## Comparison of the air-kerma x-ray standards of the NRPA, the STUK, the SSM and the LNE-LNHB in the ISO 4037 narrow spectrum series in the range 40 kV to 300 kV

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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 ISO 4037 narrow spectrum series in the range 40 kV to 300 kV. 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 12, 14 and 8 parts in 10<sup>3</sup> for the NRPA, the 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 standards of the LNE-LNHB (Laboratoire National Henri Becquerel), France, in the x-ray range from 40 kV to 300 kV. The 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. One cavity ionization chamber was used as transfer instrument. The measurements at the national dosimetry laboratories took place during 2016. The ISO 4037 [1] narrow-spectrum x-ray radiation qualities used for this comparison cover the range from 40 kV to 300 kV and are characterized by the half-value layers from about 0.08 mm Cu to 6 mm Cu as described by Büermann et al. [2] for the comparison EURAMET.RI(I)-S3. All results were submitted by November 2016. The current comparison is identified as EURAMET project # 1388 and registered in the BIPM key comparison data base (KCDB) as EURAMET.RI(I)-S3.2 supplementary comparison [3]. 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. The radiation qualities were taken from the ISO 4037 narrow beam x-ray series from 40 kV to 300 kV.

#### 2.2. Radiation qualities and reference conditions

The narrow spectrum series from ISO 4037 [1] were the radiation beams chosen for this comparison, and the selected qualities were N-40, N-60, N-120 and N-300. Table 7 and Table 8 describe the characteristics of the laboratories' x-ray beams.

The calibration coefficients for the transfer chamber were specified in terms of air kerma per charge in units of Gy/C and referred to standard conditions 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 the results were corrected to standard conditions. The SSDLs use the substitution method, and the short time between the measurements of different instruments introduce small changes in the ambient conditions.

#### 2.3. Participants and course of comparison

Four participants, listed in Table 1, were included in the comparison. 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 chamber was circulated from the NRPA to the STUK, then to the SSM and the LNE-LNHB and back to the NRPA. The NRPA calibrated the chamber in April 2016 and August/September 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 chamber, which represents the relative uncertainty in  $N_K$  arising from the stability of the transfer chamber.

The chamber 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 and reference standards used by the participants and the calibration results were filled in. The uncertainties were given in accordance with the ISO Guide to the Expression of Uncertainties in Measurements [4].

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

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

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

where  $\rho_{air}$  is the density of air under reference conditions, V is the measuring volume,  $I_{cor}$  is the ionization current under reference conditions (corrected for temperature, pressure and humidity),  $W_{air}$  is the mean energy expended by an electron to produce an ion pair in air,  $g_{air}$  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*<sub>e</sub>, correction for electron losses,
- $k_{\rm a}$ , correction for air attenuation,
- $k_{\text{dia}}$ , 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 [5] are not included, nor is the recommended increase in the uncertainty of  $W_{air}$ .

The values used for the physical constants  $\rho_{air}$  and  $W_{air}/e$  are given in Table 2. For use with this dry-air value for  $\rho_{air}$ , 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.

Constant	Value	<i>u</i> i
$ ho_{ m air}$	1.2048 kg m <sup>-3</sup>	0.0001
$W_{\rm air}/e$	33.97 J C <sup>-1</sup>	0.0015
g	1.0000	0.0001

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

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 characteristics of this free-air ionization chamber are given in Table 3.

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 <sup>3</sup>	4782.7
Polarizing voltage / V	+5000

## Table 3 Main characteristics of the free-air chamber

## 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 mono-energetic 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, except the ISO N-300. 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 replace 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's photon cross-section database [7]. For the N-300, no measured spectrum was available. The correction factors  $\prod k_i$  for N-300 was determined by using a calculated spectrum. Table 4 shows the new correction factors for the standard evaluated using these measured spectra. The change in the LNHB standard resulting from the use of new spectra is 0.9980, 0.9988, 0.9982 and 0.9762 for the radiation qualities N-40, N-60, N-120 and N-300, respectively.

