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Efficiency calibration of an HPGe X-ray detector for quantitative PIXE analysis

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ABSTRACT

Particle Induced X-ray Emission (PIXE) is an analytical technique, which provides reliably and accurately quantitative results without the need of standards when the efficiency of the X-ray detection system is calibrated. The ion beam microprobe of the Ion Beam Modification and Analysis Laboratory at the University of North Texas is equipped with a 100 mm^2 high purity germanium X-ray detector (Canberra GUL0110 Ultra-LEGe). In order to calibrate the efficiency of the detector for standard less PIXE analysis we have measured the X-ray yield of a set of commercially available X-ray fluorescence standards. The set contained elements from low atomic number $Z = 11$ (sodium) to higher atomic numbers to cover the X-ray energy region from 1.25 keV to about 20 keV where the detector is most efficient. The effective charge was obtained from the proton backscattering yield of a calibrated particle detector.

1. Introduction

A key factor in quantitative X-ray analysis using HPGe detector is the efficiency of the detector in use. This requires the calculation or measurements of detection efficiency ε, as a function of X-ray energy. The absolute efficiency of a detector can generally be defined as the measure of how many pulses occur for a given number of X-rays striking its window. This efficiency is related to the specific source–detector geometry and the transmission and absorption properties of the detector [2,3]. It generally denotes the ratio of the number of counts produced by the detector to the total number of radiations emitted by the source. The intrinsic efficiency on the other hand is the ratio of the number of pulses produced by the detector to the actual number of X-rays striking the detector window. It takes into account the X-ray transmission through the beryllium entrance window of the cryostat, a possible ice layer due to the cooling of crystal by liquid nitrogen and the absorption in the germanium active volume. For each experimental situation, it is paramount to consider the solid angle of detection and any additional absorbing layers (75 μm polyethylene layer for our case) placed in front of the detector window [4].

Ge-detectors were normally used for medium and high energy γ-ray analysis while Si(Li) detectors had been widely used for low energy X-ray analysis. However with the improvement of the fabrication properties, Ge-detectors are now made for low X-ray energy applications [8]. This has been made possible due to improved resolution and large solid angle together with advantages like room temperature storage. In the present work, we have calibrated the efficiency of the GUL0110-HPGe detector for standard less PIXE analysis of biological samples by directly measuring the X-ray yield with proton beams of a set of commercially available X-ray fluorescence standards.

2. The solid angle verses detector position

The solid angle of the detector can be derived from the yield measurements of an X-ray source from a standard radioactive source detected at different detector positions for a given detector angle. We used Fe-55 radioactive source of Mn X-rays, mounted at the center of the chamber to determine the total counts under the measured spectrum by the detector and the pulse counter readings from the output of the detector amplifier for different detector positions. The measurements at each detector distance were taken for 60 s. The data for the total counts under the spectrum and pulse counter readings were plotted against the detector distance from the source using Origin. A fit function for the yield $Y(d)$ against detector distance d (mm), was generated with fit parameters including the Ge-crystal distance from the front of the detector d_{off}, and the source activity after 60 s, A. The resulting plot (Fig. 1a) was fitted to obtain solid angle at various detector distances (Fig. 1b). The usable detector solid angle for measurements using proton

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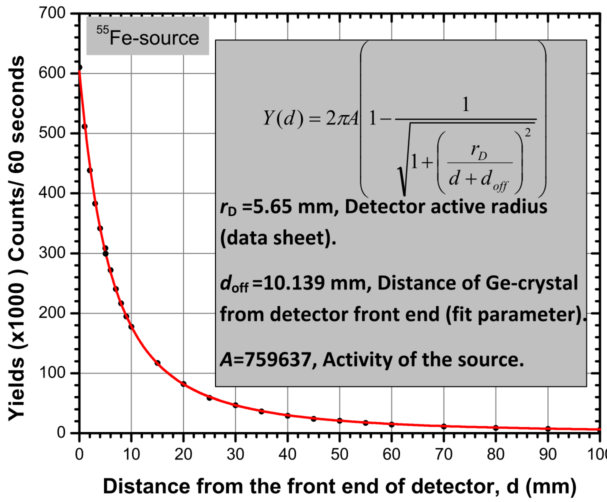

Fig. 1a. Fitted plot of Yields from ^{55}Fe source against distance from the front end of the detector. The figure shows a fit curve of the X-ray yield against detector distance with the fitting parameters as defined above. Since yield is proportional to the solid angle, the fit curve was used to produce the solid angle against detector distance graph.

