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# Radionuclide-free efficiency calibration of an HPGe detector using monochromatic photon beams calibrated with a cryogenic radiometer

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## Abstract

A cryogenic electrical-substitution radiometer was used for the primary calibration of monochromatic photon beams at synchrotron, which are in turn employed to determine the intrinsic efficiency of an HPGe detector between 3.5 keV and 8 keV. This allowed the determination of the thickness of the dead layer, which enabled the calculation of the efficiency up to 55 keV, where the active thickness is not determinant. The geometrical efficiency was determined through the measurement of a radioactive source at variable distances.

## Keywords:

Semiconductor detector, efficiency calibration, photon emission intensities, cryogenic detector, synchrotron radiation

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## 1. Introduction

The Laboratoire National Henri Becquerel (LNHB) maintains and disseminates to users recommended data characterizing the decay schemes of radionuclides. The "Radionuclide Table" (LNHB, 2023), which gathers atomic and nuclear data related to radioactive decay (half-life, branching modes and ratios, energies and intensities of the various emissions, etc.), is fed by the work of an international working group (Decay Data Evaluation Project, DDEP). Among these data, the photon emission intensities are crucial, widely used for the calibration of gamma and X-ray spectrometers, with the aim of quantifying the activity of radionuclides measured by this technique. In the energy range below 50 keV, the photons emitted are mainly X-rays, resulting from atomic relaxation following internal conversion or electron capture processes. This mode of electronic rearrangement competes with Auger electron emission, the split between the two being quantified by the fluorescence yield of the daughter element. The X-ray emission intensities recommended by DDEP are determined by calculation, using internal conversion coefficients or electron capture probabilities and tabulated fluorescence yields: they are therefore fully correlated and the relative standard uncertainties associated with these intensities are of the order of 2 % at best, compared with a few per thousand for gamma emissions. The fluorescence yields used by DDEP (Schönfeld and Janssens, 1996) are derived from compilations based on old measurements. A recent study of the germanium K-fluorescence yield, combining an experimental approach with theoretical calculations (Sampaio *et al.*, 2014) showed a difference of about 4 % between the new values and those of the published bases. Whether for the X-ray analysis techniques or for medical applications (imaging, diagnosis or therapy), accurate knowledge of X-ray emission intensities and fluorescence yields is crucial. Similarly, to calculate radionuclide interactions for many applications, Monte Carlo simulation also uses the characteristics of radionuclide decay schemes, and the reliability and precision of the results are directly proportional to the accuracy of these data. For the spectrometry applications, in the conventional approach, the full-energy peak efficiency calibration is established using standard sources with radionuclides whose emission intensities are well known and whose activity is determined by an absolute method. However, in the

energy range below 100 keV, there are few usable radionuclides, so the experimental points are scattered and it is difficult to determine precisely the position of the maximum of the curve, which leads to large uncertainties in the fitted curve (Lépy *et al.*, 2018). Reciprocally, new photon (X or gamma) emission intensities,  $I(E)$  can be derived from these efficiency calibration curves but the measurement procedure ultimately relies on the same parameters as those one aims to determine. In order to avoid this circular dependency and to make new measurements, independent of the tabulated emission intensities, it is necessary to calibrate the detector according to a procedure which does not require prior knowledge of any emission intensity. With this aim, we have developed a new, radionuclide-free, calibration method to apply it to the characterization of spectrometers in the critical energy range, i.e. between 3 keV and 50 kV. This method is based on the primary determination of the intensity of a monochromatic photon beam by means of a cryogenic electrical-substitution radiometer, BOLUX (BOLometer for Use in the field of X-rays), initially developed by Troussel and Coron (2010), which has been updated and described by Elvira *et al.* (2022), and, in a second step, the measurement of the same photon flux with the spectrometer to be calibrated, using a photodiode as transfer standard.

## 2. Experimental

### 2.1 The cryogenic electrical-substitution radiometer

The measurement principle of a cryogenic detector is based on the temperature change experienced by an absorber when exposed to photon radiation. If the absorber is heated by a continuous flow of photons, when the system reaches equilibrium, the temperature increase is proportional to the total beam power and inversely proportional to the heat transfer coefficient between the absorber and its support. The principle of electrical substitution is to determine the electrical power that must be dissipated in the absorber so that the temperature rise is the same as that induced by the radiant power (Fig. 1).

