



Primary calibration of photodiodes with monochromatic X-ray beams using an electrical-substitution radiometer

Víctor H. Elvira, Marie-Christine Lépy, Yves Ménesguen

► To cite this version:

Víctor H. Elvira, Marie-Christine Lépy, Yves Ménesguen. Primary calibration of photodiodes with monochromatic X-ray beams using an electrical-substitution radiometer. X-Ray Spectrometry, 2022, pp.10.1002/xrs.3318. 10.1002/xrs.3318 . cea-04089813

HAL Id: cea-04089813

<https://cea.hal.science/cea-04089813>

Submitted on 5 May 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial - NoDerivatives 4.0 International License

RESEARCH ARTICLE

Primary calibration of photodiodes with monochromatic X-ray beams using an electrical-substitution radiometer

Víctor H. Elvira  | Marie-Christine Lépy  | Yves Ménesguen 

Université Paris-Saclay, CEA, LIST,
Laboratoire National Henri Becquerel
(LNE-LNHB), Palaiseau, France

Correspondence

Víctor H. Elvira, Université Paris-Saclay,
CEA, LIST, Laboratoire National Henri
Becquerel (LNE-LNHB), Palaiseau F-
91120, France.

Email: victor.hernandez-elvira@cea.fr

Funding information

INSNU project

Abstract

The electrical-substitution cryogenic detector *BOLometer for Use in the range of X-rays (BOLUX)*, which was developed some years ago at CEA/DAM, has been set up and restarted now at LNHB. It has been used for the primary measurement of the intensity (total energy per unit time) of monochromatic synchrotron beams in the energy range from 3 to 30 keV. These well-determined photon beams have been employed for the efficiency calibration of two photodiodes in terms of current induced per unit optical power at different photon energies. In a final step, we explored the possibility to use these primary-calibrated photodiodes to determine the efficiency curve of an energy-dispersive spectrometer based on a semiconductor detector (Silicon Drift Detector) using less intense monochromatic photon fluxes. The characteristics of the radiometer BOLUX and its principle of operation are described, and the measurements carried out at the synchrotron beamline are presented, including the determination of the beams' intensities, the direct calibration of photodiodes with respect to BOLUX and the use of one of those photodiodes as a standard transfer for the calibration of the SDD.

KEYWORDS

cryogenic detector, efficiency calibration, photodiodes, synchrotron radiation, X-rays

1 | INTRODUCTION

Accurate measurement of fundamental parameters that characterize the radionuclide decay schemes, such as X-ray emission intensities, require well-calibrated spectrometers, the critical point being their efficiency calibration. In the conventional approach, efficiency calibration is carried out by using radionuclides standards, characterized by activity (Bq), and previously known (tabulated) photon emission intensities. Therefore, the new emission intensities derived from such a calibration procedure are

directly related to those initially used in the reference calibration: the determination of emission intensities is then subject to a circular dependency.

A solution to this limitation is to use well-known photon fluxes whose calibration is independent of any previous knowledge of radionuclide emission intensities. The primary measurement of photon fluxes intensities, therefore, represents a major goal in ionizing radiation metrology.

To this end, cryogenic electrical-substitution radiometers have proven to be a very suitable choice. They are

This is an open access article under the terms of the [Creative Commons Attribution-NonCommercial-NoDerivs](https://creativecommons.org/licenses/by-nc-nd/4.0/) License, which permits use and distribution in any medium, provided the original work is properly cited, the use is non-commercial and no modifications or adaptations are made.

© 2022 The Authors. *X-Ray Spectrometry* published by John Wiley & Sons Ltd.

well-established standards that have been used in the UV-visible range from decades ago,^{1,2} and more recently the energy range has been extended up to X-ray range.^{3–4}

In this work, we present the primary measurement of photon fluxes with the cryogenic electrical-substitution radiometer *BOLUX* (*BOLometer for Use in the range of X-rays*), which had been used in the past, especially in the low-energy range,⁵ and has now been restarted and upgraded for its application to the analysis of energies up to 50 keV. This article presents a general description of the device and its principle of operation, the theoretical detector response, established by Monte Carlo simulations, and the experimental measurements. They have been carried out at the Metrology beamline of the SOLEIL synchrotron, which provides monochromatic photon beams with enough radiative power to comply with the sensitivity of the radiometer. These measurements required the accurate characterization of the photon beam in terms of spectral purity, in order to define the optimal experimental conditions.

The primary measurement of photon fluxes is applied to the calibration of two photodiodes with different characteristics. This provides a transfer standard that can be used at a further stage for the calibration of spectrometers. Primary calibration of photodiodes also plays an important role in all those applications that rely on well-characterized photon fluxes, such as reference-free X-ray spectrometry.^{6,7}

Since both the radiometer's response and the photodiode's efficiency depend on the energy of the photons, monochromatic beams of several energies must be used in order to determine the calibration curve.

According to the International Vocabulary of Metrology,⁸ we hereafter refer to a measurement procedure as “primary” if is not related to any measurement standard for a quantity of the same kind, and we call “primary standard” a measurement standard established using a primary reference measurement procedure. Therefore, we use *BOLUX* as a primary standard; the measurement of the power of the photon beam with *BOLUX* is a primary measurement, and the photodiode calibrated using *BOLUX* as a primary standard becomes a reference standard.

To conclude, a first attempt to determine the efficiency of an energy-dispersive spectrometer (SDD, silicon drift detector) by comparison with one of the reference photodiodes is presented as an outcome of the primary calibration. The reference photodiode is used as “transfer device”, which is required because the range of photon fluxes that can be measured by the SDD and by *BOLUX* are extremely different.

2 | OPERATING PRINCIPLE OF A CRYOGENIC ELECTRICAL-SUBSTITUTION RADIOMETER

2.1 | Principle of cryogenic detectors

Cryogenic detectors characterize an incident photon flux by recording the temperature rise experienced by an absorber when the radiation interacts with it. Under the assumption that the energy transfer is complete, the temperature of the absorber increases proportionally to the energy of the incident photon flux, the proportionality ratio being the reciprocal of the material's heat capacity of the material of the absorber:

$$\Delta T = \frac{E_{in}}{C}$$

If the heat capacity is well known, the energy of the photon flux can therefore be determined by measuring the temperature rise.

