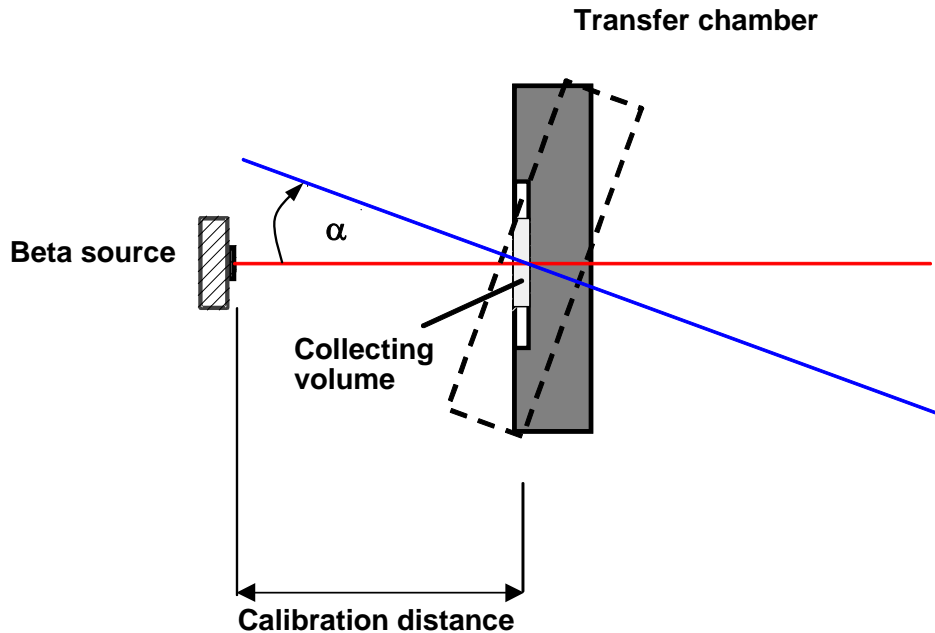


Figure 6: Experimental set-up for the calibration measurements

The beta-particle reference fields of the participants are based

- on the Beta Secondary Standard BSS 1 for one partner, see Table 4,
- on area beta sources in a custom-made irradiation facility for one partner, see Table 5,
- on the BSS 2 sources in a custom-made irradiation facility for one partner (ENEA-INMRI), see Table 6, and
- on the Beta Secondary Standard BSS 2 for five partners, see Table 6.

Table 4: Beta-particle reference fields of the BSS 1 at STUK

Parameters	Specifications of the parameters
Radionuclide / Nominal activity	$^{90}\text{Sr}/^{90}\text{Y}$ / 74 MBq (1981)
Source type	M238Sr07
Source number: STUK	29
Approximate density of source window	$(50 \pm 5) \text{ mg cm}^{-2}$ silver
Filter material	Polyethylene terephthalate (PET)
Dimensions of the beam flattening filter mounted at 10 cm distance from the source	3 concentric circular foils 190 μm thick, 2 cm, 3 cm and 5 cm in radius
Mean beta energy	0.8 MeV
Calibration distance y_0	30 cm

Table 5: Data for the VNIIM reference beta-particle radiation fields

Radionuclide	Source type	Protective material and its thickness	Active area cm^2	Nominal activity GBq	Calibration distance cm
$^{90}\text{Sr}/^{90}\text{Y}$	BIS-50	0.1 mm stainless steel	19.6	0.38 (1969)	30
^{204}Tl	BIT-40	0.2 mm Al-alloy AB	12.6	11 (1987)	20
^{147}Pm	BIP-50	0.0015 mm titanium dioxide	19.6	11 (2004)	20

Table 6: Beta-particle reference fields of the BSS 2 (sources and irradiation facility) at PTB, LNE-LNHB, NIST, NRC, and NMIJ, and beta-particle reference fields (only BSS 2 sources) at ENEA-INMRI.

Parameters	Specifications of the parameters		
	¹⁴⁷ Pm	⁸⁵ Kr	⁹⁰ Sr/ ⁹⁰ Y
Radionuclide			
Source type	AEAT PHRB4809, PHFB1028	AEAT KARB4810	AEAT SIRB4568
Source number:			
PTB	KB 467	KB 397	KB 437
LNHB	KB 468	KB 398	KB 438
NIST	FU 989	5828 BX	FU 991
NRC	LE 493	LB 232	KB 441
ENEA-INMRI	MR 424	MH 984	MR 429
NMIJ	MR 423	MH 983	MR 428
Nominal activity	3.7 GBq	3.7 GBq	460 MBq
PTB	2001	2001	2001
LNE-LNHB	2001	2001	2001
NIST	1998	1998	1998
NRC	2003	2003	2003
ENEA-INMRI	2003	2003	2003
NMIJ	2004	2004	2004
Areal mass of source window	(2.22 ± 0.5) mg cm ⁻² titanium	(22.5 ± 1.0) mg cm ⁻² titanium	(79 ± 8) mg cm ⁻² stainless steel
Filter material	Polyethylene terephthalate		
Dimensions of the beam flattening filters mounted at 10 cm distance from the source	1 circular foil 5 cm in radius and 100 µm thick with one hole, 0.975 cm in radius, in the centre	1 circular foil 4 cm in radius and 50 µm thick, and 1 foil 2.75 cm in radius and 190 µm thick	3 concentric circular foils 190 µm thick, 2 cm, 3 cm and 5 cm in radius
Mean beta energy	0.06 MeV	0.24 MeV	0.8 MeV
Calibration distance y ₀	20 cm	30 cm	30 cm

6 Primary Standards for realizing the unit of the absorbed dose rate to tissue for beta radiation at the participants' laboratory

The primary standards for realizing the unit of absorbed dose rate to tissue for beta radiation used by the participating laboratories are based on extrapolation chamber measurements in beta-particle reference fields.

Five participants used an extrapolation chamber developed at PTB and produced by PTW Freiburg.

Two participants used extrapolation chambers constructed at their labs.

In Table 7, the main characteristics of the beta primary standard measurement device used by each participant for the comparison measurements are summarized.

Differences exist in the

- thickness of the entrance window,
- conducting layer material of the entrance window (graphite or aluminium),
- use of an additional absorber for realizing the reference thickness of 0.07 mm tissue,
- area of the collecting electrode,
- range of chamber depths used, and the
- charge measurement system employed.

