Comparison of the air-kerma standards of the NRPA, the STUK, the SSM and the LNE-LNHB in medium-energy x-rays

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Abstract A supplementary comparison has been made between the air-kerma standards of the NRPA, Norway, the STUK, Finland, the SSM, Sweden and the LNE-LNHB, France in the medium-energy x-ray range. The technical protocol was approved by the CCRI(I) in April 2016. The results show the standards to be in agreement at the level of the standard uncertainty of the comparison of 4.4, 9.1 and 4.5 parts in 10³ for the NRPA, STUK and the SSM, respectively. The results are analysed and presented in terms of degrees of equivalence.

1. Introduction

A supplementary comparison has been made between the air-kerma secondary dosimetry standards of the NRPA (Norwegian Radiation Protection Authority), Norway, the STUK (Radiation and Nuclear Safety Authority), Finland, the SSM (Swedish Radiation Safety Authority) and the primary dosimetry standard of the LNE-LNHB (Laboratoire National Henri Becquerel), France in the x-ray range from 100 kV to 250 kV. NRPA was the pilot laboratory and the results were sent to a coordinator at the BIPM (Bureau International des Poids et Mesures). Once the whole set of data were received the coordinator sent the results to the NRPA. Two cavity ionization chambers were used as transfer instruments. The measurements at the national dosimetry laboratories took place during 2016 using the reference conditions recommended by the CCRI (CCEMRI 1972) [1]. All results were submitted by November 2016. The current comparison is identified as EURAMET project # 1386 and registered in the BIPM key comparison data base (KCDB) as EURAMET.RI(I)-S15 supplementary comparison [2]. The technical protocol was approved by the CCRI(I) in April 2016.

2. Comparison procedure

2.1. Object of the comparison

The object of the comparison is to support the ionizing radiation Calibration and Measurement Capabilities (CMCs) of Norway, Sweden and Finland for the quantity air kerma rate in x-rays and to validate the calibration practice of these SSDL participants.

2.2. Radiation qualities and reference conditions

Table 7 and Table 8 describe the characteristics of the laboratories' x-ray beams.

The calibration coefficients for the transfer chambers were specified in terms of air kerma per charge in units of Gy/C and referred to standard condition of air temperature, pressure and humidity; T = 293.15 K, P = 101.325 kPa and h = 50 %. The ambient conditions (temperature, pressure and humidity) during the calibrations were monitored continuously, and results corrected to standard conditions. The SSDLs use the substitution method; the SSM do not correct for ambient conditions assuming an insignificant change in T and P in the short time between the measurements of different instruments and therefore the P_{TP} cancel.

2.3. Participants and course of comparison

Four participants, listed in Table 1, were included in the comparison. The NRPA was the pilot laboratory.

Participant	Institute	Country
NRPA	Norwegian Radiation Protection Authority	Norway
LNE-LNHB	Laboratoire National Henri Becquerel	France
STUK	Radiation and Nuclear Safety Authority	Finland
SSM	Swedish Radiation Safety Authority	Sweden

Table 1 Participating Institutes

The chambers were circulated from the NRPA to the STUK, then to the SSM and the LNE-LNHB and back to the NRPA. The NRPA calibrated the chambers in April 2016 and August 2016. The values $N_{K,NRPA}$ measured before and after the measurements at the other laboratories give rise to the relative standard uncertainty s_{tr} for the chambers, which represent the relative uncertainty in N_K arising from the stability of the transfer chambers. The values $N_{K,L,NHB}$ served as reference values for the supplementary comparison.

The chambers stayed with each participant for about 3 weeks. The results were reported to the coordinator within 6 weeks of each calibration. An Excel sheet was provided by the pilot laboratory in which information about the radiation qualities, reference standards used by the participants and the calibration results were filled in. The uncertainties were given in accordance with the Guide to the Expression of Uncertainties in Measurements [3].

3. Determination of the air-kerma rate at the LNE-LNHB

For a free-air ionization standards, the air-kerma rate is determined by the equation:

$$\dot{K} = \frac{I_{\text{cor}}}{\rho_{\text{air}}V} \frac{W_{\text{air}}}{e} \frac{1}{1 - g_{\text{air}}} \prod k_i \tag{1}$$

where $\rho_{\rm air}$ is the density of air under reference conditions, V is the measuring volume, $I_{\rm cor}$ is the ionization current under reference conditions (corrected for temperature, pressure and humidity), $W_{\rm air}$ is the mean energy expended by an electron to produce an ion pair in air, $g_{\rm air}$ is the fraction of the initial electron energy lost through radiative processes in air, and $\prod k_i$ is the product of the correction factors to be applied to the standard.

The correction factors included in the product $\prod k_i$ are

- $k_{\rm sc}$, correction for scattered radiation,
- $k_{\rm e}$, correction for electron losses,

- k_a , correction for air attenuation,
- k_{dia}, correction for diaphragm effect,
- $k_{\rm s}$, correction for ion recombination,
- k_{pol} , correction for polarity,
- $k_{\rm d}$, correction for field distortion,
- k_p correction for shield transmission.

The factors k_{ii} (initial ionization) and k_{W} (energy dependence of W) introduced in the Report ICRU 90 [4] are not included, nor is the recommended increase in the uncertainty of W_{air} .

The values used for the physical constants ρ_{air} , W_{air}/e and g are given in Table 2. For use with this dry-air value for ρ_{air} , the ionization current I must be corrected for humidity and for the difference between the density of the air of the measuring volume at the time of measurement.

