

# Energy and Efficiency Calibrations for High Purity Germanium GEM30195 Coaxial Detector USING $k_0$ -IAEA Software

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**Abstract:** We have applied  $k_0$ -IAEA software in this study to calibrate the high-purity coaxial detector (HPGe) GEM 30195 (ORTEC ©) at the Nigeria Research Reactor-1 (NIRR-1) laboratory, Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria for use in the analysis of various samples. In order to achieve this, we first performed the energy calibration, plotted the energy calibration curves, and after that performed the full peak energy efficiency (FEPE) of the detector using two standard sources  $^{137}\text{Cs}$  and  $^{152}\text{Eu}$  at 15cm and 2cm geometries, and obtained values within the energy range of 121.78 – 1528.12 keV and 244.69 – 1408.00 keV respectively for 15 cm and 2 cm source-detector geometries with each recording the chi-square values of 1.0 and 3.6, where their z-values shows that all doublets at the photopeaks were correctly resolved. Besides, the results we obtained generally agrees with the limit of acceptance level of the software, and also shows good agreement with that carried out previously by other researchers.

**Keywords:** Detector calibration,  $k_0$ -IAEA software, NIRR-1, HPGe detector, FEPE

## 1. Introduction

In most facilities available for radioactivity measurements,  $\gamma$ -ray spectrometers are widely used worldwide [1], [2] because it is a non-destructive method and in general does not require sample preparation [3]. More so, it allows qualitative and quantitative analysis of radioactive material in all forms [4], [5]. Generally,  $\gamma$ -ray spectrometer consists of a semiconductor detector, coupled with associated electronics, and a computer-based, multi-channel analyzer [6], [7]. Reports indicated that many neutron activation analysis (NAA) laboratories operate one or more hyper-pure or intrinsic germanium (HPGe) detectors at liquid nitrogen temperatures (77 degrees K) for optimum performance [8] by mounting the germanium crystal in a vacuum cryostat, thermally connected to a copper rod or "cold finger" [9], [10]. Majority of this type of detectors are the coaxial detector which in NAA is useful for measurement of gamma-rays with energies ranging from about 60 keV to 4.0 MeV [11].

Though, to ensure accurate quantification as is the case for all analytical techniques,  $\gamma$ -ray spectrometry requires standard samples to establish an experimental efficiency calibration which is the most accurate method. However, standard samples are in general costly or most times unavailable and also the standard samples would need to be renewed occasionally, for example due to the short half-life of some radionuclides. Besides, according to Simonits, *et al.*, [12] during the Instrumental Neutron Activation Analysis (INAA) (relative) method quantification analysis is impossible for unexpected elements since no standards are provided; such that since standards are not provided, the calculation of detection limits (maximum possible concentration) is impossible; sometime difficulties of identical irradiation conditions may arise during the

irradiation of standards and samples or due to flux inhomogeneities along the irradiation can, or it may be lack of space in the irradiation capsule, among others; and handling of a large number of standards causes great difficulties in computer-coupled, automated activation analysis.

It was in order to overcome some of these challenges that the  $k_0$ -IAEA software was developed and distributed to various laboratories [13] after the development of the  $k_0$ -standardization technique of NAA in the 1970s [12] for multi-element analysis. For this reason,  $k_0$ -standardization in NAA is increasingly being used in many laboratories worldwide as reported during the various Proceedings of the International  $k_0$ -Users Workshop [14] – [18]. The  $k_0$ -NAA standardized Method has also been employed in the Nigeria Research Reactor-1 (NIRR-1), where the characterization of the irradiation channels and the experimental protocols has been developed and described [10], [19] & [20]. The  $k_0$ -IAEA software developed (IAEA-TECDOC-1565)[21] have the capability for the determination of peak position in a spectrum, performs energy calibration and plots energy calibration curves, determinations of peak width (Full Width Half Maximum, FWHM) and efficiency calibration coefficients, amongst others. This work therefore focuses on the energy and efficiency calibrations of the high-purity coaxial detector (HPGe) GEM 30195 at the Nigeria Research Reactor-1 (NIRR-1) laboratory, Centre for Energy Research and Training, ABU Zaria, which of course is an important step to achieving accurate results when  $k_0$ -NAA standardized method is adopted for sample analysis. The measured parameters needed for calibration of this detector has been reported [30].

