Calibration and Performance Testing of Sodium Iodide, NaI (Tl), Detector at the Food and Environmental Laboratory of the Radiation Protection Institute of the Ghana Atomic Energy Commission

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Abstract

The performance testing of a newly acquired sodium iodide detector (NaI), (Tl)) at Ghana Atomic Energy Commission (GAEC) was investigated by carrying out energy and efficiency calibration on the detector, as well as validation of its calibration. The energy and efficiency calibrations were performed using mixed radionuclides standard containing ^{241}Am, ^{109}Cd, ^{57}Co, ^{137}Cs and ^{60}Co in the energy range of 60–1333 keV. The energy and efficiency calibration curves obtained compare well with what is available in the literature. Results of the validation of the calibration showed that there were no significance differences between the measured and the calculated activities of the standard radionuclides with activity ratios in the range of 0.90-0.98 and the corresponding percentage deviation in a range of 1.43–10.47%. The average MDA of ^{238}U, ^{232}Th, ^{40}K and ^{137}Cs from background counting rates were estimated to be 0.099 ± 0.055 Bq, 0.061 ± 0.037 Bq, 0.727 ± 0.300 Bq and 0.014 ± 0.006 Bq, respectively.

Introduction

Quantitative analysis of radionuclides in materials requires the use of reliable gamma-ray detection system such as NaI (Tl). The quality of the measurements depends on the calibration and the performance testing of the detector among others like sampling methods, contamination, accuracy in weighing of samples, interference from background radiation, etc.

Analysis of gamma emitting radionuclides in a given material is usually performed by the well-established method of gamma-ray spectrometry [Gilmore *et al,*, 1995; IAEA, 1989; ICRU, 1994]. Gamma spectrometers commonly used are made up of sodium iodide NaI (Tl) scintillation detector or germanium (lithium-doped or intrinsic-high pure germanium) semiconductor detector coupled to a multichannel analyzer. The energies of gamma-rays emitted from radionuclides in a sample of the material are usually displayed in a spectrum as a function of the pulse height. Depending on the energy resolution of the detector type used, the spectrum of a mixture of a number of different radionuclides measured in a material may contain peaks which may overlap, thus, making it difficult to determine the separate activities of the overlapping individual radionuclides in the spectrum.

NaI (Tl) scintillation detectors are known to have comparatively poorer resolution than

germanium detectors, but are preferable in some specific types of analyses due to their relatively higher efficiency and, besides, operates under room temperature conditions (i.e. without the use of liquid nitrogen as in germanium detectors). The full width at half maximum (FWHM) which is a measure of the resolution of the counting system for the NaI (Tl) detector is typically in a range of 7–9% in contrast with HPGE detectors with resolution in a range of 0.1–0.2%. The smaller the figure for the energy resolution, the better the detector will be able to resolve between two radionuclides whose energies lie close to each other. For instance, in the analysis of materials or samples containing a mixture of ^{134}Cs and ^{137}Cs using NaI (Tl) scintillation detector, the peaks from the 604.7 keV of ^{134}Cs and the 661.6 of ^{137}Cs are not well resolved.

There are methods which have been developed and published for deconvolution of unresolved spectra to extract the different data from measured overlapping peaks [Horowitz *et al.*, 1995; Law *et al.*, 1984; Graver *et al*., 1966]. Some of these methods require the use of complicated mathematics and computer programmes. Spectral fitting and unfolding using Fourier series, Bessels function, Cherbyshev series (Carles, 1994), stepwise statistical method (Young *et al.*, 1966), vector and least square, strip-off, incremental and iterative methods (O'Kelly, 1962), and stepwise multiple regression (Shumway, 1963) are used in the analysis of mixed radionuclides. Each of these methods is designed to meet specific needs and accuracy. The thallium activator, which is present as an impurity (about 0.2%) in the crystal structure of NaI (Tl) detector,

converts the energy absorbed in the crystal to light. In addition to the high density of the crystal (about 3.67 gcm^{-3}), the high effective atomic number accounts for the high efficiency of the NaI (Tl) than HPGE detectors (Cember, 1996). A typical NaI (Tl) scintillation detector is shown in Fig. 1.

In general, there are two calibration steps to determine the performance of a gamma spectrometry system. Firstly, energy calibration is performed, where the pulse heights scale is calibrated in terms of gamma-ray energy to enable the exact identification of photopeaks present in the spectrum produced by the detector system. Energy calibration of the detector system is performed by measuring mixed standard sources of known radionuclides with well defined energies within the energy range of interest, usually 60–2000 keV (IAEA, 1989). The energy calibration relates the channel number of the spectrometer system to energy of the standard reference material. The relationship between energy and the channel number is given as follows (Darko *et al*., 2007).

$$E \sum_{i\ 0}^{n} A_i (C_n)^i \qquad (1)$$

where E is the energy (keV) of the corresponding gamma line; C_n is the channel number; i = 0, 1, 2, 3… *n*, which is associated with the energy E; A_i is a calibration constant.

