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# Calibration of a Gamma-Ray Spectrometer using Electronic Spreadsheet Package and Dedicated Spectral Analysis Software

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## ABSTRACT

As a first step after installation of new software and hardware in the Neutron Activation Analysis (NAA) laboratories of Nigeria Research Reactor-1 (NIRR-1) the calibration of the spectrometer was carried out before any spectral analysis could be done. Both electronic spreadsheet and dedicated spectral analysis software ($k_0$-IAEA) were employed and the results obtained from both methods were compared in this study. The efficiency curves were established from measurement and interpretation of several spectra from nine standard gamma-ray calibration sources (Na-22, Mn-54, Co-57, Co-60, Y-88, Cs-137, Eu-152, Ra-226, Am-241) whose activities are known to better than ±3%. The sets of values obtained for the full-energy peak detection efficiency from the two approaches are close at higher geometries with less than 10% variation.

**Keywords:** *Detector calibration; Efficiency; $k_0$-standardization; $k_0$-IAEA program; NIRR-1; Neutron activation analysis.*

## I. INTRODUCTION

The search for a versatile Neutron Activation Analysis (NAA) Standardization method that meets the criteria of experimental simplicity, analytical accuracy and flexibility (with respect to activation and counting conditions) has led to acceptability and the adoption of the $k_0$-based NAA standardization method for routine analysis in many laboratories all over the world [11]. These advantages come at a price of accurate characterization of the neutron flux parameters in all the irradiation channels and full calibration of the detectors [1-2]. For quantification of the radioactive products using the traditional relative method, samples and standard materials of known elemental concentrations are irradiated and counted under the same experimental conditions [3]. The physical parameters for the element of interest being identical in both the analyte and standard cancel out in the concentration calculations. This approach therefore eliminates the need for accurate determination of neutron fluxes and detector calibration. Furthermore, the $k_0$-based NAA standardization method has no room for compromise in full determination of neutron fluxes and detector calibration. The interpretation of gamma ray spectra in terms of identifying the elements present, net peak area calculation to determine activity and subsequently the elemental concentration as well as detector calibrations could be performed by the "spreadsheet" approach using any commercial electronic spreadsheet package provided all the input parameters are known. But the complexity of most γ-ray spectra coupled with the computation requiring large data set such as neutron spectrum parameters, detector efficiencies, nuclear data, time parameters (irradiation, decay, counting times), sample-specific data (mass, matrix type), spectrum-specific data (peak energies and net peak areas, dead time) necessitated computerization and automation with special spectral analysis software. The acceptability and the computation demands of the $k_0$-method led to the development of several in-house computer programs in many different laboratories all over the world [4]. To ensure harmonization of the software used for the implementation of the $k_0$-standardization of NAA and to further assist member States in their quality output from nuclear analytical laboratories, the International Atomic Energy Agency (IAEA) initiated the development of another spectral analysis software, the $k_0$-IAEA. As a prerequisite for the implementation of the $k_0$-NAA Standardized Method in the Nigeria Research Reactor-1 (NIRR-1), the characterization of the neutron flux parameters in the irradiation channels, development of the experimental protocols for the facilities and calibration of the detectors at different source-detector geometries have to be carried out. This characterization of the irradiation channels and the development of the experimental protocols have been described elsewhere [4-8]. This paper therefore focuses on the calibration of the detector. Proper, efficient and accurate calibration is critical to achieving accurate analytical results from $k_0$-NAA Standardized laboratories by ensuring sample spectra are accurately interpreted. The calibration exercise allows the spectroscopic instrument to determine the composition of future samples, whose composition is completely unknown initially [9].

## II. EXPERIMENTAL

**A.** Gamma ray spectrometer calibration using the $k_0$-IAEA spectral analysis software involved configuration of the spectral acquisition system and the actual analysis and interpretation of various spectra from standard gamma ray calibration sources.

**(i) Configuration of the spectral acquisition system**

The High-Purity Germanium detector (HPGe) GEM 30195 was coupled to the integrated digital gamma-ray spectrometer hardware DSPEC jr 2.0 (Plate 1)

which is completely computer-controlled with the MAESTRO-32 multi-channel analyzer (MCA) emulation software. Complete computer control of the front end electronics means there are no knobs to turn or buttons to push, eliminating the possibility of accidental misadjustment. Precise adjustment of all front end electronics is accomplished via emulated controls - right on the screen. The DSPEC jr 2.0 is connected via a USB port to the host personal computer (Plate 2) which is used for configuring the hardware settings (such as high voltages, presets and amplifier gain) through the multichannel buffer (MCB) interface of the MAESTRO-32 software [10-14].