Radiation quality	N-40	N-60	N-120	N-300
Air attenuation $k_a$	1.0126	1.0084	1.0058	1.0043
Scattered radiation $k_{\rm sc}$	0.9924	0.9940	0.9963	0.9981
Electron loss $k_e$	1.0000	1.0000	1.0049	1.0303
Ion recombination <i>k</i> <sub>s</sub>	1.0005	1.0007	1.0018	1.0003
Diaphragm effect k <sub>dia</sub>	0.9992	0.9988	0.9985	0.9801
Shield transmission $k_p$	1.0000	1.0000	1.0000	1.0000
Polarity k <sub>pol</sub>	1.0001	1.0001	1.0004	1.0000
Humidity <i>k</i> <sub>h</sub>	0.9980	0.9980	0.9980	0.9980
$\prod k_{i}$	1.0027	0.9999	1.0057	1.0107
Uncertainty Type A	0.0005	0.0005	0.0005	0.0005
Uncertainty Type B	0.0019	0.0019	0.0019	0.0019
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## Table 4 Correction factors for the LNE-LNHB standards for the ISO narrow beams

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#### 5. The transfer instrument

5.1. Determination of the calibration coefficient of the transfer chamber

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

$$N_{K,\text{lab}} = \frac{K}{I_{\text{tr}}}$$
(2)

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

To derive a comparison result from the calibration coefficients  $N_{K,LNHB}$  and  $N_{K,SSDL}$  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 at each institute, but the halfvalue 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

$$R_{K,\text{SSDL}} = \frac{N_{K,\text{SSDL}}}{N_{K,\text{LNHB}}} \tag{3}$$

#### 5.2. Details of the transfer instrument

A cavity ionization chamber belonging to the SSM served as transfer instrument for the comparison. Its main characteristics are given in Table 5. The chamber was oriented with the ring on the wall and the mark on the stem toward the source. An image of the chamber is reproduced in Appendix A.

Chamber type	Exradin A6
Serial number	XQ152602
Geometry	spherical
Wall material	C-552
Wall thickness / g cm <sup>-2</sup>	0.53
External diameter / mm	120
Cavity height / mm	120
Nominal volume / cm <sup>3</sup>	800
Reference point (on chamber wall)	Ring on wall and mark on stem toward source
Polarising voltage / V	+300

Table 5 Technical data of the transfer chamber for ISO Narrow series

## 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 3 m distance from the focus was 27 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  $20^{\circ}$ . 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 m distance from the focus was 20 cm and assumed to be 50 cm at 2.5 m, which was the distance for measurements.

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  $20^{\circ}$ . 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 2.5 m distance from the focus was 47.5 cm.

The STUK x-ray laboratory houses two x-ray sets having high-stability generators. X-ray equipment used for the N-40 and N-60 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 N-120 and N-300 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 2 m distance from the focus was 28 cm.

For an overview of the different laboratories set-ups, Table 6 gives the distance, field size, tube current and air-kerma rate at the radiation quality N-60.

Institute	Distance Source -	Field size,	Anode	Air-kerma rate
	Ionization chamber / mm	diameter / mm	current / mA	$/ mGy h^{-1}$
NRPA	3000	270	6	3.8
LNHB	2500	500	3,1	2.977
STUK	2000	280	2.3	3.03
SSM	2500	475	3.43	2.873

#### Table 6 Field size, beam characteristics and distances for the calibrations at N-60

#### 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 summarise the measurements radiation conditions for the generating potential, anode current, air-kerma rate, filtration (Al, Cu, Sn and Pb) and the HVL (expressed in mm of Cu).