The second step is the efficiency calibration, which involves measuring reference standard sources (i.e. sample) to obtain efficiency over the region of interest. Efficiency of the detector, in general, is defined as the ratio of the actual events registered by the detector to the events emitted by the source, and absolute efficiency is the ratio of events emitted by the
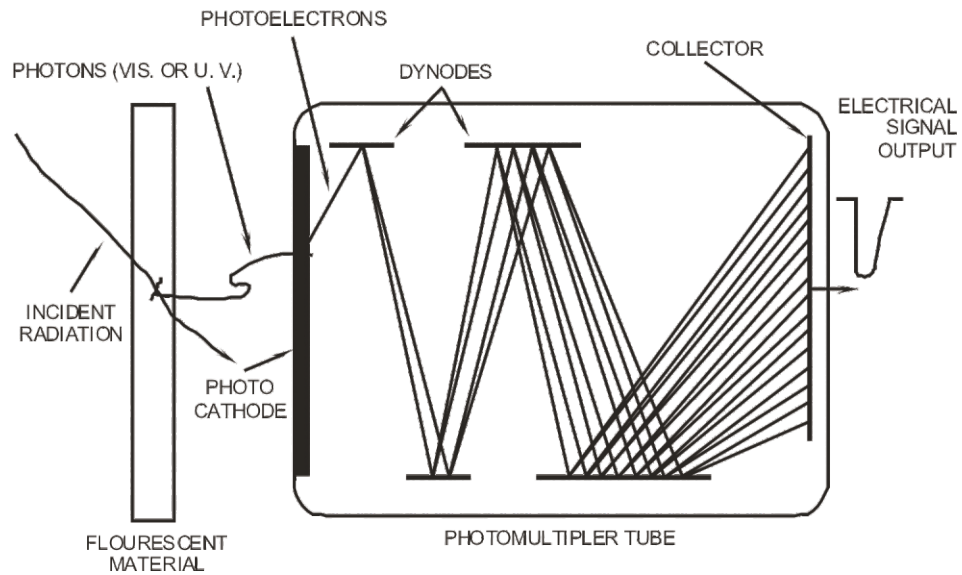
Fig. 1. Schematic diagram of the sequence of events in scintillation detector

source that is actually recorded by the detector.

$$\epsilon_{abs} = \frac{\text{Events recorded}}{\text{Events emitted by source}} \quad (2)$$

This is dependent on the properties of the detector and the design of the counting geometry. Intrinsic efficiency is also defined as the fraction of the events actually incident on the detector to that which are recorded.

$$\epsilon_{int} = \frac{\text{Events recorded}}{\text{Events impinging on the detector}} \quad (3)$$

Intrinsic efficiency is a basic parameter of the detector. It is independent of the source and detector geometry. It is a function of the type of radiation, the energy of radiation, the detector material, and the physical thickness of the detector in the direction of the incident radiation. The issue of efficiency is generally more important for neutral particles because they must first interact to produce charged particles. The interactions are often rare; so

that it is important to capture a good fraction of the incident neutral particles. Efficiency calibration of the gamma detection system is necessary for the quantification of the radionuclides present in a sample. In efficiency calibration, a source with known nuclide activity and gamma emission probability over the region of interest is required. In order to calculate activities of the nuclides, the user has to supply the computer program with detector efficiency as a function of the gamma-ray energy (efficiency curve) (Hubert *et al.,* 2001). The following factors are taken into account during the efficiency calibration; sample counting geometry, calibration method, calibration sources and the analytical efficiency expression.

It is recommended that the geometry of the reference standard materials should be the same as those used for the samples, e.g.

Marinelli beakers used for the standard mixed radionuclide and also for the samples. Several theoretical calibration methods exist for use, but it is recommended that efficiency calibration be determined experimentally for environmental measurements. Appropriate radionuclides must be selected as standards to perform efficiency calibration. The certificate accompanying the certified mixed radionuclides should state the following: uncertainty associated with activity, reference date, purity, mass or volume, chemical composition, values of half-lives, branching fractions for all modes of decay and the method of measurement of the radionuclides in the standard.

Efficiency calibration curves are usually represented by expressing the efficiency as a function of energy. The generally accepted and simple expression is as follows (IAEA, 1989).

$$\ln \varepsilon = a_1 + a_2 \ln E \qquad (4)$$

where ε is the absolute full energy peak efficiency; a_1 and a_2 are fit parameters; E is the energy (keV) of the corresponding gamma line. This expression is suitable for deter-mining efficiency of gamma energies from 200 to 2000 keV. To determine the efficiency for radionuclide with energies below 200 keV, a term with polynomial expression would improve the fit on the data [IAEA, 1989]. Samples should be counted in the same type of container used to obtain the efficiency calibration. The density, volume and height of sample in the container must be the same as that of the standards used for the calibration.

The minimum detectable activity (MDA) is defined as the smallest quantity of radioactivity that could be measured under specified conditions, and is another factor which is an important concept in environmental level measurement. The MDA depends on the lower limit of detection (LLD) and the counting efficiency of a counting system. The MDA is very important, particularly in environmental level systems, where the count rate of a sample is almost the same as the count rate of the background. Under these conditions, the background is counted with a blank, such as sample holder, and everything else that may be counted with an actual sample.