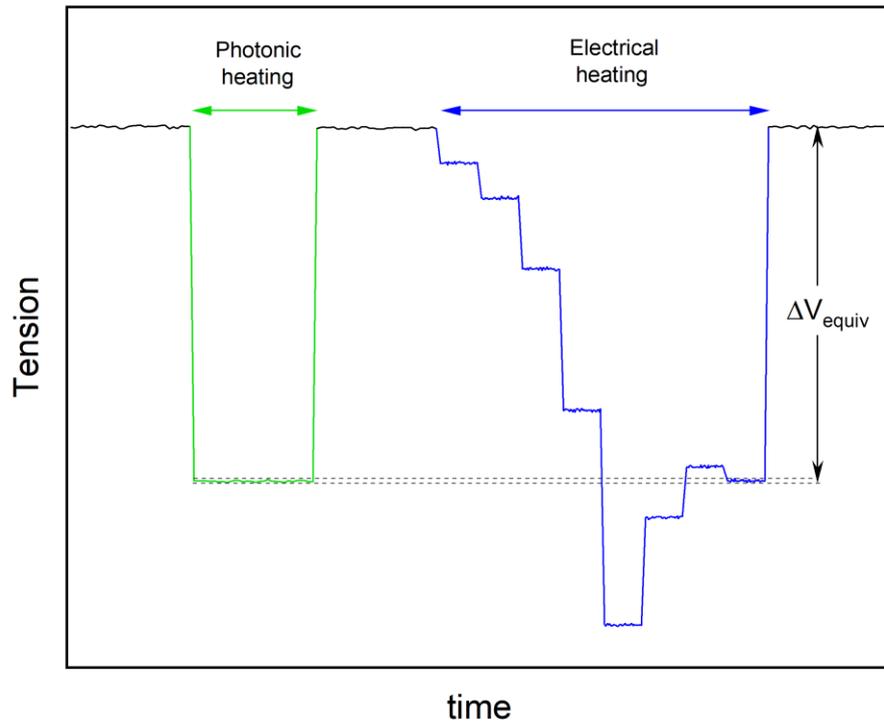


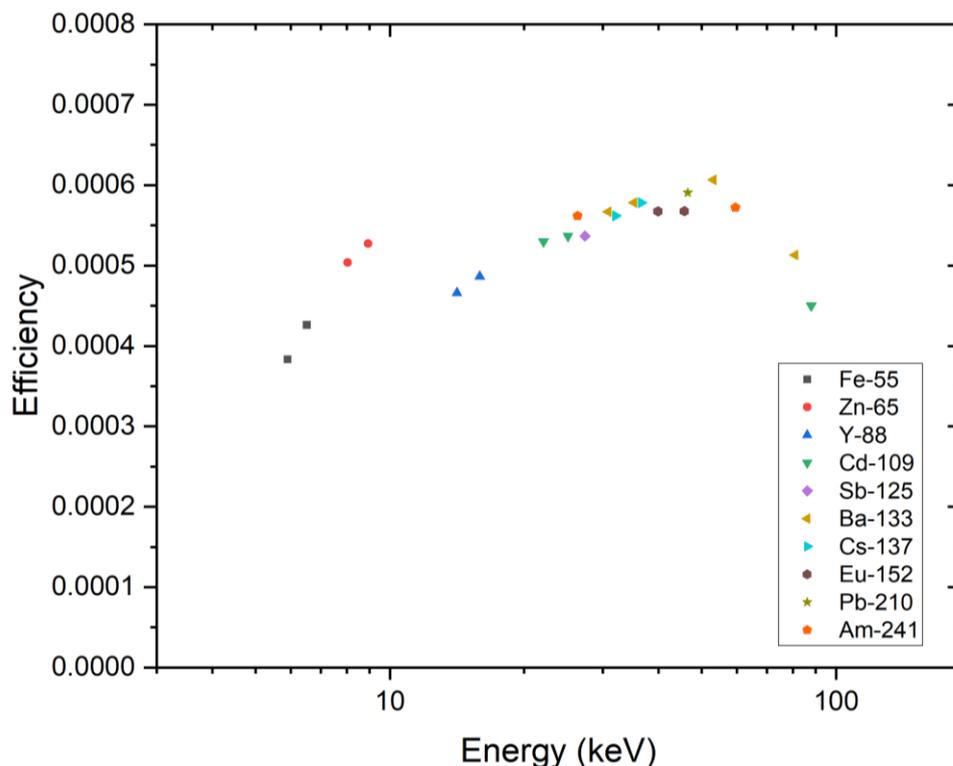
Figure 1. Principle of radiant power measurement with an electrical-substitution radiometer

The temperature changes are monitored by means of a thermistor, stuck to the absorber that is polarized with a constant current, so the change in resistance provoked by the temperature rise induces a variation in the thermistor voltage. The more pronounced the temperature dependence of the resistance, the more sensitive the technique. For this reason, the most sensible option is to use a semiconductor material as a thermistor and to operate at very low temperature. The absorber of the radiometer BOLUX, used in this work, is a 300- $\mu\text{m}$ -thick germanium disc. The thermistor employed to monitor the temperature changes is also made of germanium, and measurements are performed at liquid helium temperature (4 K), in order to steepen the dependence of the resistance with temperature.

## 2.2 The high-purity germanium spectrometer

The spectrometer calibrated in this work is based on a High-Purity Germanium (HPGe) detector supplied by Mirion (Mirion, 2023). It consists of a 6-mm-thick and 10-mm-diameter germanium cylinder, with three successive collimators (1-mm-thick aluminum, 1-mm-thick copper and 0.4-mm-

thick tungsten) with a diameter of 8 mm. The detector window is made of beryllium, with a thickness of 125  $\mu\text{m}$ . The signal is first processed by an integrated preamplifier, and then digitized and analyzed by a *Labzy* nano-MCA device (Yantel, 2023). Each pulse is treated by two different shapers: the fastest one is only used to determine the total incoming count rate, and the slowest one makes the pulse adequate for the spectroscopic analysis. Both of them give the pulse a trapezoidal shape. Given the high count rates measured in this work, we selected the fastest shaping times that do not perturb the shape of the peaks: 0.5  $\mu\text{s}$  as rise time and 0.2  $\mu\text{s}$  as the flat top for the slow shaper, and 0.1  $\mu\text{s}$  as rise time and flat top for the fast shaper. No gain was needed for the signal amplification. For a preliminary characterization of the detector, the efficiency curve was determined by the conventional procedure, using several radionuclide standard sources (Fig. 2). As it can be seen, this approach leads to inconsistencies between the calibration points.