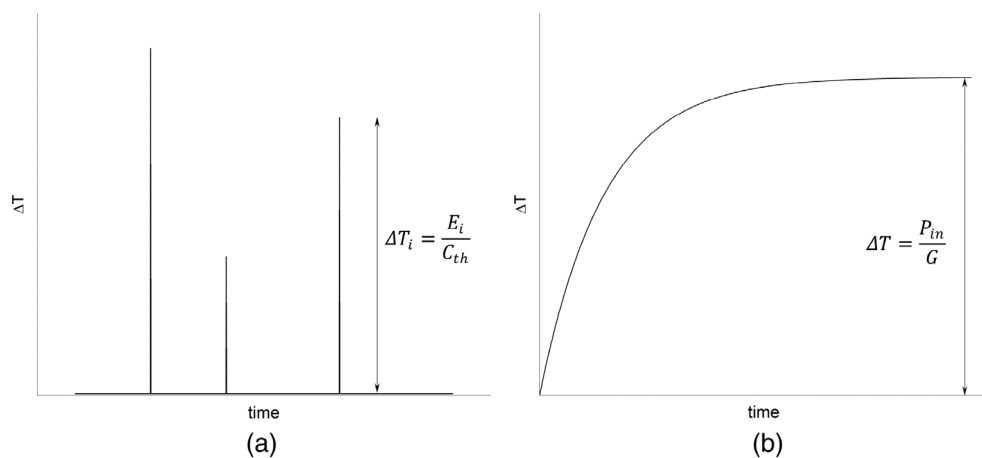
The sensitivity of the technique is inversely proportional to the material's heat capacity, which decreases at low temperatures. In order to obtain a detectable temperature rise due to the interaction of a single photon, an extremely low heat capacity is required, which implies extremely low temperatures (typically not higher than several tens of mK). This entails major technical difficulties but allows the use of cryogenic detectors as spectrometers since they allow the discrimination of photons by energy.^{9,10} This operation mode is called “pulse mode” (Figure 1a). Apart from the technical difficulties arising from extreme cooling, there is an intrinsic limitation to the count rate that can be recorded, which is related to the time required for the system to return to thermal equilibrium after the interaction with each photon. Therefore, this operation mode is not suitable for measuring very intense photon fluxes.

Both the technical difficulties of extreme cooling and the impossibility of measuring intense photon fluxes can be circumvented if the “continuous mode” is employed (Figure 1b). When an absorber is subjected to continuous radiation, the evolution of its temperature is the balance of the energy deposited by the radiation and the heat dissipated towards the thermalized environment:

$$\frac{dT}{dt} = \frac{P_{in}}{C} - \frac{G(T - T_0)}{C} \quad (1)$$

where P_{in} is the power of the incident radiation, C is the absorber's heat capacity, G is the heat transfer coefficient from the absorber to the environment and T_0 is the temperature of the environment.

FIGURE 1 Response of a cryogenic detector operating in pulse mode (a) and in continuous mode (b).



The solution to this differential equation

$$T(t) = T_0 + \frac{P_{in}}{G} \left(1 - e^{-\frac{G}{C}t}\right) \quad (\text{Eq2})$$

shows that the temperature rise $\Delta T = T(t) - T_0$ tends asymptotically towards P_{in}/G regardless of the material's heat capacity. Therefore, if we measure at equilibrium, that is, when the absorber has been receiving the radiation for a sufficiently long period, the temperature rise is proportional to the total power of the incident beam and inversely proportional to the heat transfer coefficient. This operation mode does not enable spectroscopy, since it does not discriminate photons by energy, but it allows the measurement of high photon fluxes.

2.2 | Principle of electrical substitution

In electrical substitution radiometers, the amount of energy deposited by the radiation is measured by determining the electrical power that needs to be injected into the absorber to obtain the same temperature rise as the one induced by the photonic heating:

$$\eta P_{in} = I R_{hr}^2$$

where η is the efficiency of photon absorption, I is the electrical current required to provoke the equivalent heating and R_{hr} is the resistance of the heating element.

Working under the principle of electrical substitution makes it possible to determine the power of the incident radiation with no actual need to quantify the temperature rise of the absorber: it suffices to verify that this temperature rise is the same during the electric and the photonic heating, with no need to know its specific value. Therefore, even though the temperature rise and the radiation power are related through the heat transfer coefficient of

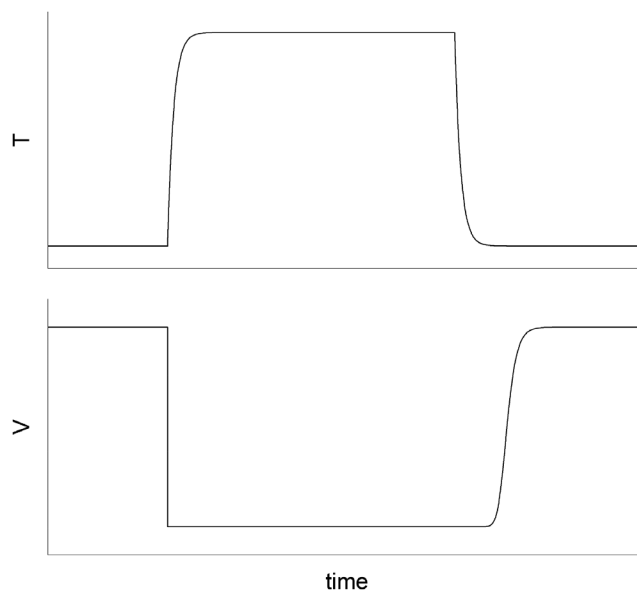


FIGURE 2 Monitoring of a temperature rise by measuring the voltage in a thermistor polarized with a constant current.

the system, it is not necessary to know the value of this coefficient, which can be difficult to determine and would add a very significant component to the uncertainty.

In order to monitor the temperature changes, a thermistor linked to the absorber is polarized with a constant current, so that the change in resistance induced by the temperature increase causes a significant change in the voltage (Figure 2). The sensitivity of the technique then depends on how steep the dependence of the thermistor's resistance to temperature is. For this reason, a judicious choice is to use a semiconductor material and to work at very low temperature (typically at the boiling point of helium at the most), to guarantee that a small increase in temperature induces a significant decrease in resistance and thus in voltage.

For measurements carried out at 4.2 K, a typical power of a few tens of microwatts as the ones measured in this work implies a temperature rise of a few tenths of kelvin.