The purpose of the study was to investigate the performance of newly-installed NaI (TI) gamma spectrometry system to be used for analysis of radionuclides in food and environmental samples at the Radiation Protection Institute (RPI) of Ghana Atomic Energy Commission (GAEC). The Food and Environmental Laboratory of the RPI is involved in the measurement of ^{137}Cs contamination in food, imported into Ghana and/or exported out of Ghana. Environmental samples such as water, soil, and air are also measured to determine levels of radioactivity for uranium and thorium decay series and potassium-40.

The studies are carried out to ascertain the potential exposures due to naturally occurring radioactive materials (NORM) in some industries in Ghana, with the aim of establishing baseline data, and to determine the levels of NORMs from past industrial activities. Some of these industries include mining and mineral processing, oil and gas extraction, fertilizer production, municipal water treatment plants, etc. For quality and reliable results to be obtained there is the need to ascertain the performance of the measurement system by ensuring that the

equipment is adequately calibrated with reliable standard calibration sources.

Material and methods

Instrumentation and calibration

The specifications of the gamma spectrometry detector system under study are cylindrical scintillation detector Model 3M3/3-X, serial number ETI 9305, with a 1.2" × 1.2" end window, and was manufactured by Saint-Gobain Crystals, USA. The detector system consists of a vertically-sealed assembly which includes a NaI (Tl) crystal and is coupled to ORTEC Multichannel Buffers (MCBs) for data acquisition and processing through Connection-32 program MAESTRO®-32. The conversion gain of the detector is up to 1024 channels. In order to reduce background gamma radiation from the room in which the detector is installed, a locally fabricated cylindrical lead shield (20 mm) with a fixed bottom and a movable lid is used to shield the detector. Within the lead shield are also copper, cadmium and plexiglass (3 mm each) to absorb x-rays and other photons that might be produced in the lead. Fig. 2 is the block diagram of the detector assembly system.

Calibration of the gamma spectrometer

The energy and efficiency calibrations of the detector were performed using multinuclide reference sources in one liter Marinelli geometry. The standard reference material source number NW 146 has reference date 1st February 2006 at 12:00 GMT. The radionuclide mixture is homogeneously incorporated in the matrix of the source, with approximate volume of 1000 ml and density of 1.0 g cm^{-3}. The relative uncertainty of the activity determined according to the DKD-3 of Germany, as reported based on the standard uncertainty multiplied by a coverage factor of k = 2 at a confidence level of 95% is 3.0% (NIST, 1995).

The mixed radionuclide, in the form of solid water used for the calibration, contained 10 radionuclides in the energy range of 60-1836 keV as ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y and ^{60}Co. However, only six radionuclides in the standard were used for the experimental energy, namely ^{241}Am, ^{109}Cd, ^{57}Co, ^{137}Cs, ^{60}Co. All the radionuclides in the mixed standard could not be used for calibration because they had decayed off due to their short half-lives. Energy calibration was performed by matching the energies of the principal gamma-rays in the spectrum of
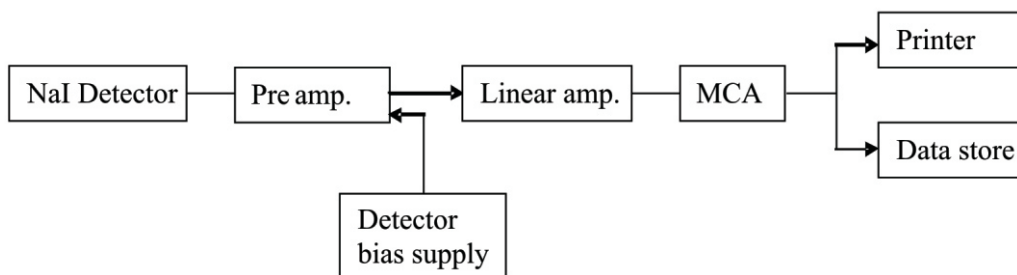

Fig. 2. Block diagram of the scintillation gamma spectrometry system

the standard reference material to the channel number of the spectrometer. Fig. 3 shows the energy calibration curve (linear), which was obtained from the plot of channel number on the X-axis and energy (keV) on the Y-axis.

Similarly, the efficiency calibration was performed by acquiring a spectrum of the calibration standard for 86400 s (24 h) i.e. until the count rate at the peak of total absorption can be calculated with statistical uncertainty of less than 1% at a confidence level of 95%. The net count rate was determined at the photopeaks for all the energies to be used for the determination of the efficiency calibration. The efficiency calibration of the NaI (Tl) detector was also carried out using the six different radionuclides obtained in the spectrum. Out of the six radionuclides, four radionuclides improved the fit of the data and the two discarded. The following radionuclides were used for the efficiency calibration: ^{57}Co (122 keV), ^{137}Cs (662 keV), ^{60}Co (1173 keV) and ^{60}Co (1333 keV).