Table 7: Main characteristics of the beta primary standard measurement device used by the participants for the comparison measurements (nominal values)

Parameter	PTB	NIST	LNE-LNHB	ENE-INMRI	STUK	NRC	VNIIM	NMIJ
Entrance window:								
Material	Graphited PET	Graphited PET	Graphited PET	Graphited PET	Graphited PET	Graphited PET	Aluminized PET	Aluminized PET
Mass per area in mg/cm ²	0.622	0.66	0.79	2.6	2.61	0.95 ± 0.1	1.433	1.67
Equivalent tissue depth d_{win} in mm	0.0057	0.0062	0.0072	0.024	0.024	0.0087	0.0132	0.0152
Additional absorber:								
Material	PET	PET	PET	none	none	none	PET	PET
Mass per area in mg/cm ²	6.963	6.9	6.835				6.749	5.41
Equivalent tissue depth d_{win} in mm	0.0654	0.0644	0.0638				0.0630	0.0493
Collector:								
Material	PMMA	PMMA	PMMA	PMMA	PMMA	PMMA	PMMA	PMMA
Thickness in mm	31	31	31	31	31	31	25	20
Diameter in mm	30.37	30.24	30.32	30.24	30.23	30.05	40.01	30.05
Area in cm ² of collecting electrode used	7.25	7.18	7.22	7.18	7.177	7.09	12.58	7.33
Guard ring width in mm	15	15	15	15	15	15	40	15
Width / depth of insulation gap in mm	0.2 / 0.2		0.2 / 0.2		0.2 / 0.2	0.3 / 0.3	0.2 / 0.2	0.5 / 20
Range of chamber depth for extrapolation in mm	0.25 to 2.5	0.25 to 2.5	0.5 to 2.0	0.25 to 2.5	0.5 to 2.5	0.25 to 2.5	0.3 to 0.7	0.5 to 2.5
Chamber voltage applied in V	± 2.5 to ± 25	± 2.5 to ± 25	± 5 to ± 20	± 2.5 to ± 25	± 25 to ± 125	± 2.5 to ± 25	30	± 5 to ± 25
Electric field strength V/mm	10	10	10	10	50	10	43 to 100	10
Charge measurement system:	Keithley 642	Keithley 642	Keithley 642	Keithley 6517A	Keithley 6517A	Keithley 642	Keithley 6517A	TR8411
Input impedance in TΩ				200				
Bias current in fA	< 1			< 3		< 1	< 10	< 1
Standard feedback capacitor, C in pF	20	100	536	500	20	20	20	100

PET: Polyethylene terephthalate
 PMMA: Polymethyl methacrylate

7 Calculation of the absorbed dose rate to tissue at a depth of 0.07 mm

The procedure used by all participants to calculate the conventionally true value of absorbed dose rate to tissue at a depth of 0.07 mm in a tissue-equivalent slab phantom in a beta-particle field for reference conditions, $\dot{D}_t(0.07; \alpha)_{ref}$, follows closely that of reference ISO 6980-2:2004 [1].

The following equation was used:

$$\dot{D}_t = \left(\frac{d}{dI}(kI) \right)_{I=0} \cdot k' \cdot \left(\frac{\overline{W}_0}{e} \right) \cdot \frac{S_{t,a}}{a \cdot \rho_{ao}}, \quad (1)$$

where

I is the ionization current;

k is the product of the correction factors [1] which are dependent on the chamber depth, including the following correction factors:

k_{ac} for attenuation of beta-particles in the collecting volume

k_{ad} for air density in the collecting volume

k_{abs} for attenuation and scattering of beta-particles between the source and the collecting volume

k_{de} for radioactive decay

k_{di} for axial non-uniformity

k_{pe} for perturbation of the beta-particle flux density by the side walls of the extrapolation chamber

k_{sat} for ionization collection losses due to ion recombination

k' is the product of correction factors [1] which are independent of the chamber depth, including the following correction factors:

k_{ba} for backscatter from the collecting electrode and guard ring

k_{br} for bremsstrahlung from the beta-particle source

k_{el} for electrostatic attraction of the entrance window

k_{hu} for variations of \overline{W}_0 for relative humidity

k_{in} for interface effects

k_{ra} for radial non-uniformity

$\left(\frac{d(kI)}{dI} \right)_{I=0}$ is the limiting value of the slope of the corrected current versus the chamber depth function;

\overline{W}_0/e is the quotient of the mean energy required to produce an ion pair in air under reference conditions and the elementary charge, e ;

$S_{t,a}$ is the quotient of mass electronic stopping powers of ICRU tissue and air;

ρ_{ao} is the air density under reference conditions; and

a is the effective area of the collecting electrode.

All participants used this formula for the calculation of the absorbed dose rates in their reference fields (see the fourth column of Table 8).

The standard uncertainties for $k = 1$ were evaluated according to the GUM [6,7] (see fifth column in Table 8).

8 Measurement results

In Table 8, the data of the calibration measurements carried out by the participants for normal incidence are summarized. The calibration coefficients N_i and their expanded standard uncertainties reported by the participants, $U_{i,\text{part}} (k = 2)$, are listed in the last two columns. The figures are rounded according to their uncertainty: In case the first significant digit of the uncertainty is 3 or larger, this digit is the one to be rounded off, in case the first significant digit of the uncertainty is 1 or 2, the digit to the right is the one to be rounded off. This rounding position was applied for both the value and the uncertainty.

For Pm-147, one value for the calibration coefficient N is very high compared with all the other results and has been excluded from the calculation of the weighted mean value as the respective participant (ENEA-INMRI) indicated that this measurement was performed at a wrong distance (as specified in Annex A). Details of the reference values and the degrees of equivalence are given in Annex A.

The repeated measurements made at PTB after each return of the comparison equipment show the reproducibility of such measurements and the higher discrepancies, particularly for Pm-147 beta radiation (see Annex B).

Only three participants carried out additional measurements in the beta-particle reference fields for angles of incidence of 45 ° and 60°. The calibration coefficients and the expanded standard uncertainties ($k = 2$) are summarized in Table 9.

