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Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the ENEA-INMRI, Italy and the BIPM in low-energy x-rays

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Abstract A key comparison has been made between the air-kerma standards of the ENEA-INMRI, Italy and the BIPM in the low-energy x-ray range. The results show the standards to be in agreement at the level of the standard uncertainty for the comparison of 1.9 parts in 10^3. No significant trend with radiation quality is observed. The results are analysed and presented in terms of degrees of equivalence, suitable for entry in the BIPM key comparison database.

1. Introduction

A direct comparison has been made between the air-kerma standards of the Istituto Nazionale di Metrologia delle Radiazioni Ionizzanti (INMRI) of the Agenzia Nazionale per le Nuove Tecnologie, l'Energia e lo Sviluppo Economico Sostenibile (ENEA), Italy and the Bureau International des Poids et Mesures (BIPM) in the x-ray range from 10 kV to 50 kV. The comparison took place at the BIPM in January 2011 using the reference conditions recommended by the CCRI [1].

2. Determination of the air-kerma rate

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

$$\dot{K} = \frac{I}{\rho_{\mathrm{air}} V} \frac{W_{\mathrm{air}}}{e} \frac{1}{1 - g_{\mathrm{air}}} \prod_i k_i \tag{1}$$

where ρ_{air} is the density of air under reference conditions, I is the ionization current under the same conditions, W_{air} is the mean energy expended by an electron of charge e to produce an ion pair in air, g_{air} is the fraction of the initial electron energy lost through radiative processes in air, and $\Pi\, k_{\mathrm{i}}$ is the product of the correction factors to be applied to the standard.

The values used for the physical constants ρ_{air} and W_{air}/e are given in Table 1. 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 and the value given in the table[1].

3. Details of the standards

Both free-air chamber standards are 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 BIPM air-kerma standard is described in [2] and the changes made to certain

[1] For an air temperature $T \sim 293$ K, pressure P and relative humidity ~50 % in the measuring volume, the correction for air density involves a temperature correction T / T_0, a pressure correction P_0 / P and a humidity correction $k_{\mathrm{h}} = 0.9980$. In addition, a factor 1.0002 is included to account for the compressibility of dry air between $T \sim 293$ K and $T_0 = 273.15$ K.

correction factors in October 2003 and September 2009 given in [3, 4] and the references therein. The ENEA standard was previously compared with the BIPM standard in a direct comparison carried out at the BIPM in 1998, the results of which are reported in [5]. The main dimensions, the measuring volume and the polarizing voltage for each standard are shown in Table 2.

Table 1. Physical constants used in the determination of the air-kerma rate

Constant	Value	u_i [a]
ρ_{air}	1.2930 kg m^{-3}	0.0001
W_{air} / e	33.97 J C^{-1}	0.0015

a u_i is the relative standard uncertainty.

Table 2. Main characteristics of the standards

Standard	BIPM	ENEA
Aperture diameter / mm	9.941	8.014
Air path length / mm	100.0	65.12 [a]
Collecting length / mm	15.466	40.738
Electrode separation / mm	70	60
Collector width / mm	71	60
Measuring volume / mm^3	1200.4	2054.9
Polarizing voltage / V	+1500	+1600

a This is the value 64.30 mm plus 0.82 mm due to three screws supporting the aperture.

3. Comparison procedure

3.1 The BIPM irradiation facility and reference beam qualities

The comparison was carried out in the BIPM low-energy x-ray laboratory, which houses a constant-potential generator and a tungsten-anode x-ray tube with an inherent filtration of 1 mm beryllium. A beryllium filter of thickness 2.16 mm is added (for all radiation qualities) so that the half-value layer (HVL) of the present 10 kV radiation quality matches that of the original BIPM x-ray tube when the same aluminium filter is used. A voltage divider is used to measure the generating potential, which is stabilized using an additional feedback system of the BIPM. Rather than use a transmission monitor, the anode current is measured and the ionization chamber current normalized for any deviation from the reference anode current. The resulting variation in the BIPM free-air chamber current over the duration of a comparison is normally not more than 3×10^{-4} in relative value. The radiation qualities used in the range from 10 kV to 50 kV are those recommended by the CCRI [1] and are given in Table 3 in ascending HVL from left to right.