Radiation quality	Institute	N-40	N-60	N-120	N-300
<i>E</i> <sub>mean</sub> / keV LNHB		33	48	100	250
	NRPA, SSM	40	60	120	300
Generating	LNHB	40.4	60.4	120.3	300.1
	STUK	40.1	60.0	119.8	300.4
Anode	NRPA	10	6	15	7
	LNHB	3.4	3.1	8.3	2.6
current / mA	STUK	5.0	2.3	10.0	3.0
	SSM	5.41	3.43	9.78	3.59
	NRPA	3.6	3.8	3.6	3.6
Air kerma rate / mGy h <sup>-1</sup>	LNHB	2.970	2.977	2.980	3.000
	STUK	3.63	3.03	3.41	3.39
	SSM	2.873	2.873	2.882	2.842

Table 7 Characteristics of the generated radiation beams

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

Radiation quality	Institute	N-40	N-60	N-120	N-300
	NRPA	3.64	4.06	4.07	4.08
Additional	LNHB		3.99	3.99	3.99
mm	STUK	4.0	4.0	5.9	5.9
	SSM	4.13	3.05	2.12	-
	NRPA	0.21	1.99	4.96	
Additional	LNHB	0.281	0.598	4.383	-
mm	STUK	0.21	0.26	5	-
	SSM	0.2	0.65	5.00	-
	NRPA			1.05	3.12
Additional	LNHB			0.999	2.994
mm	STUK			1.0	3.0
	SSM	-	-	1.01	3.0
	NRPA				5.04
Additional	LNHB				4.501
mm	STUK	-	-	-	5
	SSM	-	-	-	5.3
	NRPA	0.085	0.241	1.737	6.3
	LNHB	0.084	0.241	1.695	6.058
	STUK	0.086	0.24	1.75	6.25
	SSM	0.086	0.24	1.7	6.2

## 7. Calibration at LNE-LNHB

The volume of the free-air ionization chamber was too small to make precise measurements for the air-kerma rate (3 mGy/h) required for this comparison. So, a cavity ionization chamber of 1 litre was calibrated against the free-air chamber at these qualities at 1200 mm. This chamber was then used to determine the air-kerma rate at 2500 mm, the reference distance for this comparison. A cavity chamber in use at a longer distance can be influenced by more attenuation of the beam and more scatter radiation. No correction was used for the effect. 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 and the cavity chamber are given in Table 9.

Radiation quality	N-	40	N	-60	N-120		N-300	
Relative standard uncertainty / %	$u_{iA}$	$u_{i\mathrm{B}}$	<i>u</i> <sub>iA</sub>	$u_{i\mathrm{B}}$	<i>u</i> <sub>iA</sub>	$u_{i\mathrm{B}}$	$u_{iA}$	$u_{i\mathrm{B}}$
Volume		0.05		0.05		0.05		0.05
Position, standard		0.10		0.10		0.10		0.10
Ionisation current	0.40	0.11	0.33	0.11	0.89	0.11	0.74	0.11
k <sub>T</sub>		0.04		0.04		0.04		0.04
kp		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	0.52		0.47		0.95		0.81	
Position, cavity chamber		0.10		0.10		0.10		0.10
Ionisation current	0.18	0.11	0.12	0.11	0.15	0.11	0.10	0.11
k <sub>T</sub>		0.04		0.04		0.04		0.04
<i>k</i> <sub>P</sub>		0.04		0.04		0.04		0.04
Air-kerma rate at 2500 mm	0.	58	0	.51	0.	98	0.	84
Calibration of transfer chamber, position, current, $k_{\rm T}$ , $k_{\rm P}$	0.10	0.16	0.10	0.16	0.10	0.16	0.16	0.16
Calibration coefficient, N <sub>K</sub>	0.	61	0	.55	1.	00	0.	87

#### Table 9 Uncertainties associated with the LNE-LNHB standard

## 8. Calibration at the SSDLs

## 8.1. Calibration at NRPA

The calibrations of the transfer chamber were performed against the NRPA reference secondary-standard chamber, type Capintec PM30 #3650, calibrated at the VSL in 2009. Its energy response is within  $\pm$  1.6 % for the beam qualities for this comparison. The distance between the focus and reference point of the chamber was 300 cm and the field size diameter 27 cm.

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, type NE2575C, calibrated at the PTB in 2014. Its energy response is from -5 % to +3 % for the beam qualities for this comparison relative to N-40. The distance between the focus and reference point of the chamber was 200 cm and the field size diameter 28 cm.