Table 8: Data of the calibration measurements carried out in the beta-particle reference fields for an angle of incidence of 0°

Partner	Radionuclide * with filter ** no filter	Calibra- tion distance m	Dose		Chamber		Calibration	
			rate	Uncert. $k = 1$	current	Uncert. $k = 1$	coefficient N	Uncert. $k = 2$
			mGy/h		fA		10 ¹³ mGy/(h A)	
PTB	Pm-147 *	0.2	4.85	0.06	299.5	0.9	1.621	0.037
LNE-LNHB	Pm-147 *	0.2	4.50	0.06	274.2	1.3	1.641	0.045
NIST	Pm-147 *	0.2	1.91	0.05	114.8	0.5	1.663	0.086
ENEA-INMRI	Pm-147 *	0.2	11.09	0.18	594.5	1.7	1.864	0.065
NRC	Pm-147 *	0.2	5.91	0.21	346.1	0.9	1.707	0.123
VNIIM	Pm-147 **	0.2	40.0	0.5	2307	14	1.736	0.059
NMIJ	Pm-147 *	0.2	5.49	0.12	329.3	1.1	1.668	0.074
PTB	Kr-85 *	0.3	127.4	1.3	8287	15	1.537	0.031
LNE-LNHB	Kr-85 *	0.3	134.3	1.2	8789	16	1.528	0.029
NIST	Kr-85 *	0.3	117.3	2.0	7631	10	1.538	0.053
ENEA-INMRI	Kr-85 *	0.3	142.6	1.7	9404	18	1.517	0.038
NRC	Kr-85 *	0.3	138.3	1.2	9112	10	1.517	0.027
VNIIM	Tl-204 **	0.2	14.39	0.17	988	6	1.457	0.044
NMIJ	Kr-85 *	0.3	130.0	1.6	8548	16	1.521	0.037
PTB	Sr-90/Y-90 *	0.3	31.7	0.3	2267	4	1.399	0.029
LNE-LNHB	Sr-90/Y-90 *	0.3	32.02	0.29	2296	4	1.395	0.025
NIST	Sr-90/Y-90 *	0.3	31.5	0.6	2244.3	2.5	1.402	0.050
ENEA-INMRI	Sr-90/Y-90 *	0.3	44.7	0.5	3287	7	1.359	0.035
NRC	Sr-90/Y-90 *	0.3	29.80	0.26	2160.5	2.9	1.379	0.025
VNIIM	Sr-90/Y-90 **	0.3	28.1	0.3	1961	12	1.431	0.042
NMIJ	Sr-90/Y-90 *	0.3	40.0	0.5	2877	5	1.391	0.033
STUK	Sr-90/Y-90 *	0.3	3.86	0.06	281.0	0.6	1.375	0.043
PTB	Sr-90/Y-90 **	0.3	49.9	0.5	3549	6	1.406	0.027
LNE-LNHB	Sr-90/Y-90 **	0.3	50.1	0.5	3597	7	1.393	0.026

Table 9: Data of the calibration measurements carried out in the beta-particle reference fields for different angles of incidence

Radio-nuclide	Angle of incidence	Calibration distance m	Participant	Dose rate mGy/h	Uncert. $k = 1$	Chamber current fA	Uncert. $k = 1$	Calibration coefficient N 10^{13} mGy/(h A)	Uncert. $k = 2$
Pm-147	0°	0.2	PTB	4.85	0.06	299.5	0.9	1.621	0.037
			NIST	1.91	0.05	114.8	0.5	1.663	0.086
			ENEA-INMRI	11.09	0.18	594.5	1.7	1.864	0.065
Pm-147	45°	0.2	PTB	3.55	0.05	219.7	0.7	1.617	0.044
			NIST	1.37	0.07	82.1	0.3	1.670	0.159
			ENEA-INMRI	7.95	0.27	415.9	2.2	1.911	0.140
Pm-147	60°	0.2	PTB	2.61	0.04	161.9	0.4	1.617	0.049
			NIST	1.01	0.05	60.4	0.3	1.672	0.16
			ENEA-INMRI	5.88	0.20	296.4	1.6	1.983	0.140
Kr-85	0°	0.3	PTB	127.4	1.3	8287	15	1.537	0.031
			NIST	117.3	2.0	7631	10	1.538	0.053
			ENEA-INMRI	142.6	1.7	9404	18	1.517	0.038
Kr-85	45°	0.3	PTB	112.1	1.1	6870	12	1.632	0.033
			NIST	103	4	6310	6	1.627	0.112
			ENEA-INMRI	126	4	7839	27	1.607	0.110
Kr-85	60°	0.3	PTB	91.7	0.9	5304	10	1.730	0.035
			NIST	84.2	2.9	4841	5	1.738	0.120
			ENEA-INMRI	103	3	5959	21	1.727	0.115
Sr-90/ Y-90	0°	0.3	PTB	31.7	0.3	2267	4	1.399	0.029
			NIST	31.5	0.6	2244.3	2.5	1.402	0.050
			ENEA-INMRI	44.7	0.5	3287	7	1.359	0.035
Sr-90/ Y-90	45°	0.3	PTB	35.5	0.4	2363	4	1.503	0.030
			NIST	35.2	1.0	2332.8	2.2	1.508	0.081
			ENEA-INMRI	50.5	1.6	3437	12	1.468	0.097
Sr-90/ Y-90	60°	0.3	PTB	36.2	0.4	2117	4	1.709	0.034
			NIST	35.8	1.0	2076.1	2.0	1.724	0.093
			ENEA-INMRI	51.9	1.7	3058	11	1.696	0.110

9 Conclusion

The EUROMET supplementary comparison of the absorbed dose rate in tissue for beta radiation (EUROMET project No. 739 and BIPM KCDB: EUROMET.RI(I)-S2) was performed successfully. The results of most of the participants are consistent within the scope of the assigned uncertainties. An extreme deviation is apparent for Pm-147 beta radiation for one participant: the calibration coefficient is 12 % higher than the reference value. In the meantime, the participant has indicated a physical reason for this, see note 2 to Table A.1.

The reproducibility of the measurements carried out with the transfer chamber and the electronic measurement device was established through repeated measurements made at PTB over the course of the comparison. The results of these measurements are taken into account in the evaluation of the comparison in terms of degrees of equivalence.

10 Acknowledgements

The authors would like to thank G. Buchholz and G. Winterbottom from PTB for developing the transfer chamber and the electronic measurement device as well as B. Koplín from PTB for the extensive calibration measurements at PTB and the logistical tasks for the shipment of the comparison equipment to the partners. In addition the authors would like to thank C. Elster from PTB for his very helpful support to take the correlations of quantities into account for the evaluation of the reference values, the degrees of equivalence and their uncertainties, see Annex A.