## 2. Experimental

The  $k_0$ -IAEA program has the following main components as shown in figure 1. The main program which uses nuclear data that do not change but are updated from time to time can write on and read from the permanent and series database. The permanent database is edited only once while the series database can be edited anytime. From an experimental viewpoint, the implementation of the  $k_0$ -IAEA software can be divided into four basic steps: (i) the first step is to edit the permanent database, by entering the Analysts name, certificates of comparators and the reference materials including the certified activities of the calibration sources, input the name of detectors, the irradiation facilities, sample capsule, material composition; (ii) the second step is to calibrate the detector; (iii) the third step is to characterize the irradiation facilities, and (iv) the fourth step is to analyze samples and report them.

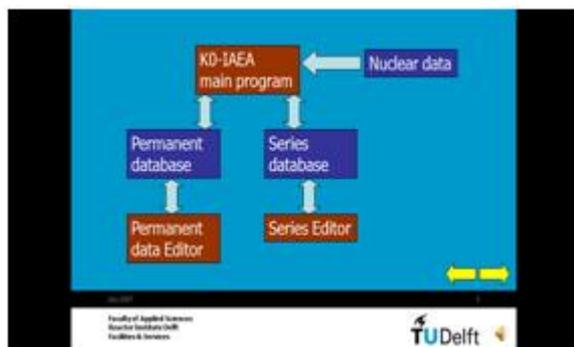


Figure 1: Overview of the main components of  $k_0$ -IAEA program [22]

All our measurements were done using high-purity coaxial germanium detector (HPGe) model number 30195 (ORTEC ©) with relative efficiency of 30% and a resolution of 1.95 keV (FWHM) for the 1.33 MeV gamma line of  $^{60}\text{Co}$ . The detector's dimensions; crystal diameter 58.8mm, crystal length 76.3mm, end cap to crystal 3mm, aluminum absorbing layer 1.27mm, inactive germanium layer 0.7mm, and the top cover diameter 72.4mm were all entered into the permanent database of the  $k_0$ -IAEA program together with the certificates of the radionuclide [1].

### 2.1 Energy Calibration

For the peak areas determination and energy calibration, we created two samples-the first is for the background count, while the second (point source, Eu-152) is for the energy and shape calibrations as well as the peak area determination. The description of the samples used for the measurement and energy calibration as well as the calibration of gamma-ray spectrometer is listed on table 1. The Photon Emission Rates (PER) from the sealed standard radioactive sources used in this work (table 2) were measured.

Table 2: Photon energies used for this work

Isotopes	Energy (keV)	Branching ratio (%)
$^{152}\text{Eu}$	121.78	28.21
	244.70	7.20
	344.20	26.41
	411.00	2.30
$^{137}\text{Cs}$	661.60	85.00

$^{152}\text{Eu}$	778.90	13.00
	964.00	14.48
	1112.00	13.55
	1408.70	20.71

(Source: [11])

The packaging (which determine the dimension of the sample) and the unpackaging actions which are part of the sample history for the  $k_0$ -IAEA program have been evaluated. The details concerning each of the sample history used for the peak area measurement and calibrations performed are indicated on tables 3 to 6. The details on tables 3 to 6 which were before the detector was calibrated comprised of the mass of the sample, duration the sample measured, geometry (source-detector distances for counting with the GEM 30195) and the time the sample was removed from the spectrometer.

Table 3: Sample History for Energy/Shape Calibration and Peak Area Measurement

History of sample: 2 (ENERGY CALIBRATION-Eu-152@15cm)  
 Jul 15 2004 12:00:0.000: 0.100 mg of sample packaged in recipient 1 (none)  
 Jul 30 2014 17:02:0.000 (01h00): with GEM30195 at 150.0 mm (efficiency unknown)  
 Aug 1 2014 12:00:0.000: sample unpacked from recipient 1 (none)

Table 4: Sample History for p/t Curve Measurement

History of sample: 3 (PEAK - TOTAL RATIO-Cs-137@15cm)  
 Jul 15 2004 12:00:0.000: 0.100 mg of sample packaged in recipient 1 (none)  
 Jul 30 2014 16:00:0.000 (01h00): with GEM30195 at 150.0 mm (efficiency unknown)  
 Aug 1 2014 12:00:0.000: sample unpacked from recipient 1 (none)