**Plate 1. Digital Spectrometer**

**Plate 2. DSPEC jr 2.0 connected to desktop computer**

**Plate 3. GEM 30195 detector mounted on 30-liter liquid nitrogen dewar for cooling to cryogenic temperatures and surrounded by lead shield to reduce the background caused by sources other than the sample.**

At the start of the day before acquisition of any spectrum, a routine energy calibration of the MAESTRO-32 spectrum acquisition software was carried out using a mixed energy gamma ray calibration sources consisting of three pure standard gamma ray calibration sources: $^{137}$Cs, $^{60}$Co and $^{88}$Y. The sources were placed on a sample holder at about 10 cm from the detector end cap (Plate 3) and the spectrum acquired long enough to clearly identify the positions of the pure photopeaks in the spectrum. This usually takes about five minutes. The corresponding peaks were seen prominently at 661.6 KeV, 1173 keV, 1332 keV and 1836 keV for $^{137}$Cs, $^{60}$Co and $^{88}$Y respectively. However, within this region, there was also a less significant impurity peak at 1460 keV due to $^{40}$K. The calibration is carried out by replacing the channel number placeholders for these peaks on the horizontal axis of the spectrum plot with the well-known actual values of the expected peak energies from these calibration sources.

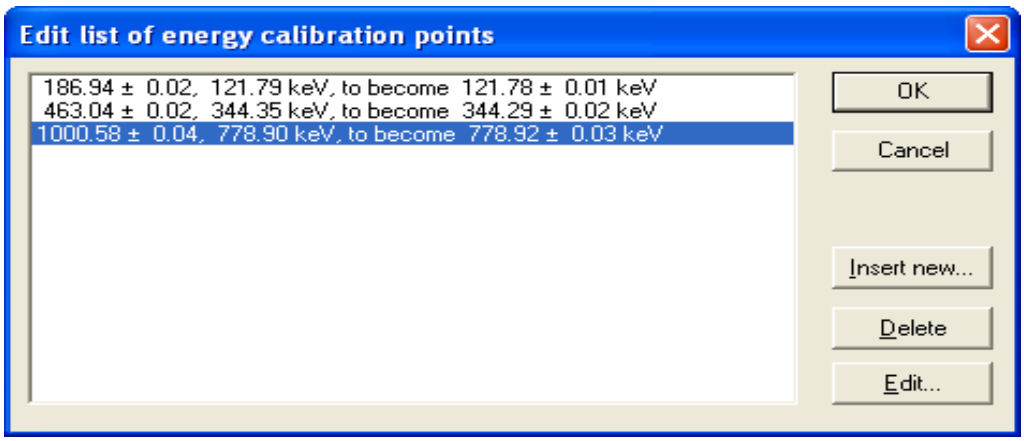


**Fig. 1. Window of the menu "Spectrum analysis/Calibration/Energy calibration" with typical values of data entered for energy calibration with Eu-152 spectrum analysed with $k_0$-IAEA program**