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Annex A
Introductory text and degrees of equivalence for the calibration coefficient of the transfer chamber for the measurand “absorbed dose rate” in a tissue-equivalent slab phantom at a depth of 0.07 mm

EUROMET comparison:

- EUROMET project No. 739; CCRI Ref No: EUROMET.RI(I)-S2

Measurand: Calibration coefficient of a transfer chamber in terms of the absorbed dose rate in tissue at a depth of 0.07 mm.

Table A.1: Results of the participants and reference values x_R that were determined from statistically consistent results, see text.

Lab i	Radio-nuclide	Calibration coefficient x_i 10^{13} mGy/(h A)	Expanded uncertainty $U_{i,part} (k = 2)$	Reference value x_R ¹⁾ 10^{13} mGy/(h A)	Expanded uncertainty $U_R (k = 2)$
PTB LNE-LNHB NIST ENEA-INMRI ²⁾ NRC VNIIM NMIJ	Pm-147	1.621 1.641 1.663 1.864 ^{*)} 1.707 1.736 1.668	0.037 0.045 0.086 0.065 ^{*)} 0.123 0.059 0.074	1.659	0.042
PTB LNE-LNHB NIST ENEA-INMRI NRC VNMI ³⁾ NMIJ	Kr-85	1.537 1.528 1.538 1.517 1.517 1.457 ^{*)} 1.521	0.031 0.029 0.053 0.038 0.027 0.044 ^{*)} 0.037	1.524	0.027
PTB LNE-LNHB NIST ENEA-INMRI NRC VNIIM NMIJ STUK	Sr-90/Y-90	1.399 1.395 1.402 1.359 1.379 1.431 ^{*)} 1.391 1.375	0.029 0.025 0.050 0.035 0.025 0.042 ^{*)} 0.033 0.043	1.386	0.024

Note 1): Details on the weighted mean values and their uncertainties are given in the text.

Note 2): The participating laboratory ENEA-INMRI has explained - after having been informed about the large deviation of more than 10% for the Pm-147 results – that they have thoroughly checked the precision of their measurement system. During this check it turned out that their alignment system worked incorrectly at source-detector distances (SSD) below approximately 25 cm. Measurements with Pm-147 were made at 20 cm SDD and were consequently wrong. The positioning system has now been repaired and a new bilateral or multilateral comparison for beta measurements with Pm-147 (at 20 cm SDD) is planned.

Note 3): At VNIIM, a TI-204 source was used instead of Kr-85. According to the ISO 6980 series, this is not expected to make any differences.

*)): These values are not statistically consistent with the others and thus are not used for the x_R .

Contents of Table A.1:

- The calibration coefficients x_i are the values, N_i , reported by the participants. The notation has been changed to match that commonly used in the KCDB and to avoid any confusion over the use of N as the number of participants.

- The reference value x_R is determined for each radiation quality separately:

1) To obtain the reference values x_R for the different radiation qualities, the corrected uncertainties $u_{i,\text{corr}} = \sqrt{u_{i,\text{part}}^2 + (v_{\text{rep.meas.}})^2}$ have to be calculated. They are corrected for the contribution of the transfer chambers instability to the uncertainty. The $u_{i,\text{corr}}$ result from the uncertainties reported by the participants, $u_{i,\text{part}}$, and the standard deviation of the repeated measurements performed at PTB, $v_{\text{rep.meas.}}$, see Annex B. The second term had to be added as the fluctuations observed during the repeated measurements are significant. Mathematically, the model function $x_i = x_{i,\text{part}} \cdot k$ is applied with $x_{i,\text{part}}$ the value reported and its uncertainty, $u_{i,\text{part}}$, and $k = 1.0$ and its uncertainty, $v_{\text{rep.meas.}}$, taking account of the fluctuations observed over the time of the comparison, see Annex B. Due to $k = 1.0$, it is $x_i = x_{i,\text{part}}$.

2) The covariance matrix, \mathbf{C} , describing the correlations between the participants is determined using the fact that all participants used equation (1) in section 7. Thus, some of the input quantities are equivalent for all participants: \overline{W}_0/e , $s_{t,a}$, ρ_{a0} , k_{hu} , k_{ba} , and k_{ra} for all three radiation qualities and in addition k_{br} for Kr-85 and Sr-90/Y-90.

Let q_l with $l = 1..L$ be the equivalent input quantities, then it follows

$$\text{for } i \neq j: [\mathbf{C}]_{ij} = u(x_i, x_j) = \sum_{l=1}^L \frac{\partial x_i}{\partial q_l} \cdot \frac{\partial x_j}{\partial q_l} \cdot u^2(q_l) = x_i \cdot x_j \cdot \sum_{l=1}^L (u_{\text{rel}}(q_l))^2 \quad \text{and}$$

for $i = j$ it is $[\mathbf{C}]_{ii} = u_{\text{corr}}^2(x_i) = u_{i,\text{corr}}^2$. The $u_{\text{rel}}(q_l)$ are the relative uncertainties for \overline{W}_0/e , $s_{t,a}$, etc.

3) The general least squares fit as a candidate for the reference value is

$$\tilde{x}_R = \left(\sum_{i=1}^N x_i \cdot \sum_{j=1}^N [\mathbf{C}^{-1}]_{ij} \right) / \left(\sum_{i=1}^N \sum_{j=1}^N [\mathbf{C}^{-1}]_{ij} \right), \quad \text{see equation (3) in Douglas and$$

Steele [8], with N the number of participants considered and \mathbf{C}^{-1} the inverse of the covariance matrix.

4) The relevant chi-square $\chi_R^2 = (N-1)^{-1} \cdot \sum_{i=1}^N \sum_{j=1}^N (x_i - \tilde{x}_R) \cdot [\mathbf{C}^{-1}]_{ij} \cdot (x_j - \tilde{x}_R)$

results from equation (4) in Douglas and Steele [8].

5) The probability that all N results are statistically consistent within the frame of their uncertainties is $p_{\text{cons}} = p(\chi^2(\nu) > \chi_R^2)$: „Probability that the value of a chi-square function with $\nu = N-1$ degrees of freedom is larger than χ_R^2 .”

When this probability is below 5 %, the values are regarded as not consistent. To find a consistent subset of $N-1$ participants, one value x_i is omitted and the corresponding χ_R^2 is determined. This is done omitting the

result of each participant one after the other. N such different subsets are available. In case more than one of these subsets has got a probability $p_{\text{cons}} > 5\%$, the subset with the largest p_{cons} is chosen.

