

Synchrotron radiation-based bilateral intercomparison of ultraviolet source calibrations

U Arp¹, R Klein², Z Li¹, W Paustian², M Richter², P-S Shaw¹ and R Thornagel²

¹ Electron and Optical Physics Division, National Institute of Standards and Technology, 100 Bureau Dr, MS 8410, Gaithersburg, MD 20899-8410, USA

² Physikalisch-Technische Bundesanstalt, Abbestraße 2-12, D-10587 Berlin, Germany

E-mail: uwe.arp@nist.gov

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Abstract

We report on a successful bilateral intercomparison between the Physikalisch-Technische Bundesanstalt (PTB) and the National Institute of Standards and Technology (NIST). In both laboratories deuterium lamps were calibrated using the calculability of synchrotron radiation. However, the methods applied in the two laboratories differed significantly. Whereas the calibrations at PTB were performed in vacuum and led to spectral radiant intensities, the measurements at NIST were done in air and resulted in spectral irradiances. Therefore, for this bilateral comparison we not only proved the equivalence of the two measurement procedures, but also the validity of the conversion between the different spectro-radiometric quantities. This successful comparison re-establishes the usefulness of deuterium lamps as transfer standards in the ultraviolet spectral range.

1. Introduction

Since its discovery by Ritter in 1801 [1], ultraviolet (UV) radiation has affected many areas of our life. Initially dubbed chemical radiation for its capability to induce change in many compounds, it was later called UV to complement the infrared radiation discovered by Herschel [2, 3]. UV radiation in the wavelength range from 200 nm to 400 nm is called air-UV, because it can propagate through air. The Schumann–Runge absorption bands [4–7] of oxygen make air opaque to UV radiation with wavelengths shorter than 200 nm and it is therefore called vacuum-ultraviolet (VUV).

First studies by Widmark in 1889 showed the effects of UV exposure of the human skin [8]. While UV-B (280 nm to 315 nm) is absorbed in the top layer of the skin, the epidermis, and causes sunburn, the more dangerous UV radiation to the human body is UV-A between 315 nm and 400 nm [9], because it penetrates into the deeper lying dermis and can cause genetic mutations and skin cancer. The germicidal effects of UV were discovered early on by Downes and Blunt [10], which led later to the discovery of phototherapy by Finsen in 1895 [11]. The shorter wavelength UV-C with wavelengths from

100 nm to 280 nm is completely absorbed by molecules in the upper atmosphere, especially ozone. This fact attracted interest when in 1974 Molina and Rowland [12] discovered that chlorofluorocarbons (CFCs) can break down atmospheric ozone under short wavelength UV irradiation and again in 1985 when Farman, Gardiner and Shanklin discovered the Antarctic ozone hole [13], potentially exposing mankind to the even greater amounts of dangerous UV radiation. This led to the establishment of environmental UV monitoring networks around the world.

But UV radiation is not only important to human health, it also is an indispensable factor in technological advancement. The microelectronics revolution that brought us personal computers [14] and hand-held devices, such as cellular telephones [15], would not have been possible without pushing the limits of photolithography by decreasing the wavelengths in order to reduce the size of electronic components. Other industrial applications of UV include the photoinitiated conversion of inks, coatings and adhesives and germicidal applications such as the disinfection of water for human consumption.

UV radiation is also crucial in many areas of science. It is the most important part of the electromagnetic spectrum

in investigations of the sun and sun–Earth interactions. Scientific applications of UV include astrophysics [16], heliophysics [17], planetary science [18, 19], plasma physics [20], atmospheric science [12, 13], biophysics [21], materials science [22] and materials processing [23].

Because of this ever increasing interest in UV radiation and its properties, national measurement laboratories like the Physikalisch-Technische Bundesanstalt (PTB) and the National Institute of Standards and Technology (NIST) provide UV source and detector transfer standards and related calibration services. In addition, not only the absolute calibration of transfer standards is important, but also the accurate conversion of different spectro-radiometric quantities into each other. The work reported in this paper is an example of a successful bilateral comparison between NIST and PTB in which deuterium lamps are used as transfer standards, but different calibrations are performed at the two laboratories. The PTB at its laboratory at the BESSY II electron storage ring [24] performs calibrations in vacuum that result in spectral radiant intensities, whereas at NIST using SURF III spectral irradiances are measured in air. Intercomparisons, like the present one, have been successfully performed for more than 30 years [25].