The irradiation area is temperature controlled at around 20 °C and is stable over the duration of a calibration to better than 0.1 °C. Two thermistors, calibrated to a few mK, measure the temperature of the ambient air and the air inside the BIPM standard. Air pressure is measured by means of a calibrated barometer positioned at the height of the beam axis. The relative humidity is controlled within the range from 47 % to 53 %.

Table 3. Characteristics of the BIPM reference radiation qualities

Radiation quality	10 kV	30 kV	25 kV	50 kVb	50 kVa
Generating potential / kV	10	30	25	50	50
Additional Al filtration / mm	0	0.208 2	0.372 3	1.008 2	3.989
Al HVL / mm	0.037	0.169	0.242	1.017	2.262
$(\mu/\rho)_{air}$[a] / $cm^2\ g^{-1}$	14.84	3.661	2.604	0.753	0.378
$\dot{K}_{BIPM}$ / $mGy\ s^{-1}$	1.00	1.00	1.00	1.00	1.00

a Measured for an air path length of 100 mm using a variable-pressure tube.

3.2 Correction factors

The correction factors applied to the ionization current measured at each radiation quality, together with their associated uncertainties, are given in Table 4 for the BIPM standard and in Table 5 for the ENEA standard.

Table 4. Correction factors for the BIPM standard

Radiation quality	10 kV	30 kV	25 kV	50 kVb	50 kVa	u_{iA}	u_{iB}
Air attenuation k_a[a]	1.195 7	1.045 1	1.031 9	1.009 1	1.004 6	0.000 2	0.000 1
Scattered radiation k_{sc}[b]	0.9962	0.9972	0.9973	0.9977	0.9979	-	0.000 3
Fluorescence k_{fl}[b]	0.9952	0.9971	0.9969	0.9980	0.9985	-	0.000 5
Electron loss k_e	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.000 1
Ion recombination k_s	1.000 6	1.000 7	1.000 7	1.000 7	1.000 7	0.000 1	0.000 1
Polarity k_{pol}	1.000 5	1.000 5	1.000 5	1.000 5	1.000 5	0.000 1	-
Field distortion k_d	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.000 7
Diaphragm effects k_{dia}[c]	0.999 9	0.999 5	0.999 6	0.998 9	0.998 4	-	0.000 3
Wall transmission k_p	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	0.000 1	-
Humidity k_h	0.998 0	0.998 0	0.998 0	0.998 0	0.998 0	-	0.000 3
$1 - g_{air}$	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.000 1

a Values for 293.15 K and 101.325 kPa; each measurement is corrected using the air density measured at the time.
b Values for k_{sc} and k_{fl} adopted in October 2003, based on Monte Carlo calculations.
c Correction factor k_{dia} for diaphragm transmission, scatter and fluorescence adopted September 2009, replacing the factor k_l. See reference [6].

The largest correction at low energies is that due to the attenuation of the x-ray fluence along the air path between the reference plane and the centre of the collecting volume. The correction factor k_a is evaluated using the measured mass attenuation coefficients $(\mu/\rho)_{air}$ given in Table 3. In practice, the values used for k_a take account of the temperature and pressure of the air in the standard at the time of the measurements. The value for k_a for the ENEA chamber at 10 kV has been increased by the factor 1.001 0 to account for the larger mean air-attenuation coefficient for an air path length of 65 mm (the values given in Table 3 were measured at the BIPM for an air path length of 100 mm). This effect is negligible at the other radiation qualities. Ionization measurements are also corrected for changes in air attenuation arising from variations in the temperature and pressure of the ambient air between the radiation source and the reference plane.

Measurements using the BIPM standard were made using positive polarity only. A correction factor of 1.000 5 was applied to correct for the known polarity effect in the standard. Similarly, measurements using the ENEA standard were made using positive polarity only. A correction factor of 0.999 9(1) was applied based on the results of additional measurements at the BIPM. These results are consistent with those made at the BIPM for the previous comparison in 1998. The mean polarity correction factor of 0.999 4 measured at the ENEA, with a statistical uncertainty approaching 1 part in 10^3, has not been used for the present comparison.