In case none of the N subsets had $p_{\text{cons}} > 5\%$, the procedure would be applied for $N-2$, $N-3$, etc. participants until a subset with $p_{\text{cons}} > 5\%$ is found. This procedure is described in detail in sections 5.1 and 7.7 in Cox [9]. In this comparison, always a subset of $N-1$ participants has $p_{\text{cons}} > 5\%$.

- 6) The consistent subsets of this comparison have $N_{\text{cons}} = 6$ for Pm-147 and Kr-85 and $N_{\text{cons}} = 7$ for Sr-90/Y-90, see ^{*} in Table A.1, and the reference

$$\text{values result to } x_R = \left(\sum_{i=1}^{N_{\text{cons}}} x_i \cdot \sum_{j=1}^{N_{\text{cons}}} [\mathbf{C}^{-1}]_{ij} \right) / \left(\sum_{i=1}^{N_{\text{cons}}} \sum_{j=1}^{N_{\text{cons}}} [\mathbf{C}^{-1}]_{ij} \right).$$

- The expanded uncertainty U_R of the reference value x_R is determined from

$$U_R = 2 / \left(\sum_{i=1}^{N_{\text{cons}}} \sum_{j=1}^{N_{\text{cons}}} [\mathbf{C}^{-1}]_{ij} \right)^{1/2}, \text{ see equation (7) in Elster and Link [10], with } N_{\text{cons}}$$

the number of participants forming the consistent subset as described above.

In Figure A.1 and Table A.2 (first three columns), the degree of equivalence of each laboratory i relative to the reference value, x_R , is given by a pair of terms:

$D_i = (x_i - x_R)$ and U_i , its expanded uncertainty ($k = 2$), where $U_i = 2 \cdot u_i = 2 \cdot (u_{i,\text{corr}}^2 - u_R^2)^{1/2}$ in the case where x_i was used to determine x_R (x_i and x_R are directly correlated) and $U_i = 2 \cdot u_i = 2 \cdot (u_{i,\text{corr}}^2 + u_R^2 - r_{iR} \cdot u_{i,\text{corr}} \cdot u_R)^{1/2}$, where x_i was not used to determine x_R (x_i and x_R are only correlated via equation (1) in

section 7). The correlation matrix is $r_{iR} = x_i \cdot x_R \cdot \sum_{l=1}^L (u_{\text{rel}}(q_l))^2 / (u_{i,\text{corr}} \cdot u_R)$; the

$u_{\text{rel}}(q_l)$ are explained above in number 1).

The degree of equivalence between two laboratories i and j is given as well in Table A.2 (further columns) by a pair of terms relative to the mean value of the two values, $x_{ij} = (x_i + x_j) / 2$:

$D_{ij} = D_i - D_j = (x_i - x_j)$ and U_{ij} , its expanded uncertainty ($k = 2$), with $U_{ij} = 2 \cdot (u_{i,\text{corr}}^2 + u_{j,\text{corr}}^2 - r_{ij} \cdot u_{i,\text{corr}} \cdot u_{j,\text{corr}})^{1/2}$ with the correlation matrix $r_{ij} = [\mathbf{C}]_{ij} / (u_{i,\text{corr}} \cdot u_{j,\text{corr}})$ and with \mathbf{C} the covariance matrix, see above.

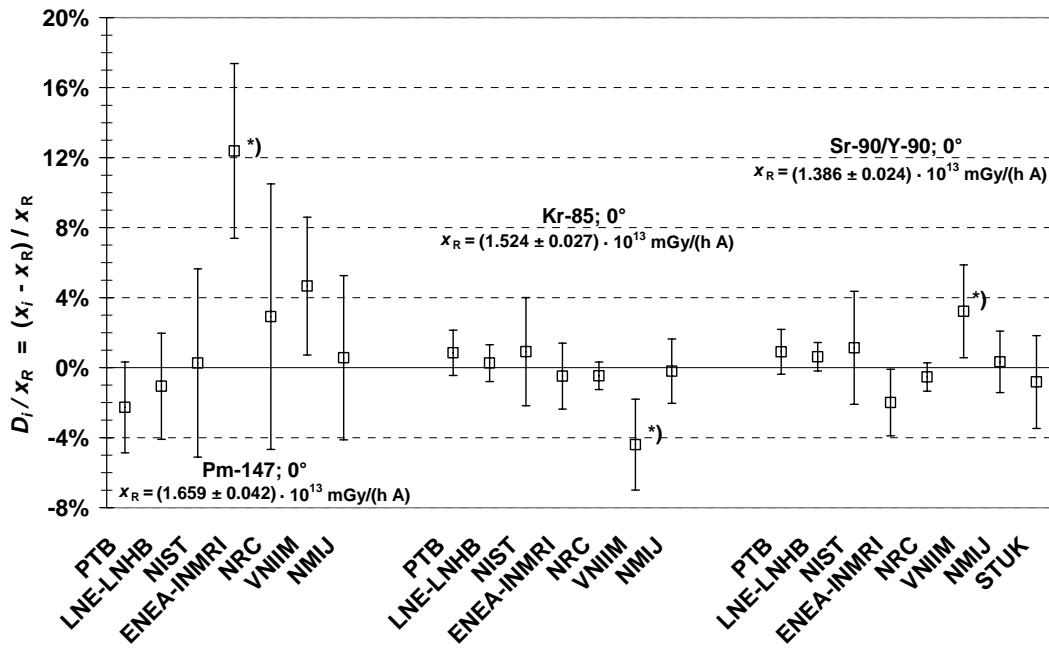


Figure A.1:

Degree of equivalence D_i of each laboratory i relative to the reference value x_R .

*) These values were not taken into account for x_R , see text explaining Table A.1.

Table A.2: Degree of equivalence D_i of each laboratory i with respect to the reference value x_R (second and third column) and D_{ij} between two laboratories i and j (the other columns). All values are given in terms of 10^{-2} .