Table 2 Physical constants used in the determination of the air-kerma rate

Constant	Value	$u_{\rm i}$
$ ho_{ m air}$	1.2048 kg m ⁻³	0.0001
$W_{ m air}$ / e	33.97 J C ⁻¹	0.0015
g	1.0000	0.0001

The ambient reference conditions for the LNE-LNHB standard were: T = 293.15 K, P = 101.325 kPa and h = 0 % (dry air). The correction factor for humidity k_h was taken equal to 0.998 for a free-air chamber with a parallel-plate design, the ambient relative humidity being kept within the range 25 % - 75 %.

4. Details of the LNE-LNHB standard

4.1. Free-air chamber

The LNE-LNHB standard is established with a free-air ionization chamber. The free-air chamber is of the conventional parallel-plate design. The measuring volume, V, is defined by the diameter of the chamber aperture and the length of the collecting region. The details of the LNHB standard, which was previously compared with the BIPM standard in 2008 [5]; the main dimensions, the measuring volume and the polarizing voltage for the LNHB standard are shown in Table 3.

Table 3 Main characteristics of the free-air chamber

Characteristic	60 - 300 keV
Aperture diameter / mm	10.074
Air path length / mm	317.9
Collecting length / mm	60.004
Electrode separation / mm	180
Collector width / mm	231
Measuring volume / mm ³	4782.7
Polarizing voltage / V	+5000

4.2. Determination of the LNE-LNHB correction factors

The pressure and temperature correction factors k_P and k_T were calculated for each measurement. The k_{pol} and k_s factors are measured for each radiation quality. The remaining correction factors are firstly calculated by the Monte Carlo method for a set of monoenergetic photon energies covering the energy range of interest. From these, the energy-fluence weighted average values of the correction factors are computed for each radiation quality.

For the radiation qualities currently available at the LNE-LNHB, the energy distributions were measured. For this purpose, the LNE-LNHB has developed a system that can be equipped with CdTe, Si or Ge spectrometers [6] to measure the spectra of its x-ray reference beams. In-house software has been developed to derive the real spectra from the measured raw spectra. This software corrects for the effects of pile-up, photon escape and Compton scattering. These spectra replaced the calculated spectra used previously in the determination of the correction factors. The k_a correction factors were calculated using these measured spectra and the air attenuation coefficients taken from the Hubbell photon cross-section database [7]. The correction factors are given in Table 4.

Table 4 Correction factors for the LNE-LNHB standards for the medium energy x-ray beams

Radiation quality	100 kV	135 kV	180 kV	250 kV	u_{iA}	u_{iB}
Air attenuation k_a	1.012	1.0079	1.0066	1.0053	-	0.0010
Scattered radiation $k_{\rm sc}$	0.9932	0.9948	0.9958	0.9970	-	0.0007
Electron loss ke	1.0000	1.0012	1.0041	1.0076	-	0.0010
Ion recombination k_s	1.0006	1.0006	1.0006	1.0006	-	0.0003
Diaphragm effect k _{dia}	0.9990	0.9986	0.9985	0.9967	-	0.0001
Shield transmission k_p	1.0000	1.0000	0.9999	0.9998	-	0.0001
Polarity k_{pol}	0.9994	0.9995	0.9992	0.9994	0.0005	-
Field distortion k_d	1.0000	1.0000	1.0000	1.0000	-	0.0010
Humidity k _h	0.9980	0.9980	0.9980	0.9980	-	0.0001
$\prod k_i$	1.0021	1.0006	1.0027	1.0044	0.0005	0.0019

It should be noted that several of the new factors resulting from the use of new spectra are different from those used at the time of the 2008 comparison with the BIPM [5]. The combined effect is a decrease in the LNHB standard by the factor 0.9992, 0.9986, 0.9992 and 0.9974 for the radiation qualities 100 kV, 135 kV, 180 kV and 250 kV, respectively. This change directly affects the LNHB-BIPM comparison result published in the KCDB.

5. The transfer instruments

5.1. Determination of the calibration coefficient of the transfer chambers

The air-kerma calibration coefficient $N_{K,lab}$ for a transfer instruments is given by the relation

$$N_{K,\text{lab}} = \frac{\dot{K}}{I_{\text{tr}}} \tag{2}$$

where \dot{K} is the air-kerma rate determined at the participating laboratory (the LNHB or an SSDL) and $I_{\rm tr}$ is the ionization current measured by the transfer instrument and associated current-measuring system. The $I_{\rm tr}$ is corrected to the standard conditions of air temperature, pressure and relative humidity chosen for the comparison ($T=293.15~{\rm K}, P=101.325~{\rm kPa}$ and $h=50~{\rm \%}$). The air-kerma rate at the LNHB is determined using the primary standard and applying eq. (1) and at the SSDLs is determined by the relation

$$\dot{K} = N_{K,\text{BIPM}} I_{\text{SSDL}} \tag{3}$$

where $N_{K,BIPM}$ is the air-kerma calibration coefficient of the SSDL secondary standard traceable to the BIPM and I_{SSDL} is the ionization current measured by the secondary standard and associated current-measuring system at the SSDL. The I_{SSDL} is corrected to the standard conditions of air temperature, pressure and relative humidity used for the evaluation of $N_{K,BIPM}$ at the BIPM.