All measured ionization currents are corrected for ion recombination. The measured values for the ion recombination correction k_s for the BIPM standard are given in Table 4. For the ENEA standard, the values for k_s given in Table 5 for the BIPM air-kerma rates are derived from the equation $k_s = 1 / (1 - a - bI_{tr})$, where $a = 4.46 \times 10^{-4}$ and $b = 1.92 \times 10^{-6}$ pA^{-1} were determined at the ENEA and I_{tr} is the measured ionization current in pA.

Table 5. Correction factors for the ENEA standard

Radiation quality	10 kV	30 kV	25 kV	50 kVb	50 kVa	u_{iA}	u_{iB}
Air attenuation k_a [a]	1.124 6	1.029 1	1.020 6	1.005 9	1.003 0	0.000 2	0.000 1
Scattered radiation k_{sc}	0.997 1	0.997 8	0.998 0	0.998 4	0.998 6	-	0.000 6
Fluorescence k_{fl}	0.996 1	0.997 2	0.997 5	0.998 3	0.998 8		0.000 6
Electron loss k_e	1.000 0	1.000 0	1.000 0	1.000 0	1.000 1	-	0.000 2
Ion recombination k_s	1.000 6	1.000 6	1.000 6	1.000 6	1.000 6	-	0.000 5
Polarity k_{pol}	0.999 9	0.999 9	0.999 9	0.999 9	0.999 9	0.000 1	-
Field distortion k_d	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.001 0
Aperture edge transmission k_l	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.000 5
Wall transmission k_p	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.000 5
Humidity k_h	0.998 0	0.998 0	0.998 0	0.998 0	0.998 0	-	0.000 3
$1 - g_{air}$	1.000 0	1.000 0	1.000 0	1.000 0	1.000 0	-	0.000 1

a Values for 293.15 K and 101.325 kPa, determined using the BIPM values for the air-attenuation coefficient; each measurement is corrected using the air density measured at the time.

3.3 Chamber positioning and measurement procedure

The ENEA chamber was positioned close to the BIPM chamber and both remained fixed throughout the comparison; the alternation of measurements between chambers was carried out by displacement of the radiation source. Alignment on the beam axis was measured to around 0.1 mm and this position was reproducible to better than 0.01 mm. No correction is applied for the radial non-uniformity of the beam; for apertures of diameter 8 mm and 10 mm, the non-uniformity correction is the same at the level of 1 part in 10^4 (included as an uncertainty in Table 6). The reference plane for each chamber was positioned at 500 mm from the radiation source for all qualities. This distance was measured to 0.03 mm and was reproducible to better than 0.01 mm. The beam diameter in the reference plane is 45 mm for all qualities.

The air temperature for the ENEA chamber was measured using a BIPM mercury thermometer calibrated to better than 0.1 K and positioned in the holder of the ENEA chamber; an additional uncertainty component of 3 parts in 10^4 is included for ionization current in Table 6. The leakage current was measured before and after each series of ionization current measurements and a correction made based on the mean of these leakage measurements. For the BIPM chamber the leakage current, relative to the ionization current of around 40 pA, was less than 1 part in 10^4 and for the ENEA chamber, with measured current around 70 pA, the relative leakage was even less.

For the ENEA standard, the standard uncertainty of the mean of a series of seven measurements, each with integration time 60 s, was around 1 part in 10^4. Two such series were made for the comparison at each beam quality. For the BIPM standard, a similar series was made for each beam quality with a standard uncertainty also around 1 part in 10^4. For each of the radiation qualities (except 50 kVa), the comparison was repeated on a subsequent day (the chambers remaining fixed in position) and the two comparison results for each quality agreed to better than 3 parts in 10^4 (included as an uncertainty for short-term stability in Table 6).

Table 6. Uncertainties associated with the comparison results

Standard	BIPM		ENEA	
Relative standard uncertainty	$u_{i\mathrm{A}}$	$u_{i\mathrm{B}}$	$u_{i\mathrm{A}}$	$u_{i\mathrm{B}}$
Ionization current	0.000 2	0.000 2	0.000 2	0.000 4[a]
Positioning	0.000 1	0.000 1	0.000 1	0.000 1
Volume	0.000 3	0.000 5	-	0.000 5
Correction factors (excl. k_h)	0.000 3	0.001 0	0.000 2	0.001 6
Humidity k_h	-	0.000 3	-	0.000 3
Physical constants	-	0.001 5	-	0.001 5
$\dot{K}_\mathrm{Standard}$	0.000 5	0.001 9	0.000 3	0.002 3[a]
	0.002 0		0.002 3[a]	
Radial non-uniformity	0.000 1			
Short-term reproducibility	0.000 3			
$\dot{K}_\mathrm{ENEA}/\dot{K}_\mathrm{BIPM}$	u_c = 0.001 9[b]			