*Figure 2. Efficiency curve of the HPGe detector through the measurement of radioactive standard sources and using tabulated photon emission intensities*

## 2.3 Description of the measurements

The goal of these measurements is to determine the intrinsic efficiency of the spectrometer as the ratio between the count rate measured and the number of incident photons per unit time. In order for the technique to be primary, the number of incident photons per unit time must be determined in an absolute way. In this work, this task is accomplished by the radiometer BOLUX. The electrical substitution radiometer (ESR) is capable of measuring the full radiant power of the beam (i.e. the sum of the energy of all the incident photons per unit time), so, provided that the radiation is monochromatic, the number of incident photons per unit time can be determined by dividing the full radiant power by the energy of each photon. For this reason, the measurements were carried out on the *Métrologie* beamline at the SOLEIL synchrotron (France), which provides monochromatic radiation in the energy range from 3.5 keV to 30 keV, at a rate high enough to be detected and quantified by the radiometer. It must be noticed that BOLUX requires a flux of the order of billions of photons per second, whereas the HPGe spectrometer can only detect up to several tens of thousands of photons per second in a reliable way. In order to circumvent this limitation, a standard transfer is needed: a device that produces a signal proportional to the radiant power and whose response is linear enough so as to be calibrated against BOLUX with a very high flux of photons and then be used to measure a much lower flux for the calibration of the HPGe spectrometer. Photodiodes have proven to provide a good linear response in a very wide range of incident radiant power, and therefore a photodiode was used as a standard transfer in this work. The determination of the intrinsic efficiency of the HPGe detector comprises the two stages:

In the first stage, the ESR is used to calibrate the photodiode in terms of current induced per unit radiant power. To do so, the full radiant power of a monochromatic photon beam is measured by BOLUX, and next the photodiode is placed in the same beam in order to measure the current induced in it by the incident radiation. The photodiode response ( $A/W$ ) is then determined as the ratio between the current induced and the radiant power measured by BOLUX;

In a second stage, with a much lower photon flux, this photodiode is used to determine the number of photons emitted per second in the beam: the current induced by the beam is measured and divided by the A/W response of the photodiode and by the energy of the photon beam. Afterwards, the same beam is measured by the HPGe detector, and its intrinsic full-energy peak (FEP) efficiency is determined as the ratio between the count rate in the FEP and the number of incident photons per second.

This procedure was repeated for several values of the photons energy in order to determine the efficiency calibration of the germanium detector. The photodiode employed as standard transfer is a classic commercial silicon-based photodiode *OptoDiode AXUV* (OptoDiode, 2023), whose response is of the order of hundreds of milliamperes per watt of incident power for low-energy X-rays up to 10 keV, as it was determined by Elvira *et al.* (2022), with uncertainties ranging from 0.4 % to 1 %. The power of the beams used for this measurements ranging from a few microwatts to a few tens of microwatts, they induce a current of the order of several microamperes.

#### 2.4 Correction factor for energy deposition in BOLUX

The determination of the beam power with the ESR requires the calculation of a correction factor to account for the fact that not all the energy of the photon beam is deposited in the absorber, since there are photons that pass through the absorber without interacting with it, and photons that do interact with the absorber but do not deposit all of their energy, as a consequence of the escape of characteristic X rays or scattering. By means of Monte Carlo simulations with PENELOPE code (Salvat, 2019), we calculated the spectrum of the energy deposited in the absorber for monochromatic beams of several energies, in order to compute the total amount of energy deposited in the absorber per incident photon. The ratio between the incident energy and the deposited energy is then the correction factor by which the measured power has to be multiplied in order to obtain the actual power of the beam. Fig. 3 shows the example of the spectrum of deposited energy when the absorber is irradiated by a monochromatic photon beam of 5 keV, where the ratio between the incident energy and the deposited energy (weighted sum of the spectrum channel contents) is 1.007. Fig. 4 shows the

correction factor for BOLUX calculated by the procedure explained above for different photon energies that represent generally a relative correction lower than 1 %.

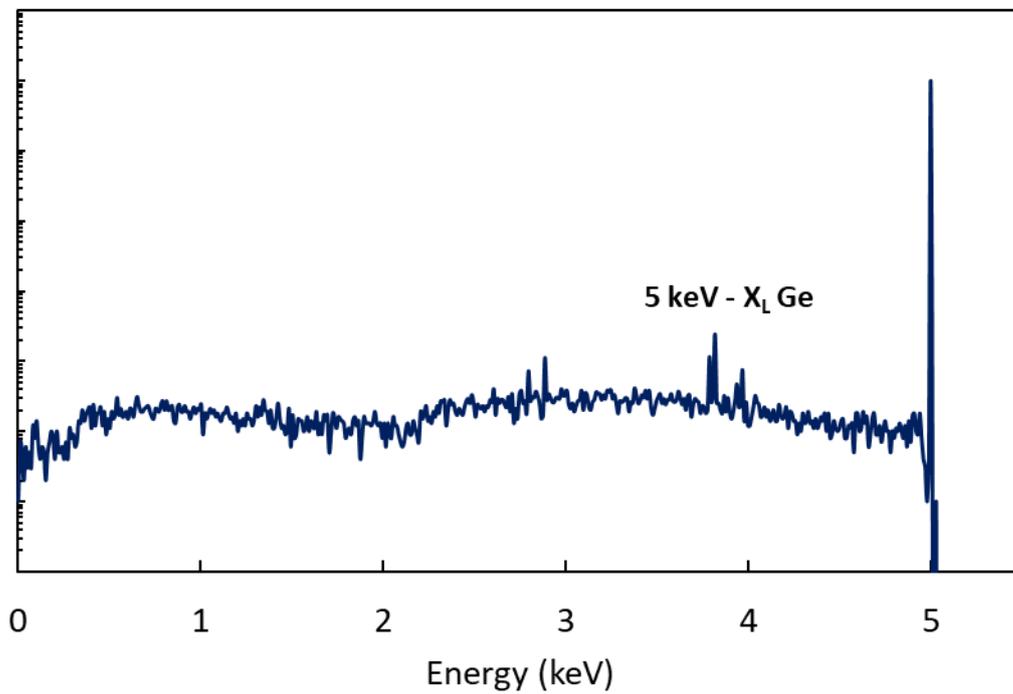


Figure 3. Simulated spectrum of energy deposited in the ESR absorber for incident photons of 5 keV