To derive a comparison result from the calibration coefficients $N_{K, \rm LNHB}$ and $N_{K, \rm SSDL}$ measured at the LNHB and at a secondary standard dosimetry laboratory (SSDL), respectively, differences in the radiation qualities must be taken into account. Normally, each quality used for the comparison has the same nominal generating potential and filtration at each institute, but the half-value layers (HVLs) can differ. A radiation quality correction factor was not derived for each comparison quality, Q, because it is assumed that the half-value layers at the laboratories differ by only a small amount (see Section 9.2). The comparison result at each quality is then taken as

$$R_{K,\text{SSDL}} = \frac{N_{K,\text{SSDL}}}{N_{K,\text{LNHB}}} \tag{4}$$

The presented result is the mean of the ratios for the two transfer instruments.

5.2. Details of the transfer instruments

Two cavity ionization chambers belonging to the NRPA served as transfer instruments for the comparison. Their main characteristics are given in Table 5. Each chamber, without build-up cap, was oriented with the line or cross marked on the stem facing the source. An image of the chambers is reproduced in Appendix A.

Table 5 Technical data of the transfer chambers for medium energy x-ray beams

Chamber type	NE2611A	IBA FC65-G
Serial number	153	3228
Geometry	thimble	thimble
Wall material	graphite	graphite
Wall thickness / g cm ⁻²	0.090	0.068
External diameter / mm	8.36	7.15
Cavity height / mm	9.2	24
Nominal volume / cm ³	0.33	0.65
Reference point (on chamber wall)	6 mm from tip	13 mm from tip
Polarising voltage / V	+300	+300

6. X-ray facilities and characteristics of the radiation beams

6.1. X-ray facilities

The NRPA x-ray laboratory houses a high-stability generator and a bipolar tungsten-anode x-ray tube of type MXR-320/26, with a 3 mm beryllium window and a W-anode angle of 20°. The high tension is produced by a GE ISOVOLT Titan E Power Module 1Ph constant potential generator, the potential verified using x-ray spectrometer measurements. The diameter of the circular beam at 1 m distance from the focus was 12 cm.

The LNE-LNHB beam qualities used in this comparison are produced with a bipolar x-ray tube of type Gulmay 320kV - CP320 with a W-anode angle of 20°. High tension is produced by a Gulmay CP320 constant potential generator. The inherent filtration is 3 mm beryllium. The diameter of the circular beam at 1.2 m distance from the focus was 12 cm.

The SSM x-ray laboratory houses a high-stability generator and a tungsten-anode x-ray tube with a 3 mm beryllium window. The bipolar x-ray tube is of type Yxlon Y.TU 320-D03 with a W-anode angle of 20°. The high tension is produced by an Yxlon MG325 constant potential generator, the potential verified using x-ray spectrometer measurements. The diameter of the circular beam at 1 m distance from the focus was 10 cm.

The STUK x-ray laboratory houses two x-ray sets having high-stability generators. X-ray equipment used for the 100 kV and 135 kV radiation qualities consists of a GE ISOVOLT Titan E 160kV three phase constant potential generator and a unipolar x-ray tube of type MBXR-161/4 having a tungsten anode with angle of 20° and a 1 mm Be window. For the 180 kV and 250 kV radiation qualities, the x-ray equipment includes two GE ISOVOLT Titan three phase constant potential generators having +160 kV and -160 kV high tensions and a bipolar MXR-320/13 x-ray tube having a tungsten anode with angle of 20° and 7 mm Be inherent filtration. The high tension is verified using x-ray spectrometer measurements. The diameter of the circular beam at 1 m distance from the focus was 14 cm.

For an overview of the different laboratory set-ups, Table 6 gives the distance, field size, tube current and air-kerma rate at the radiation quality 100 kV.

Table 6 Field size, beam characteristics and distances for the calibrations at 100 kV.

Institute	Distance Source -	Field size,	Tube current	Air-kerma rate /
	Ionisation chamber / mm	diameter / mm	/ mA	mGy s ⁻¹
NRPA	1000	120	6	0.56
LNHB	1200	120	3,1	0.501
STUK	1000	140	5.0	0.52
SSM	1200	126	7.36	0.496

6.2. Characteristics of the generated radiation beams

The four radiation beams at the four laboratories had the characteristics given in Table 7 and Table 8. The tables summarize the measurement radiation conditions for the generating potential, anode current, filtration (Al and Cu), HVL (expressed in mm of Cu) and the air-kerma rate.

Table 7 Characteristics of the generated radiation beams

Radiation quality	Institute	100 kV	135 kV	180 kV	250 kV
E _{mean} / keV	LNHB	50.97	67.81	85.33	129.21
	NRPA, SSM	100	135	180	250
Generating potential / kV	LNHB	100.4	135.4	180.3	250.2
potentiar / K v	STUK	100.2	135.4	180.1	250.2
	NRPA	6	6	4.5	3
Anode	LNHB	7.7	8.1	5.9	4.7
current / mA	STUK	5.0	5.0	4.5	3.4
	SSM	7.36	8.06	5.62	4.32
	NRPA	0.56	0.55	0.56	0.51
Air kerma rate / mGy s ⁻¹	LNHB	0.501	0.503	0.509	0.507
	STUK	0.52	0.50	0.49	0.50
	SSM	0.496	0.498	0.497	0.493

7. Calibration at LNE-LNHB

The distance between the x-ray tube focus and the reference plane of the free-air chamber is 1200 mm. The geometrical centre of the chambers to be calibrated was placed at the same distance from the x-ray tube focus. For all radiation qualities the relative standard uncertainties Type A, u_{iA} , and Type B, u_{iB} , associated with the use of the LNHB free-air chamber are given in Table 9.