Ionization currents were measured using Keithley 6517A electrometers. A monitor chamber was used to measure the beam output, but no corrections were made relative to the monitor chamber measurements. The electrometer was calibrated 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 A4 #Sh304, calibrated at the PTB in 2016. The distance between the focus and reference point of the chamber was 250 cm and the field size diameter 47.5 cm.

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. A relative uncertainty component of 5 parts in  $10^4$  is introduced in Table 13 for this effect. The transfer chamber was used with the same polarity at each laboratory and so no corrections are applied for polarity effects in the transfer chamber.

No correction is applied at any of the laboratories for the radial non-uniformity of the radiation fields. A relative uncertainty component of 5 parts in  $10^4$  is introduced in Table 13 for this effect.

The reference distance is not the same at the four laboratories, ranging from 2 m to 3 m. The field size of 475 mm at the SSM is larger than that of 270 mm at the NRPA. It is known that longer distance give more air absorptions and more scatter radiation, and wider field size more scatter radiation, showing some sensitivity to the calibration coefficients. Furthermore, these effects might change with HVL. A relative uncertainty component of 1 part in 10<sup>3</sup> is introduced in Table 13 for the effects.

#### 9.2. Radiation quality correction factors

Slight differences in radiation qualities might require a correction factor  $k_Q$ . However, from Table 7 and Table 8 it is evident that the radiation qualities at the laboratories are very closely matched in terms of HVL and so the correction factor  $k_Q$  is taken to be unity for the different qualities. The NRPA made no corrections from the calibration qualities at the VSL to the HVL measured at the SSDL, but the SSM and the STUK applied the corresponding correction for the calibration qualities at the PTB. 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 from ISO 4037 [1] narrow-spectrum x-ray radiation qualities) resulted in the mean of  $k_Q$  values to be 0.9999 with a standard deviation 0.00048. A relative uncertainty component of 5 parts in 10<sup>4</sup> is introduced in Table 13 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. The values  $N_{K,NRPA}$  measured before and after the calibrations at the other laboratories give rise to the relative standard uncertainty  $s_{tr}$  for the chamber, 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 [11].

Radiation quality	N-40	N-60	N-120	N-300
Exradin A6 #XQ152602				
$N_{K,\text{NRPA}}(\text{April})) / \mu \text{Gy nC}^{-1}$	39.32	37.10	37.44	38.05
$N_{K,\text{NRPA}}$ (September) ) / $\mu$ Gy nC <sup>-1</sup>	39.39	37.13	37.51	38.12
<i>s</i> <sub>tr</sub> (relative) <sup>a</sup>	0.0013	0.0006	0.0014	0.0014
$N_{K,STUK}$ / $\mu$ Gy nC <sup>-1</sup>	39.55	36.66	37.12	37.93
$N_{K,SSM}$ / $\mu$ Gy nC <sup>-1</sup>	39.60	36.94	37.07	38.15
$N_{K,LNHB}$ / $\mu$ Gy nC <sup>-1</sup>	39.784	36.68	36.839	36.461

Table 10	Calibration	coefficients for	• the transfer	chamber

<sup>a</sup> For each pre-post pair of  $N_{K,NRPA}$  values with half-difference *d*, the standard uncertainty of the mean is taken to be  $s_{tr} = d / \sqrt{(n-1.4)}$ , 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,  $s_{tr} = 1.3d$  [8]



Figure 1 Calibration coefficients for the transfer chamber determined by the participating laboratories. Indicated uncertainties are the stated standard deviation

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

It can be seen from Table 10 that the LNHB calibration coefficient for the N-300 quality is 4.2% lower than the mean of the three SSDL results ( $\overline{N_{K,SSDL}} = 38.06 \,\mu\text{Gy.nC}^{-1}$ ). 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 coefficient for the N-300 quality determined by 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) for this quality.

The mean value of  $s_{tr}$  for the four qualities,  $s_{tr,comp} = 0.0012$ , is a global representation of the comparison uncertainty arising from the transfer chamber 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 September.

