

Self-absorption correction and efficiency calibration for radioactivity measurement of environmental samples by gamma-ray spectrometry

Ryszard Misiak,
Roman Hajduk,
Marcin Stobiński,
Mirosław Bartyzel,
Katarzyna Szarłowicz,
Barbara Kubica

Abstract. In this work empirical functions which relate the full-energy peak efficiency with sample height, energy, matrix composition and bulk density for voluminal samples in a cylindrical counting geometry were found. Accurate determination of the radioactivity of gamma-emitting radionuclides in environmental samples requires taking into account self-absorption. For the obtained self-absorption correction factor, a direct transmission method was chosen. Finally, this paper proposes a simple correlation between the self-absorption correction factor and the bulk density and height of the measured sample for a given energy.

Key words: gamma-spectrometry • efficiency calibration • self-absorption • environmental

R. Misiak[✉], R. Hajduk, M. Stobiński, M. Bartyzel
Division of Applications of Physics
and Interdisciplinary Research,
The Henryk Niewodniczański Institute of Nuclear
Physics, Polish Academy of Sciences,
152 Radzikowskiego Str., 31-342 Kraków, Poland,
Tel.: +48 12 662 8390, Fax: +48 12 662 8458,
E-mail: ryszard.misiak@ifj.edu.pl

K. Szarłowicz
Department of Analytical Chemistry,
Faculty of Chemistry,
Jagiellonian University,
3 Ingardena Str., 30-060 Kraków, Poland
and Division of Applications of Physics and
Interdisciplinary Research,
The Henryk Niewodniczański Institute of Nuclear
Physics, Polish Academy of Sciences,
152 Radzikowskiego Str., 31-342 Kraków, Poland

B. Kubica
Division of Applications of Physics
and Interdisciplinary Research,
The Henryk Niewodniczański Institute of Nuclear
Physics, Polish Academy of Sciences,
152 Radzikowskiego Str., 31-342 Kraków, Poland
and Faculty of Fuels and Energy,
AGH University of Science and Technology,
30 A. Mickiewicza Ave., 30-059 Kraków, Poland

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Introduction

Gamma-ray spectrometry is widely used for the determination of radionuclide activity in nature. This technique allows to carry out measurements without the routinely used radiochemical methods and multielemental analysis [1, 10, 12, 17, 26, 27, 29].

In order to obtain correct results, voluminal samples shall be counted under the same measuring conditions (geometric setup, chemical composition, density) as those under which the γ -spectrometer has been calibrated. However, in many cases this is not possible, above all when we want to measure environmental samples. The latter have different chemical compositions and density. This is the reason why attenuation correction is an important factor. In the last three decades a number of ways of comprehensive calibration of Ge detectors were developed, especially concerning measurements of environmental volume samples. Generally, we have two approaches: mathematical [9, 11, 13–15, 23, 28, 30] and experimental [2–7, 16, 18–22, 24, 25].

Accurate determination of the activity of radionuclides emitting γ -rays in environmental samples requires a knowledge of the efficiencies of full-energy peaks of the detector and self-absorption corrections for these full-energy peaks. Value of self-absorption of γ -rays in the sample depends on many factors: chemical composition of the sample, density, weight of the sample and gamma-emission energy. Another difficulty of the calibration process is the variable sample size that we have to measure [4, 5, 21, 22].

The aim of this study was to carry out a comprehensive calibration of the three spectrometers, allowing de-

Table 1. Parameters of HPGe detectors

Number of spectrometer	Type	Relative efficiency	Resolution – FWHM ^a at 1.33 MeV	Producer
1	Coaxial – <i>p</i>	7%	2.5 keV	Institute of Nuclear Physics, Kraków
2	Coaxial – <i>p</i>	16%	2.3 keV	
3	Coaxial – <i>n</i>	22%	2.3 keV	Eurysis Measures (now Canberra Eurysis)

^aFWHM – full width at half maximum.

termination of radioactivity of ¹³⁷Cs and ⁴⁰K in different environmental samples (moss, bilberry, grass, soil and sediment) investigated in our laboratory.

Experimental

The calibration procedure was developed for three coaxial high purity germanium (HPGe) detectors. Parameters of the detectors are presented in Table 1.

Detectors were connected to the standard setup: Canberra or Ortec Amplifiers and High Voltage modules, and then to an ORTEC EtherNIM Multichannel Buffer 919E (high-speed, 4-input multiplexer/router, with individual start/stop/preset/conversion gain control of each input). The spectrometers were shielded by 5 cm lead bricks with an inner 2 mm Cu layer. Additionally, between the spectrometer shield and wall of the room was built a wall of 5 cm lead bricks, due to decreasing background of ⁴⁰K.