The LNHB has reported lower uncertainties of the air-kerma calibration coefficients for the IBA FC-65-G chamber than for that of the NE2611A. Therefore both values are given in the last row of Table 9. The Type A uncertainty for the current measurements with the IBA FC65-G was half the Type A uncertainty of the other chamber, because the IBA chamber is twice as big as the second. Only the larger uncertainties were used in further uncertainty calculations.

Table 8 Characteristics of the generated radiation beam filters and HVLs.

Radiation quality	Institute	100 kV	135 kV	180 kV	250 kV
	NRPA	3.52	2.03	3.034	2.03
Additional	LNHB	3.402	2.498	2.968	2.968
Al filtration / mm	STUK	3.552	2.351	2.201	2.200
	SSM	3.14	3.18	3.59	1.02
	NRPA		0.247	0.485	1.560
Additional Cu filtration /	LNHB		0.225	0.467	1.624
mm	STUK	-	0.232	0.484	1.570
	SSM	-	0.19	0.4	1.69
	NRPA	0.157	0.495	1.000	2.481
Cu HVL / mm	LNHB	4.038*	0.49	0.974	2.477
Cu HVL/IIIII	STUK	0.149	0.485	1.024	2.403
	SSM	0.141	0.471	0.93	2.49

^{*}expressed in mm of Al

Table 9 Uncertainties associated with the LNE-LNHB standards. Last row gives uncertainties for the chambers IBA FC65-G / NE2611A.

Radiation quality	100	kV	135	5 kV	180 kV		250 kV	
Relative standard uncertainty / %	u_{iA}	$u_{i\mathrm{B}}$	u_{iA}	$u_{i\mathrm{B}}$	u_{iA}	$u_{i\mathrm{B}}$	u_{iA}	$u_{i\mathrm{B}}$
Volume		0.05		0.05		0.05		0.05
Position, standard		0.10		0.10		0.10		0.10
Ionisation current	0.12	0.11	0.12	0.11	0.10	0.11	0.12	0.11
k_{T}		0.04		0 .04		0.04		0.04
$k_{ m P}$		0.04		0.04		0.04		0.04
Correction factors	0.05	0.19	0.05	0.19	0.05	0.19	0.05	0.19
Physical constants		0.15		0.15		0.15		0.15
Air-kerma rate at 1200 mm	0.	32	0	.32	0.	31	0	32
Calibration of transfer chamber, position, current, k_T , k_P	0.22	0.16	0.20	0.16	0.20	0.16	0.25	0.16
Calibration IBA FC65-G coefficient, N _K NE2611A		37 42	_	.37 .41		36 41		37 44

8. Calibration at the SSDLs

8.1. Calibration at NRPA

The calibrations of the transfer chambers were performed against the NRPA reference secondary-standard chamber, type Capintec PR-06G #8429, calibrated at the BIPM in 2013. Its energy response is within \pm 1.9 % for the beam qualities for this comparison. The distance between the focus and reference point of the chamber was 1000 mm and the field size diameter 120 mm.

Ionization currents were measured using Keithley 6517A electrometers. No monitor chamber was used. The current mode ranges of the electrometers were calibrated using a Keithley electrometer resistor standard type 5156 and a voltage standard, both traceable to the Justervesenet (JV), Norway. The uncertainties associated with the calibrations at the NRPA are reported in Appendix B.

8.2. Calibration at STUK

The calibrations of the transfer chamber were performed against the STUK reference secondary standard chamber, NE 2561, calibrated at the BIPM in 2014. Its energy response is within \pm 0.52 % for the beam qualities for this comparison.

The distance between the focus and reference point of the chamber was 1000 mm and the field size diameter 140 mm.

Ionization currents were measured using a Keithley 6517A electrometer. A monitor chamber was used to measure the beam output, but no corrections were made relative to the monitor chamber measurements. The charge mode ranges together with the indicated charge collection time of the electrometers were calibrated in 2016 at National Standards Laboratory, MIKES. The uncertainties associated with the calibrations at the STUK are reported in Appendix B.

8.3. Calibration at SSM

The calibrations of the transfer chamber were performed against the SSM reference secondary-standard chamber, type Exradin A3 #Sh169, calibrated at the BIPM in 2012. The distance between the focus and reference point of the chamber was 1200 mm and the field size diameter 126 mm.

Ionization currents were measured using SSM-build electrometer based on charge measurement. Voltage and capacitor standards were calibrated in 2016 at the Research Institute of Sweden (RISE), former Technical Research Institute of Sweden (SP), Sweden. No monitor chamber was used. The uncertainties associated with the calibrations at the SSM are reported in Appendix B.

9. Additional corrections to transfer chamber measurements

9.1. Ion recombination, polarity, radial non-uniformity, distance and field size

As can be seen from Table 7, the air-kerma rates were similar at the laboratories and so no corrections are applied for ion recombination. The transfer chambers were used with the same polarity at each laboratory and so no corrections are applied for polarity effects in the transfer chambers.

No correction was applied at the SSDL laboratories for the radial non-uniformity of the radiation fields. A relative uncertainty component of 3 parts in 10⁴ is introduced in Table 13 for this effect.