#### **Table 11 Comparison results**

Radiation quality	N-40	N-60	N-120	N-300
EXRADIN A6 #XQ152602				
N <sub>K,NRPA</sub> / N <sub>K,LNHB</sub>	0.9892	1.0118	1.0173	1.0446*
N <sub>K,STUK</sub> / N <sub>K,LNHB</sub>	0.9941	0.9995	1.0077	1.0403*
N <sub>K,SSM</sub> / N <sub>K,LNHB</sub>	0.9955	1.0071	1.0062	1.0464*
<i>s</i> tr (relative)	0.0013	0.0006	0.0014	0.0014
<i>R<sub>K,NRPA</sub></i>	0.9892	1.0118	1.0173	1.0008
<i>R<sub>K,STUK</sub></i>	0.9941	0.9995	1.0077	0.9967
R <sub>K,SSM</sub>	0.9955	1.0071	1.0062	1.0014
Str mean	0.0012			

\* These ratios were not used because the chi-squared test failed for the LNHB result. The SCRV for the N-300 was taken as the mean of the SSDLs' results from Table 10.

## 11. Uncertainties

The uncertainties associated with the LNHB primary and the secondary standards are listed in Table 9 and those for the transfer chamber calibrations in Table 12. 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. All Type B uncertainties for the PTB, the VSL and the LNHB measurements cancelled in the evaluation of the ratios. The Type B uncertainty was determined to 0.0037 and used in the calculations.

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Combined relative standard uncertainty	N-40	N-60	N-120	N-300
$N_{K,\mathrm{NRPA}}$ / $u_\mathrm{i}$	0.0090	0.0090	0.0090	0.0130
$N_{K,\text{LNHB}}$ / $u_{\text{i}}$	0.0061	0.0055	0.0100	0.0087
$N_{K,\mathrm{STUK}}$ / $u_{\mathrm{i}}$	0.0130	0.0130	0.0130	0.0130
$N_{K,\rm SSM}$ / $u_{\rm i}$	0.0057	0.0057	0.0057	0.0057

Combined relative standard uncertainty	N-40	N-60	N-120	N-300	Mean
N <sub>K,NRPA</sub> / N <sub>K,LNHB</sub>	0.0109	0.0105	0.0135	0.0156	0.0126
N <sub>K,STUK</sub> / N <sub>K,LNHB</sub>	0.0144	0.0141	0.0164	0.0156	0.0151
N <sub>K,SSM</sub> / N <sub>K,LNHB</sub>	0.0083	0.0079	0.0115	0.0104	0.0095
Transfer chamber	0.0013	0.0006	0.0014	0.0014	0.0012
Recombination	0.0005	0.0005	0.0005	0.0005	0.0005
Non-uniformity of radiation fields	0.0005	0.0005	0.0005	0.0005	0.0005
Distance and field size	0.0010	0.0010	0.0010	0.0010	0.0010
Radiation correction factor	0.0005	0.0005	0.0005	0.0005	0.0005
$R_{K,\text{NRPA}} / u_{c}$	0.0097	0.0093	0.0125	0.0149	0.0116
$R_{K,STUK} / u_c$	0.0135	0.0132	0.0157	0.0149	0.0143
$R_{K,SSM} / u_c$	0.0068	0.0061	0.0104	0.0092	0.0081

Table 13 Uncertainties associated with the comparison results



Figure 2 Degrees of equivalence for each SSDL laboratory *i* with respect to the supplementary comparison reference value. The brackets show the SSDL traceabilities.

## 12. Degrees of Equivalence and discussion

Burns [8] described the analysis of the results of key comparisons in x-ray beams in terms of degrees of equivalence and this analysis was applied for the present comparison. The LNHB determination of the air-kerma calibration coefficient is taken as the supplementary comparison reference value (SCRV), for each radiation quality, except for the N-300 beam as discussed in Section 10. 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 are shown in Table 14 and in Figure 2.