Table 5: Sample History for Efficiency Measurement at 15 cm

History of sample: 4 (EFFI. CALIBRATION-Eu-152 @15cm)  
 Jul 15 2004 12:00:0.000: 0.100 mg of sample packaged in recipient 1 (none)  
 Jul 30 2014 17:02:0.000 (01h00): with GEM30195 at 150.0 mm (efficiency unknown)  
 Aug 1 2014 12:00:0.000: sample unpacked from recipient 1 (none)

Table 6: Sample History for Efficiency Measurement at 2 cm

History of sample: 5 (EFFI. CALIBRATION-Eu-152 @ 2cm)  
 Jul 15 2004 12:00:0.000: 0.100 mg of sample packaged in recipient 1 (none)  
 Jul 31 2014 13:31:51.000 (01h05): with GEM30195 at 20.0 mm (efficiency unknown)  
 Aug 1 2014 12:00:0.000: sample unpacked from recipient 1 (none)

After obtaining spectra, (where for this study we obtained our spectra from the MAETRO software in the NAA counting room at Centre for Energy Research and Training, CERT, Ahmadu Bello University, Zaria), the procedure to perform the spectra analysis has been described [22]. The choice of Europium-152 for the energy calibration is because it is a multi-gamma ray standard source that allows for a rapid, precise energy and efficiency calibration. We have presented the results of the spectra using GEM 30195 coaxial germanium detector of energy calibration, shape calibration as well as speak area analysis for the  $^{152}\text{Eu}$  point source sample in figures 2 to 5.

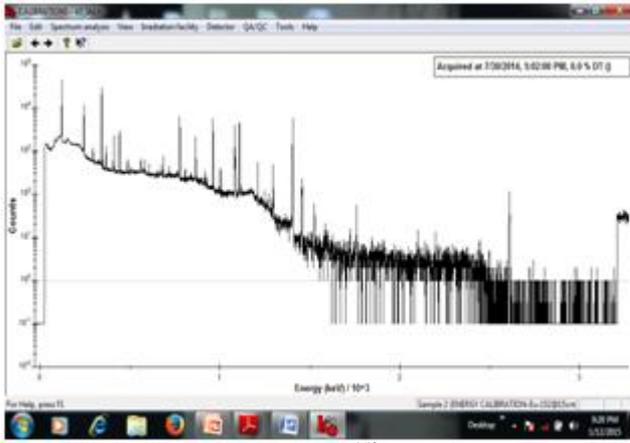


Figure 2: Acquired spectrum for <sup>152</sup>Eu geometry H2 (15 cm) Raw View

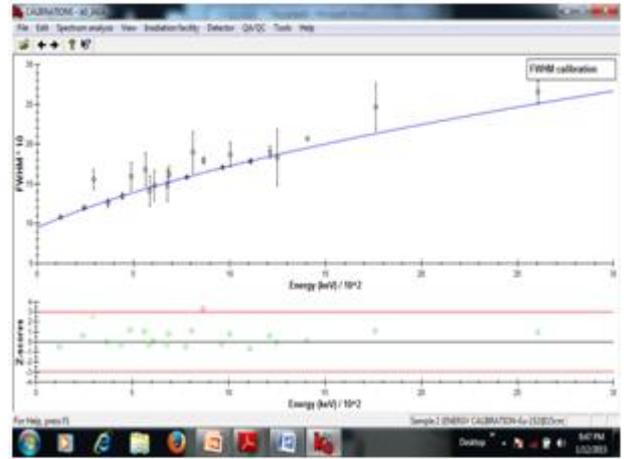


Figure 5: Curve for Peak Shape calibration of <sup>152</sup>Eu at geometry H2 (15 cm)

During the calibration, we selected the spectrum analysis command, performed peak search with known peak energies of 121.78, 244.69, 778.92 and 1112.08 keV with gamma abundance of 28.21, 7.51, 12.96 and 13.50% respectively, and the entire results were stored in the permanent database. The background spectrum analysis was only performed after the energy calibration had been completed.