a For ionization current measurements at the ENEA, the $u_{i\mathrm{B}}$ component of 0.000 4 is replaced by 0.001 6. Consequently, the uncertainty of the air-kerma determination at the ENEA is 0.002 8, rather than the value 0.002 3 tabulated here. It is the higher value that appears as $u_{\mathrm{Lab}i}$ in the KCDB.
b Takes account of correlation in the type B uncertainties.

4. Uncertainties

The uncertainties associated with the primary standards and with the results of the comparison are listed in Table 6. The uncertainties associated with the measurement of the ionization current and with chamber positioning are those which apply to measurements at the BIPM.

The combined standard uncertainty u_c of the ratio $\dot{K}_{\mathrm{ENEA}} / \dot{K}_{\mathrm{BIPM}}$ takes into account correlation in the type B uncertainties associated with the humidity correction and the physical constants. Correlation in the values for k_{sc} and k_{fl} is taken into account in an approximate way by assuming half of the uncertainty value for each factor at each laboratory. This is consistent with the analysis presented in [7].

5. Results and discussion

The comparison results are given in Table 7. General agreement at the level of 2 parts in 10^3 is observed, consistent with the standard uncertainty for the comparison of 1.9 parts in 10^3 given in Table 6. There is no evidence of a significant trend in the results for the different radiation qualities, although the result for 30 kV is notably around 1 part in 10^3 lower which is curious in view of the smooth variation of the correction factors with radiation quality. A more likely explanation is a smooth decrease in the comparison result with decreasing HVL combined with a slight increase at 10 kV due to the difficulty in correcting for air attenuation at this radiation quality. It is noted that a diaphragm correction $\underline{k}_{dia}$ is applied to the BIPM standard (Table 4), which includes the effects of photon scatter and fluorescence from the diaphragm [6]. No corresponding corrections are currently applied to the ENEA standard; such corrections would have a tendency to reduce the observed variation between 30 kV and 50 kVa.

Also shown in Table 7 are the results of the direct comparison in 1998 between the two standards [5]. These results have been updated for the changes made to the standards in the interim, notably the adoption of the Monte Carlo method for the evaluation of k_{sc} and k_{fl} and the inclusion of the diaphragm correction k_{dia} for the BIPM standard. This analysis shows the very high stability of the standards over the past twelve years.

Table 7. Comparison results

Radiation quality	10 kV	30 kV	25 kV	50 kVb	50 kVa
$\dot{K}_{\mathrm{ENEA}} / \dot{K}_{\mathrm{BIPM}}$	**0.997 8**	**0.996 8**	**0.997 6**	**0.998 0**	**0.997 9**
Revised 1998 comparison	0.998 9	0.997 4	0.998 2	0.998 3	0.998 5

6. Degrees of Equivalence

The analysis of the results of BIPM comparisons in low-energy x-rays in terms of degrees of equivalence is described in [7]. Following a decision of the CCRI, the BIPM determination of the air-kerma rate is taken as the key comparison reference value, for each of the CCRI radiation qualities. It follows that for each laboratory i having a BIPM comparison result x_i with combined standard uncertainty u_i, the degree of equivalence with respect to the reference value is the relative difference $D_i = (K_i - K_{\mathrm{BIPM},i}) / K_{\mathrm{BIPM},i} = x_i - 1$ and its expanded uncertainty $U_i = 2\,u_i$. The results for D_i and U_i, expressed in mGy/Gy and including those of the present comparison, are shown in Table 8 and in Figure 1.