The calibration process was repeated with a different geometry of mixed standard source number NW 147. The standard was constructed by homogenously distributing radionuclide mixture onto an active area of a plastic foil. The reference date of the source is 1st January 2006 at 12:00 GMT. The standard source contained ^{133}Ba, ^{57}Co, ^{139}Ce, ^{85}Sr, ^{137}Cs, ^{54}Mn, ^{88}Y and ^{65}Zn radionuclides in the energy range of 81–1836 keV. The reported uncertainties on the activity was based on standard uncertainty multiplied by a coverage factor of k = 2, at 95% confidence interval [NIST, 1995]. The efficiency curve was obtained by plotting the detection efficiency of NaI (Tl) as a function of energy. The calibration curves are shown in Fig. 5 and 6.

The full-energy-peak (FEP) efficiency was determined as follows.

$$Eff = \frac{100 . N_p}{A . I . T_c} \qquad (5)$$

where Np is the net peak area at E, I is the intensity of emitted gamma-ray (%), Tc is the time of counting (s), and A is the standard activity of the radionuclide at the beginning of counting.

The standard activity (A) was also calculated from the following decay equation:

$$A = Ao . \exp(-\lambda . Td) \qquad (6)$$

where Ao is the initial activity of the radionuclide at the reference date, λ is the decay constant of the individual radionuclide and Td is the time between the reference date of the radionuclide and the time of counting the standard. The absolute efficiency at each photopeak was plotted as a function of the peak energy and extrapolated to determine the efficiencies at other peak energies for the measurement geometry used.

The resolution of the detector was determined by measuring a standard ^{137}Cs source on the detector and a spectrum of the full energy peak (FEP) located at 662 keV for 600 seconds. The channel number and the corresponding counts were determined from FEP. A plot of the channel number on the X-axis and the counts on the Y-axis is showed in Fig. 7. A formal definition of the energy resolution is given in terms of the full width at half maximum (FWHM) divided by the location of the peak centroid E shown in Fig. 7. The resolution was determined from the equation.
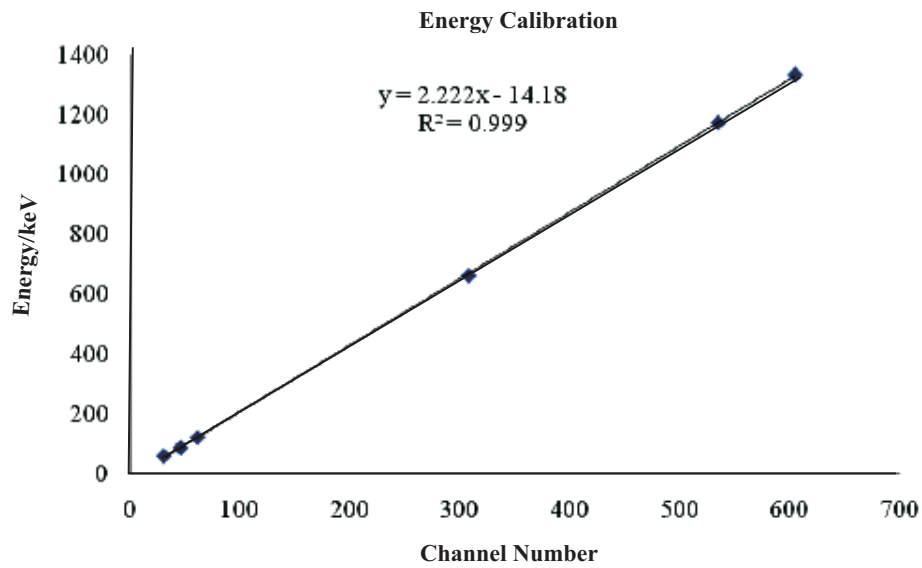
Energy Calibration

$y = 2.222x - 14.18$
$R^2 = 0.999$

Fig. 3. Energy calibration curve for a NaI (Tl) gamma detection system using mixed standar radionuclides in solid water form in one liter Marinelli beaker
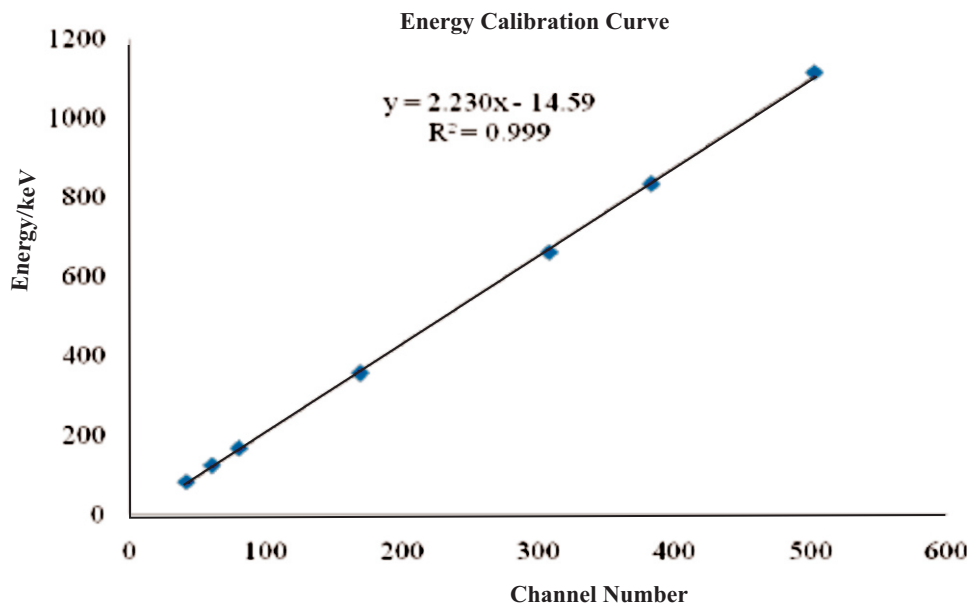
Energy Calibration Curve