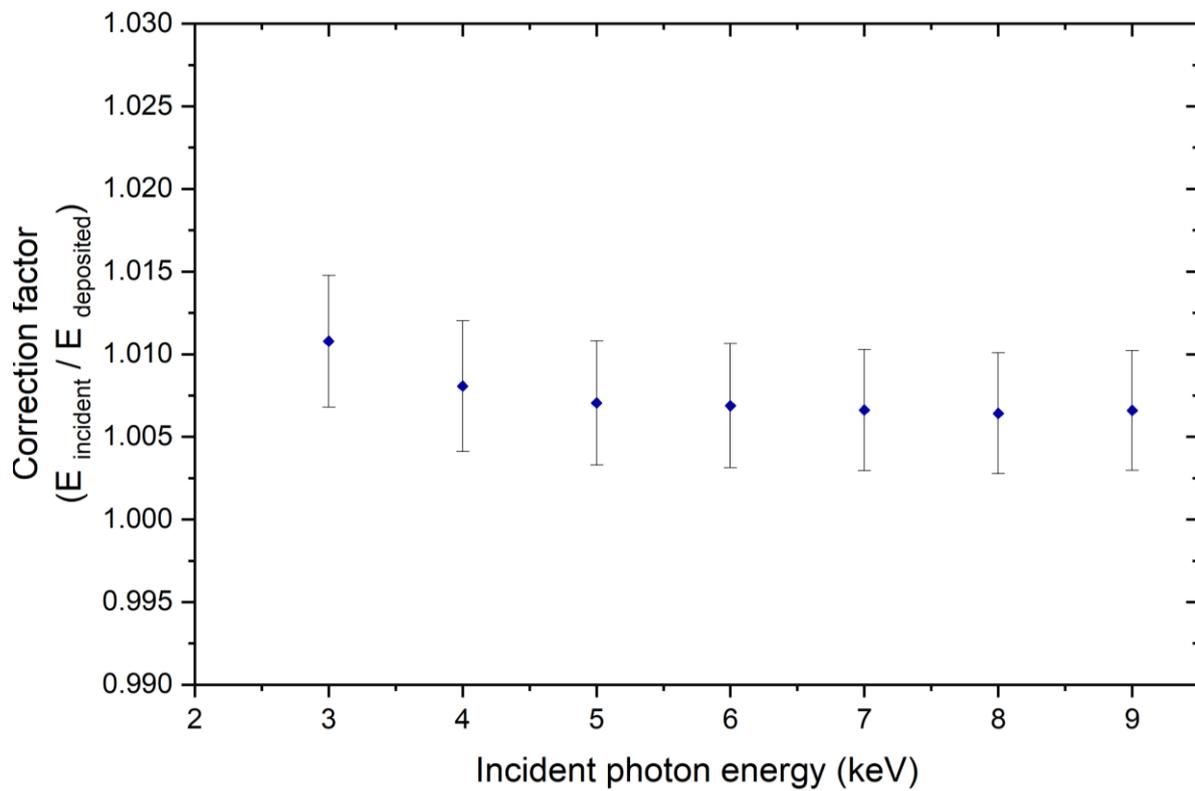


Figure 4. Calculated correction factor for the energy deposition in BOLUX

## 2.5 Optimization of the beam in terms of spectral purity

The correction factor for BOLUX and the response of the photodiode depend on the energy of the radiation. However, neither of them are able to discriminate photons by energy. For this reason, it is essential that the radiation is actually monochromatic, so that one can divide the full power by the energy of the photons in order to obtain the number of photons emitted per unit time. However, a synchrotron beamline selects the radiation's wavelength according to Bragg's law:

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

so, when a given energy  $E_i$  is aimed, all its multiple integers  $n E_i$  are selected as well. It is then necessary to minimize the fraction of photons with  $n$  higher than 1 (higher order harmonics).

The monochromator in the *Métrologie* beamline at the SOLEIL synchrotron is equipped with an additional crystal that can be detuned with respect to the first one. As the detuning angle is increased, the photon flux of the main energy decreases, but the flux of higher order harmonics decreases in a greater proportion, so it is possible to find a detuning angle for which the contribution of higher order harmonics is negligible while keeping a significant flux of photons of the targeted energy. All measurements therefore require a preliminary step of finding a suitable detuning angle for each energy. To do so, the energy spectrum of the beam was measured with the HPGe detector for increasing values of the detuning angle in order to determine the fraction of photons of each harmonic. An example of this result at 4 keV is shown in Fig. 5 (notice that the harmonic  $n=2$  is not permitted by the selection rules for the crystal Si111 used at *Métrologie* beamline). It must be pointed out that the accurate determination of the distribution of harmonics would require to know the detection efficiency of the spectrometer for each energy beforehand, but at this stage the spectrometer had not been calibrated yet. Therefore, a mere estimation of the detection efficiency, based on the specification of the manufacturers, was used, since the only goal of these measurements was to make sure that the contribution of higher order harmonics was negligible (below  $10^{-3}$ ) at the selected detuning angle. For example, at the maximum intensity (angle = 0.567 mrad), the intensities

of harmonics 3, 4 and 5 relative to the main energy are respectively 2.3 %, 0.9 % and 0.2 %, while when the angle is detuned to 0.58 mrad, these decrease to 0.1 %, 0.02 % and  $10^{-3}$  %, respectively.