The degree of equivalence of laboratory i with respect to each laboratory j that has taken part in a BIPM comparison is the difference $D_{ij} = D_i - D_j = x_i - x_j$ and its expanded uncertainty $U_{ij} = 2\,u_{ij}$. The combined standard uncertainty u_{ij} is mainly the combined uncertainty of the air-kerma rate determinations for laboratories *i* and *j*. In evaluating each u_{ij}, correlation between the standards is removed, notably that arising from k_{sc} and k_{fl}. As described in [7], if correction factors based on Monte Carlo calculations are used by both laboratories, or by neither, then half the uncertainty value is taken for each factor. Note that the uncertainty of the BIPM determination of air-kerma rate does not enter in u_{ij}, although the uncertainty arising from the comparison procedure is included. The results for D_{ij} and U_{ij} when *j* represents the ENEA are also given in Table 8 and in Figure 2. Note that the data presented in the tables, while correct at the time of publication of the present report, become out of date as laboratories make new comparisons with the BIPM. The formal results under the CIPM MRA are those available in the BIPM key comparison database.

7. Conclusions

The key comparison BIPM.RI(I)-K2 for the determination of air kerma in low-energy x-rays shows the standards of the ENEA and the BIPM to be in agreement at the level of the standard uncertainty for the comparison of 1.9 parts in 10^3. The results are in very close agreement with those of the 1998 comparison between the two standards when the changes made to the standards are taken into account. Tables and graphs of degrees of equivalence, including those for the ENEA, are presented for entry in the BIPM key comparison database.

Table 8. Degrees of equivalence. For each laboratory i, the degree of equivalence with respect to the key comparison reference value is the difference D_i and its expanded uncertainty U_i, and with respect to laboratory j is the difference D_{ij} and its expanded uncertainty U_{ij}. Here j represents the ENEA. Tables formatted as they appear in the BIPM key comparison database.

10 kV

Lab i	D_i /(mGy/Gy)	U_i /(mGy/Gy)	D_{ij} /(mGy/Gy)	U_{ij} /(mGy/Gy)
GUM	-0.9	5.3	1.3	5.9
VSL	0.0	4.7	2.2	5.3
NPL	1.1	4.8	3.3	5.4
NIST	-4.4	5.1	-2.2	5.6
METAS	2.2	3.5	4.4	4.3
VNIIM	-4.0	5.4	-1.8	5.8
PTB	2.8	5.0	5.0	5.5
BEV	-1.2	4.8	1.0	5.4
MKEH	1.0	4.0	3.2	4.6
NMIJ	0.9	3.6	3.1	4.2
NRC	0.3	5.5	2.5	6.0
ARPANSA	-1.5	14.0	0.7	14.1
ENEA	-2.2	3.8		

30 kV

Lab i	D_i /(mGy/Gy)	U_i /(mGy/Gy)	D_{ij} /(mGy/Gy)	U_{ij} /(mGy/Gy)
GUM	-1.0	5.3	2.2	5.9
VSL	0.1	4.7	3.3	5.3
NPL	-0.3	4.8	2.9	5.4
NIST	-5.2	5.1	-2.0	5.6
METAS	1.0	3.5	4.2	4.3
VNIIM				
PTB	-3.1	5.0	0.1	5.5
BEV	-2.4	4.8	0.8	5.4
MKEH	0.4	4.0	3.6	4.6
NMIJ	1.0	3.6	4.2	4.2
NRC	1.3	5.5	4.5	6.0
ARPANSA	-2.5	7.5	0.7	7.8
ENEA	-3.2	3.8		

25 kV

Lab i	D_i /(mGy/Gy)	U_i /(mGy/Gy)	D_{ij} /(mGy/Gy)	U_{ij} /(mGy/Gy)
GUM				
VSL				
NPL	1.4	4.8	3.8	5.4
NIST	-4.9	5.1	-2.5	5.6
METAS	1.3	3.5	3.7	4.3
VNIIM	-3.3	5.4	-0.9	5.8
PTB	-1.8	5.0	0.6	5.5
BEV	-1.2	4.8	1.2	5.4
MKEH	1.0	4.0	3.4	4.6
NMIJ	3.2	3.6	5.6	4.2
NRC	0.7	5.5	3.1	6.0
ARPANSA	-2.6	7.5	-0.2	7.8
ENEA	-2.4	3.8		