$y = 2.230x - 14.59$
$R^2 = 0.999$

Fig. 4. Energy calibration curve for a NaI (Tl) gamma detection system using mixed standard radionuclides in a plastic foil

Efficiency calibration curve
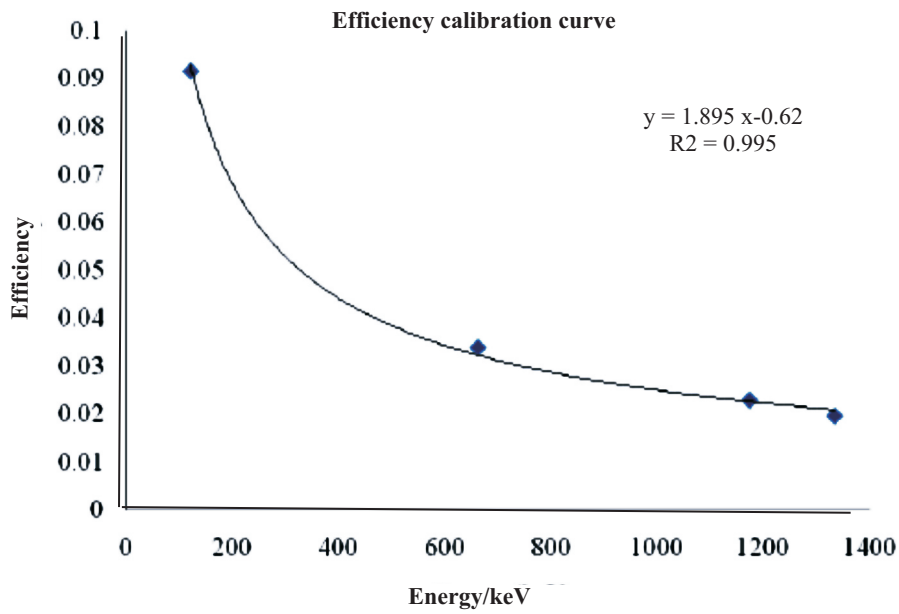
$$y = 1.895\,x{-}0.62$$
$$R2 = 0.995$$

Fig. 5. Efficiency calibration curve of a NaI (Tl) detection system using mixed standard radionuclide in solid
    water form in a 1litre Marinelli beaker

Efficiency calibration curve
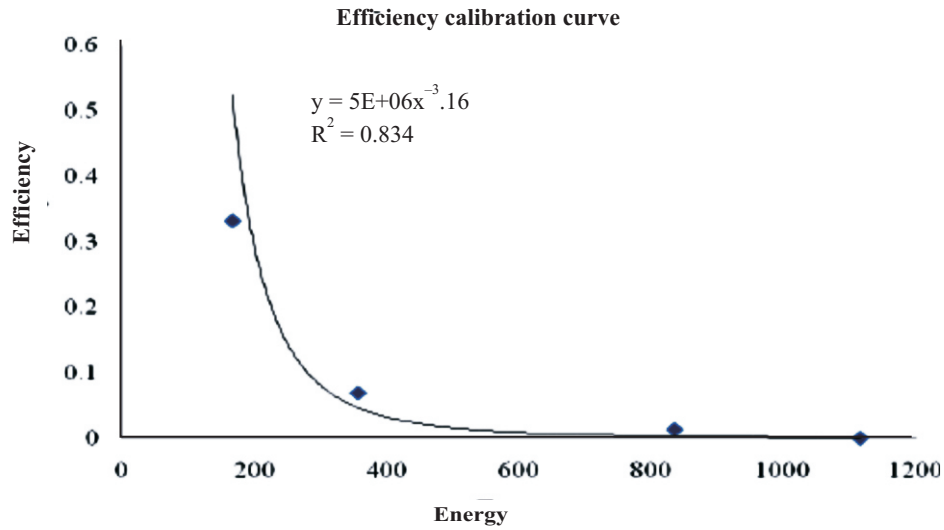
$$y = 5E{+}06x^{-3}.16$$
$$R^2 = 0.834$$