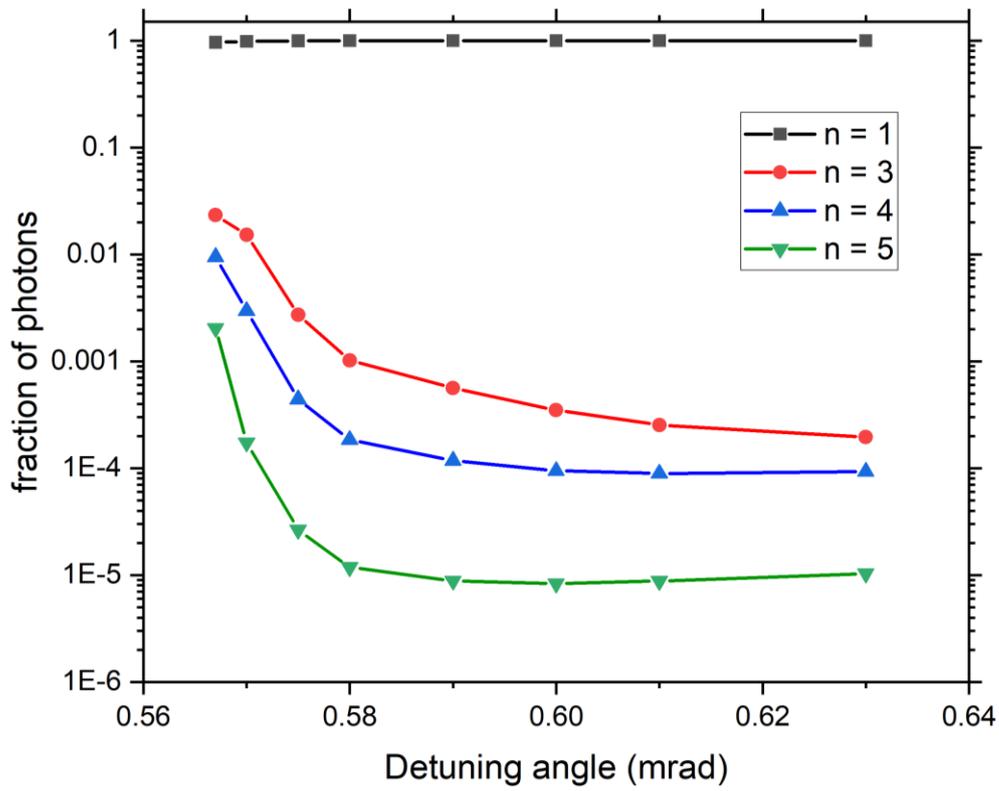


Figure 5. Fraction of harmonics emitted for a selected energy of 4 keV as a function of the detuning angle

### 3. Experimental results

By the procedure explained above, the intrinsic efficiency of the HPGe was determined in the energy range from 3.5 keV to 8 keV, where it was possible to achieve a good compromise between the photon flux required by the detection limit of the photodiode and the one tolerated by the HPGe. Fig. 6 represents the results obtained in two different measurement campaigns with a good reproducibility. This energy range does not cover the whole range of interest. Nevertheless, it allows the determination the thickness of the dead layer though Monte Carlo simulations.

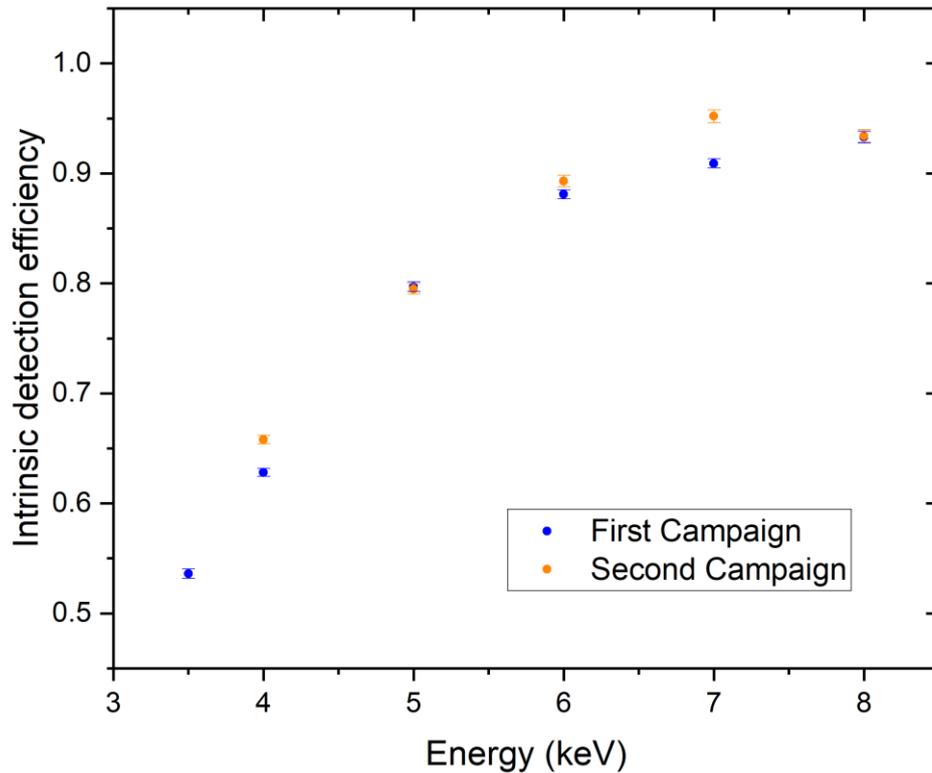


Figure 6. Experimental intrinsic detection efficiency of the HPGe detector determined at the synchrotron beamline in two measurement campaigns

The uncertainty in the determination of the intrinsic efficiency arises from the uncertainty in the measured count rate and the uncertainty in the determination of the number of photons emitted per second.