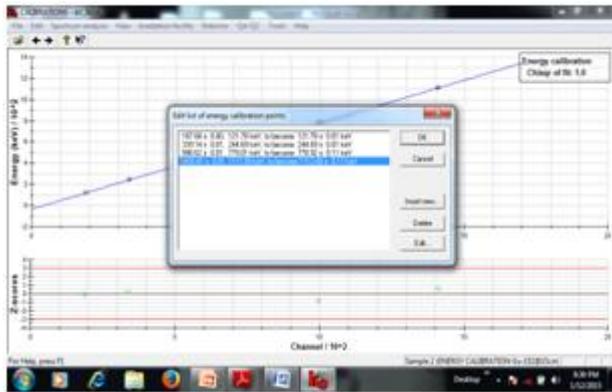


Figure 3: Curve for energy calibration of <sup>152</sup>Eu at H1(15 cm) showing Edit list of energy

## 2.2 Efficiency Calibration

In order to calibrate the efficiency of GEM 30195 coaxial detector used, we first edited the permanent database as described earlier. We have also entered the detector and its dimensions as well as the certificates of the sources. The Efficiency calibration is quite necessary because it establishes the relationship between the peak energy and the probability of the detector recording a count in the full energy peak.

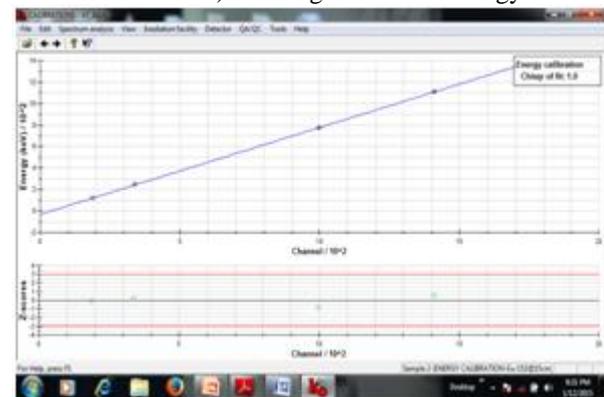


Figure 4: Curve for energy calibration of <sup>152</sup>Eu at geometry H2 (15 cm) showing successful energy calibration

The detected activities of the radionuclides were measured with the detector and calculated with equation 1 using the peak areas in the spectra acquired with MAESTRO-32 software:

$$Activity = \frac{PeakNetArea}{LiveTime} \quad (1)$$

The actual current activities of the radionuclides were calculated using equation 2 given by

$$A = A_0 e^{-\lambda t} \quad (2)$$

where  $A_0$  = Initial activity of the radionuclide at the time of packaging,  $t$  is the duration of decay from time of packaging to time of measurement and  $\lambda$  = decay constant defined by the equation

$$\lambda = \frac{\log_e}{T_{1/2}} = \frac{0.693}{T_{1/2}} \quad (3)$$

where  $T_{1/2}$  = half-life of the source.

The efficiency of each gamma line is calculated from equation 4 given by:

$$Efficiency = \frac{Experimental \ value \ of \ activity \ measured \ with \ the \ detector}{(Actual \ activity \ of \ calibration \ source) \times (branching \ ratio \ of \ \gamma \text{-ray})} \quad (4)$$

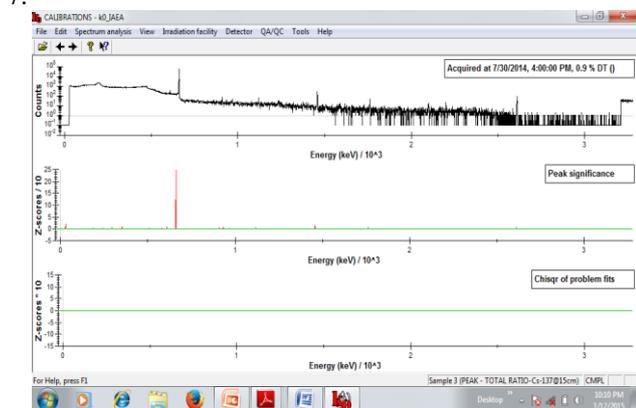
Establishing the efficiency curves from measurement involves using two spectra from two separate standard calibration sources whose activities are known accurately,

the first being <sup>137</sup>Cs (a standard source that emits only one photo peak at 661.6 keV energy according to Rossbach, *et al.*, [23] at a time with half-life of 30.070 years), the second source is a multiple gamma ray emitter <sup>152</sup>Eu (half-life of