The lamps used in this comparison were three Cathodeon V04³ lamps with serial numbers AR094, BQ051 and BQ056. The first test was done with the lamp BQ056, which was calibrated at BESSY II in November 2002.

2. Spectro-radiometric quantities

2.1. Spectral irradiance, spectral radiance and spectral radiant intensity

This comparison depends on the reliable conversion of different spectro-radiometric quantities into each other. Measurements at SURF deliver spectral irradiances E_λ and measurements at BESSY yield spectral radiant intensities I_λ . Therefore, it is necessary to introduce these different spectro-radiometric quantities and their relation to each other (Φ is the radiant power and λ the wavelength).

The *spectral radiant intensity* I_λ is the spectral radiant power $d\Phi(\lambda)/d\lambda$ of a point source per solid angle Ω [26, 27]:

$$I_\lambda = \frac{dI(\lambda)}{d\lambda} = \frac{d^2\Phi(\lambda)}{d\Omega d\lambda}, \quad (1)$$

where I is the radiant intensity. The *spectral radiance* L_λ is the spectral radiant power $d\Phi(\lambda)/d\lambda$ per solid angle Ω and per effective source area A_S of an extended source:

$$L_\lambda = \frac{dL(\lambda)}{d\lambda} = \frac{d^3\Phi(\lambda)}{dA_S d\Omega d\lambda}, \quad (2)$$

where L is the radiance. The *spectral irradiance* E_λ is the spectral radiant power $d\Phi(\lambda)/d\lambda$ per unit irradiated area A of

³ Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for the purpose.

Table 1. Horizontal full width at half maximum (FWHM_x) and vertical (FWHM_y) source sizes for the three deuterium lamps, measured at 334 nm.

Lamp	FWHM _x /mm	FWHM _y /mm
AR094	1.266 ± 0.002	1.227 ± 0.003
BQ051	1.242 ± 0.007	1.192 ± 0.005
BQ056	1.203 ± 0.002	1.136 ± 0.001

Table 2. Error in the solid angle determination for different distances. The source size is assumed to be 2.5 mm and the aperture radius is $r = 3.9895$ mm. $d\Omega$ is the exact solid angle, which takes into account the finite source and detector sizes [29], and $d\Omega_{ps} = \frac{\pi r^2}{d^2}$ is the point source approximated solid angle.

Distance d /mm	$d\Omega/10^{-4}$ sr	$d\Omega_{ps}/10^{-4}$ sr	$\frac{d\Omega - d\Omega_{ps}}{d\Omega_{ps}}$
372	3.612 932	3.613 274	$-9.471\ 807 \times 10^{-5}$
472	2.244 281	2.244 413	$-5.883\ 740 \times 10^{-5}$
500	1.999 973	2.000 077	$-5.243\ 253 \times 10^{-5}$
572	1.528 190	1.528 251	$-4.006\ 408 \times 10^{-5}$

any source:

$$E_\lambda = \frac{dE(\lambda)}{d\lambda} = \frac{d^2\Phi(\lambda)}{dA d\lambda}, \quad (3)$$

where E is the irradiance.

2.2. Conversion of spectro-radiometric quantities

The relevant quantities for this comparison are the spectral irradiance E_λ and the spectral radiant intensity I_λ . For a point source at distance d we obtain:

$$I_\lambda = \frac{d^2\Phi(\lambda)}{d\Omega d\lambda}. \quad (4)$$

Assuming $d\Omega = \frac{dA}{d^2}$ this results in

$$I_\lambda = \frac{d^2\Phi(\lambda)}{dA d\lambda} d^2 = E_\lambda d^2. \quad (5)$$

The error in the solid angle, introduced by the point source assumption, is less than 10^{-4} and negligible, as seen in table 2.

2.3. Point source approximation

In order to determine the error introduced by the assumption of a point source in the conversion of the spectral irradiance into spectral radiant intensity, the source sizes of the three lamps were measured (see table 1). The measurement system was the same as used in the determination of the electron beam size at SURF [28]. All the measurements were performed using an interference filter with a centre wavelength of $\lambda_0 = 334$ nm and a bandwidth of $\Delta\lambda = 10$ nm.