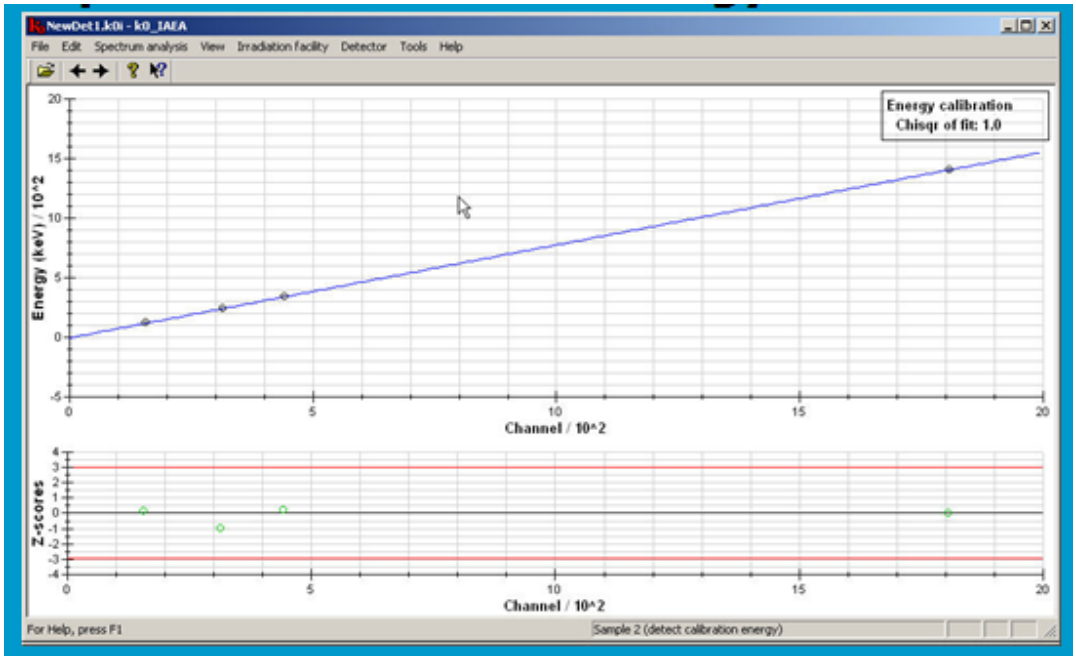


**Fig. 2. Typical curve for energy calibration in $k_0$-IAEA program.**

**(ii) Analysis and interpretation of various spectra from standard gamma ray calibration sources :**

The Efficiency calibration establishes the relationship between the peak energy and the probability of the detector recording a count in the full energy peak. Establishing the efficiency curves from measurement involves using two spectra from two separate standard calibration sources whose activities are known accurately, the first being Cs-137 (a radionuclide that emit only one gamma-ray at a time), the second source is a multiple gamma ray emitter Eu-152 radionuclide calibration source. Interpreting the single-radionuclide spectrum produces the peak-to-total curve while interpreting the multi-gamma line spectrum produces a full-energy peak efficiency curve [14].

In this study, the farthest source-detector distance was used for the peak-to-total ratio calibration, where true-coincidence effects are negligible. 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. Cs-137 standard source emits only one photo peak at 661.6 KeV energy [17]. 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. 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 [12,15].

The last set of calibration samples interpreted were the full-energy peak efficiency spectra for all the various geometries to be used. Eu-152 calibration source was used for this study at 2 cm and 15 cm; the two optimum source-detector distances for the counting with the GEM 30195 detector. The acquired spectrum for each source-detector distance was loaded into the calibration series and interpreted using the command Edit → interpret selected sample. The calibration source certificate entered into the permanent database indicated the nuclide was a multiple gamma line emitter. Therefore, the program recognized that it was to calculate the full-energy peak efficiency curves during the interpretation of the spectrum. After fitting the curve to the points, the program converts the computed efficiencies from real non-point source to ideal mathematical point source geometry that have no mass, size and γ-ray self-absorption. "Ideal" calibration sources should be point sources [14]. 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. This correction is determined by the matrix type, the geometry and the filling height [12,14, 16]. After the computation, the numerical results can be viewed by clicking on the command View → numerical results. Furthermore, the fitted and the referenced efficiency curves can also be viewed using View → fitted efficiency curve and View → efficiency curve for the spectra respectively. The efficiency curve is finally stored by clicking on Detector → efficiency curve → store efficiency curves. 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. The detector's dimensions including the thickness of the dead layer and the distance from the germanium crystal to the housing were entered into the permanent database for this purpose. Generally, it is a good practice to perform full-energy peak detection calibration for all the geometries to be used for measurement during sample analysis [14]. This improves accuracy because of much smaller correction factors for the conversion from reference geometry efficiency to actual sample geometry efficiency. [14,16] Efficiency calibrations were done for the two geometries used in this study over the operating range of approximately 100 - 3000 KeV. (Fig. 3 and Fig. 5)