All spectrometric measurements were performed for samples being enclosed in polyethylene cylindrical containers with a 47 mm bottom diameter and a 60 mm top diameter. The reference material IAEA-154 (whey powder, bulk density 0.76 g/cm³) was chosen to perform the efficiency calibration process for ¹³⁷Cs ($E_\gamma = 661.6$ keV) and ⁴⁰K ($E_\gamma = 1460.8$ keV).

Different environmental samples (moss, bilberry, grass, soil and sediment) were used to the determination of self-absorption correction factor. Samples bulk density ranging from 0.15 to 1.8 g/cm³. Samples and reference materials heights varying from 10 to 70 mm with intervals of 5 mm.

Gamma spectra were collected five times for each measuring point (for a certain type of matrix, density and height).

The method

The activity concentration, A , of a given radionuclide in the comparative method, when the calibration and real samples have almost identical geometrical setup, apparent density and chemical composition, is calculated from the formula:

$$(1) \quad A_s = \frac{A_{\text{ref}} \times (\text{CPS}_s - \text{CPS}_b)}{(\text{CPS}_{\text{ref}} - \text{CPS}_b)}$$

where: CPS = N/t , N is the net area under the full-energy peak corresponding to the photon of energy E_γ with an emission probability P_γ and t is the counting time. Subscripts refer to: *s* – sample; ref – reference material; *b* – background.

Therefore, we do not have to know the full-energy peak efficiency and self-absorption factor.

The real samples are characterized by the different apparent density and chemical composition in a wide range. It is very difficult to obtain calibration samples with identical listed physicochemical properties as the real samples. In the case of real voluminal sample the activity concentration of the radionuclide of interest, A_s , is calculated from the expression:

$$(2) \quad A_s = \frac{(\text{CPS}_s - \text{CPS}_b) \times 1000}{\varepsilon(E_\gamma, h) \times P_\gamma \times T_z(E_\gamma, h, \rho) \times m_s} \quad [\text{Bq / kg}]$$

where: $\varepsilon(E_\gamma, h)$ is the full-energy peak efficiency corresponding to E_γ and h (sample height); $T_z(E_\gamma, h, \rho)$ – self-absorption factor dependent on E_γ , h and ρ (sample bulk density); m_s – mass of the sample [g].

For the calculation of the activity of radionuclides for different real samples and their various height, we need to determine the functional dependence for $\varepsilon(E_\gamma, h)$ and $T_z(E_\gamma, h, \rho)$.

Results and discussion

$\varepsilon(E_\gamma, h)$ determination

The experimental efficiency $\varepsilon(E_\gamma)$ at energy E_γ and a selected geometry is calculated through the equation:

$$(3) \quad \varepsilon(E_\gamma) = \frac{(\text{CPS}_{\text{ref}} - \text{CPS}_b)}{A_{\text{ref}} \times P_\gamma \times m_{\text{ref}}}$$

where: A_{ref} – the radioactivity of radionuclide of interest in reference material; m_{ref} – mass of reference material.

The value of $\varepsilon(E_\gamma)$ depends on the density of the sample for the same measuring conditions. Some authors use an approach based on the determination of full-energy peak efficiencies for different densities and a given geometric setup [5, 18–20, 24]. In recent years, Monte Carlo methods have been applied to compute the full-energy peak efficiency [9, 11, 13, 14, 30].

In our laboratory practice, we often deal with variable sample sizes (from 15 to 200 g). Therefore, we had to make a calibration for obtaining a full-energy peak efficiency $\varepsilon(E_\gamma, h)$ curve reflecting the variability in the sample height h . In this study, reference material IAEA-154 (whey powder, $\rho = 0.76$ g/cm³) was used to perform needed measurements for the determination of full-energy peak efficiency of ¹³⁷Cs ($E_\gamma = 661.6$ keV) and ⁴⁰K ($E_\gamma = 1460.8$ keV) in the range height from 10 to 70 mm (with intervals of 5 mm), independently

of any spectrometer. Spectra were taken during 72 or 96 h in order to decrease the counting uncertainties below 2%.