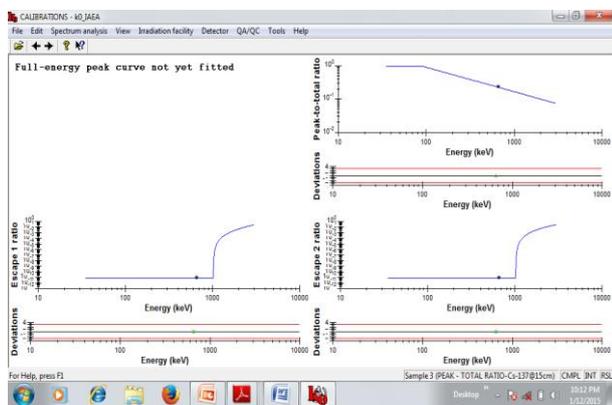
13.51 years) radionuclide calibration source. Interpreting the single-radionuclide spectrum produces the peak-to-total (p/t) curve while interpreting the multi-gamma line spectrum produces a full-energy peak efficiency curve [24]. The two sources must be measured with the detector, long enough so that the peak statistics in each main peak of each radionuclide are better than 0.5%. The Cs-137 source was measured at about 10 cm but the precise position is not very important. The mixed source was counted at the precisely known distances from the detector [24].

We have adopted the H1 (15 cm) source-detector distance for the peak-to-total ratio calibration, because the true-coincidence effects are negligible at this distance. The acquired spectrum for Cs-137 was loaded into the calibration series and interpreted using the command Edit/interpret selected sample. Since the calibration source certificate entered into the permanent database indicated that the nuclide was a single gamma line emitter, the program recognized that it would be calculating the peak-to-total ratio during the interpretation of its spectrum. The efficiency curve was then fitted, and viewed with the command View/fitted efficiency curve and finally stored by clicking on the menu command Detector/efficiency curve/store efficiency curves option. Storing the fitted efficiency curve in the permanent database of the  $k_0$ -IAEA program requires specifying the name of the detector and detector-source distance in the dialog box. The spectrum is presented in figure 6, while the plot of the p/t ratio curve and the deviation from the energy position is also presented in figure 7.

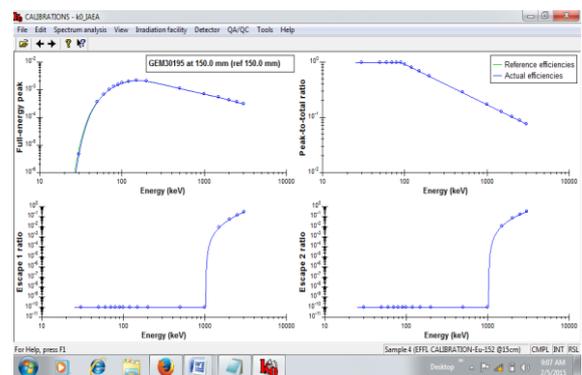
We finally calibrated the detector with the interpretation of  $^{152}\text{Eu}$  so as to obtain the full-energy peak efficiency (FEPE) spectra for all the various geometries to be used. Eu-152 calibration source was used for this study at 15 cm and 2 cm; which are the two source-detector distances adopted for the counting with the GEM 30195 detector at the NAA laboratory, Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria. The acquired spectrum for each source-detector distance was loaded into the calibration series and interpreted using the command Edit/interpret selected sample option. The calibration source certificate entered into the permanent database indicated the nuclide was a multiple gamma line emitter. Hence, the program will recognize that it ought to calculate the full-energy peak efficiency (FEPE) curves during the interpretation of the spectrum. While fitting the curve to the points, the  $k_0$ -IAEA program converts the computed efficiencies from real non-point source to ideal mathematical point source geometry that does not have size, mass and  $\gamma$ -ray self-absorption. It is this point source referenced efficiency data that is stored in the permanent database so that appropriate corrections is made for the geometrical conditions used in the actual analysis for measurement of bulky or extended samples, varying comparator shapes and sizes. After the computation, the numerical results can be viewed by clicking on the command View/numerical results option (figure 8). Furthermore, the fitted and the referenced efficiency curves can also be viewed using View/fitted efficiency curve option and View/efficiency curve for the spectra option (figure 9) respectively. The efficiency curve is finally stored by clicking on Detector/efficiency curve/store efficiency curves option. The program prompts the user to specify the name of the detector and respective detector-source distances at this point. During routine analysis of real samples, the reference efficiency curve that is the closest match to the sample counting geometry is used. This is converted to the actual sample counting geometry using a semi-empirical approach based on the calculation of effective solid angles with a Monte Carlo method for which knowledge of the geometrical parameters of the source-detector configuration is required.