Lab i ↓	Lab j ⇒																	
	D_i/x_R U_i/x_R		PTB D_{ij}/x_R U_{ij}/x_R		LNE-LNHB D_{ij}/x_R U_{ij}/x_R		NIST D_{ij}/x_R U_{ij}/x_R		ENEA D_{ij}/x_R U_{ij}/x_R		NRC D_{ij}/x_R U_{ij}/x_R		VNIIM D_{ij}/x_R U_{ij}/x_R		NMIJ D_{ij}/x_R U_{ij}/x_R		STUK D_{ij}/x_R U_{ij}/x_R	
Pm-147																		
PTB	-2	3	---	---	-1	5	-3	6	-15	6	-5	8	-7	5	-3	6	---	---
LNE-LNHB	-1	3	1	5	---	---	-1	7	-13	6	-4	9	-6	6	-2	6	---	---
NIST	0	5	3	6	1	7	---	---	-12	7	-3	10	-4	7	0	8	---	---
ENEA-INMRI *)	12	5	15	6	13	6	12	7	---	---	9	9	8	6	12	7	---	---
NRC	3	8	5	8	4	9	3	10	-9	9	---	---	-2	9	2	9	---	---
VNIIM	5	4	7	5	6	6	4	7	-8	6	2	9	---	---	4	7	---	---
NMIJ	1	5	3	6	2	6	0	8	-12	7	-2	9	-4	7	---	---	---	---
Kr-85																		
PTB	1	1	---	---	1	2	0	3	1	2	1	2	5	3	1	2	---	---
LNE-LNHB	0	1	-1	2	---	---	-1	3	1	2	1	2	5	3	0	2	---	---
NIST	1	3	0	3	1	3	---	---	1	4	1	3	5	4	1	4	---	---
ENEA-INMRI	0	2	-1	2	-1	2	-1	4	---	---	0	2	4	3	0	3	---	---
NRC	0	1	-1	2	-1	2	-1	3	0	2	---	---	4	3	0	2	---	---
VNIIM *)	-4	3	-5	3	-5	3	-5	4	-4	3	-4	3	---	---	-4	3	---	---
NMIJ	0	2	-1	2	0	2	-1	4	0	3	0	2	4	3	---	---	---	---
Sr-90/Y-90																		
PTB	1	1	---	---	0	2	0	4	3	2	1	2	-2	3	1	2	2	3
LNE-LNHB	1	1	0	2	---	---	-1	3	3	2	1	1	-3	3	0	2	1	3
NIST	1	3	0	4	1	3	---	---	3	4	2	3	-2	4	1	4	2	4
ENEA-INMRI	-2	2	-3	2	-3	2	-3	4	---	---	-1	2	-5	3	-2	3	-1	3
NRC	-1	1	-1	2	-1	1	-2	3	1	2	---	---	-4	3	-1	2	0	3
VNIIM *)	3	3	2	3	3	3	2	4	5	3	4	3	---	---	3	3	4	4
NMIJ	0	2	-1	2	0	2	-1	4	2	3	1	2	-3	3	---	---	1	3
STUK	-1	3	-2	3	-1	3	-2	4	1	3	0	3	-4	4	-1	3	---	---

*) These values were not taken into account for x_R , see text explaining Table A.1.

Annex B

Repeated measurements of the calibration coefficient for the transfer chamber at PTB

The reproducibility of the calibration measurements was studied at PTB, where several sets of measurements were carried out, see Table 1.

In Table B.1, the data of these calibration measurements at PTB are summarized for an angle of incidence of 0°. The strong correlations of these repeated measurements are taken into account by assuming the correlated quantities, e.g. reference dose rate at the beginning of the comparison, to have no uncertainty. The mean values and the remaining standard deviations (times 2) are given for every nuclide. Figure B.1 shows the data. It can easily be seen that, especially for Pm-147, unknown effects influence the measurements. This fact is considered in the data evaluation of the comparison, see Annex A.

Table B.1: Reproducibility of the calibration coefficients of the transfer chamber obtained at PTB over the period from February 2004 to April 2007 for Pm-147, Kr-85, and Sr-90/Y-90 with beam-flattening filter.

Date	Radio-nuclide	Calibration distance m	Dose rate mGy/h	Uncertainty $k = 1$	Chamber current fA	Uncertainty $k = 1$	Calibration coefficient 10^{13} mGy/(h A)	Uncertainty $k = 2$
2004-02	Pm-147	0.2	4.85	0.03	299.5	0.9	1.621	0.022
2004-07	Pm-147	0.2	4.51	0.03	272.3	2.0	1.655	0.031
2004-10	Pm-147	0.2	4.34	0.03	259.1	0.8	1.677	0.022
2005-02	Pm-147	0.2	3.87	0.02	236.6	0.7	1.634	0.022
2005-04	Pm-147	0.2	3.56	0.02	218.4	0.7	1.628	0.022
2005-08	Pm-147	0.2	3.40	0.02	202.8	1.1	1.679	0.027
2006-01	Pm-147	0.2	2.51	0.01	155.6	0.9	1.610	0.026
2007-02	Pm-147	0.2	2.38	0.01	145.2	0.6	1.636	0.024
2007-04	Pm-147	0.2	2.12	0.01	129.4	0.6	1.638	0.024
Mean value and standard deviation times two							1.642	2.9%
2004-02	Kr-85	0.3	127.4	0.8	8287	15	1.537	0.020
2004-07	Kr-85	0.3	124.6	0.8	8127	15	1.533	0.020
2004-10	Kr-85	0.3	122.8	0.8	7973	15	1.541	0.020
2005-02	Kr-85	0.3	119.8	0.7	7813	14	1.534	0.020
2005-04	Kr-85	0.3	118.4	0.7	7731	14	1.532	0.020
2005-08	Kr-85	0.3	116.5	0.7	7670	14	1.518	0.019
2006-01	Kr-85	0.3	112.4	0.7	7351	13	1.530	0.020
2007-02	Kr-85	0.3	105.8	0.6	6938	13	1.525	0.020
2007-04	Kr-85	0.3	104.0	0.6	6802	12	1.529	0.020
Mean value and standard deviation times two							1.531	0.9%
2004-02	Sr-90/Y-90	0.3	31.7	0.2	2267	4	1.399	0.016
2004-07	Sr-90/Y-90	0.3	31.3	0.2	2255	4	1.389	0.016
2004-10	Sr-90/Y-90	0.3	31.1	0.2	2240	4	1.388	0.016
2005-02	Sr-90/Y-90	0.3	30.9	0.2	2222	4	1.390	0.016
2005-04	Sr-90/Y-90	0.3	30.8	0.2	2209	4	1.393	0.016
2005-08	Sr-90/Y-90	0.3	30.6	0.2	2205	4	1.386	0.016
2006-01	Sr-90/Y-90	0.3	30.2	0.2	2171	4	1.393	0.016
2007-02	Sr-90/Y-90	0.3	29.4	0.2	2122	5	1.386	0.016
2007-04	Sr-90/Y-90	0.3	29.3	0.2	2109	4	1.390	0.016
Mean value and standard deviation times two							1.390	0.6%