50 kVb

Lab i	D_i /(mGy/Gy)	U_i /(mGy/Gy)	D_{ij} /(mGy/Gy)	U_{ij} /(mGy/Gy)
GUM	-1.4	5.3	0.6	5.9
VSL	0.2	4.7	2.2	5.3
NPL				
NIST	-5.2	5.1	-3.2	5.6
METAS	0.2	3.5	2.2	4.3
VNIIM	-1.6	5.4	0.4	5.8
PTB	-1.3	5.0	0.7	5.5
BEV	-1.0	4.8	1.0	5.4
MKEH	1.6	4.0	3.6	4.6
NMIJ	4.6	3.6	6.6	4.2
NRC	4.3	5.5	6.3	6.0
ARPANSA	-1.0	7.5	1.0	7.8
ENEA	-2.0	3.8		

50 kVa

Lab i	D_i /(mGy/Gy)	U_i /(mGy/Gy)	D_{ij} /(mGy/Gy)	U_{ij} /(mGy/Gy)
GUM	-0.7	5.3	1.4	5.9
VSL	-2.1	4.7	0.0	5.3
NPL	-0.7	4.8	1.4	5.4
NIST	-3.5	5.1	-1.4	5.6
METAS	0.1	3.5	2.2	4.3
VNIIM	-0.1	5.4	2.0	5.8
PTB	-0.2	5.0	1.9	5.5
BEV	-1.0	4.8	1.1	5.4
MKEH	1.2	4.0	3.3	4.6
NMIJ	5.4	3.6	7.5	4.2
NRC	3.3	5.5	5.4	6.0
ARPANSA	0.2	7.5	2.3	7.8
ENEA	-2.1	3.8		