The measurement of radiation power by electrical substitution, therefore, involves two steps: in the first stage, the absorber is exposed to the radiation beam in

order to register the decrease in voltage (i.e., the photon flux power); in the second stage, the absorber is no longer exposed to the radiation, but an electrical current is introduced instead, and this current is adjusted until the decrease in voltage matches the decrease obtained in the first step (Figure 3).

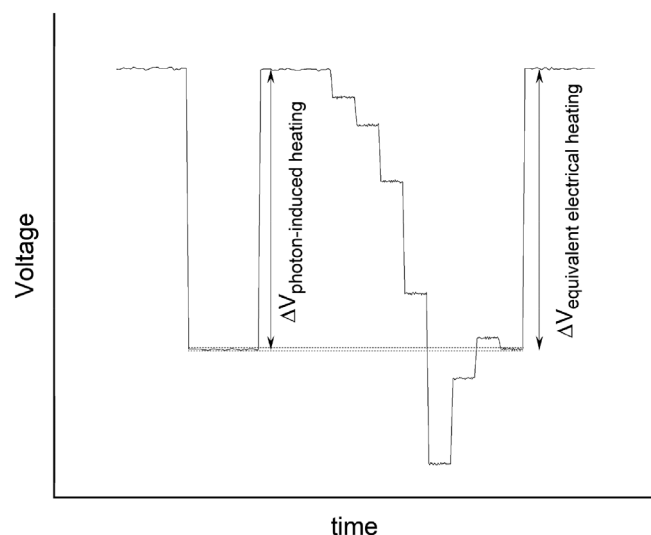


FIGURE 3 Principle of radiation power measurement with an electrical-substitution radiometer

3 | INSTRUMENTATION AND MEASUREMENT PROCEDURES

3.1 | Description of the cryogenic electrical-substitution radiometer

The cryogenic electrical-substitution radiometer *BOLUX* (*BOLometer for Use in the field of X-rays*) was initially developed at CEA/DAM. This instrument was successfully used in the past to characterize monochromatic photon fluxes in the energy range up to 11 keV.⁵ It has been recently handed over to the Laboratoire National Henri Becquerel (LNHB) at CEA, where it has been restarted and upgraded.

The measuring bolometer is integrated in a cryostat (Figure 4), which consists of a 39-cm-high and 20-cm-diameter cylinder that comprises a two-liter tank for liquid helium isolated from the outside by three concentric walls, between which void is created, and a smaller

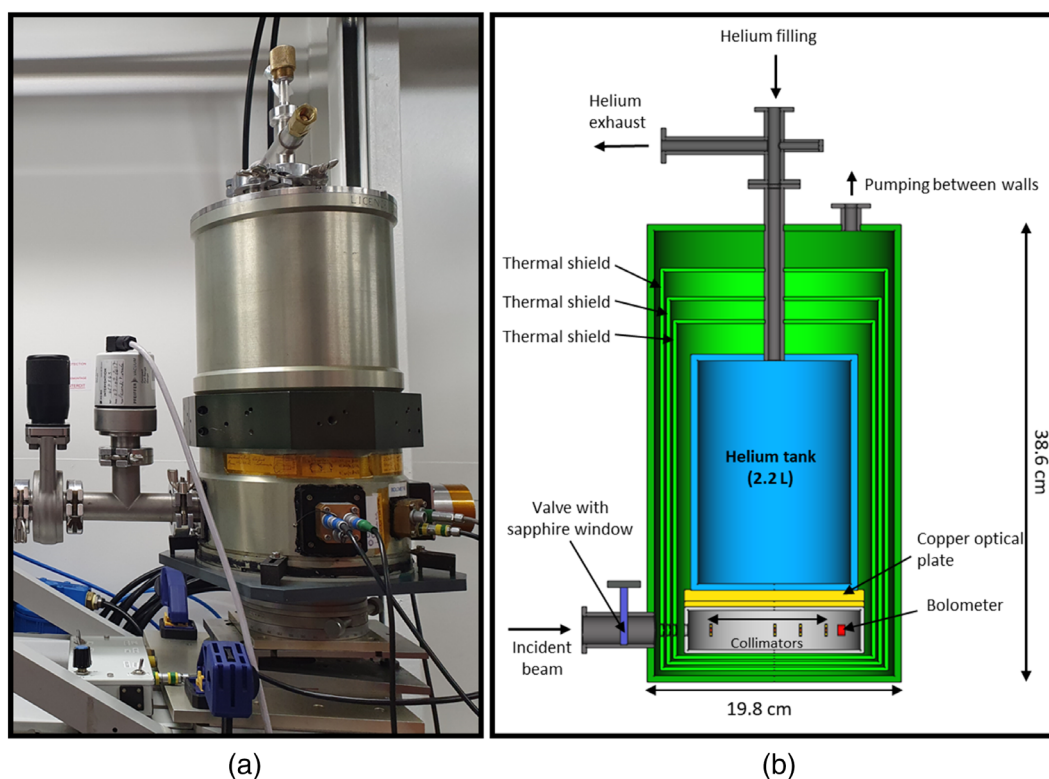


FIGURE 4 Photograph (left) and diagram (right) of the radiometer BOLUX used in this work

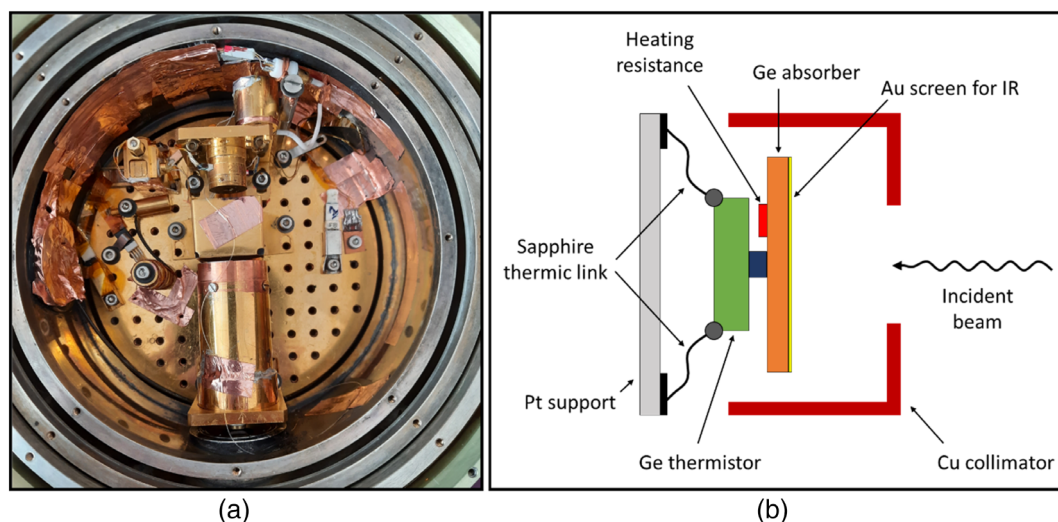


FIGURE 5 Up-side-down photograph of the inner part of the chamber that hosts the measuring bolometer (left) and diagram of the measuring bolometer (right)