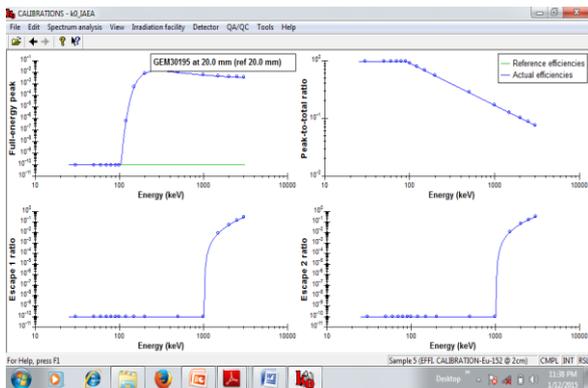
**Figure 6:** Single energy peak spectrum for p/t curve analysis ( $^{137}\text{Cs}$  at 15 cm)



**Figure 7:** Fitted Efficiency curve of the single energy peak for  $^{137}\text{Cs}$



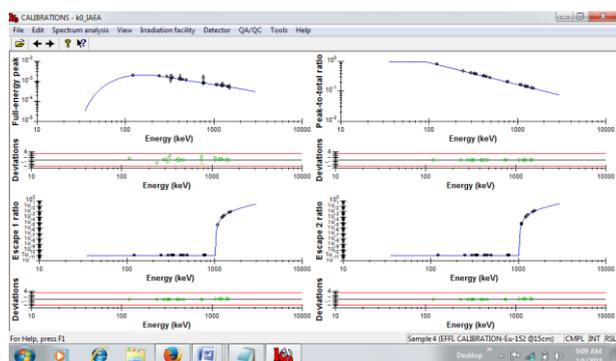
**Figure 8:** Full energy peak efficiency and the peak-to-total curves for the GEM 30195 Detector at 15 cm source-detector geometry



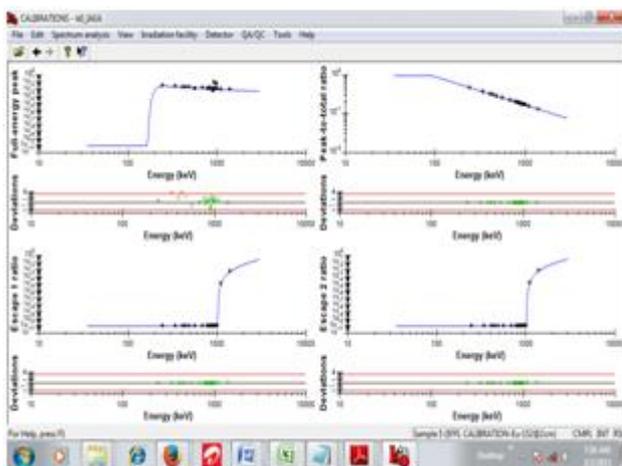
**Figure 9:** Full energy peak efficiency and the peak-to-total curves for the GEM 30195 Detector at 2 cm source-detector geometry

### 3. Results And Discussion

The automated plots of the  $k_0$ -IAEA software of log-log full-energy peak efficiency versus energy (keV), efficiency deviation against energy (keV), Peak-Total Ratio curve against energy (keV) and deviation for the p/t against energy (keV) for the 15 cm is shown in figure 9 and that for the 2 cm is shown in figure 10 with a recorded chi-square value of 1.0 and 3.6 respectively. The p/t ratio shows a good agreement with the theoretical solid line and the deviation ratio tends approximately to unity.