The reference distance is not the same at the four laboratories ranging from 1 m to 1.2 m, and the field size have a variation from 120 mm at the NRPA to 140 mm at the STUK. It is known that transfer chambers respond to scattered radiation in a way that free-air chambers do not, so that calibration coefficients can show some sensitivity to field size. Furthermore, the effect of field size might change with HVL. A relative uncertainty component of 5 parts in 10⁴ is introduced in Table 13 for distance and field size.

9.2. Radiation quality correction factors

Slight differences in radiation qualities might require an additional correction factor k_Q . It can be seen from Table 7 and Table 8 that there are some differences in the radiation qualities expressed in terms of HVL, despite the use of the same calibrated generating potentials and similar filtration. The NRPA made no corrections from the BIPM radiation qualities to the HVL measured at the SSDL, but SSM and STUK applied this correction. A calculation of the correction factors k_Q to correct calibration coefficients from the laboratory stated HVL, to the comparison reference x-ray qualities (HVLs recommended by the CCRI for medium energy x-rays) resulted in the mean of k_Q values to be 1.0000 with a standard deviation 0.00028. Because no correction factor k_Q is applied, a relative standard uncertainty of 5 part in 10^4 is introduced in Table 13 to account for this effect.

10. Comparison results

The calibration coefficients $N_{K,NRPA}$, $N_{K,LNHB}$, $N_{K,STUK}$, and $N_{K,SSM}$ for the transfer chamber are presented in Table 10, and in Figure 1 and Figure 2. The values $N_{K,NRPA}$ measured before and after the calibrations at the other laboratories give rise to the relative standard uncertainties $s_{tr,1}$ and $s_{tr,2}$ for the two chambers, which represent the uncertainty in N_K arising from transfer chamber stability. The analysis of uncertainties of this comparison is similar to that described in the BIPM.RI(I)-K3 comparison report of the NMIJ, Japan and the BIPM [8].

Table 10 Calibration coefficients for the transfer chambers

Radiation quality	100 kV	135 kV	180 kV	250 kV
NE2611A #153				
$N_{K,NRPA}$ (April)) / μ Gy nC ⁻¹	91.32	92.53	93.11	93.92
$N_{K,NRPA}$ (August)) / μ Gy nC ⁻¹	91.49	92.63	93.31	94.05
$s_{\rm tr,1}$ (relative) ^a	0.0012	0.0007	0.0014	0.0009
$N_{K,STUK}$ / μ Gy nC ⁻¹	91.25	92.26	92.97	93.64
$N_{K,SSM}$ / μ Gy nC ⁻¹	90.82	92.19	92.78	93.64
$N_{K,LNHB}$ / μ Gy nC ⁻¹	93.818	91.985	92.660	93.995
IBA FC65-G #3228				
$N_{K,NRPA}$ (April)) / μ Gy nC ⁻¹	45.766	44.996	44.380	43.692
$N_{K,NRPA}$ (August)) / μ Gy nC ⁻¹	45.784	45.028	44.367	43.723
$s_{\rm tr,2}$ (relative) ^a	0.0003	0.0005	0.0002	0.0005
N _{K,STUK} / μGy nC ⁻¹	45.77	44.91	44.33	43.68
$N_{K,SSM}$ / μ Gy nC ⁻¹	45.69	45.00	44.39	43.66
$N_{K,LNHB}$ / μ Gy nC ⁻¹	46.044	44.030	43.543	43.292

^a For each pre-post pair of $N_{K,NRPA}$ values with half-difference d, the standard uncertainty of the mean is taken to be $s_{tr,i} = d / \sqrt{(n-1.4)}$, where the term (n-1.4) is found empirically to be a better choice than (n-1) to estimate the standard uncertainty for low values of n. For n = 2, $s_{tr,i} = 1.3d$ [8][11].

For each radiation quality, the calibration coefficients $N_{K,SSDL}$ are used to evaluate the comparison results $R_{K,SSDL}$ given in Table 11. For the NRPA, the mean of the results determined before and after circulation of the transfer chambers is used.

It can be seen from Table 10 that the NE2611A calibration coefficient determined by the LNHB for the quality 100 kV is 2.9 % greater than the mean of the three SSDL results ($\overline{N}_{K,SSDL}$ = 91.16 μ Gy. nC⁻¹). Regarding the IBA FC65-G, the calibration coefficients for the qualities 135 kV and 180 kV determined by the LNHB are around 2 % lower than the mean of the three SSDL results ($\overline{N}_{K,SSDL}$ = 44.97 μ Gy. nC⁻¹ and $\overline{N}_{K,SSDL}$ = 44.37 μ Gy. nC⁻¹ for 135 kV and 180 kV, respectively).

A chi-squared test (procedure A from Cox 2002 [9]) was performed to analyse these results: we have 4 observations that give the degree of freedom equal to 3. Using a p-value of 0.05, the test failed for a square sum above the critical value 7.82. The chi-squared test failed for the calibration coefficients for the NE2611A at 100 kV and the IBA FC65-G at 135 kV and 180 kV determined at the LNHB. Because of this, it was decided to use the mean value of the three SSDL calibration coefficients as the supplementary comparison reference value (SCRV) to evaluate the comparison results for the three measurements failing the chi-squared test.

The final results $R_{K,SSDL}$ in Table 11 are evaluated as the mean for the two transfer chambers.