Fig. 6. Efficiency calibration curve of a NaI (Tl) detection system using mixed standard radionuclide in a plastic
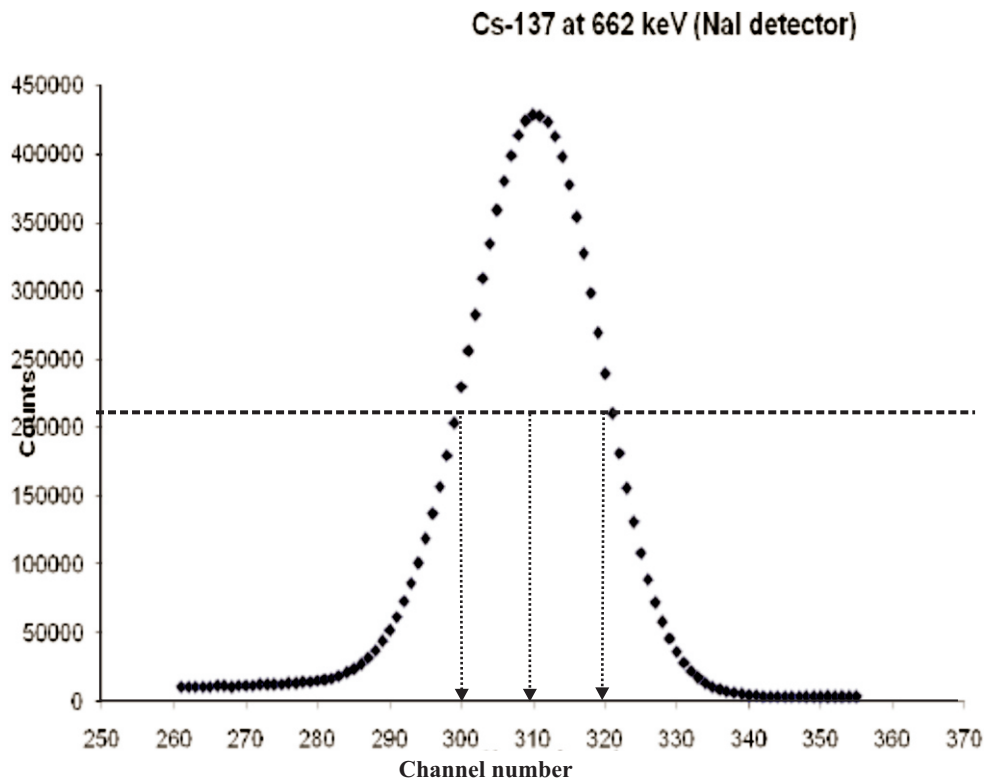   foil.

Fig. 7: Resolution of NaI (Tl) detector at 662 keV of [137]Cs.

$$\text{Resolution} = \frac{\text{Full width at half maximum}}{E} \quad (7)$$

Determination of lower limit of detection (LLD) and minimum detectable activity (MDA)

In order to determine the background distribution in the environment around the detector, 10 empty Marinelli beakers were counted in the same manner and in the same geometry as the samples. Each Marinelli beaker was thoroughly washed with detergents and dried in an oven at a temperature of 75 °C. They were then wiped with acetone and dried again in an oven. Each Marinelli beaker was sealed and counted on the NaI (Tl) detector for 86400 s to determine the background counts of the detection system. The average background counts of radionuclide that occurred in the background spectrum were used to determine the LLD and MDA (Cember, 1996). In the spectrum of the background counting, the following radionuclides [214]Pb (295.21 and 351.92 keV), and [214]Bi (609.31 and 1764.49 keV) were used to determine [238]U. To determine [232]Th the following radionuclides were used: [212]Pb (238.63 keV), [208]Tl (583.19 keV) and [228]Ac (911.21keV). In the background spectrum, the photopeaks of [40]K and ^{137}Cs, which occurred at 1460.83 and 662 keV, respectively, were used to determine their LLD and MDA.

Background counts were used to correct the net peak area of gamma-rays of the measured standard isotopes. The LLD and the MDA of ^{238}U, ^{232}Th, ^{40}K and ^{137}Cs were determined using equations 8 and 9, respectively (Cember, 1996; Curie, 1968).

$$LLD \quad 4.66 \;_b \quad 3 \tag{8}$$

$$MDA \quad \frac{4.66 \;_b \quad 3}{.\;.t} \tag{9}$$

where $\acute{o}_b$ is the standard deviation of the background in the region of interest and it is equal to the square root of the number of counts for the background spectrum, ε is the absolute efficiency of the detector, Y is the absolute gamma emission probability of the gamma decay, and t is the counting time in seconds.

Validation

The calibration and performance of the NaI (Tl) detector system was validated by measuring standard reference material containing mixed radionuclides of known activities. The peak area of each radionuclide determined was treated as unknown and then applying the efficiency values determined from Fig. 5 to calculate the activity. The calculated absolute efficiency of each radionuclide from Equation 5 and the net peak areas were used to calculate the activity of each radionuclide in the sample. The measured and the calculated activities from Equation 6 were compared. If the detector were performing well then the measured and the calculated activity values should not differ significantly.