The calculated experimental full-energy peak efficiency for different height of calibration sample was fitted for one-phase exponential decay function. The following equations were obtained:

Spectrometer no. 1

$$(4) \quad \varepsilon_{\text{Cs-137}} = 0.005 * \exp(-0.33 * h/h_o) + 0.00154$$

$$R^2 = 0.99$$

$$(5) \quad \varepsilon_{\text{K-40}} = 0.00184 * \exp(-0.227 * h/h_o) + 0.00038$$

$$R^2 = 0.98$$

Spectrometer no. 2

$$(6) \quad \varepsilon_{\text{Cs-137}} = 0.01 * \exp(-0.249 * h/h_o) + 0.0018$$

$$R^2 = 0.99$$

$$(7) \quad \varepsilon_{\text{K-40}} = 0.005 * \exp(-0.264 * h/h_o) + 0.0009$$

$$R^2 = 0.99$$

Spectrometer no. 3

$$(8) \quad \varepsilon_{\text{Cs-137}} = 0.016 * \exp(-0.284 * h/h_o) + 0.0038$$

$$R^2 = 0.99$$

$$(9) \quad \varepsilon_{\text{K-40}} = 0.0068 * \exp(-0.24 * h/h_o) + 0.00136$$

$$R^2 = 0.99$$

where R^2 – coefficient of determination and $h_o = 1$ cm.

Bolivar *et al.* [4] and Perez-Moreno *et al.* [22] found a similar experimental dependence of the full-energy peak efficiency $\varepsilon(E_\gamma, h)$ on the sample height h . It was described by an exponential growth function:

$$(10) \quad \varepsilon(E_\gamma, h) = \alpha e^{-\beta h}$$

where α and β are the parameters which depend on E_γ .

$T_z(E_\gamma, h, \rho)$ determination

Gamma rays on passing through matter disappears exponentially. The phenomenon is often referred to as sample self-absorption or sample attenuation. A relation of gamma-ray beam I after passing the absorbent of linear attenuation coefficient η [cm^{-1}] and thickness x [cm] can be expressed as:

$$(11) \quad I = I_0 e^{-\eta x} = I_0 e^{-\mu \rho x}$$

where: I_0 – initial beam intensity; μ – mass attenuation coefficient [cm^2/g]; ρ [g/cm^3] – density.

In measurements by gamma spectrometry this value of η should be estimated for different measured environmental samples.

To determine the self-absorption corrections a transmission method proposed by Cutshall *et al.* [6] and modified by Joshi [16] and Bolivar *et al.* [3] was chosen. In this method the direct transmission coefficient T_γ is calculated as the ratio between the transmission

rates, when a radioactivity source is placed above the container with the sample (CPS_s), and the empty container (CPS_{bl}). T_γ for the gamma-ray energy E_γ is then given by

$$(12) \quad T_\gamma = \frac{\text{CPS}_s}{\text{CPS}_{bl}} = e^{-\mu \rho x}$$

The direct transmission coefficients T_γ were calculated for different environmental samples (moss, bilberry, grass, soil and sediment) including also the following IAEA reference materials of CRM code: 154, 315, 326, 373, 375 and their different height. Samples bulk density ranging from 0.15 to 1.8 g/cm^3 and samples height varying from 10 to 70 mm. ^{152}Eu source was used in the transmission method measurements, giving the range of gamma-ray energy from 100 to 1500 keV. Dependence of the direct transmission coefficients T_γ on energy for the measuring point (for a given density and height of the measured sample) were described by the power series function:

$$(13) \quad T_\gamma(E_\gamma) = AE_\gamma^B + CE_\gamma^D$$

where: A, B, C, D – fitting parameters. The coefficients of determination (R^2) for each dependency ranged from 0.87 to 0.99.

Obtained dependencies allowed for the determination of T_γ for ^{137}Cs ($E_\gamma = 661.6$ keV) and ^{40}K ($E_\gamma = 1460.8$ keV) for the measured points in experiments.

The full-energy peak efficiency $\varepsilon(E_\gamma, h)$ for ^{137}Cs (661.6 keV) and ^{40}K (1460.8 keV) was determined from the measurements of reference material IAEA-154 (bulk density – 0.76 g/cm^3). It appeared necessary to calculate a self-absorption correction factor $T_z(E_\gamma, \rho, h)$, expressed as the ratio of the direct transmission coefficient of the sample $T_s(E_\gamma)$ and the reference material $T_{\text{ref}}(E_\gamma)$:

$$(14) \quad T_z(E_\gamma) = T_s(E_\gamma)/T_{\text{ref}}(E_\gamma)$$

for the tested range of bulk density ρ and height h .