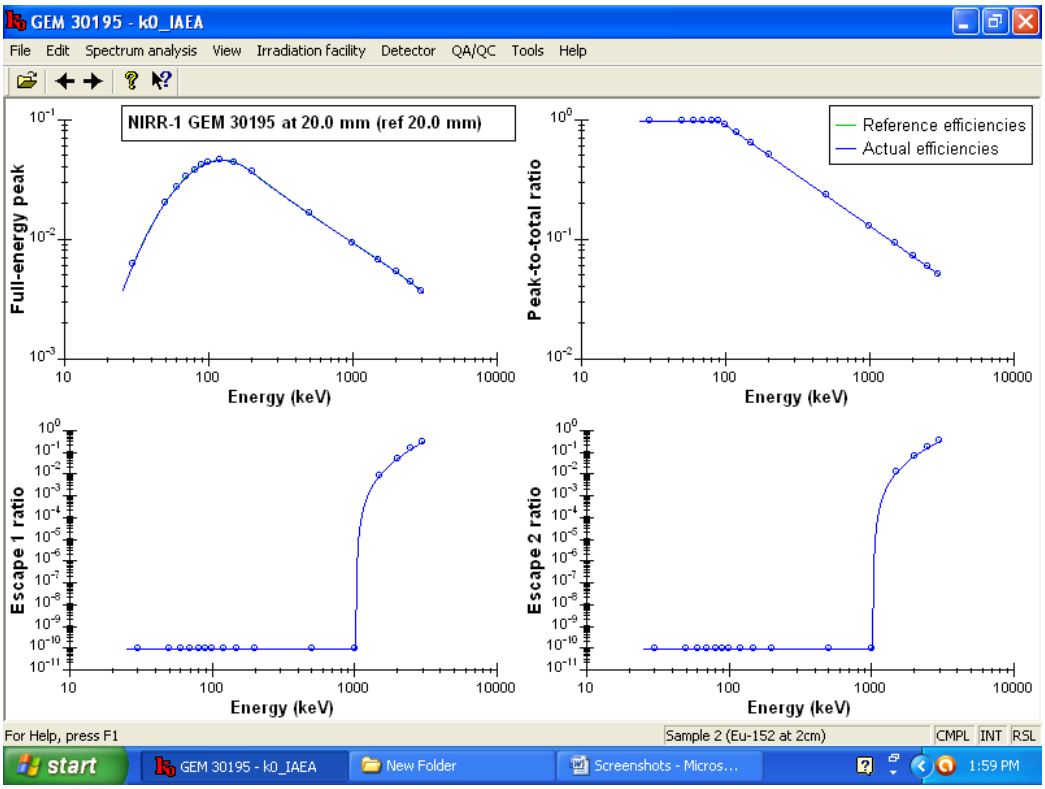


**Fig. 3. Full energy efficiency and the peak-to-total curves for the GEM 30195 detector at 2 cm source-detector geometry.**