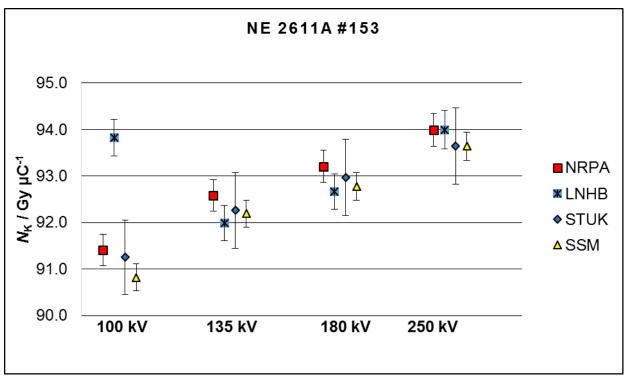


Figure 1 Calibration coefficients for the NE2611A #153 determined by the participating laboratories. Indicated uncertainties are the stated standard uncertainty

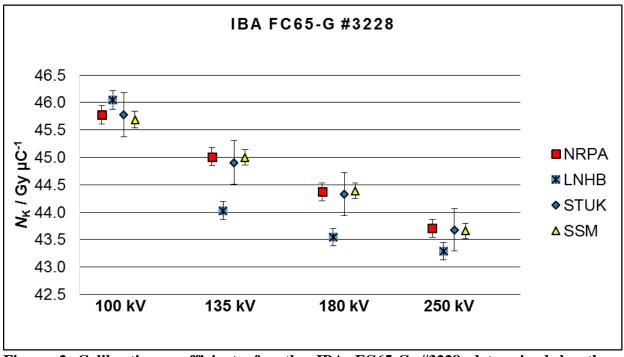


Figure 2 Calibration coefficients for the IBA FC65-G #3228 determined by the participating laboratories. Indicated uncertainties are the stated standard uncertainty.

For each quality, the corresponding uncertainty s_{tr} is the usual standard uncertainty of the mean, or taken as

$$s_{\rm tr} = \sqrt{(s_{\rm tr,1}^2 + s_{\rm tr,2}^2)/2} \tag{5}$$

if this is larger (on the basis that the agreement between transfer chambers should, on average, not be better than their combined stability estimated using $s_{tr,1}$ and $s_{tr,2}$ from Table 10). The mean value of s_{tr} for the four qualities, $s_{tr,comp} = 0.0006$, is a global representation of the comparison uncertainty arising from the transfer chambers and is included in Table 11.

For each quality, the corresponding uncertainty s_{tr} is the standard uncertainty of measurements at NRPA in April and August 2016.

Table 11 Comparison results

Radiation quality	100 kV	135 kV	180 kV	250 kV
NE2611A #153				
$N_{K,NRPA}$ / SCRV	1.0027*	1.0065	1.0059	0.9999
$N_{K,STUK}$ / SCRV	1.0010*	1.0030	1.0033	0.9963
$N_{K,SSM}$ / SCRV	0.9963*	1.0022	1.0013	0.9962
IBA FC65-G #3228				
$N_{K,NRPA}$ / SCRV	0.9942	1.0009*	1.0002*	1.0096
$N_{K,STUK}$ / SCRV	0.9941	0.9985*	0.9993*	1.0090
$N_{K,SSM}$ / SCRV	0.9923	1.0006*	1.0006*	1.0085
S _{tr} ,	0.0006	0.0004	0.0007	0.0005
$R_{K,\mathrm{NRPA}}$	0.9984	1.0037	1.0031	1.0048
$R_{K,STUK}$	0.9976	1.0007	1.0013	1.0026
$R_{K, SSM}$	0.9943	1.0014	1.0009	1.0024
S _{tr,mean}		0.0	006	

^{*} The SCRV for these chambers and radiation qualities were taken as the mean of the SSDL results from Table 10 as explained above.

11. Uncertainties

The uncertainties associated with the LNHB primary standard are listed in Table 9 and those for the transfer chamber calibrations in Table 12 and in the uncertainty budgets in Appendix B. The combined standard uncertainty u_c for the comparison results $R_{K,SSDL}$ is presented in Table 13. This combined uncertainty takes into account correlation in the type B uncertainties associated with measurements at the LNHB and the BIPM. The LNHB type B uncertainty from Table 9 is 0.0029 and from Appendix B the BIPM's is 0.0020. The combined value of the correlation was estimated to 0.0026 when accounting for the total uncertainty associated with the physical constants and half the uncertainties of the Type B for all other corrections associated with the primary standards. This is consistent with the analysis of the results of BIPM comparisons in medium- energy x-rays in terms of degrees of equivalence described in Burns (2003) [10], and the analysis in the comparison of air kerma standards for medium-energy x radiation described by Csete et al [11].

Table 12 Uncertainties associated with the calibration of the transfer chambers

Combined relative standard uncertainty	100 kV	135 kV	180 kV	250 kV
$N_{K,\mathrm{NRPA}} / u_{\mathrm{i}}$	0.0037	0.0037	0.0037	0.0037
$N_{K,\mathrm{LNHB}}$ / u_{i}	0.0042	0.0041	0.0041	0.0044
$N_{K,\mathrm{STUK}} / u_{\mathrm{i}}$	0.0088	0.0088	0.0088	0.0088
$N_{K, SSM} / u_i$	0.0039	0.0039	0.0039	0.0039