- The uncertainty in the count rate is a statistical uncertainty, which barely amounts to a few hundredths of a percent, since the total number of counts in the FEP of the measured spectra is of several millions.
- The uncertainty in the determination of the number of photons per second is mainly due to the measurement of the beam power, since the uncertainty in the energy of the photons (spectral purity) in the synchrotron is completely negligible. The uncertainty in the measurement of the beam power has two main components:

- The uncertainty in the determination of the photodiode's response. This component has been analyzed in Elvira *et al.* (2022), and it ranges from 0.8 % at the lowest part of the curve to 0.4 % at 8 keV.
- The uncertainty in the determination of the current induced in the photodiode. This uncertainty component can be estimated from the standard deviation of a series of measurements of the current induced by the beam and of the background current. It amounts to 0.2 % at the lowest part of the curve, where the photodiode response is higher, to 0.4 % where the photodiode is less performant.

### 3.1 Determination of the germanium dead layer

By means of Monte Carlo simulations with PENELOPE code (Salvat, 2019), the intrinsic efficiency of the detector was calculated for several values of the thickness of the dead layer (Fig. 7). The model for the simulation consisted of a 6-mm-thick germanium cylinder used as the detector body (active zone), a thin germanium disc of variable thickness (dead layer) in front of it, and a 125- $\mu\text{m}$ -thick beryllium disc in front of them (detector window). The geometrical model is immersed into void, in order to recreate the conditions of the synchrotron beamline where the measurements were carried out. The simulated beams were perfectly collimated and of infinitesimal section, impinging on the center of the cylinder. From the simulations performed for several thicknesses of the germanium dead layer, it was concluded that the one that best describes the experimental results is 1.1  $\mu\text{m}$ .

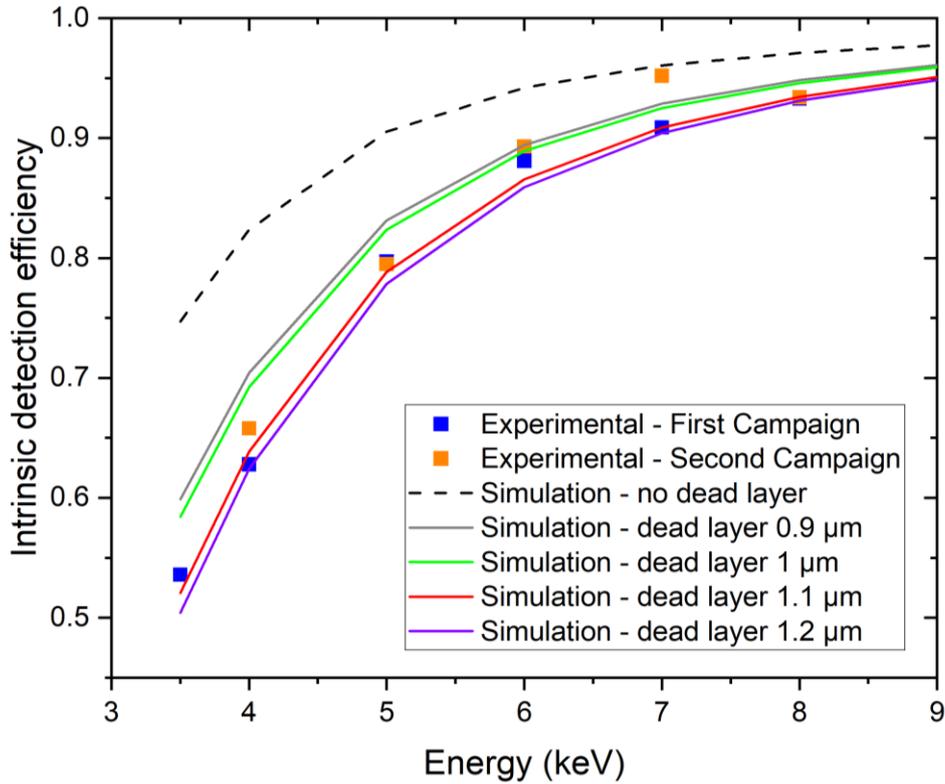
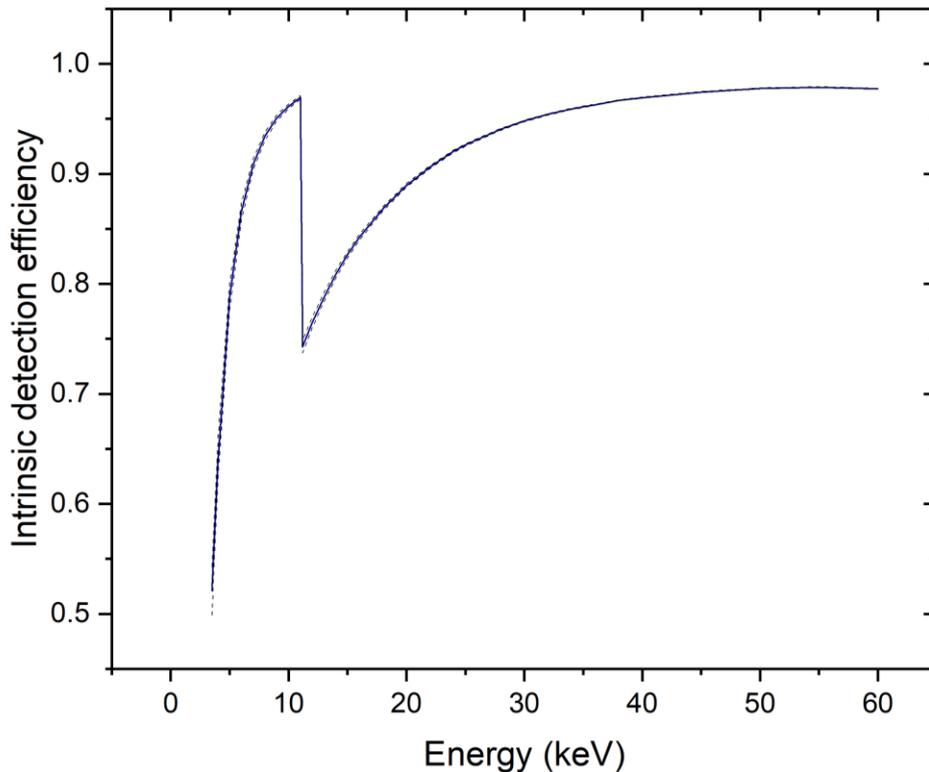