chamber in the bottom part (Figure 5a) that hosts the measuring bolometer, its electronic components and optical collimators. The bolometer (Figure 5b) comprises a cylindrical germanium absorber (4.65 mm diameter and 300 μm thickness), covered in the front part by a thin gold layer to shield it against infrared radiation, and linked in the rear side to a germanium thermistor. The heating resistance is directly coupled to the rear side of the absorber. The whole system is surrounded by a copper collimator with a 4.11-mm-diameter aperture through which the incident radiation enters the bolometer. The thermistor is linked by a sapphire fiber to a platinum support, which is in contact with the copper optical plate, thermalized by the helium bath contained in the tank just above it.

In order to monitor the temperature, the thermistor is polarized by a constant current. To this end, a constant voltage is supplied from outside the cryostat by means of a polarization box based on a system of batteries, and turned into a constant current by means of a charging resistor. The thermistor voltage is continuously measured with a multimeter Keithley2000. During the electrical heating stage, the heating resistance is polarized by means of an external current source Keithley220, and the voltage induced is measured by a different multimeter Keithley2000, in order to deduce the electrical power as the product $I \times V$.

3.2 | Electrical substitution procedure

The instrument is operated in continuous mode, that is, it measures the total amount of energy that reaches the

bolometer per unit time. As mentioned above, it does not allow spectrometry, but it does allow the measurement of intense photon fluxes. Since it is not possible to discriminate photons by energy, it is a convenient solution to use a monochromatic radiation source from synchrotron, which in addition is powerful enough to rise to the sensitivity of the technique.

An automated system coordinates the sequence of measurements of the photon flux intensity for selected incident energy (monochromator), as follows:

- i. First, the beam shutter is closed and the thermistor's voltage in the absence of any heating (baseline) is measured.
- ii. Next, the beam shutter is opened and, once the thermal equilibrium is reached, the thermistor's voltage change due to the photonic heating is registered.
- iii. Afterwards, the shutter is closed again to repeat the measurement of the baseline, and then, while keeping the beam closed, the external current source is turned on. A searching algorithm has been developed to adjust the supplied current until the equivalent current is found, that is, until the thermistor's voltage change induced by the electric current is the same as the one induced by the photonic beam (within the sum of standard deviations [Figure 3]).
- iv. Once the equivalent current is reached, the voltage in the heating resistance is measured. The power deposited in the absorber then corresponds to the product of the equivalent current and the voltage induced by it. The energy of incoming photons being known, the beam intensity (number of photons per second) can be derived.

3.3 | Procedure for primary calibration of photodiodes

Once the power of the beam is determined, it can be used for the calibration of a photodiode.

The efficiency calibration of a photodiode consists in determining the amount of electric current generated in it when it is exposed to a radiation of unit power. Since it depends on the photon energy, the determination of its efficiency curve implies the measurement of its efficiency for a set of fixed values of this energy.

The determination of the photodiode's efficiency at a certain energy involves two steps. In the first step, the flux power of a monochromatic photon beam was measured with BOLUX as described in the previous section: the thermistor's voltage change is measured when the absorber is exposed to the radiation, and afterwards an electrical current is introduced and modified until the same voltage change is reached. In the second step, the photodiode is exposed to the same photon beam and the current induced is measured. The background current is also measured: the current generated in the photodiode in the absence of radiation, which is subtracted from the total current. The photodiode's efficiency is then the ratio between the current induced by the radiation (after subtracting the background) and the power of the beam. Once the whole cycle was completed, the photodiode was withdrawn again, the energy of the monochromator was changed and the new cycle started again in order to determine the photodiode's efficiency at the new energy. The operation is repeated for different energies between 3.5 and 30 keV.

4 | DESCRIPTION OF THE MEASUREMENTS

In this work, measurements were performed at the *Métrologie* beamline at *SOLEIL* synchrotron (France), more specifically at the hard X-ray branch, which is equipped with a Si-111 double-crystal monochromator and provides monochromatic photons covering an energy range from 3 to 30 keV.

By the procedure described in the previous section, the radiometer BOLUX was used for the primary calibration of two photodiodes in terms of efficiency: OptoDiode AXUV¹¹ and Hamamatsu S8193.¹² They are classic commercial silicon-based photodiodes, although they are slightly different because S8193 has a thicker silicon foil and is covered with a ceramic scintillator to increase its efficiency at high photon energies. Afterwards, the AXUV photodiode was used as a reference standard for the

calibration of an energy-dispersive spectrometer based on SDD.

Two previous actions are necessary to ensure the reliability of measurements: the calculation of the correction factor for the energy deposition of radiation in the cryogenic detector and the optimization of the parameters of the beamline to ensure spectral purity.

4.1 | Correction factor for BOLUX

The radiometer measures the energy deposited in the absorber. However, part of the energy of the incident radiation is not deposited, either due to photons that do not interact with the absorber, or photons that do interact but do not deposit all of their energy, as a consequence of scattering processes or escape of characteristic X-rays.

Monte Carlo simulations were carried out to compute the average energy deposited by each photon in the absorber for different values of the photon's energy. The emission of monochromatic X-ray towards a 300- μm thick germanium disk was simulated with *PENELOPE* code¹³ in order to obtain the spectrum of deposited energy and calculate the amount of energy deposited per emitted photon. The ratio between the incident energy and the energy deposited per photon (i.e., the reciprocal of the fraction of deposited energy) constitutes the correction factor by which the measured power has to be multiplied in order to get the actual beam power. Figure 6 shows the correction factor computed for different values of the photon energy.