Table 13 Uncertainties associated with the comparison results

Combined relative standard uncertainty	100 kV	135 kV	180 kV	250 kV	Mean
$N_{K,\mathrm{NRPA}}$ / $N_{K,\mathrm{LNHB}}$	0.0056	0.0055	0.0055	0.0057	0.0056
$N_{K,\mathrm{STUK}}$ / $N_{K,\mathrm{LNHB}}$	0.0098	0.0097	0.0097	0.0098	0.0098
$N_{K, SSM} / N_{K, LNHB}$	0.0057	0.0057	0.0057	0.0059	0.0057
Transfer chambers	0.0006	0.0004	0.0007	0.0005	0.0006
Non-uniformity of radiation fields	0.0003	0.0003	0.0003	0.0003	0.0003
Different distances	0.0005	0.0005	0.0005	0.0005	0.0005
Radiation quality correction factor	0.0005	0.0005	0.0005	0.0005	0.0005
$R_{K,\mathrm{NRPA}} / u_{\mathrm{c}}$	0.0044	0.0043	0.0043	0.0045	0.0044
$R_{K,STUK} / u_c$	0.0091	0.0091	0.0091	0.0092	0.0091
$R_{K,SSM} / u_c$	0.0045	0.0044	0.0044	0.0047	0.0045

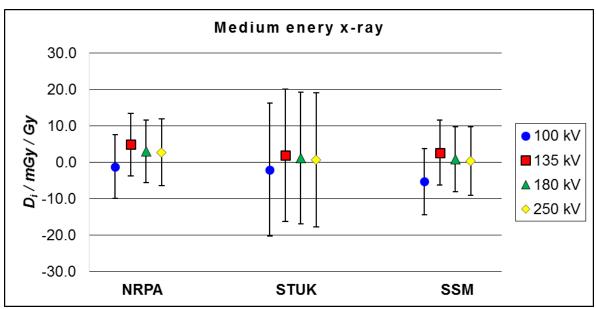


Figure 3 Degrees of equivalence for each SSDL laboratory i with respect to the supplementary comparison reference value.

12. Degrees of Equivalence and discussion

Burns [8] has described the analysis of the results of key comparisons in medium energy x-rays beams in terms of degrees of equivalence, and this analysis was applied. The LNHB determination of the air-kerma calibration coefficient was taken as the supplementary comparison reference value (SCRV). It follows that for each laboratory i the degree of equivalence with respect to the reference value is the relative difference $D_i = R_{K,SSDL} - 1$ and its expanded uncertainty $U_i = 2 u_i$. The results for D_i and U_i , expressed in mGy Gy⁻¹ are shown in Table 14 and Figure 3.

All the SSDLs have traceability to the BIPM, and the uncertainty budgets reported in Appendix B show for all radiation qualities a standard uncertainty from the PSDL (the BIPM) not more than 0.21 %. In Table 9 the LNHB uncertainties are in the range 0.36 % - 0.44 %. The STUK has much larger uncertainties for the long-term stability, the spectral differences and the effect of beam non-uniformity, see Appendix B. The uncertainty budgets were prepared individually and STUK's approach has been conservative. This comparison likely supports lower uncertainty estimates for the STUK CMC claims in the future. As discussed in Section 10, for three calibration coefficients determined by the LNHB, the chi-squared test failed and the mean of the SSDL determination was used as the SCRV for these measurement results. The LNHB received notice from the pilot laboratory of their discrepant results for the three calibration coefficients NE2611A 100 kV and IBA FC65-G 135 kV and 180 kV. The LNHB can find no reason for the mistakes and see only a possibility due to mistake of filter, HV or current. The logbooks have been investigated without fault. The last LNHB K3-key comparison with BIPM was in 2008. The LNHB plan to make a new K3 comparison in 2018. Further information on the LNHB standard will be given there.

13. Conclusions

The comparison results presented in Table 11 and Figure 3 show agreement between the SSDLs and SCRV at the level of the standard uncertainty of the comparison of 4.4, 9.1 and 4.5 parts in 10³ for NRPA, STUK and SSM, respectively. These uncertainties are the mean calculated from the four radiation qualities uncertainties given in the last three rows of Table 13.

The supplementary comparison EURAMET.RI(I)-S15 for the determination of air kerma in medium energy x-rays beams shows the standards of the NRPA, the STUK, the SSM and the LNHB to be in general agreement at the level of the standard uncertainty of the comparison, except for three results from the LNHB that did not pass the chi-squared test. Tables and graphs of degrees of equivalence are presented.

14. References

- [1] CCEMRI 1972 Qualités de rayonnement *Comité Consultatif pour les Étalons de Mesures des Rayonnements Ionisants (Section I)* 2nd meeting R15–16
- [2] KCDB 2014 The BIPM key comparison database is available online at http://kcdb.bipm.org/
- [3] Evaluation of measurement data Guide to the Expression of Uncertainty in Measurement. JCGM 100:2008. http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf
- [4] ICRU International commission on radiation units and measurements. Key data for ionizing-radiation dosimetry: Measurement Standards and Applications. Journal of ICRU 14 No 1(2014) Report 90, Oxford 2017.