The comparison results presented in Table 11 and in Figure 2 show agreement between the SSDLs and the SCRV at the level of the standard uncertainty of the comparison of 12, 14 and 8 parts in 10<sup>3</sup> for the NRPA, STUK and the SSM, respectively. These uncertainties are the mean calculated from the four radiation qualities uncertainties given in the last three rows of Table 13. The LNHB result for the N-300 quality is around 4.2 % lower than the SSDL results. The spectrum for the N-300 radiation quality at the LNHB is not measured so the correction factors are based on an older evaluation using a calculated spectrum. The clear discrepancy for N-300 seen in Figure 1 and that the SSDLs are traceable to two different primary laboratories (VSL and PTB) make it reasonable to use the mean of the three SSDL results as the SCRV for N-300 quality.

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.

The STUK participated in the supplementary comparison EUROMET.RI(I)-S3 [2] for radiation protection narrow spectrum x-ray beams. The LS01 chamber (1000 cm<sup>3</sup>) was one of the chambers used in the comparison. The differences,  $D_i$ , reported from measurement of LS01 chamber, for radiation qualities N-40, N-60, N-120 and N-300 for STUK were 1.2, 2.9, 1.0 and 0.5 per cent, respectively. This comparison likely supports lower uncertainty estimates for the STUK CMC claims in the future.

## 13. Conclusions

This is the third supplementary comparison at radiation protection level x-ray beams. The first performed in 2004, the EUROMET.RI(I)-S3 [2] and the second in 2011, the EURAMET.RI(I)-S3.1 [10]. The STUK took part in the first comparison. Both the PTB and the VSL, that are the traceable laboratories for the SSDLs, also participated in EUROMET.RI(I)-S3. The NRPA, the LNHB and the SSM have not participated in this type of comparison before.

The current supplementary comparison EURAMET.RI(I)-S3.2 for the determination of air kerma in ISO narrow beam x-rays 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 one result (N-300) from the LNHB that did not pass the chi-squared test. The present results for the STUK show lower deviations than those for the 2004 comparison.

## 14. References

- [1] ISO International Organisation for Standardisation (1996). "X and gamma reference radiation for calibrating dosemeters and doserate meters and for determining their response as a function of photon energy Part 1: Radiation characteristics and production methods." ISO 4037-1:1996(E).
- [2] Büermann L, O'Brien M, Butler D, Csete I, Gabris F, Hakanen A, Lee J-H, Palmer M, N. Saito N and Vries W de, *Comparison of national air kerma standards for ISO* 4037 narrow spectrum series in the range 30 kV to 300 kV, <u>Metrologia, 2008, 45</u>, <u>Tech. Suppl., 06013</u>.
- [3] KCDB 2014 The BIPM key comparison database is available online at <a href="http://kcdb.bipm.org/">http://kcdb.bipm.org/</a>
- [4] Evaluation of measurement data Guide to the Expression of Uncertainty in Measurement. JCGM 100:2008.

- [5] 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.
- [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 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>
- [9] Cox M. G., The evaluation of key comparison data, Metrologia, 2002, 39, 589-595
- [10] Csete I, Czap L and Gomola I, *Comparison of air kerma standards for mediumenergy x radiation between MKEH and IAEA*, <u>Metrologia, 2012, 49</u>, <u>Tech. Suppl.</u>, <u>06011</u>.
- [11] 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 <u>Metrologia 53 Tech. Suppl. 06002</u>.

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



Figure 3 The transfer chamber Exradin A6

## Table 14 Degree of equivalence for the SSDLs

Differences  $D_i$  and their expanded (k = 2) uncertainties  $U_i$  expressed in mGy / Gy. For each laboratory *i*, the degree of equivalence with respect to the SCRV the difference  $D_i$  and its expanded uncertainty  $U_i$ , were calculated.

Laboratory <i>i</i>	N-40 N-60		60	N-120		N-300		
$D_i$ and $U_i$ / mGy Gy <sup>-1</sup>	$D_i$	$U_i$	$D_i$	$U_i$	$D_i$	$U_i$	$D_i$	$U_i$
NRPA	-10.8	19.4	11.8	18.5	17.3	25.1	0.8	29.7
STUK	-5.9	27.0	-0.5	26.4	7.7	31	-3.3	29.7
SSM	-4.5	13.5	7.1	12.2	6.2	20.8	2.5	18.4

## 16. Appendix B Uncertainty budgets for the SSDLs

## *16.1.* SSDL of the NRPA

In the uncertainty budget for the radiation quality N-300 the uncertainty from the primary calibration coefficient is 2.5 % (k=2). The total combined standard uncertainty,  $1\sigma$  for this radiation quality is therefore 1.3 %.