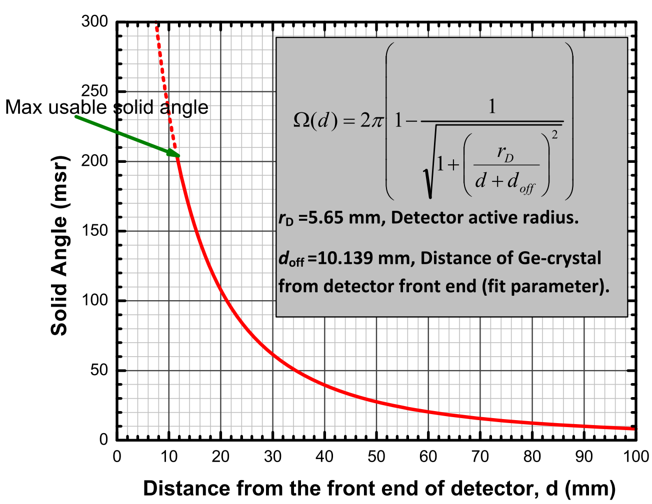

Fig. 1b. Solid angle derived from 55Fe-source measurements in Fig. 1a.

beams was later determined as 205 msr with the front of the detector positioned at about 11 mm from the sample. This distance is the closest limit so as not to block the beam and touch the sample with the detector that is mounted at 135°.

3. Intrinsic efficiency – energy relationship

The intrinsic detection efficiency ε depends on: solid angle Ω subtended by the detector, transmission $T(E)$ between X-ray source and detector crystal through absorbers (detector window, potential ice layer, gold-front contact, germanium dead layer in the crystal), and the absorbance $A(E)$ in the active volume of the Ge-crystal. For each experimental situation, additional absorbing layers in front of the detector, like a proton stop filter have to be considered (we used 75 µm polyethylene). The intrinsic efficiency of the detector as a function of energy following Hansel model is thus given by equation [1,7].

$$\varepsilon_i(E) = [T(E) \cdot A(E) \cdot Fc] \quad (1)$$

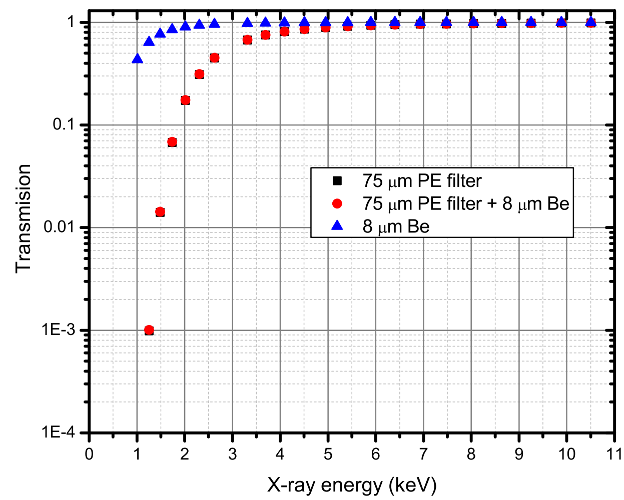

Fig. 2. Effect of filter on transmission against X-ray energy.

The transmission and absorption factors are expressed as follows:

$$T(E) = \exp\left(-\sum_{\text{layers}} \mu_l \cdot d_l\right) \quad \text{and} \quad A(E) = 1 - \exp(-\tau_{Ge} \cdot d_{Ge}) \quad (2)$$

where μ_l is the total linear attenuation coefficient for X-rays with energy E for a material of layer l, d_l is the layer thickness, τ_{Ge} is the photo-absorption coefficient of germanium and d_{Ge} is the detector thickness. The transmission of various X-rays through the 75 µm polyethylene filter and the 8 µm Beryllium in front of the detector are shown in Fig. 2. When no polyethylene filter is used, the Beryllium that comes with the detector allows low energy X-rays including Na to be transmitted. However, the detector cannot be used without the Polyethylene filter since backscattered protons will damage the detector crystal.

4. PIXE method

A 2 MeV proton beam delivered by the 3 MV 9SDH-2 (NEC) tandem accelerator at the IBMAL, in the physics department of University of North Texas was used to excite X-rays in thick standard samples (acquired from Geller Micro-analytical laboratory, Inc) of single and multi-element composition with energy range from 1.25 to 25 keV. The standards included Al, Mg, KCl, ZnS, GaP, Sc, Ti, CaF2, Mn, Cr, V, Ni, Co, Fe, Zr, InAs, and Cu. Simultaneous PIXE and RBS measurements were taken. The samples were positioned at the center of the chamber with the X-ray detector placed at 135°, at a distance 12 mm corresponding to the solid angle of 205.335 msr. A 75 µm polyethylene filter was interposed in front of the detector to stop the back scattered protons. Partially depleted PIPS (series: PD-25-10-500 AM) particle detector was mounted at 170°, 70 mm from the sample for RBS measurements. Due to low solid angle (5.1×10^{-3} sr) of the PIPS detector, the simultaneous PIXE and RBS data were collected for between (45 and 75 min) to obtain significant statistics at least 1000 counts for the respective samples. The detector under investigation was made by Canberra Industries Inc. and it is an ultra low energy HPGe-detector which came equipped with transistor reset preamplifier (model ITRP). The detector has an active thickness of 10 mm and an area of 100.287 mm^2 with a bias voltage of −800 V. The detector crystal distance from the window, which is made of Beryllium of thickness 8 µm, is 10.139 mm. The resolution is 154 eV FWHM at 5.9 keV with optimum shaping time constant of 12 µs as provided in the manufacturer's manual [8].

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