Figure 7. Determination of the dead layer thickness by the comparison of Monte Carlo simulations to experimental results

### 3.2 Extension of the efficiency curve

The determination of the dead layer enables the extension of the efficiency curve to a much wider energy range via Monte Carlo simulation. The intrinsic efficiency is limited, in the lowest part of the curve, by the photons absorbed in the beryllium window and in the dead layer, and above a certain energy, by the photons that pass through the detector without interacting (thus determined by the thickness of the active layer). Therefore, once the thickness of the dead layer has been determined, the efficiency can be calculated in the whole energy range where the transmission of photons is negligible, that is to say where the thickness of the active part is not critical. Fig. 8 shows the intrinsic efficiency calculated by means of Monte Carlo simulations with PENELOPE code for a 6-mm thick detector with a dead layer of 1.1  $\mu\text{m}$  in the front part. Complementary simulations have been performed for a much thinner active part (5 mm instead of 6 mm) in order to confirm that this thickness is not critical below 55 keV.



*Figure 8. Intrinsic efficiency curve of the HPGe detector obtained by Monte Carlo simulations taking into account the dead layer thickness experimentally determined*

In the whole energy range where the transmission through the germanium crystal is negligible, the uncertainty in the calculated efficiency is governed by the uncertainty in the transmission through the beryllium window and the transmission through the germanium dead layer. Each of them stems from the uncertainty in the thickness and the uncertainty in the interaction cross-section used in the simulation. The contribution of the relative uncertainty in the cross-section and the thickness to the relative uncertainty in the transmission is weighted, in a good approximation, by the logarithm of the transmission itself.

- The beryllium window being a flat element of significant size, it is quite a conservative estimation to limit it to 10 %.
- Simulations were made for different thickness to the dead layer, and it was possible to discriminate the results for thicknesses 0.1  $\mu\text{m}$  apart, so it is judicious to estimate half of that interval as the uncertainty in the thickness.

- The uncertainty in the cross-section used by PENELOPE code are estimated to be of a few percent for energies above 1 keV (Cullen, 1997; NEA 2015), so as a conservative approach a 5 % uncertainty can be considered for the entire energy range considered

This gives a total uncertainty in the intrinsic efficiency of a few percent in the lowest part of the curve (3 keV to 5 keV), of a few per-thousand up to 20 keV and nearly negligible above that energy up to 55 keV.

### 3.3 Determination of the geometrical efficiency

It should be noted that the experiments to determine the intrinsic efficiency at the synchrotron were carried out with a very thin collimated beam (of the order of tenths of a millimeter), so all the photons impinge on a very small central part of the germanium crystal. However, when the detector is used to measure a radioactive source that emits isotropic radiation, photons impinge on a much wider surface of the crystal. For this reason, it is only possible to extrapolate the results of the intrinsic efficiency measured at the synchrotron if it is verified that the detector response is homogenous over the entire area defined by the detector collimator. To this end, the count rate of the same collimated photon beam was measured by varying the position of the detector relative to the beam in both perpendicular directions, in order to scan the detection surface. A good homogeneity was found: the count rate is always within 0.5 % the count rate at the center for measurements along the horizontal axis and within 0.3 % for measurements along the vertical axis.

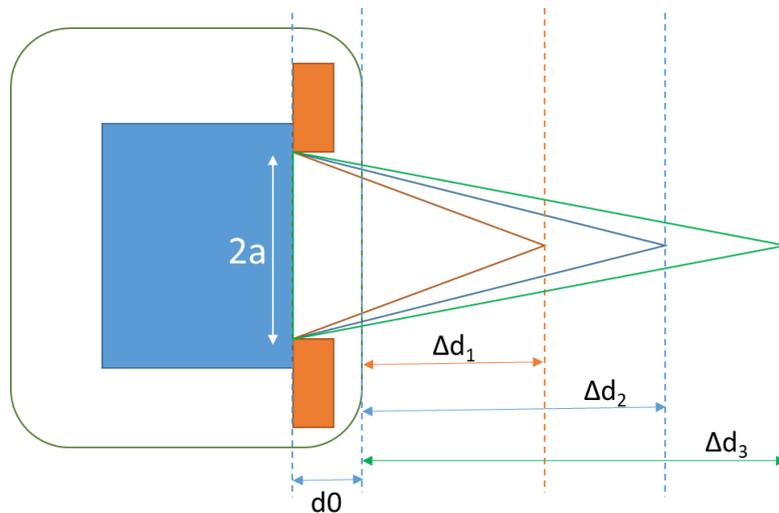
Therefore, if the goal of the detector is to measure the emission rate of radionuclide sources, it must be characterized in terms of its overall efficiency,  $\varepsilon(E)$ , which also comprises the geometrical efficiency,  $\varepsilon_{geom}$ , i.e., the solid angle subtended by the collimator and the source, as a fraction of the full sphere:

$$\varepsilon(E) = \varepsilon_{intr}(E) \cdot \varepsilon_{geom} = \varepsilon_{intr}(E) \cdot \Omega/4\pi$$

To determine the geometrical efficiency, the same  $^{133}\text{Ba}$  source was measured at variable distances, and the count rate of the  $K\alpha$  peak was fitted to the equation of the solid angle subtended by a circle at a point laying on its axis:

$$r = r_{4\pi} 0.5 \left( 1 - \frac{(d_0 + \Delta d)}{\sqrt{(d_0 + \Delta d)^2 + a^2}} \right)$$

where (Fig. 9)  $a$  is the radius of the collimator,  $d_0$  is an unknown offset to the source-to-collimator distances,  $\Delta d$  is the additional source-to-collimator distance (which is well known for each measurement),  $r$  is the count rate measured at the distance  $d_0 + \Delta d$  and  $r_{4\pi}$  is the extrapolation of the count rate to the whole sphere. Both  $r_{4\pi}$  and  $d_0$  are left as free parameters in the non-linear fit, and the radius of the collimator,  $a$ , is fixed to 0.4 mm. The source-to-collimator distances are long enough for the source to be considered as a point source. The count rates shown in Fig. 10, used for the fit, were corrected for the attenuation in the excess of path length into air with respect to the closest measurement.