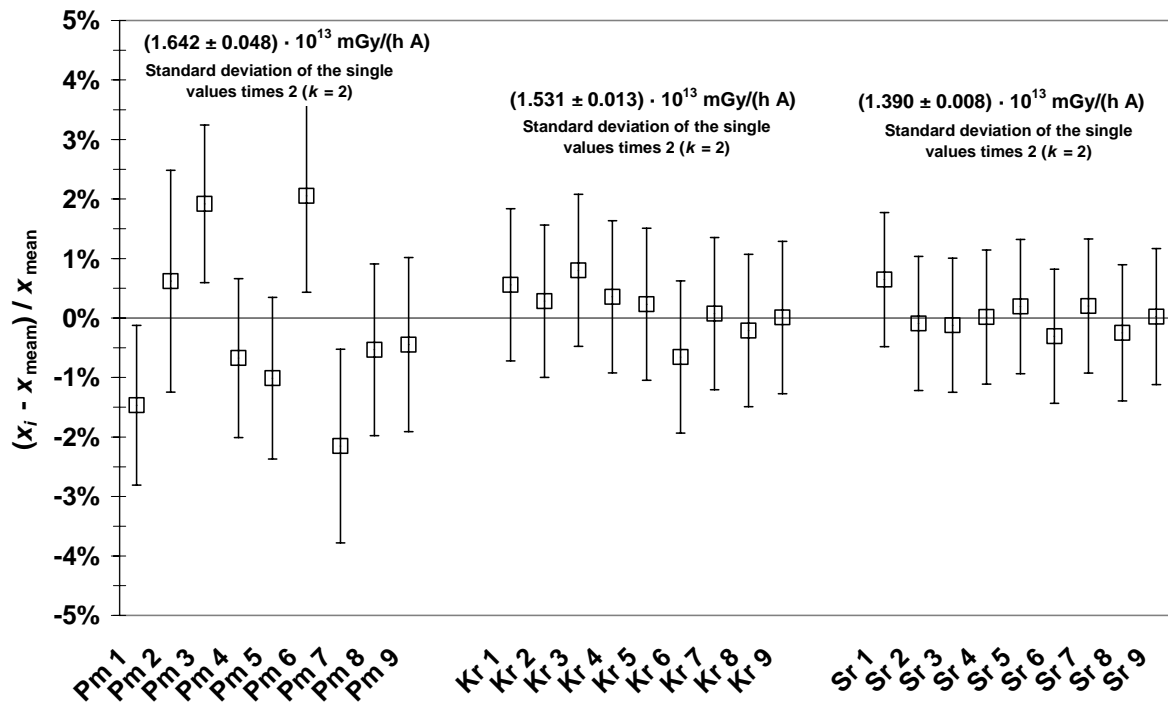


Figure B.1: Reproducibility of the measurements at PTB during the comparison. The error bars represent the uncorrelated contributions to the expanded uncertainty of these measurements ($k = 2$).

Annex C

Check of electronic device and transfer chamber

After receipt of the measurement system, the participant checked the electronic device and the chamber for damage.

The test procedure, which was performed automatically by the test program, was as follows: through variation of the chamber voltage, charge was transferred to the connected electrometer as a result of the chamber capacitance. The value of the chamber capacitance was determined by variation of the chamber voltage, the electrometer capacitance (calibrated against standards at PTB), and by variation of the output voltage of the integrator. The test program compared the measured chamber capacitance with a reference value determined at PTB prior to supplying the measurement system (chamber and electronic device) to each participant. If all measurement units worked correctly and all instruments (including the chamber) were connected properly, the chamber capacitance measured by means of the test program agreed within 1 % with the reference value stored in a parameter file on the laptop.

At PTB, the chamber capacitance was determined at the beginning of the comparison to be (1.7524 ± 0.0003) pF.

In Figure C.1, the chamber capacitance values measured at each participant's laboratory and the subsequent repeated measurements carried out at PTB are

summarized. A good stability of the system during the entire period of the comparison is evident. The variation is within $\pm 0.3\%$.

The electrometer feedback capacitors were also re-calibrated at PTB when the comparison equipment returned from each partner institute. The reproducibility of these feedback capacitors during the comparison period was within $\pm 0.35\%$, see Figure C.2.

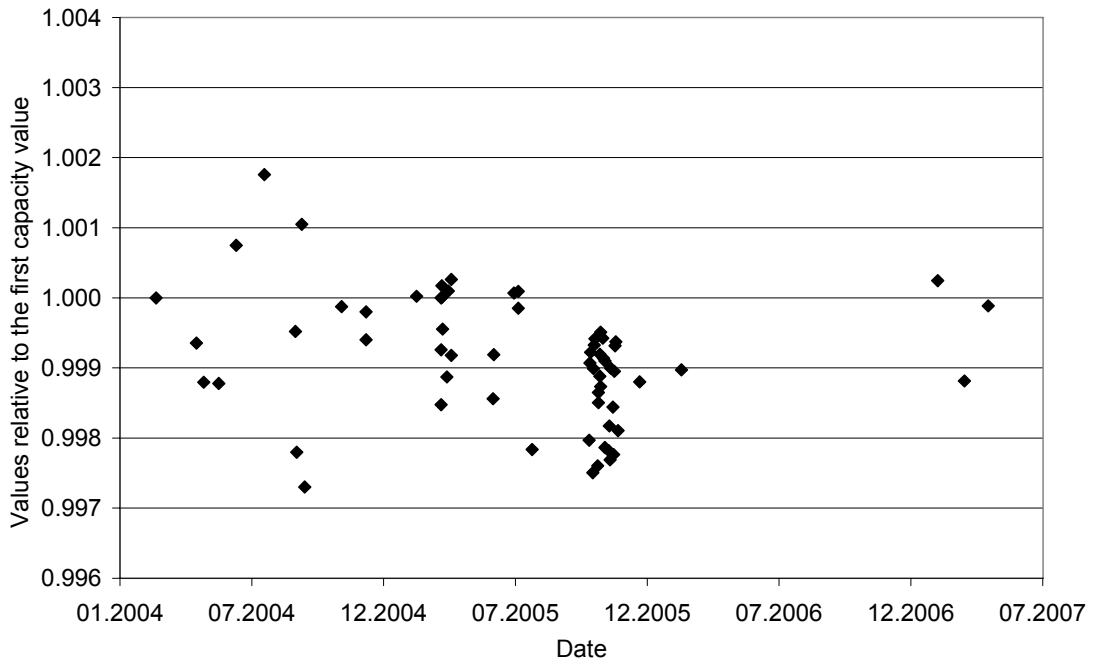


Figure C.1: Stability of the chamber capacitance during the comparison period.