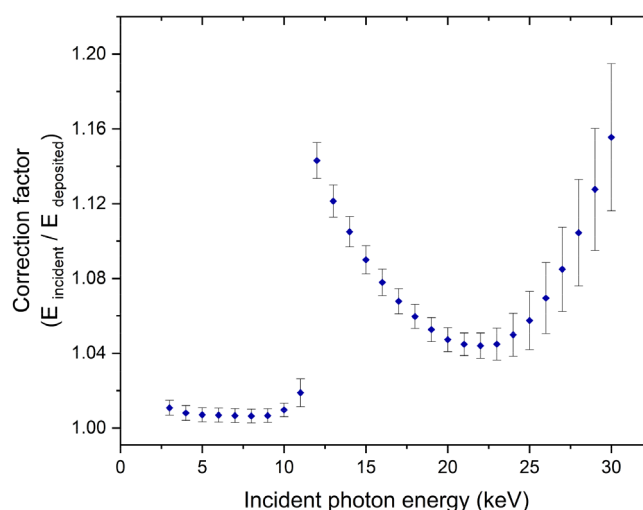


FIGURE 6 Correction factors for the measurement of the beam power with BOLUX, computed by means of Monte Carlo simulations with *PENELOPE* code

4.2 | Search for optimal beam purity

The energy in the beamline is selected by a monochromator that diffracts radiation according to Bragg's law:

$$n\lambda = 2d \sin \theta$$

where n is an integer number, λ is the radiation wavelength, d is the distance between two successive layers in the crystallographic lattice and θ is the angle of the incident radiation with respect to the surface of the crystal. For a given angle, not only photons of a single energy are reflected, but all those whose energy is an integer multiple of the lowest one. If an energy E_i needs to be selected, the beamline will actually emit a set of energies nE_i , the harmonics of the main energy (selection rules for the crystallographic structure of the crystal employed in *Métrologie* beamline allow all natural numbers for n except $n = 2$).

However, in order to determine the efficiency of a photodiode at a specific energy, it must be guaranteed that the radiation that reaches the photodiode is actually monochromatic, that is to say that higher harmonics must be suppressed or at least their contribution must be negligible.

The beamline is equipped with two crystals (Figure 7) for a finer selection of the wavelength. The second crystal can be detuned with respect to the first one by a certain angle in a process customarily called “pitch”. As the detuning angle increases, the photon flux of all the harmonics decreases, but this decrease is more pronounced the higher the harmonic's order. Therefore, the flux of photons of the targeted energy decreases, but the fraction of higher harmonics decreases in a greater proportion, so for a sufficiently high angle, the contribution of higher harmonics can be negligible. Figure 8 shows the emission spectrum of *Métrologie* beamline, measured by a high-purity germanium (HPGe) spectrometer, when the monochromator is set to 4 keV, for different values of the detuning angle. (Note that the peaks at 2 E do not correspond to a harmonic since $n = 2$ is not permitted for the crystal used, but it is the pile-up of the main energy due to the high count rate).

The first step of the experimental work then consisted in characterizing the beam for different values of the

detuning angle in order to select the lowest value that makes higher harmonics negligible at each energy.

5 | RESULTS

5.1 | Primary calibration of photodiodes with BOLUX

In *Métrologie* beamline at synchrotron SOLEIL, we carried out the primary calibration of two different photodiodes in the energy range from 3.5 to 30 keV.

As described above, for each value of the energy, the pitch was set so as to make high-order harmonics negligible. The beamline power was measured with BOLUX,

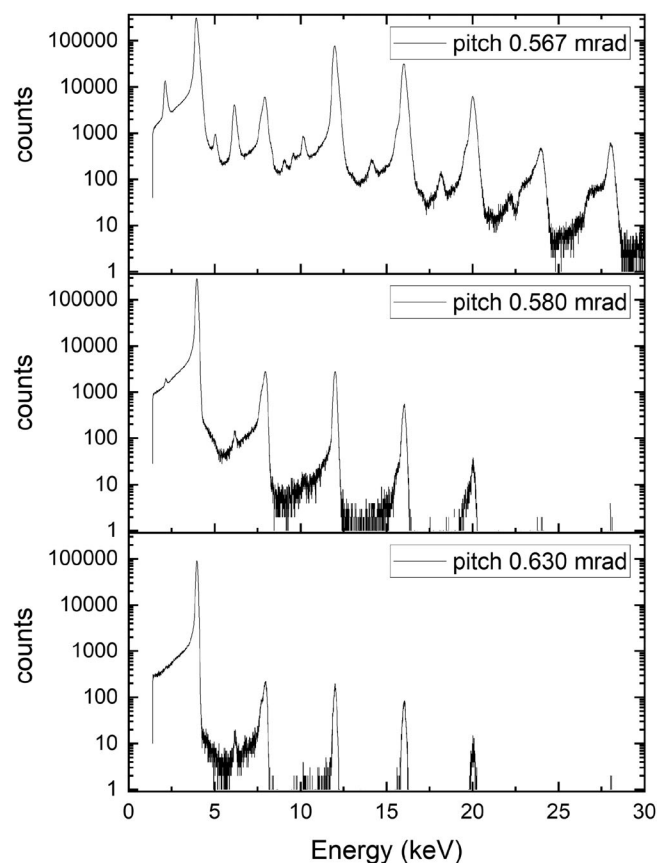


FIGURE 8 Emission spectra of *Métrologie* beamline, measured by an HPGe spectrometer, when the monochromator is set to 4 keV, for different values of the detuning angle.