Following [29] one can estimate the error introduced by the finite source size. This error introduced in the solid angle determination by the point source approximation is negligible, as shown in table 2.

Table 3. Relative expanded ($k = 2$) uncertainty for the measurements performed at NIST (see table A1 for more details).

SURF radiation	0.72%
Wavelength	0.27%
Stray light and fluorescence	0.5%
Lamp distance	0.13%
Random uncertainty	0.6%
Lamp stability	2.0%
<i>Combined</i>	2.4%

3. Experiments

3.1. SURF III/NIST

3.1.1. Measurement procedure. The beamline used for synchrotron radiation-based source calibrations at SURF was recently described in great detail [30]. This new experimental station was the basis for the NIST contribution to the Consultative Committee for Photometry and Radiometry (CCPR) key comparison CCPR-K1.b ‘Spectral irradiance from 200 nm to 350 nm’ [31, 32]. In short, polychromatic or white synchrotron radiation is used to calibrate a monochromator-detector system, which is subsequently used to determine the irradiance of a deuterium lamp.

3.1.2. Setup. Great care has been taken to ensure reproducible lamp positioning and conditioning. Therefore, the lamps are positioned by centring a laser in the exit window. After that, the lamp direction is adjusted, so that the laser retroreflects onto itself. This is in general an iterative process that has to be repeated a few times. Then, the distance between the entrance aperture of the integrating sphere and the exit window of the lamp is determined using a tubular inside micrometer. Before the lamp is started, the front window is wiped with isopropanol. After that the lamp is switched on and operated for about 60 min to ensure stable operation.

3.1.3. Uncertainties. The uncertainty budget for the setup is described in detail in appendix A.1 and [30]. The main difference in comparison is that the Cathodeon lamps are less stable than the lamps used in the CCPR key comparison. Therefore the combined relative expanded uncertainty for the spectral irradiance measurements at SURF is 2.4% (see table 3).

3.2. BESSY II/PTB

3.2.1. Measurement procedure. The spectral intensity calibration of the lamps was performed at the PTB laboratory at BESSY II [24]. The employed normal-incidence monochromator station is dedicated to the calibration of radiation sources in the spectral range from 40 nm to 400 nm and is described in detail in [33, 34]. It consists—in addition to apertures, slits and filters—of a spherical mirror and a spherical grating that images the source point on to a photomultiplier detector. The station can be equipped with different optical components depending on the spectral range covered. Operated to cover the spectral range of interest for

Table 4. Relative expanded ($k = 2$) uncertainty for the measurements performed at PTB.

BESSY II radiation	0.6%
Wavelength	0.7%
Inhomogeneity of mirror reflectance	2.8%
Distance measurements, angular acceptance of station	1.4%
Stability of station, polarization effects, detector noise and non-linearity	2.3%
Lamp stability	2.0%
<i>Combined</i>	4.5%

this comparison, a MgF₂ coated Al mirror and a MgF₂ coated Al grating with 600 lines mm⁻¹ and 150 nm blaze angle were used. The station is calibrated traceably to the calculable synchrotron radiation of the BESSY II electron storage ring operated as a primary source standard [35]. Moreover, the entire station can be rotated by 90° in order to take into account the polarization of the synchrotron radiation. The calibration with synchrotron radiation is performed with a reduced stored electron beam current in order to achieve similar photomultiplier signals as with the deuterium lamp. After calibration, the station can be translated to the deuterium lamp. This calibration principle is described in more detail in [36].

3.2.2. Setup. All calibrations are performed under vacuum conditions. The alignment of the lamp to the optical axis of the monochromator station must be done with great care. For this a laser beam, which defines the optical axis, is used and the lamp is positioned in such a manner that the laser beam hits the centre of the entrance window and is retroreflected. The lamp is operated for at least 45 min before the calibration is performed.