**Figure 10:** Fitted Efficiency curve of the multiple energy peaks for  $^{152}\text{Eu}$  at 15 cm geometry



**Figure 11:** Fitted Efficiency curve of the multiple energy peaks for  $^{152}\text{Eu}$  for GEM 30195 at 2 cm

We can see from the results in figures 12 & 13, that z-values here shows that all the doublet at the photopeaks have been

resolved correctly, and that the photons detected by GEM 30195 HpGe coaxial detector have the highest efficiency values within an energy range of 122 keV which is in agreement with the investigation of the FEPE for a high aspect ratio (1.29) detector reported by Ewa, *et al.*, (2002). Our work is also in good agreement with report presented by other researchers such as [7], [25] – [29]. We noticed particularly that FEPE values increases from low energy region, followed by an optimum peak of efficiency value and then shows further decrease as the energy increases. Though, the photopeaks at 344.29 keV and 443.89 keV in figure 13 which shows high z-values are part of doublets that may have been resolved incorrectly. These discrepancies were previously reported by Debertain[6] and recently by Blaauw[1], and this may indicate that some property of the decay scheme of  $^{152}\text{Eu}$  remains as yet unknown. A further explanation could be that these discrepancies may be due to the coincidence of  $^{154}\text{Sm}$  X-rays penetrating Zr absorber.

### 4. Conclusions

In the use of this software, we have succeeded in the energy and efficiency calibrations, determination of peak area (background inclusive). We first performed the energy calibration, plotted the energy calibration curves, and then performed the full peak energy efficiency (FEPE) of GEM 30195 coaxial HpGe detector using two standard sources  $^{137}\text{Cs}$  and  $^{152}\text{Eu}$  and obtained values within the energy range of 121.78 – 1528.12 keV and 244.69 – 1408.00 keV respectively for 15 cm and 2 cm source – detector geometries with their z-values showing that all doublets at the photopeaks were correctly resolved. The automated plots of the  $k_0$ -IAEA software of log-log full-energy peak efficiency versus energy (keV), efficiency deviation against energy (keV), Peak-total Ratio curve against energy (keV) and deviation for the p/t against energy (keV) for the 15 cm and 2 cm both recorded chi-square values of 1.0 and 3.6 respectively. The p/t ratio shows a good agreement with the theoretical solid line and the deviation ratio tends approximately to unity. We also noticed that the photons detected by GEM 30195 HpGe coaxial detector have the highest efficiency values within an energy range of 122 keV. More so, we noticed particularly that FEPE values increases from low energy region, followed by an optimum peak of efficiency value and then shows further decrease as the energy increases.

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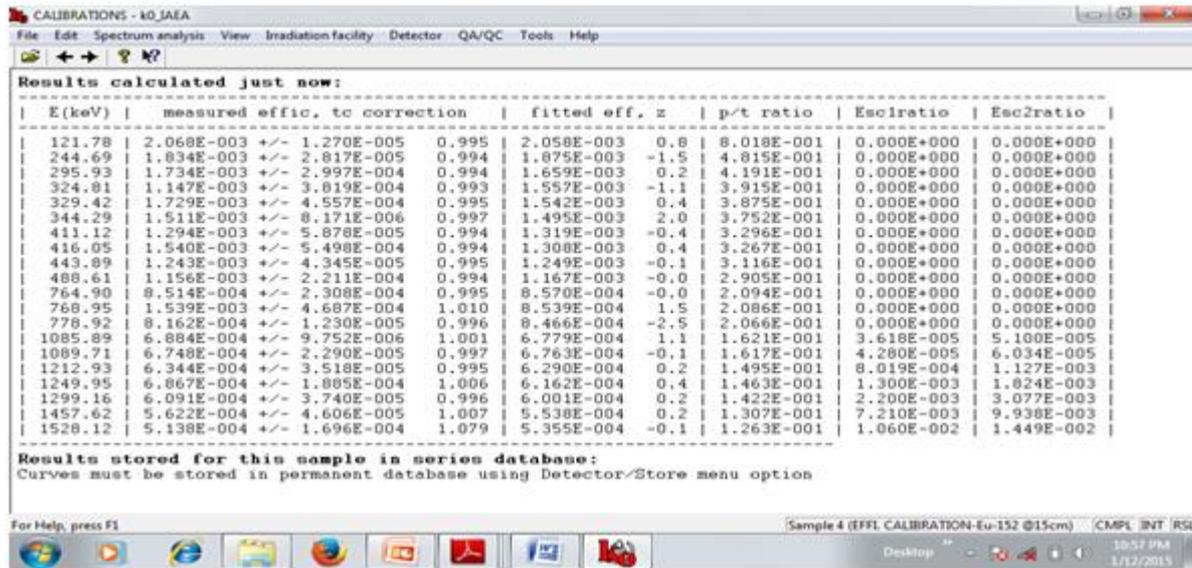