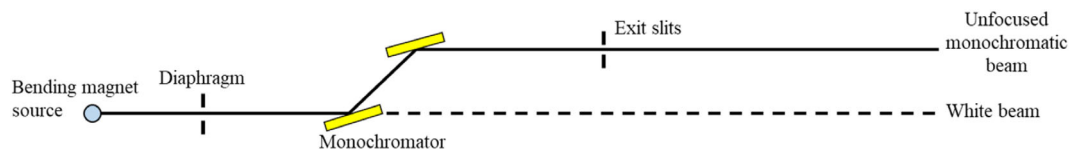


FIGURE 7 Diagram of the monochromator in *Métrologie* beamline

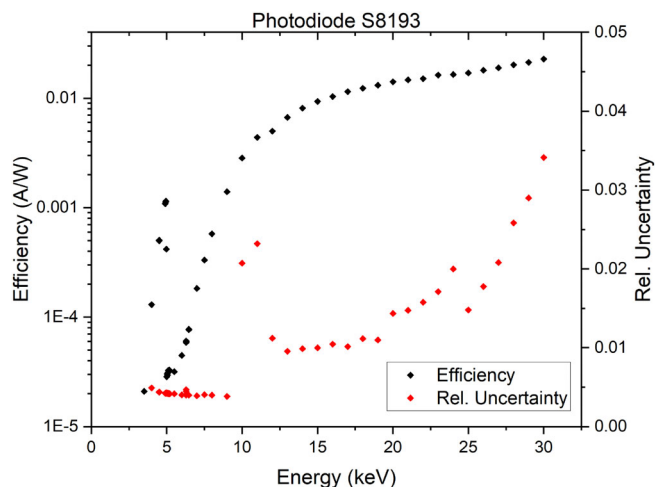


FIGURE 9 Efficiency of photodiode S8193 determined by measuring the current induced by a photon beam whose power has been measured by BOLUX in a primary way

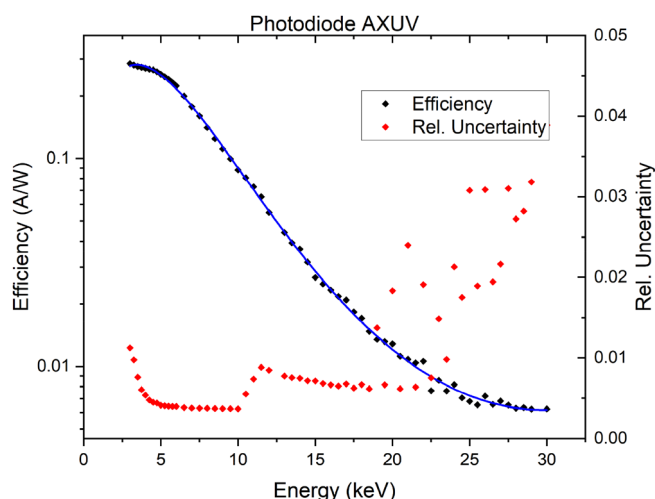


FIGURE 10 Efficiency of photodiode AXUV determined by measuring the current induced by a photon beam whose power has been measured by BOLUX in a primary way. A polynomial fit in the log-log scale has been superimposed to account for the oscillations mainly in the range where the efficiency is lower and the current induced is lower, thus leading to a greater uncertainty in the efficiency.

applying the corresponding correction factor for the fraction of deposited energy. Then the photodiode was placed in the beam pathway, and the current induced was measured, subtracting the background current (current measured in the absence of beam), in order to determine the current generated in the photodiode per unit power. The process was repeated for several energies between 3.5 and 30 keV.

Figures 9 and 10 show the efficiency of both photodiodes (current generated per unit power) as a function of

the energy, and the associated relative uncertainties in red.

The uncertainty in the photodiode's efficiency is the combination of the uncertainty in the beam power measured by BOLUX and in the current generated by the photodiode.

- The uncertainty in the beam power has two kinds of sources: the uncertainty in the measurement itself and the uncertainty in the correction factor.
 - The uncertainty in the measured beam power stems from the uncertainty in the determination of the equivalent current, which in turn arises from the uncertainty in the size of the voltage change ΔV . This uncertainty is propagated approximately in a linear way since ΔV follows a nearly quadratic tendency with the applied current, and the electrical power is proportional to the square of the current (given that the resistance of the heating element is essentially constant). Therefore, the relative uncertainty of the measured power can be approximated to be the relative uncertainty of ΔV , which can be taken as the combination of the standard deviation of the voltage baseline and the voltage when exposed to heating, divided by the size of ΔV . This contribution ranges from a few tenths of a percent in those cases when the measured beam power (and therefore the voltage change) is higher to around 1.5% for the weakest beam measured.
 - The uncertainty in the calculated correction factor has a contribution that arises from the statistics of the simulations and another one due to the uncertainties in the interaction cross sections used by the code. There is also a major source of uncertainty due to the uncertainty in the thickness of the absorber.
- The statistical contribution is provided by the code itself, and depends on the number of simulated events. The simulations carried out in this work were done with a million events and led to uncertainties of around 0.3% for the energies below the germanium K-edge and around 0.5% above the edge.
- Uncertainty in the thickness of the germanium absorber. It is the main source of uncertainty at those energies for which the transmission is significant. It is difficult to estimate the uncertainty in the manufacturing of a germanium disc, so as a conservative estimation we assumed a 10% uncertainty, which should be enough to account for any possible irregularities as well. In order to infer its contribution to the total uncertainty in the correction factor,

simulations were carried out for absorbers 270 and 330 μm thick in order to evaluate the difference with the correction factor at 300 μm . It could be established that this contribution to the total uncertainty is less than 1% below 25 keV, and even a few tenths of a percent below 20 keV (except right below germanium's K-edge). The contribution begins to be significant at 25 keV and it reaches several percent in the highest part of the considered interval.

- The interaction cross-sections from the database used by PENELOPE have an uncertainty of a few percent above 1 keV,^{14,15} so as a conservative estimation one can assume 5% in the whole interval considered. The contribution of the relative uncertainty in the interaction cross section to the relative uncertainty in the correction factor is weighted, in a good approximation, by the absolute value of the natural logarithm of the correction factor itself.

The combined uncertainties in the correction factor for each energy are included in Figure 6.

- The uncertainty in the photodiode's current is taken from the standard deviation of the sequence of twenty measurements made for each energy. It is affected by the noise of the photodiode, but also by small oscillations in the beam power arising from oscillations in the synchrotron current. In the regular operation mode, oscillations in the synchrotron current are around $\pm 0.2\%$, but sometimes there can be occasional fluctuations when the current drops more steeply for a short period of time, and if this happens during the time of measurement, it can lead to significantly higher standard deviations in the photodiode's current. This is the case of some of the measurements with photodiode AXUV in the highest energy range and S8193 at 10 and 11 keV, where the standard deviation of the photodiode's current accounts for the high uncertainties in the photodiode's efficiency, which is not only explained by the uncertainty in BOLUX's correction factor.