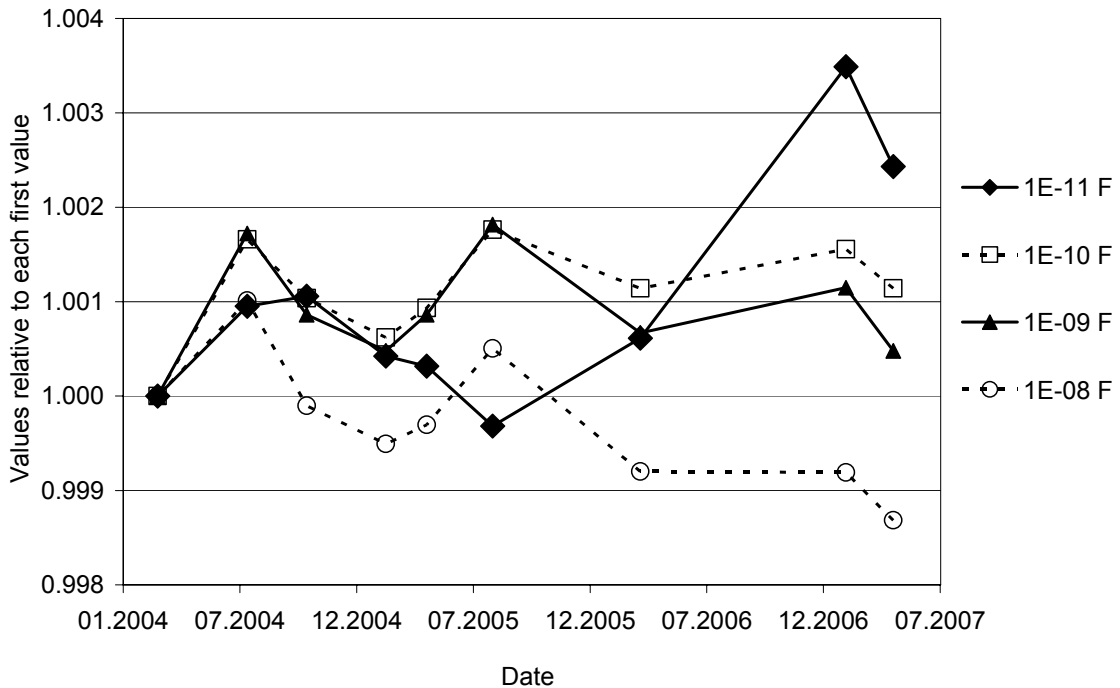


Figure C.2: Reproducibility of the feedback capacitance of the electrometer during the comparison period.

Annex D Addresses of the participants

Pilot laboratory

PTB / DE

Physikalisch-Technische Bundesanstalt (PTB)
Department 6.3 'Radiation protection dosimetry'
Working Group 6.34 'Beta dosimetry'
Bundesallee 100
38116 Braunschweig
Germany

<http://www.ptb.de>

Contact person: Dr. Rolf Behrens

Tel.: +49 531 592 6212

Fax: +49 531 592 69 6212

e-mail: rolf.behrens@ptb.de

LNE-LNHB / FR

LNE-LNHB
CEA/SACLAY, bat 534
FR - 91191 Gif sur Yvette Cedex
France

<http://www.cea.fr>

Contact person: Dr. Jean-Marc Bordy

Tel.: +33 1 69 08 4189

Fax: +33 1 69 08 4773

e-mail: jean-marc.bordy@cea.fr

ENEA-INMRI / IT

Istituto Nazionale di Metrologia delle Radiazioni Ionizzanti
ENEA-INMRI- CR Casaccia
Via Anguillarese 301.
I-00060 SM Galeria (Roma)
C.P. 2400, I - 00100 Roma A.D.
Italy

<http://www.casaccia.enea.it/inmri/>

Contact person: Dr. Maurizio Bovi

Tel.: +39 06 3048 4524

Fax: +39 06 3048 3558

e-mail: bovi@casaccia.enea.it

STUK / FI

Radiation and Nuclear Safety Authority (STUK)
P.O. Box 14
FI - 00881 Helsinki
Finland

<http://www.stuk.fi>

Contact person: Mr. Ilkka Jokelainen

Tel.: +358 9 7598 8451

Fax: +358 9 7598 8450

e-mail: Ilkka.Jokelainen@stuk.fi

VNIIM / RU

All-Russian Institute for Metrology
Ionizing Radiation Laboratory
19 Moscovsky prosp.
St. Petersburg. 198005
Russia
<http://www.vniim.ru>

Contact person: Dr. I. A. Kharitonov
Tel.: 007 812 323-96-11
Fax: 007 812 323-96-17
e-mail: khia@vniim.ru

NIST / US

National Institute of Standards & Technology
100 Bureau Dr., Stop 8460
Gaithersburg, MD 20899-8460 U.S.A.
<http://www.nist.gov>

Contact person: Dr. Christopher G. Soares.
Tel.: +001 301 975 5589
Fax: +001 301 869 7682
e-mail: csoares@email.nist.gov

NRC / CA

National Research Council of Canada
Ionizing Radiation Standards
1200 Montreal Road, M-35
Ottawa. Canada
K1A 0R6
<http://www.nrc.ca>

Contact person: Dr. Patrick R. B. Saull
Tel.: +001 613 993 2715
Fax: +001 613 952 9865
e-mail: patrick.saul@nrc.ca

NMIJ / JP

National Metrology Institute of Japan NMIJ
National Institute of Advanced Industrial Science and Technology, AIST
Quantum Radiation Division
1-1-1 Umezono, Tsukuba, Ibaraki 305-8568
Japan
<http://www.nmij.jp>

Contact person: Tadahiro Kurosawa
Tel.: +81-29-861-5927
Fax: +81-29-861-5673
e-mail: tadahiro-kurosawa@aist.go.jp