Figure 12: Efficiency calibration datapoints for GEM 30195 at 15 cm source-detector geometry

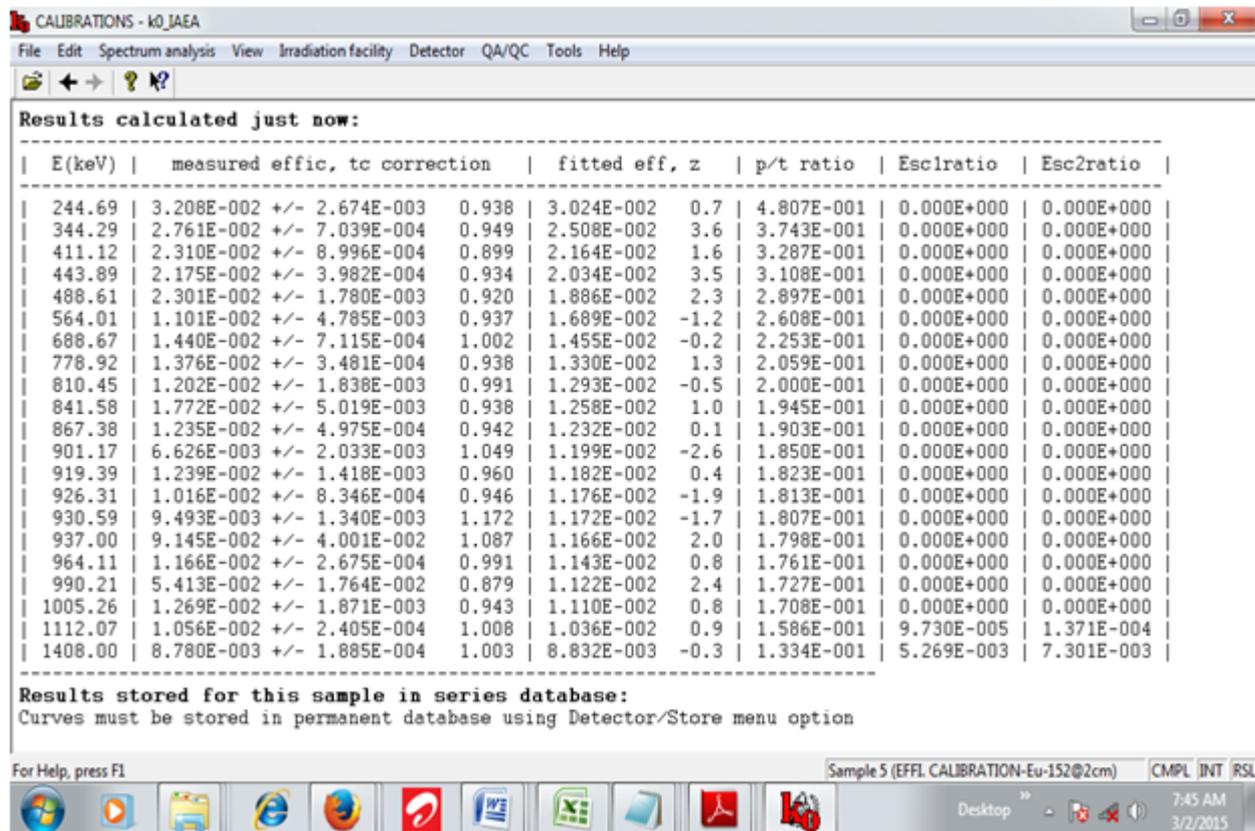


Figure 13: Efficiency calibration datapoints for GEM 30195 at 2 cm source – detector geometry

Table 1: Description of the samples used for the calibration of gamma-ray spectrometer

Sample ID	Sample type	Activity (kBq)	Packaging Date	Counting Date	T <sub>begin</sub> (s)	T <sub>cool</sub> (s)	T <sub>live</sub> (s)	T <sub>real</sub> (s)	Geometry
Cs-137	Source	36.9	15/07/2004	30/07/2014	16:00:00	314380800	3600	3632.86	15 cm
Eu-152	Source	38.0	15/07/2004	30/07/2014	17:02:00	314384520	3600	3639.06	15 cm
Eu-152	Source	38.0	15/07/2004	31/07/2014	13:32:00	314458320	3600	3956.68	2 cm