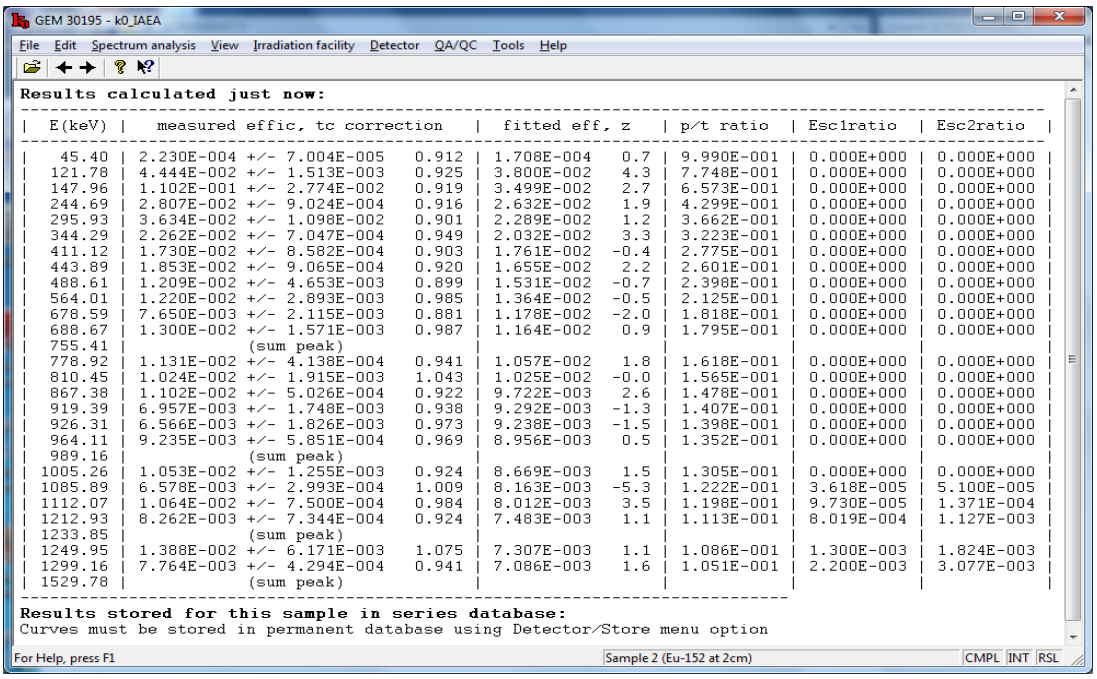


**Fig. 4. Efficiency calibration datapoints for GEM 30195 at 2 cm source-detector geometry**

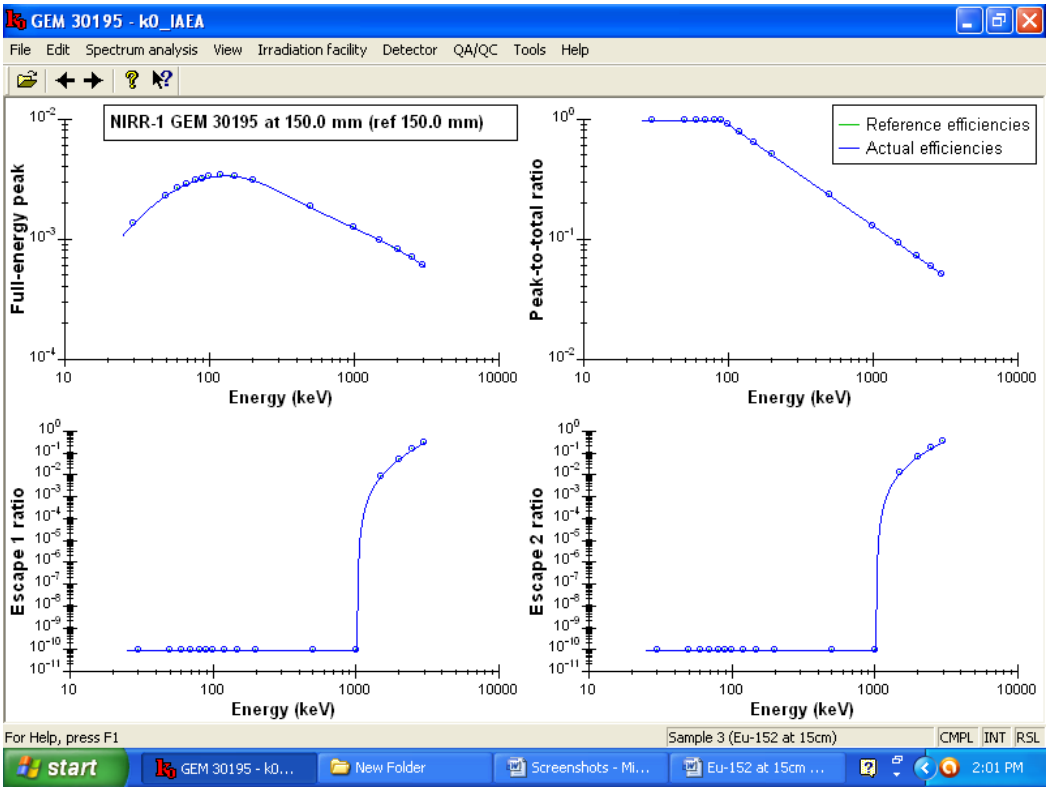


**Fig. 5. Full energy efficiency and the peak-to-total curves for the GEM 30195 detector at 15 cm source-detector geometry**