- [5] Burns D T, Kessler C, Denozièr M, and Ksouri W, 2008, Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the LNE-LNHB, France and the BIPM in the medium-energy x-rays *Metrologia* 45 *Tech. Suppl.* 06004.
- [6] Plagnard J., Comparison of the measured and calculated spectra emitted by the x-ray tube used at the Gustave Roussy radiobiological service, X-Ray Spectrom. 2014, 43, 298-304.
- [7] Berger M.J., Hubbell J.H., *XCOM: Photon Cross Sections on a Personal Computer*, NBSIR 87-3597, 1987.
- [8] Burns D T, Kessler C, Tanaka T, Kurosawa T and Saito N, 2016, Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the NMIJ, Japan and the BIPM in the medium-energy x-rays *Metrologia* 53 *Tech. Suppl.* 06002.
- [9] Cox M. G., The evaluation of key comparison data, Metrologia, 2002, 39, 589-595
- [10] Burns D T 2003 Degrees of equivalence for the key comparison BIPM.RI(I)-K3 between national primary standards for medium-energy x-rays <u>Metrologia 40 Tech.</u> <u>Suppl. 06036</u>
- [11] Csete I, Czap L and Gomola I. Comparison of air kerma standards for mediumenergy x radiation between the MKEH and the IAEA. <u>Metrologia 49 Tech. Suppl.</u> 06011

15. Appendix A. Image of the transfer chamber and degree of equivalence



Figure 4 The transfer chambers IBA FC65-G and NE2611A

Table 14 Degree of equivalence. For each laboratory i, the degree of equivalence with respect to the key comparison reference value D_i and its expanded uncertainty U_i , Here the LNE-LNHB represents the SCRV, but as explained above, for three calibration coefficients determined by the LNHB the chi-squared test failed and the mean of the SSDL determinations were used as the SCRV for these measurement results.

Laboratory i	100 kV		135 kV		180 kV		250 kV	
D_i and U_i / mGy Gy ⁻¹	D_i	U_i	D_i	U_i	D_i	U_i	D_i	U_i
NRPA	-1.6	8.9	3.7	8.7	3.1	8.7	4.8	9.3
STUK	-2.4	18.3	0.7	18.2	1.3	18.2	2.6	18.5
SSM	-5.7	9.2	1.4	9.1	0.9	9.1	2.4	9.6

16. Appendix B Uncertainty budgets for the SSDLs

16.1. SSDL of the NRPA

Air kerma rate	Type A	Туре В
	Uncerto	ainty (%)
1 Reference standard, set-up and radiation field		
Calibration coefficient reported by PSDL	0.05	0.19
Long term stability of reference standard	0.03	-
Spectral difference of SSDL and PSDL	-	0.20
Difference in radial non-uniformity of the beam and field size	0.15	0.10
Combined uncertainty of reference standard and setup	0.16	0.29
2 Use of reference standard		
Chamber positioning (distance, orientation)	-	0.06
Current/charge measurement including leakage	0.07	0.05
Air temperature correction	-	-
Air pressure correction	-	-
Combined uncertainty in measuring with reference standard	0.07	0.08
Combined uncertainty in air-kerma determination, K _{std} (1+2)	0.18	0.30
3 Use of transfer chamber		
Chamber positioning (distance, orientation)	-	0.06
Current/charge measurement including leakage	0.07	0.05
Air temperature correction	-	-
Air pressure correction		_
Combined uncertainty in measuring with tranfer chamber	0.07	0.08
Relative combined standard uncertainty (1+2+3)	0.19	0.31
Total uncertainty for the air-kerma calibration coefficient, 1σ	0.	37

16.2. SSDL of the STUK

Air kerma rate	Type A	Туре В
	Uncerto	ninty (%)
1 Reference standard, set-up and radiation field		
Calibration coefficient reported by PSDL	0.06	0.19
Long term stability of reference standard		0.29
Spectral difference of SSDL and PSDL	-	0.32
Difference in radial non-uniformity of the beam and field size		0.29
Combined uncertainty of reference standard and setup	0.06	0.55
2 Use of reference standard		
Chamber positioning (distance, orientation)		0.40
Current/charge measurement including leakage		0.21
Air temperature correction		0.09
Air pressure correction		0.03
Humidity		0.12
Combined uncertainty in measuring with reference standard	0.00	0.48
Combined uncertainty in air-kerma determination, K std (1+2)	0.06	0.73
3 Use of transfer chamber		
Chamber positioning (distance, orientation)		0.40
Current/charge measurement including leakage		0.21
Air temperature correction		0.09
Air pressure correction		0.03
Humidity		0.12
Combined uncertainty in measuring with tranfer chamber	0.00	0.48
Relative combined standard uncertainty (1+2+3)	0.06	0.87
Total uncertainty for the air-kerma calibration coefficient, 1σ	0.	88

16.3. SSDL of the SSM

Air kerma rate	Type A	Type B
	Uncerto	ainty (%)
1 Reference standard, set-up and radiation field		
Calibration coefficient reported by PSDL	0.06	0.20
Long term stability of reference standard	0.10	
Spectral difference of SSDL and PSDL	-	0.10
Difference in radial non-uniformity of the beam and field size		0.17
Drift in HVL		0.22
Short time stability in air kerma rate	0.02	
Combined uncertainty of reference standard and setup	0.12	0.36
2 Use of reference standard		
Chamber positioning (distance, orientation)		0.06
Current/charge measurement including leakage	0.02	0.02
Uncertainty in HVL		0.05
Interpolating N_K (there are diference in HVL between SSM and PSDL)		0.03
Air temperature correction	-	-
Air pressure correction	-	-
Combined uncertainty in measuring with reference standard	0.02	0.09
Combined uncertainty in air-kerma determination, K std (1+2)	0.12	0.37
3 Use of transfer chamber		
Chamber positioning (distance, orientation)		0.06
Current/charge measurement including leakage	0.02	0.02
Air temperature correction	-	-
Air pressure correction	-	-
Combined uncertainty in measuring with tranfer chamber	0.02	0.06
Relative combined standard uncertainty (1+2+3)	0.12	0.37
Total uncertainty for the air-kerma calibration coefficient, 1σ	0.	39