3.2.3. Uncertainties. The combined relative expanded uncertainty for the spectral intensity calibration at PTB sums up to 4.5% as can be seen in table 4. The first contribution is due to the uncertainty in the calculation of the synchrotron radiation of BESSY II [35] and is dominated by the uncertainty in the measurement of the electron beam current, which typically is below 1 mA for these kinds of calibrations. The second contribution includes the uncertainty in the wavelength determination for the measurement with synchrotron radiation and the deuterium lamp. The third contribution is given by the fact that the distances of the electron storage ring and the deuterium lamp to the station largely differ. This has the drawback that different areas of the optical components are illuminated. An aperture is used to ensure that the illuminated area of the grating does not depend on the source distance, but the illuminated areas of the mirror are different. Thus, the inhomogeneity of the mirror reflectance has to be taken into account and contributes largely to the uncertainty budget. The uncertainty contribution due to the distance measurement during the deuterium lamp calibration and due to the flux defining aperture during synchrotron radiation calibration is listed in the fourth line. The fifth line of table 4 summarizes contributions from the temporal stability of the station, polarization effects, detector non-linearity and detector noise.

Table 5. Relative expanded ($k = 2$) uncertainty for the conversion of spectral irradiances measured at NIST/SURF into spectral radiant intensities measured at PTB/BESSY.

PTB measurements	4.5%
NIST measurements	2.4%
Conversion	
Point source approximation	0.01%
Distance (internal and external)	0.5%
Atmospheric conditions	1.0%
Alignment	1.0%
Diffraction	1.0%
<i>Combined</i>	5.4%

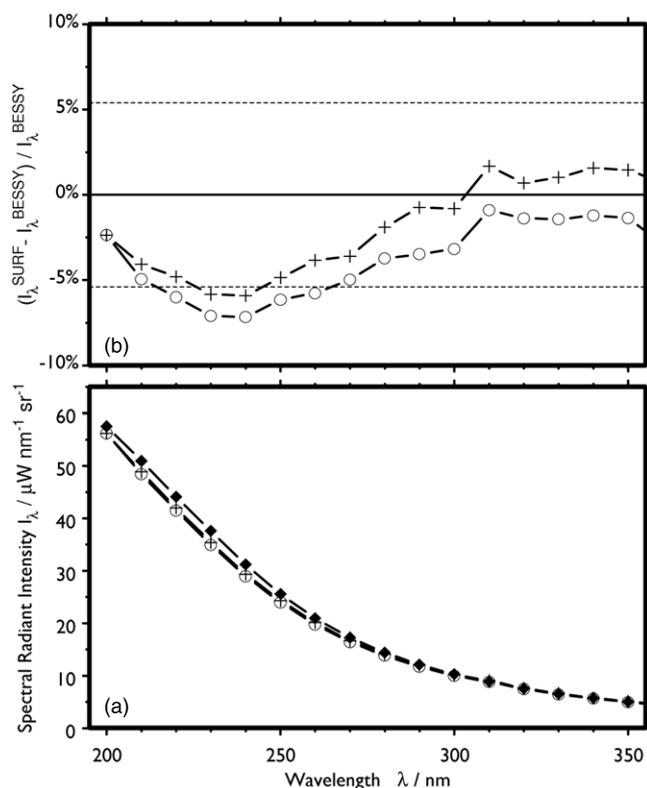


Figure 1. Results for the lamp designated AR094. (a) Measured spectral radiant intensity, PTB measurement May 2006 (\blacklozenge), NIST June 2007 (+), NIST April 2008 (\circ). (b) Percentage difference between NIST and PTB measurements.

4. Conversion and comparison

4.1. Uncertainties in the conversion of spectral irradiance into spectral radiant intensity

As we have explained earlier, the conversion of the spectral irradiance into spectral radiant intensity is based on the assumption that equation (5) is valid. In table 5 the combined relative expanded uncertainty of the comparison is listed. It contains both the measurement uncertainties of the measurements performed at NIST and PTB, and the uncertainty resulting from the conversion.

4.2. Distance determination for the conversion

In order to convert the spectral irradiances measured at SURF III into spectral radiant intensities the distance between the