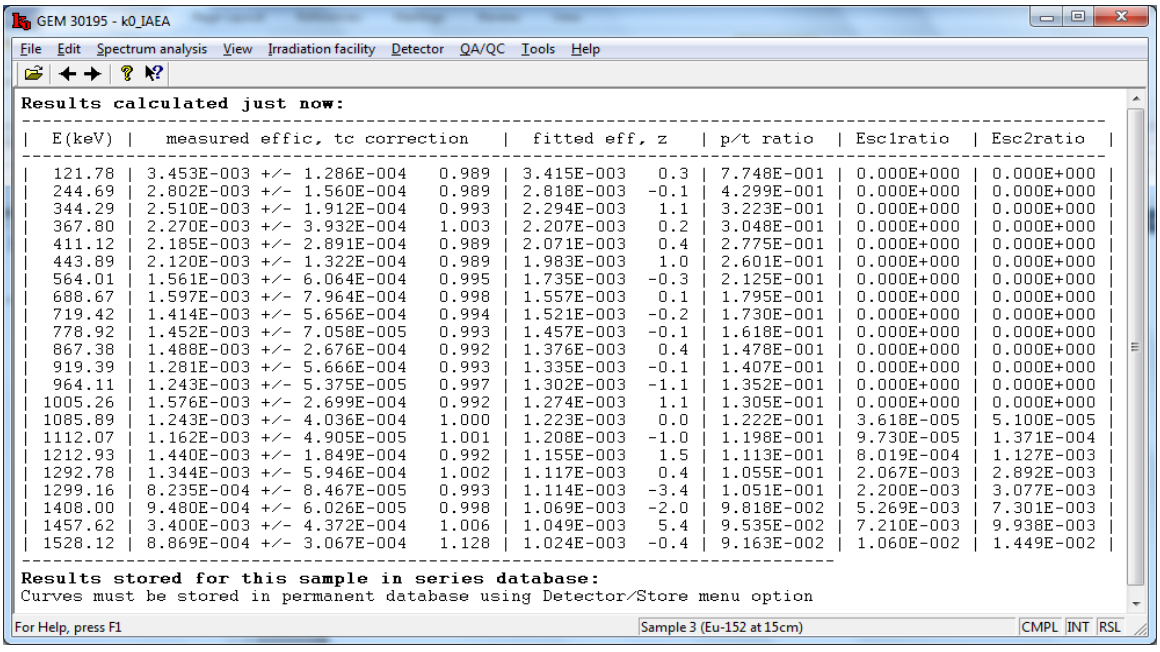


**Fig. 6. Efficiency calibration datapoints for GEM 30195 at 15 cm source-detector geometry.**

**B. Full-energy peak efficiency curve fitting with electronic spreadsheet:**

The detected activities of the radionuclides was measured with the detector and calculated using equation 1

$$Activity = \frac{Peak \text{ Net Area}}{Live \text{ Time}} \quad \text{.....(1)}$$

using the peak areas in the spectra acquired with MAESTRO-32 software. The actual current activities of the radionuclides were calculated using equation

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

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 λ = decay constant defined by equation 3.

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

where $T_{½}$ = half-life of the source.

The efficiency of each gamma line is calculated from equation 4

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

This experiment was concerned with finding out how the value of the efficiency varies with the gamma-ray energy. Therefore, the graph of efficiency versus gamma-ray energy was plotted using the Microsoft Excel on a log-log scale as shown in Fig. 7. Since a non-linear relationship was obtained, a polynomial function of degree 3 was fitted to the experimental data points of the detector's efficiency curve. An interpolation of efficiency at any required gamma-ray energy between two tabulated and plotted data points could then be calculated using the equation obtained from the trend line and regression analysis. The least square method was used as a criterion for determining the best fitting curve which is as close as possible to the experimental data. The correlation coefficient given by the electronic spreadsheet is a measure of how the two variables relate.

**Table 1. Data from the certificates of the standard gamma ray calibration sources used.**

| Radionuclide | Activity | Uncertainty | Half-life | Packaging Date | Time | Material |
|---|---|---|---|---|---|---|
| Co-60 | 38400 | 1100 | 5.27 years | 15-Jul-04 | 12:00 | DKD Co-60 |
| Cs-137 | 36900 | 1100 | 30.14 years | 15-Jul-04 | 12:00 | DKD Cs-137 |
| Mn-54 | 38700 | 1100 | 312.50 days | 15-Jul-04 | 12:00 | DKD Mn-54 |
| Y-88 | 35800 | 1100 | 106.6 days | 15-Jul-04 | 12:00 | DKD Y-88 |
| Na-22 | 36900 | 1100 | 2.60 years | 15-Jul-04 | 12:00 | DKD Na-22 |
| Ra-226 | 37400 | 1100 | 1,600 years | 1-Jul-04 | 12:00 | DKD Ra-226 |
| Eu-152 | 38000 | 1100 | 13.33 years | 15-Jul-04 | 12:00 | DKD Eu-152 |
| Am-241 | 36500 | 1100 | 432.2 years | 1-Jul-04 | 12:00 | DKD Am-241 |
| Co-57 | 37700 | 1100 | 271.80 days | 15-Jul-04 | 12:00 | DKD Co-57 |