As a verification measurement, in a further stage, the current generated by the two photodiodes was measured when exposed to the same beam, in order to directly determine the ratio of their efficiencies and compare it to the ratio of the efficiencies determined with the primary calibration with BOLUX. As it is shown in Figure 11, the ratio of efficiencies determined both ways are in good agreement, thus confirming the reliability of the measurements.

5.2 | Calibration of a spectrometer traced to BOLUX

An SDD was calibrated in terms of efficiency using one of the photodiodes (AXUV) calibrated with BOLUX as a reference standard.

It must be noticed that an energy-dispersive spectrometer as the one calibrated in this work is not capable of reliably measuring photon fluxes as high as the ones required by a radiometer like BOLUX. The solution is to use a "transfer device", that is to say a reference standard that shows a good linearity in a wide range of fluxes, so it can be calibrated in terms of efficiency using a strong flux measured with BOLUX, and can then be used to determine the power of a much weaker beam, which will be measured by the energy-dispersive spectrometer. Photodiodes have proven to have a linear response in a wide range of beam powers, so they are a very suitable choice as transfer devices when it comes to calibrate an energy-dispersive spectrometer with a radiometer.

For each value of the energy, the current induced by the beam in the reference photodiode was measured. Since the photodiode's efficiency was known at this point, the beam power (total amount of energy per unit time) could be determined. Given that the beam is monochromatic, by dividing the beam power by the energy of each photon, the number of photons emitted in the beam per unit time is determined. Subsequently, the same photon beam was measured by the SDD. The ratio between the detector's count rate and the total number of emitted

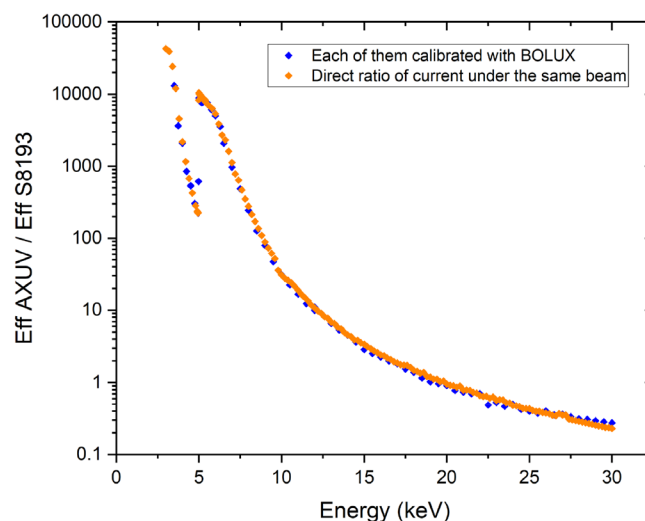


FIGURE 11 Ratio between the efficiency of photodiodes S8193 and AXUV. Blue dots represent the ratio between the efficiencies obtained from their primary calibration against BOLUX; orange dots show the direct ratio of the current generated by the same photon flux in both photodiodes.

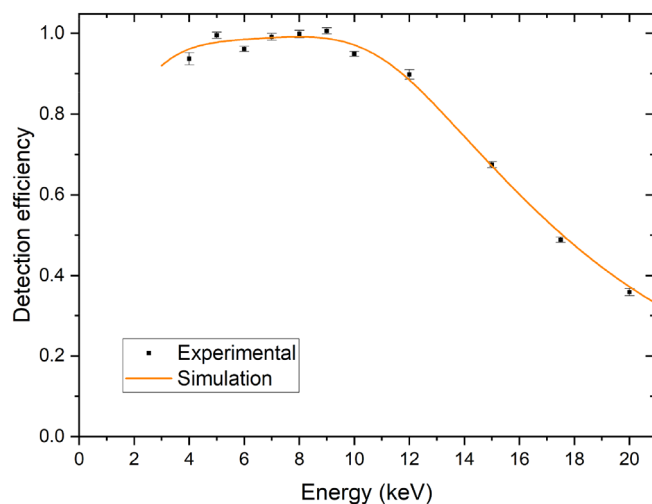


FIGURE 12 Efficiency curve of an SDD spectrometer calibrated against photodiode AXUV, in turn calibrated against BOLUX, along with the efficiency calculated by Monte Carlo simulations with PENELOPE code.

photons is the intrinsic efficiency of the detector. The efficiency curve of the SDD determined by this procedure is shown in Figure 12. The experimental results obtained by this procedure were compared with the result of Monte Carlo simulations with PENELOPE code, relying on the geometric parameters obtained by optimization of other measurements that had been carried out in the laboratory using a tunable monochromatic source based on an X-ray tube and a proportional counter as a reference detector¹⁶: 488- μm -thick silicon disc, with a 70-nm dead layer and a 15- μm -thick beryllium window.

In this case, there are two kinds of sources of uncertainty:

- Uncertainty in the determination of the photon flux, which is due to:
 - The uncertainty in the photodiode's efficiency, which has been determined in previous sections, and it is close to or below 1% in this energy range, except at 20 keV, where it was occasionally high due to the reason mentioned above.
 - Uncertainty in the current induced in the photodiode. It has been taken from the standard deviation of the sequence of measurements, as was the case in the determination of the photodiode's efficiency. However, this time a much weaker photon flux had to be used, so the induced current is smaller, which led to a lower signal-to-noise ratio. The relative uncertainty of the photodiode's current ranges from a few tenths of a percent to 1.5%.
- Uncertainty in the determination of the count rate measured by the SDD.

- There is a statistical component arising from the stochastic nature of the radiation emission itself, but it is below 0.1% in the whole interval, given the high number of counts in the measured spectra.
- The main source of uncertainty regarding the determination of the count rate stems from the difficulty to fit the full-energy peaks to a clear curve, since, precisely due to the high count rates, the shape of the peaks deviates from the ideal Gaussian. This contribution of the uncertainty was taken as the difference between the area of the Gaussian curve that best fits the peaks (which largely underestimates the area), and the total gross count of the peak, which is by definition an overestimation. This contribution was a few tenths of a percent for all the measurements.

The overall uncertainty in the detection efficiency is around 1% in most of the intervals. It is occasionally higher at 4 keV because of a high relative standard deviation in the photodiode's current, and at 20 keV, on account of the high uncertainty in the determination of the photodiode's efficiency, which was also due to an occasionally high standard deviation of the photodiode's current in the measurements that led to its calibration. As we can see, it is occasional instabilities in the beam that account for the cases when the uncertainty of measurements is above 1%. Therefore it can be concluded that the uncertainty of this calibration technique is close to 1%.