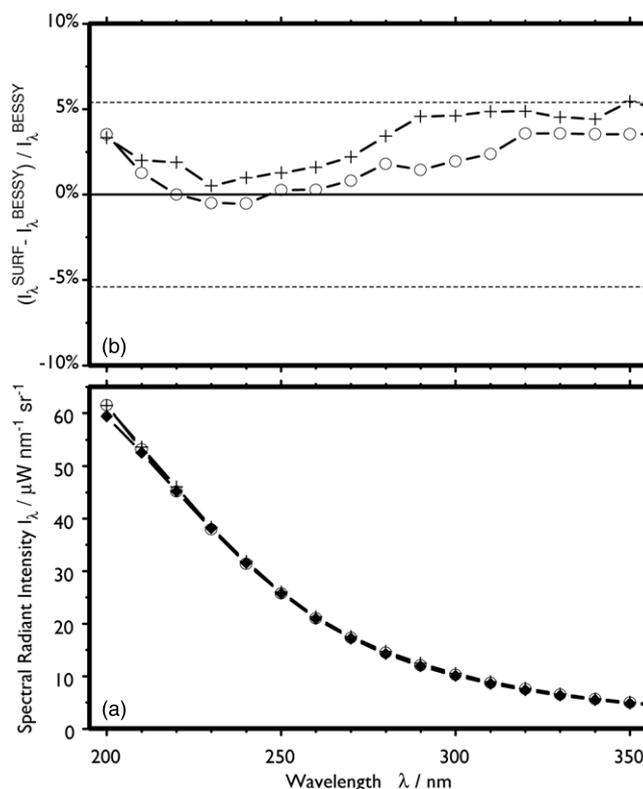


Figure 2. Results for BQ051. (a) Measured spectral radiant intensity, PTB measurement May 2006 (\blacklozenge), NIST June 2007 (+), NIST April 2008 (\circ). (b) Percentage difference between NIST and PTB measurements.

entrance aperture and the source point of the deuterium lamp has to be known. Only the external distance d_e between the front of the exit window and the aperture can be measured. The internal distance from the deuterium arc to the front of the window is given by the lamp manufacturer to be $d_i = 72 \text{ mm}$. The optical distance also has to be corrected for the dispersion of the window material (window thickness $\Delta w = 1 \text{ mm}$), which in the case of the Cathodeon V04 lamps is Spectrosil, also known as synthetic fused silica. The total optical distance is

$$d(\lambda) = d_e + d_i - \Delta w + \Delta w \cdot n_{\text{SiO}_2}(\lambda), \quad (6)$$

where $n_{\text{SiO}_2}(\lambda)$ is the refractive index of silicon dioxide [37].

4.3. The influence of atmospheric effects

The fact that the measurements at NIST are performed in air is potentially the most significant difference between the two experiments. Even though the measurements reported here were done in the spectral range commonly called air-UV, oxygen does absorb in this spectral range. The Schumann–Runge bands extend to about 205 nm [6, 38] and the Herzberg continuum [39–41] spans from about 187 nm to 242 nm. In addition, there is a pressure-dependent absorption continuum that involves a weakly bound oxygen dimer [6, 40]. All these processes dissociate oxygen molecules and can lead

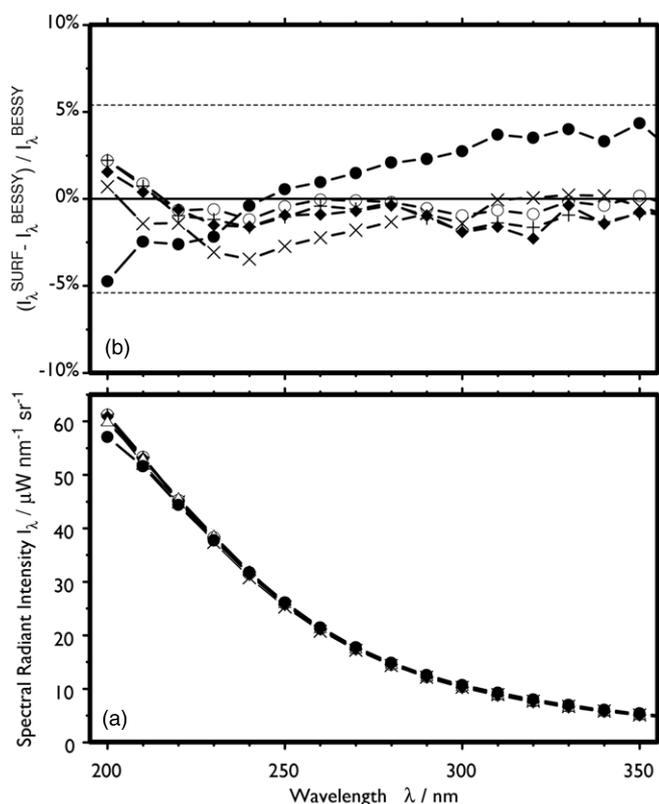


Figure 3. Results for BQ056. (a) Measured spectral radiant intensity, PTB measurement May 2006 (Δ), NIST November 2005, 30 cm distance (\circ), 40 cm ($+$), 50 cm (\blacklozenge), NIST April 2008 (\times), NIST June 2006 (\bullet). (b) Percentage difference between NIST and PTB measurements.

subsequently to the formation of ozone [38, 40], which has very strong absorption features in the air-UV spectral range [42, 43].