Results and discussion

Fig. 3 and 4 are the energy calibration curves obtained when the standard radionuclides in two different matrices were counted on the NaI (Tl) detector. The energy calibration curve indicates the correlation between the energy of radionuclide and the corresponding channel number at the centroid of a full energy peak. The plot fitted a linear function with equation as shown in Fig. 3, which indicates that the detector system is performing well. Even when the matrices for the standard radionuclides were different the energy calibration curves appear identical with the same correlation of $R^2 = 0.999$.

The efficiency calibration curves were obtained by plotting the full energy peak efficiency as a function of energy with the calibration equations as power functions shown in Fig. 5 and 6. The calibration curves are valid for energies greater than 100 keV up to about 1400 keV. The efficiency curve was obtained by discarding two out of the six radionuclides which were used for the energy calibration, since the data points of the four radionuclides fitted well into the efficiency curve. The correlation in Fig. 5 and 6 were $R^2 = 0.995$ and $R^2 = 0.834$ respectively indicating the correlation in Fig. 5 is better than that in Fig. 6. This also indicates that the standard radionuclides in the solid water, used to carry out the efficiency calibration, seem to give better results than the standard radionuclides in the plastic foil.

Fig. 7 is an illustration of the determination of the resolution of NaI detector at 662 keV of a standard ^{137}Cs. The resolution was estimated to be 7.4%, which compared quite well with the manufacturer's recommended value of 7.5% for ^{137}Cs.

Resolution at 662 keV of ^{137}Cs

$$= \frac{\text{FWHM}}{\text{Peak Count Channel Number}} \text{ x } 100\% \quad (10)$$

Table 1 shows the result of the LLD and MDA that were determined from the background counting of 10 empty Marinelli beakers. The LLD and the MDA were calculated from Equations 8 and 9. The average MDAs of ^{238}U, ^{232}Th, ^{40}K and ^{137}Cs were estimated to be 0.099 ± 0.055 Bq, 0.061 ± 0.037 Bq, 0.727 ± 0.300 Bq and 0.014 ± 0.006 Bq, respectively.

Table 2 shows the results from the validation of the calibration of the NaI detector. The results of the certified and the measured activity concentrations compared quite well with the percentage deviation range from 1.43 to 10.47%. The ratios of the measured to the certified values of the activities were in a range of 0.90–0.98, which indicates an insignificant difference between the measured and the certified values.

The results of the validation are also provided in Fig. 8 and 9. In Fig. 8 the percentage deviation and the energy of the radionuclides used for the calibration and their error bars are compared. Fig. 9 shows the comparison of the measured and the calculated activities of the radionuclides used

TABLE 1
LLD and MDA from background counting

Nuclide	Energy keV	Background counts	LLD(counts)	MDA (Bq) $\pm$ Uncertainty
U-238	295.21	38	31.73	0.026 ± 0.011
	351.92	49	35.62	0.045 ± 0.014
	609.31	50	35.95	0.043 ± 0.036
	1764.49	13	19.80	0.293 ± 0.157
Average		37.5	31.54	0.099 ± 0.055
Th-232	238.63	35	30.57	0.011 ± 0.006
	583.19	50	35.95	0.059 ± 0.026
	911.21	40	32.47	0.113 ± 0.081
Average		41.7	33.09	0.061 ± 0.037
K-40	1460.83	80	44.68	0.727 ± 0.300
Cs-137	662	13	19.80	0.0140 ± 0.006

TABLE 2
Calculated and measured activities of standards radionuclides used for energy and efficiency calibration

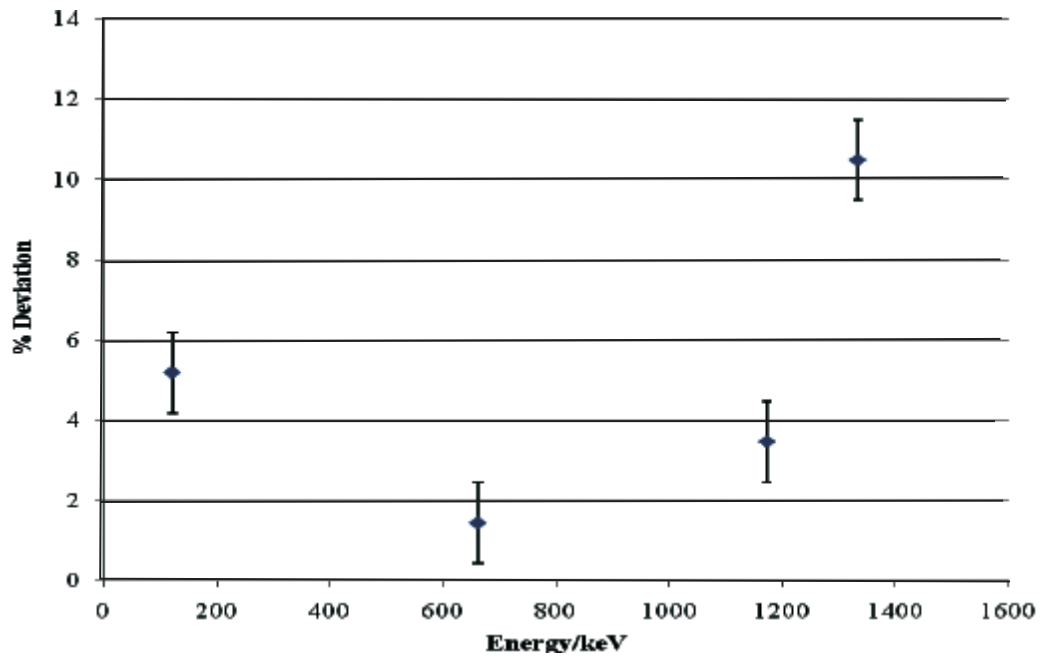| Nuclide	Energy (keV)	Net Area	Calculated activity (Bq)	Measured activity (Bq)	Percent deviation	Ratio of measured to calculated
Co-57	122	209282	3.10E+01	2.94E+01	5.18	0.95
Cs-137	662	625772	2.56E+03	2.52E+03	1.43	0.98
Co-60	1173	417998	2.12E+03	2.04E+03	3.46	0.96
Co-60	1333	358561	2.12E+03	1.90E+03	10.47	0.90