Air kerma rate	Type A	Туре В	
	Uncertainty (%)		
1 Reference standard, set-up and radiation field			
Calibration coefficient reported by PSDL	-	0.80	
Long term stability of reference standard	0.25	-	
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	0.29	0.83	
2 Use of reference standard			
Chamber positioning (distance, orientation)	-	0.06	
Current/charge measurement including leakage	0.10	0.05	
Air temperature correction	-	-	
Air pressure correction	-	-	
Combined uncertainty in measuring with reference standard	0.10	0.08	
Combined uncertainty in air-kerma determination, <i>K</i> <sub>std</sub> (1+2)	0.31	0.83	
3 Use of transfer chamber			
Chamber positioning (distance, orientation)	-	0.06	
Current/charge measurement including leakage	0.10	0.05	
Air temperature correction	-	-	
Air pressure correction	-	-	
Combined uncertainty in measuring with tranfer chamber	0.10	0.08	
Relative combined standard uncertainty (1+2+3)	0.32	0.84	
Total uncertainty for the air-kerma calibration coefficient, $1\sigma$	0.	90	

# *16.2. SSDL of the STUK*

Air kerma rate	Туре А	Туре В
	Uncertainty (%)	
1 Reference standard, set-up and radiation field		
Calibration coefficient reported by PSDL	_	0.60
Long term stability of reference standard		0.58
Spectral difference of SSDL and PSDL	-	0.58
Difference in radial non-uniformity of the beam and field size		0.58
Combined uncertainty of reference standard and setup		1.17
2 Use of reference standard		
Chamber positioning (distance, orientation)	-	0.29
Current/charge measurement including leakage	0.20	0.06
Air temperature correction		0.08
Air pressure correction	-	0.02
Combined uncertainty in measuring with reference standard	0.20	0.31
Combined uncertainty in air-kerma determination, $K_{\rm std}$ (1+2)	0.20	1.21
3 Use of transfer chamber		
Chamber positioning (distance, orientation)	-	0.29
Current/charge measurement including leakage	0.20	
Air temperature correction	-	0.08
Air pressure correction	-	0.02
Combined uncertainty in measuring with tranfer chamber	0.20	0.30
Relative combined standard uncertainty (1+2+3)	0.28	1.25
Total uncertainty for the air-kerma calibration coefficient, $1\sigma$	1.	28

# 16.3. SSDL of the SSM

Air kerma rate	Туре А	Туре В		
	Uncertainty (%)			
1 Reference standard, set-up and radiation field				
Calibration coefficient reported by PSDL		0.39		
Long term stability of reference standard	0.10			
Spectral difference of SSDL and PSDL	-	0.10		
Drift in HVL		0.06		
Short time stability in air kerma rate	0.02			
Difference in radial non-uniformity of the beam and field size		0.17		
Combined uncertainty of reference standard and setup	0.10	0.44		
2 Use of reference standard				
Chamber positioning (distance, orientation)		0.06		
Current/charge measurement including leakage	0.20	0.17		
Uncertainty in HVL		0.05		
Interpolating $N_{\kappa}$ (there are diference in HVL between SSM and PSDL)		0.03		
Air temperature correction	-	-		
Air pressure correction	-	-		
Combined uncertainty in measuring with reference standard	0.20	0.19		
Combined uncertainty in air-kerma determination, K <sub>std</sub> (1+2)	0.22	0.48		
3 Use of transfer chamber				
Chamber positioning (distance, orientation)		0.06		
Current/charge measurement including leakage	0.20	0.02		
Air temperature correction	-	-		
Air pressure correction	-	-		
Combined uncertainty in measuring with tranfer chamber	0.20	0.06		
Relative combined standard uncertainty (1+2+3)	0.30	0.48		
Total uncertainty for the air-kerma calibration coefficient, 1o 0.		57		