The abundance of water and carbon dioxide, and the possibility that water could be adsorbed to surfaces [44], could cause problems as well. Liquid water absorbs in the air-UV spectral range [45, 46] as does carbon dioxide [47–49].

4.4. Results

The results for the comparison of the three lamps are shown in figures 1, 2 and 3. The spectral radiant intensities are displayed in the bottom part of each graph for the three lamps. In the top part the relative differences between the NIST measurements and the PTB results are shown. Within the established standard uncertainties, the overall agreement is good. The differences for the lamps AR094 (figure 1) and BQ051 (figure 2) exhibit a similar wavelength-dependent behaviour.

5. Conclusion and outlook

We have proven that we can reliably convert spectral irradiances into spectral radiant intensities, within the established experimental uncertainties. In addition, we have re-established the usefulness of deuterium lamps as transfer standards in the ultraviolet spectral range [50].

The combined relative uncertainty of the described comparison is much below that of a former comparison of

Table A1. Components of the relative expanded ($k = 2$) uncertainty of spectral irradiance of synchrotron radiation at beamline 3 at SURF III.

Source of uncertainty	Nominal value	Relative uncertainty	Sensitivity coefficient	Uncertainty in irradiance
Electron energy	380 MeV	0.02%	0.057	0.0011%
Electron beam current	15 mA	0.4%	1.0	0.4%
Orbital radius	837.08 mm	10^{-10}	0.66	6.6×10^{-10}
Window transmittance	0.8	0.5%	1.0	0.5%
Distance	6926.0 mm	0.086%	2.0	0.17%
Detector alignment	$\pm 0.6 \text{ mm}^a$		0.0011	0.066%
Aperture radius	3.9895 mm	6.3%	0.015	0.096%
Air absorption ^b			1.0	0.24%
Combined				0.72%

^a Absolute uncertainty.

^b For wavelengths below 205 nm, <0.1% for wavelengths longer than 205 nm.

the spectral radiance of deuterium lamps in the same spectral range [51].

Currently the PTB is setting up a new station for the calibration of radiation sources in the spectral range between 7 nm and 400 nm [52] at the Metrology Light Source (MLS) [53], the dedicated electron storage ring of PTB. This station avoids the difficulties arising from different distances for the synchrotron radiation calibration or the deuterium lamp calibration and thus will lead to improved relative uncertainties in the spectral intensity calibration.

In addition, NIST is planning to extend its synchrotron radiation-based source calibration capabilities into the previously covered [25, 54, 55] vacuum-ultraviolet spectral range, thus also eliminating atmospheric effects in future intercomparisons.

A few years from now, it will be useful to compare the new source calibration capability of the PTB at the MLS with the improved setup at NIST.

Appendix. Uncertainties

A.1. Spectral irradiance measurements at SURF III/NIST

The relative expanded uncertainty ($k = 2$) for the measurement of the spectral irradiance of synchrotron radiation at beamline 3 is given in table 1 of [30]. This content of this table is shown in table A1. The combined relative expanded uncertainty leads to the value for SURF radiation in table 3.

A.2. Spectral radiant intensity measurements at BESSY II/PTB

The relative uncertainty ($k = 1$) for the measurements of the spectral radiant intensity at BESSY II is given in table 1

Table A2. Components of the relative expanded ($k = 2$) uncertainty of spectral radiant intensity of synchrotron radiation at BESSY II.

Source of uncertainty	Nominal value	Relative uncertainty
Electron energy	1718.60 MeV	0.02%
Magnetic induction	1.29932 T	0.02%
Electron beam current	10.0 mA	0.6%
Vertical source size	100 μm	0.02%
Vertical angular displacement	0.0 μrad	0.01%
<i>Combined</i>		0.6%

of [35]. The content of this table is displayed in table A2. The combined relative expanded uncertainty leads to the value for BESSY II radiation in table 4.

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