Fig. 8. Plot of % deviation of measured to calculated activity concentrations versus Energy of radionuclides use for calibration
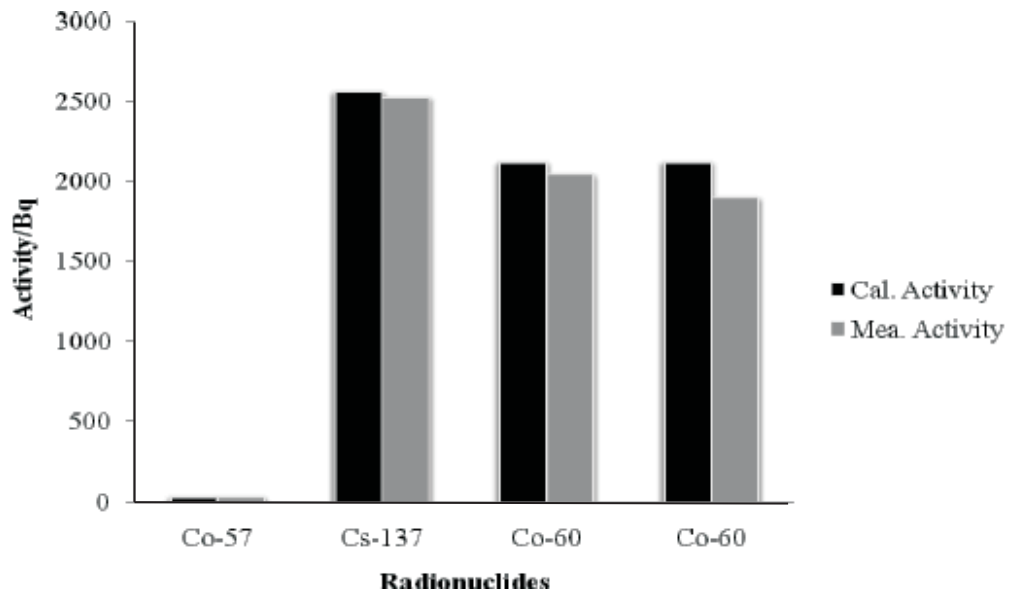

Fig. 9. Comparison of calculated and measured activities of radionuclides

in the calibration of the NaI (Tl) scintillation detector. From Fig. 9 and 10 no significant difference between the measured and the calculated activities of the standard radionuclides were observed.

Conclusion

The study was carried out with respect to the energy and efficiency calibrations, and validation of the calibration results. This was done by using standard reference materials in the energy range of interest. The accuracy of the efficiency calibration was verified by counting the mixed radionuclide standard and treating the full energy peaks as unknown to determine the activities of the standard radionuclides. The results of the measured and the calculated activities of the standard radionuclides were compared. The ratio of the measured to the calculated activity values of reference radionuclides were in the range of 0.90–0.98, which are almost to unity, indicating that there was no significant difference between the measured and the calculated reference standards. The percentage deviation between the measured and the calculated activity values were estimated to be in the range of 1.43–10.47%. The resolution of the NaI detector system was estimated to be 7.4% which compares well with the manufacturer's recommended value of 7.5% at 662 keV of ^{137}Cs.

The MDA of ^{238}U, ^{232}Th, ^{40}K and ^{137}Cs were estimated to be 0.099 ± 0.055 Bq, 0.061 ± 0.037 Bq, 0.727 ± 0.300 Bq and 0.014 ± 0.006 Bq, respectively. The results of the evaluated parameters: energy calibration, efficiency calibration, the resolution of the detector, validation of the calibration and the estimated MDAs show that the NaI (Tl) detector system is working quite well. The detector system is suitable for use in the quantitative determination of radionuclides in food and environmental samples.

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