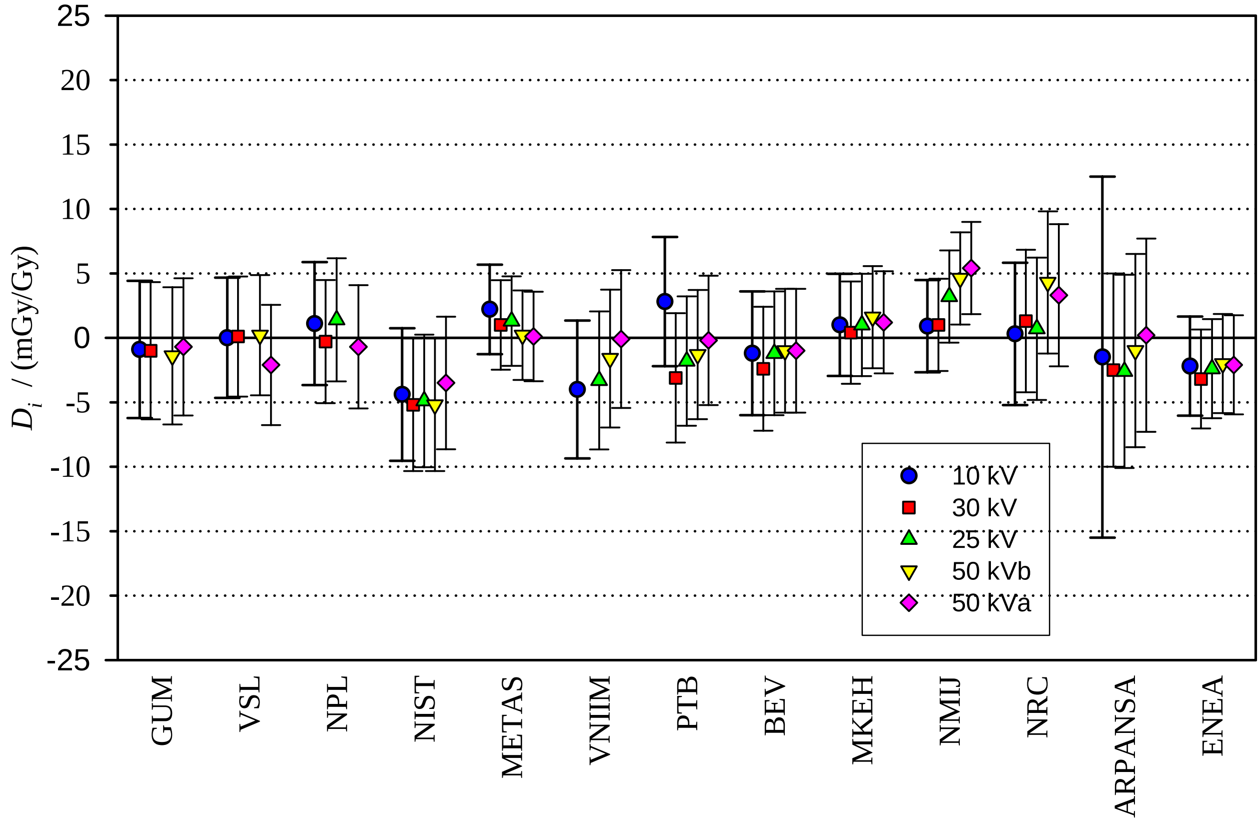

Figure 1. Degrees of equivalence for each NMI *i* with respect to the key comparison reference value

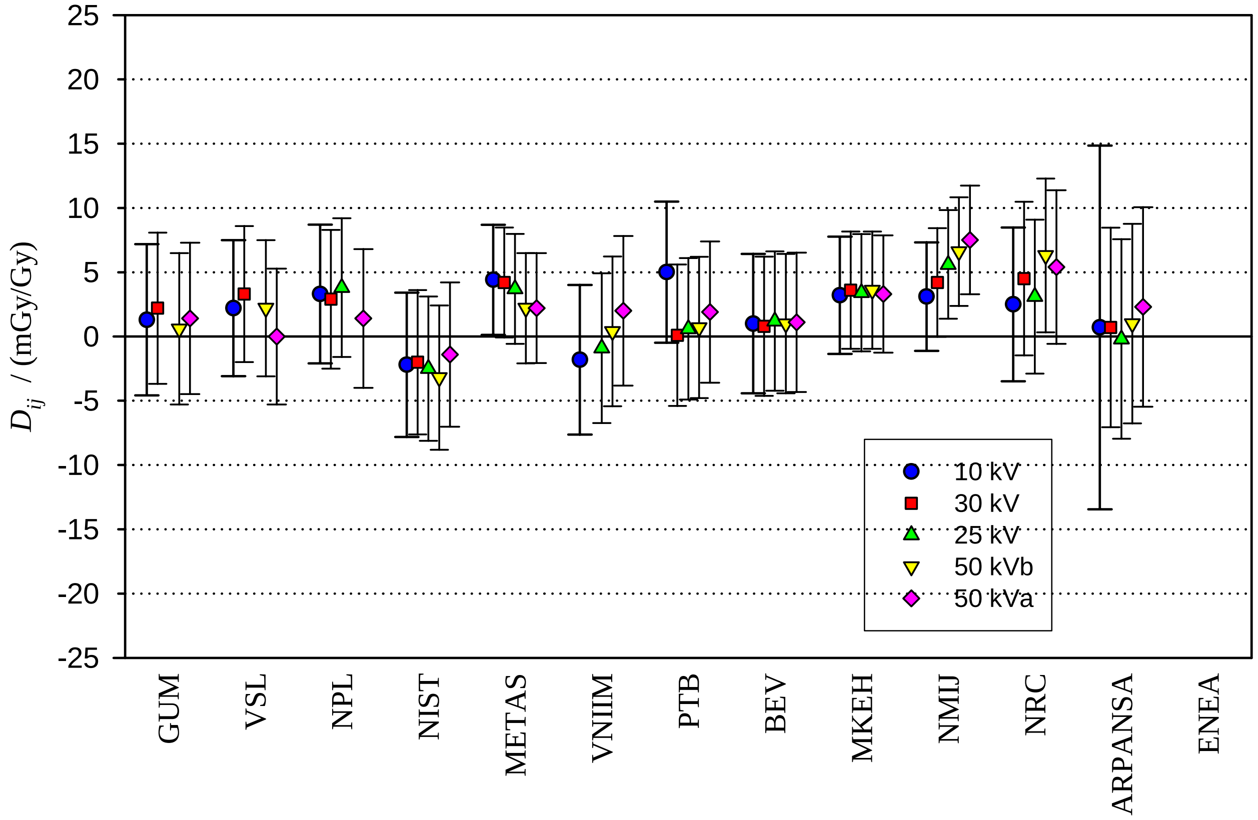

Figure 2. Degrees of equivalence for each NMI *i* with respect to the ENEA

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