**Table 2. Efficiency computed and fitted with electronic spreadsheet for GEM 30195 with sources at 15 cm from detector end cap.**

| Nuclide | Energy (KeV) | Efficiency |
|---|---|---|
| Am-241 | 59.54 | 0.000613337 |
| Eu-152 | 121.78 | 0.003553117 |
| Eu-152 | 244.7 | 0.00295213 |
| Eu-152 | 344.28 | 0.002491134 |
| Eu-152 | 367.76 | 0.002412776 |
| Eu-152 | 411.3 | 0.00216846 |
| Eu-152 | 444 | 0.002089715 |
| Eu-152 | 778.9 | 0.00132318 |
| Eu-152 | 867.38 | 0.001252362 |
| Eu-152 | 964 | 0.001204472 |
| Eu-152 | 1112 | 0.001111782 |
| Eu-152 | 1212.94 | 0.001109479 |
| Eu-152 | 1408.03 | 0.000931153 |
| Ra-226 | 1729.6 | 0.000770708 |
| Ra-226 | 1764.51 | 0.000740934 |
| Ra-226 | 2118.54 | 0.000589674 |
| Ra-226 | 2204.12 | 0.000595507 |
| Ra-226 | 2447.71 | 0.000574499 |

**Table 3. Efficiency computed and fitted with electronic spreadsheet for GEM 30195 with sources at 2 cm from detector end cap**

| Nuclide | Energy (KeV) | Efficiency |
|---|---|---|
| Am-241 | 59.54 | 0.009084383 |
| Eu-152 | 121.78 | 0.045945262 |
| Eu-152 | 244.7 | 0.032750254 |
| Eu-152 | 344.28 | 0.026325629 |
| Eu-152 | 367.76 | 0.023403456 |
| Eu-152 | 444 | 0.019739155 |
| Eu-152 | 778.9 | 0.012700505 |
| Eu-152 | 867.38 | 0.012206212 |
| Eu-152 | 1212.94 | 0.00773334 |
| Eu-152 | 1298.7 | 0.007228203 |
| Ra-226 | 1509.19 | 0.005574393 |
| Ra-226 | 1661.28 | 0.004065313 |
| Ra-226 | 2118.54 | 0.003298883 |
| Ra-226 | 2204.12 | 0.003446851 |
| Ra-226 | 2293.36 | 0.003573887 |
| Ra-226 | 2447.71 | 0.003721455 |

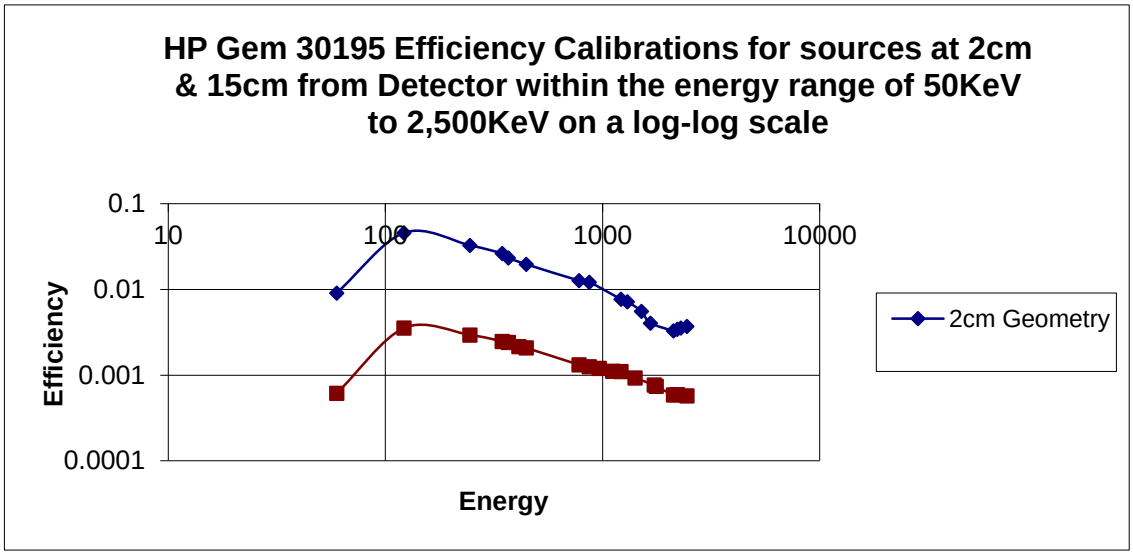


**Fig. 7. Efficiency computed and fitted with electronic spreadsheet for GEM 30195.**

## III. RESULTS AND DISCUSSION

Comparison of the full-energy peak efficiency calibrations using the spreadsheet and $k_0$_IAEA software for the two geometries shows good agreement. This is evident from the quality of the two sets of values of the computed efficiency which is within the range of 10% of each other for the 15cm geometry as shown in Table 4. The higher variations at close geometry of 2cm as seen in Table 5 could be attributed to the disregard of the peak-to-total ratio in the spreadsheet. The peak-to-total ratio which is required for true coincidence effect correction is especially important when the source-detector distance becomes small.