*Figure 9. Principle of the determination of the geometrical efficiency through the measurement of a radioactive source at variable distances*

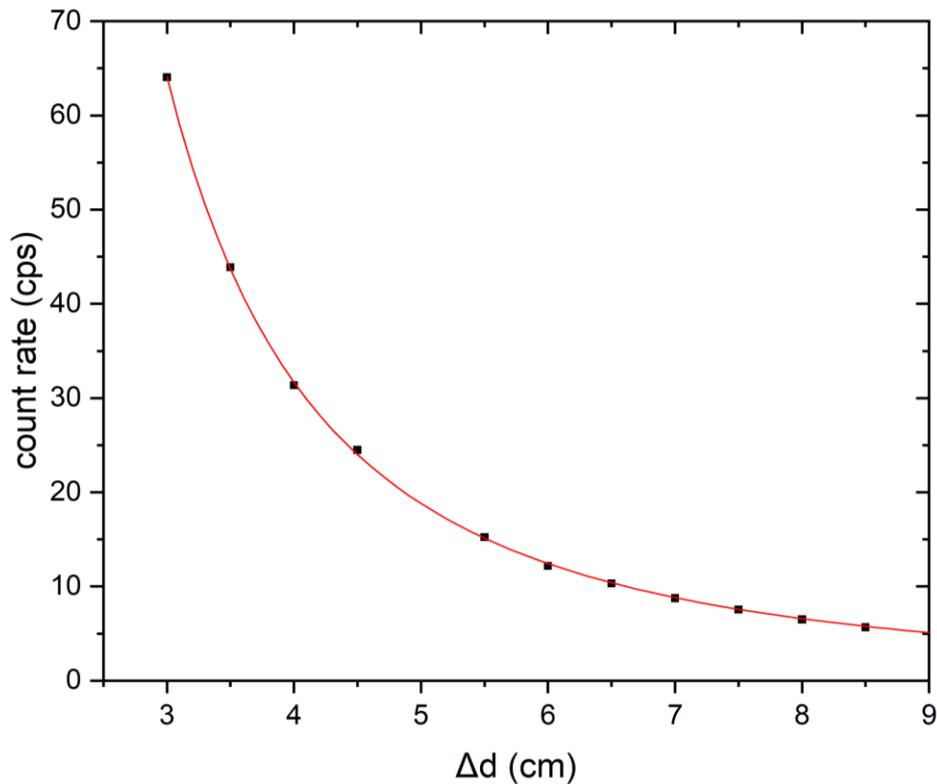


Figure 10. Count rate of the  $K\alpha$  peak of a  $^{133}\text{Ba}$  source measured at variable distances and fit to the equation of the solid angle

The distance offset obtained from the fit is:

$$d_0 = 0.6767 \pm 0.0165$$

This allows us to determine the solid angle at each position of the source. For the source-to-detector distance (about 4.5 cm) as the one customarily used in our set-up, the resulting geometrical efficiency is  $5.76 \times 10^{-4}$ , with a relative uncertainty of 0.4 %.

Finally, the total uncertainty in the overall detection efficiency, taking into account the uncertainties in the intrinsic and geometrical efficiency, is then around 1 % in the lowest part of the curve and around 0.4 % above 20 keV (up to 55 keV), where the only significant contribution is the uncertainty in the solid angle.

## Conclusions

The cryogenic electrical-substitution radiometer BOLUX, which has been recently set up and restarted at CEA/LNHB (Elvira *et al.*, 2021, 2022) has been used for the primary measurement of the radiant beam power of monochromatic photon fluxes of several energies at SOLEIL synchrotron. Those photon beams of known power allowed to determine the response of a photodiode in terms of current induced per unit radiant power. The calibrated photodiode was used to determine the number of photons emitted per second in a much weaker photon beam, which was employed to determine the intrinsic efficiency of an HPGe detector. Consistent and reproducible results were obtained between 3.5 keV and 8 keV, where it was possible to find a good agreement between the photon flux required by the photodiode and the one tolerated by the HPGe detector. This allowed us to determine the thickness of the detector dead layer, which made it possible to derive the intrinsic efficiency through Monte Carlo simulations in the whole energy interval where the thickness of the active layer is not determinant (up to 55 keV), with uncertainties of a few per-thousand up to 20 keV and nearly negligible above that energy up to 55 keV. The geometrical efficiency was determined with a relative uncertainty of around 0.4 % by the procedure of measuring the same source at variable distances and fitting the count rate to the equation of the solid angle. This enabled us to determine the overall FEP efficiency of the detector (for its practical use with radioactive sources) with uncertainties close to 1 % in the lowest part of the curve and down to 0.4 between 20 keV and 55 keV. The determination of the detector efficiency with a small uncertainty by a procedure that is independent of any previous knowledge of any radionuclide emission intensities will allow the application of this detector to the measurement of the emission intensities of radionuclides of interest for X-ray analysis techniques, medical applications or radiological protection.

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## Figure captions

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