6 | CONCLUSIONS

Electrical-substitution cryogenic radiometer *BOLUX* has been successfully set up and restarted at LNHB/CEA. It allows the primary measurement of high photon flux intensities. In this work, it has been used for the measurement of monochromatic photon beams of different energies between 3 and 30 keV at synchrotron SOLEIL, which were in turn used to determine the efficiency curve of two photodiodes in terms of current generated per unit optical power.

The uncertainties in the determination of beam intensities are close to 1% or below up to 20 keV, but they are significantly higher (up to 3%–4%) in the highest part of the interval, especially due to the important corrector factor that needs to be applied. This is generally the main contribution to the total uncertainty in the photodiode's efficiency, so we can say that this technique allows the determination of photodiode's efficiencies with an uncertainty close or below 1% up to 20 keV. However, the uncertainty in the measurement of the photodiode's

current itself was occasionally higher due to the punctual instabilities of the beam, and in those cases, this one becomes the dominant contribution.

In the highest part of the range, the uncertainty of the correction factor reduces the accuracy of measurements. For this reason, the possibility to change the absorber will be explored in the mid-term future in order to increase the fraction of energy deposited in it, thus reducing the contribution of the uncertainty in the correction factor, allowing the extension of the energy range.

A good agreement has been found between the comparison of the efficiency of two photodiodes determined by direct calibration with BOLUX and the direct comparison of the current induced in both photodiodes by the same beam, thus confirming the reproducibility of the technique.

In a further step, we explored the possibility to apply the primary measurement of photon fluxes with BOLUX to the calibration of energy-dispersive spectrometers, that is, with no need to rely on previously measured photon emission intensities, which is a major goal of ionizing radiation metrology. The detection efficiency of SDD was determined at several photon energies by measuring the count rate of monochromatic beams whose intensity was measured with one of the photodiodes calibrated with BOLUX. Experimental results reproduce in a good approximation the efficiency curve calculated by means of Monte Carlo simulations relying on the geometric parameters obtained by other measurements carried out in the laboratory with a different primary technique. This promising result opens the door to the calibration of energy-dispersive spectrometers using well-known monochromatic photon beams measured in a primary way by a non-dispersive radiometer, via a transfer measurement device with a sufficiently good linearity to be calibrated with intense photon fluxes as the ones required by the radiometer, and which, at the same time, is sensitive enough to measure much weaker fluxes as the ones tolerated by the energy-dispersive spectrometer. Spectrometers calibrated by this procedure will allow, in combination with primary measurements of activity, the determination of photon emission probabilities that will be independent of any previous measurement of these parameters.

ACKNOWLEDGEMENTS

This work has been supported by the INSNU project (CEA-DES/DPE/GEN23). We thank the CEA/DAM and all the researchers and technicians that participated in the design and development of BOLUX, led by Philippe

Troussel and Noël Coron. We very much appreciated the kind help of Philippe Troussel and Gilles Lidove in the installation of BOLUX at the LNHb and in getting acquainted with the instrument. We acknowledge SOLEIL for provision of synchrotron radiation facilities and we would like to thank Pascal Mercere and Paulo Da Silva for assistance in using beamline *Métrologie*.

ORCID

Victor H. Elvira  <https://orcid.org/0000-0002-3581-2178>

Marie-Christine Lépy  <https://orcid.org/0000-0002-1917-6802>

Yves Ménesguen  <https://orcid.org/0000-0003-2505-979X>

REFERENCES

- [1] J. E. Martin, N. P. Fox, P. J. Key, *Metrologia* **1985**, *21*, 147.
- [2] F. Lei, J. Fischer, *Metrologia* **1993**, *30*, 297.
- [3] H. Rabus, V. Persch, G. Ulm, *Appl. Opt.* **1997**, *36*, 5421.
- [4] M. Gerlach, M. Krumrey, L. Cibik, P. Müller, G. Ulm, *Nucl. Instr. Meth. A* **2007**, *580*, 218.
- [5] P. Troussel, N. Coron, *Nucl. Instr. Meth. A* **2010**, *614*(2), 260.
- [6] B. Beckhoff, *J. Anal. Spectrom.* **2008**, *23*, 845.
- [7] J. Lubeck, B. Beckhoff, R. Fliegau, I. Holfelder, P. Hönicke, M. Müller, B. Pollakowski, F. Reinhardt, J. Weser, *Rev. Sci. Instrum.* **2013**, *84*, 045106.
- [8] *Basic and General Concepts and Associated Terms (VIM)*, 3rd ed., Vol. JCGM 200, BIPM, Sèvres (France) **2012**.
- [9] M. Rodrigues, R. Mariam, M. Loidl, *EPJ Web Conf.* **2017**, *146*, 10012.
- [10] J. W. Fowler, G. C. O'Neil, B. K. Alpert, D. A. Bennett, E. V. Denison, W. B. Doriese, G. C. Hilton, L. T. Hudson, Y. I. Joe, K. M. Morgan, D. R. Schmidt, D. S. Swetz, C. I. Szabo, J. N. Ullom, *Metrologia* **2021**, *58*, 015016.
- [11] OptoDiode. <https://optodiode.com/photodiodes-axuv-detectors.html>, last visited in July 2022.
- [12] Hamamatsu. <https://www.hamamatsu.com/jp/en/product/optical-sensors/photodiodes/si-photodiodes/S8193.html>, last visited in July 2022.
- [13] F. Salvat, PENELOPE-2014: A Code System for Monte Carlo Simulation of Electron and Photon Transport, OECD/NEA Data Bank. **2015**.
- [14] NEA/NSC/DOC(2015)3, PENELOPE-2014 A Code System for Monte Carlo Simulation of Electron and Photon Transport.
- [15] D. E., Cullen, J. H. Hubbell, L. Kissel, EPDL97, The Evaluated Data Library, '97 Version, Technical Report UCRL-50400, Lawrence Livermore National Laboratory, Livermore, California.
- [16] Y. Ménesguen, M.-C. Lépy, *Nucl. Instr. Meth. A* **2012**, *695*, 193.

How to cite this article: V. H. Elvira, M.-C. Lépy, Y. Ménesguen, *X-Ray Spectrom* **2022**, *1*. <https://doi.org/10.1002/xrs.3318>