**Table 4. Comparison of the full-energy peak efficiency calibration results obtained from spreadsheet and $k_0$_IAEA software for 15 cm source-detector distance.**

| Energy (KeV) | Efficiency | | Ratio Spreadsheet/$k_0$_IAEA |
|---|---|---|---|
| | Spreadsheet | $k_0$_IAEA | |
| 121.78 | 3.553E-03 | 3.415E-03 | 1.04 |
| 244.7 | 2.952E-03 | 2.818E-03 | 1.05 |
| 344.28 | 2.491E-03 | 2.294E-03 | 1.09 |
| 367.76 | 2.413E-03 | 2.207E-03 | 1.09 |
| 411.3 | 2.168E-03 | 2.071E-03 | 1.05 |
| 444 | 2.090E-03 | 1.983E-03 | 1.05 |
| 778.9 | 1.323E-03 | 1.457E-03 | 0.91 |
| 867.38 | 1.252E-03 | 1.376E-03 | 0.91 |
| 964 | 1.204E-03 | 1.302E-03 | 0.93 |
| 1112 | 1.112E-03 | 1.208E-03 | 0.92 |
| 1212.94 | 1.109E-03 | 1.155E-03 | 0.96 |
| 1408.03 | 9.312E-04 | 1.069E-03 | 0.87 |

**Table 5. Comparison of the full-energy peak efficiency calibration results obtained from spreadsheet and $k_0$_IAEA software for 2 cm source-detector distance.**

| Energy (KeV) | Efficiency | | Ratio Spreadsheet/$k_0$_IAEA |
|---|---|---|---|
| | Spreadsheet | $k_0$_IAEA | |
| 121.78 | 4.595E-02 | 3.800E-02 | 1.21 |
| 244.7 | 3.275E-02 | 2.632E-02 | 1.24 |
| 344.28 | 2.633E-02 | 2.032E-02 | 1.30 |
| 444 | 1.974E-02 | 1.655E-02 | 1.19 |
| 778.9 | 1.270E-02 | 1.057E-02 | 1.20 |
| 867.38 | 1.221E-02 | 9.722E-03 | 1.26 |
| 1212.94 | 7.733E-03 | 7.483E-03 | 1.03 |
| 1298.7 | 7.228E-03 | 7.086E-03 | 1.02 |

The $k_0$-IAEA software was purposely developed for the implementation of the $k_0$-standardization of NAA making it more suitable for spectral analysis. This software has several advantages over the use of electronic spreadsheet in detector efficiency calculations. It has an internal provision for the conversion of the reference geometry efficiency to actual sample geometry efficiency leading to improved accuracy of analytical results due to smaller correction factors. The dynamic nature of the $k_0$-IAEA software makes it possible for the analyst to easily jump back and forth between the concentration calculation and peak fit; when troubleshooting is necessary since the program remembers all the peak fitting parameters once it has performed them. The $k_0$-IAEA software always corrects for gamma-ray self-absorption in the sample/calibration sources and their recipients as long as the information about the matrix composition and sample/source dimensions have been entered correctly in the software [13]. On the other hand, the electronic spreadhseet is more tedious and labour intensive in carrying out all the necessary calculations [11]. Furthermore, the $k_0$-IAEA software package has several other routines in its suite for energy calibration, storing of the detector background spectrum, peak shape calibration and peak-to-total ratio curves. All these advantages make its analytical results more accurate and dependable.

However, there is still further need for eenhancement of the code to make it capable of resolving multiplet or overlapping peaks into individual components for accurate net peak area determination without the user intervention. This will reduce the manual selection of the region of interest (ROI) for some photopeaks before peak area analysis.

## IV. CONCLUSIONS

Two approaches ($k_0$_IAEA software and electronic spreadsheet) for calculating the full-energy peak efficiency for the high purity germanium detector GEM 30195 have been compared. On the whole, the performance of the two approaches in terms of accuracy and effectiveness are close at higher geometries with less than 10% variation. However, $k_0$-IAEA software holds out great prospects with exciting possibilities given its several advantages over the spreadsheet method which have been earlier enumerated in results and discussion. Finally, this study was carried out as part of preliminary studies needed for the implementation of the $k_0$-

standardized NAA method for routine elemental analysis in NIRR-1 NAA laboratories. [4, 11]

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