For a certain gamma-ray energy, the self-absorption correction factor $T_z(E_\gamma, \rho, h)$ depends on the density of sample (ρ_s) and reference material (ρ_{ref}) and height of sample. For this reason, an additional variable X was introduced and expressed by the equation:

$$(15) \quad X = \log\left(\frac{\rho_s}{\rho_{\text{ref}}}\right) * \frac{h}{2}$$

The variation of $T_z(E_\gamma)$ with ρ_s , ρ_{ref} and h was described by the polynomial fourth order:

$$(16) \quad T_z(\rho_s, \rho_{\text{ref}}, h) = a + b * X + c * X^2 + d * X^3 + e * X^4$$

where a, b, c, d, e – fitting parameters.

As it can be seen in Figs. 1 and 2 the following functional dependencies of $T_z(E_\gamma)$ on X were obtained:

Spectrometer no. 1

$$(17) \quad T_{\text{Cs-137}} = 0.994 + (-0.1) * X + (-0.0377) * X^2 + (-0.0184) * X^3 + (-0.00235) * X^4$$

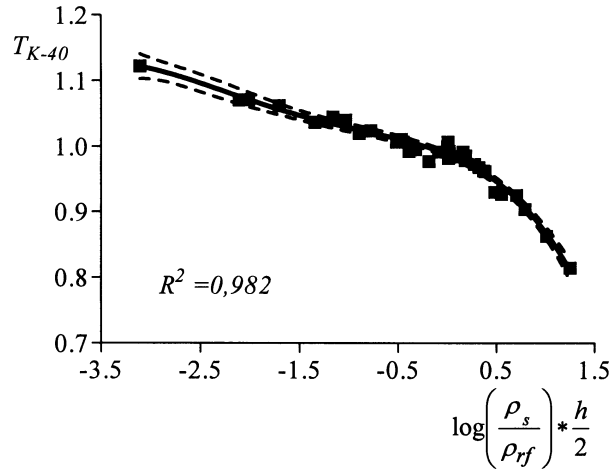


Fig. 1. Correction factor T_{K-40} vs. X for $E_\gamma = 1460.8$ keV and spectrometer no. 1.

$$(18) \quad T_{K-40} = 0.99 + (-0.0643) * X + (-0.0215) * X^2 + (-0.0186) * X^3 + (-0.00455) * X^4$$

Spectrometer no. 2

$$(19) \quad T_{Cs-137} = 1.0 + (-0.085) * X + (-0.0311) * X^2 + (-0.0238) * X^3 + (-0.00418) * X^4$$

$$(20) \quad T_{K-40} = 0.99 + (-0.0604) * X + (-0.0366) * X^2 + (-0.0197) * X^3 + (-0.00313) * X^4$$

Spectrometer no. 3

$$(21) \quad T_{Cs-137} = 0.998 + (-0.1168) * X + (-0.0257) * X^2 + (-0.0039) * X^3 + (-0.00017) * X^4$$

$$(22) \quad T_{K-40} = 1.0 + (-0.0813) * X + (-0.0188) * X^2 + (-0.0038) * X^3 + (-0.00014) * X^4$$

The coefficients of determination R^2 for a set of equations are greater than 0.97. The variable X is equal to 0.0 when $\rho_s = \rho_{ref}$ and $T_z(E_\gamma)$ takes obviously the value 1.0.

In papers using the gamma-ray transmission method [2–4, 18, 25], the authors do not take to the calculations $T_z(E_\gamma)$ the direct transmission coefficient, but the self-absorption factor $F(E_\gamma)$ is written by Cutshall [6] as:

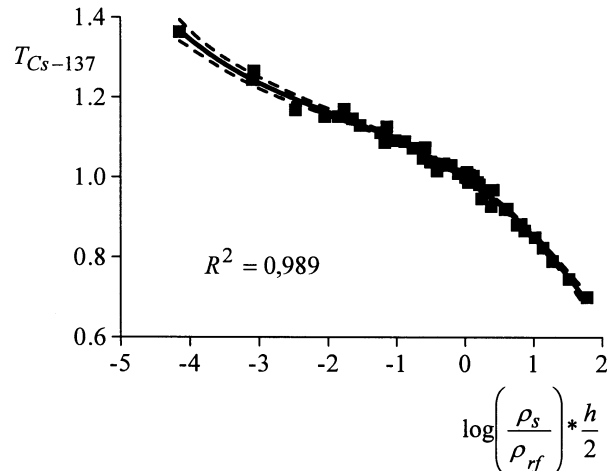


Fig. 2. Correction factor T_{Cs-137} vs. X for $E_\gamma = 661.6$ keV and spectrometer no. 3.

$$(23) \quad F(E_\gamma) = \frac{T_\gamma - 1}{\ln(T_\gamma)}$$

and then

$$(24) \quad T_z(E_\gamma) = F_s(E_\gamma)/F_{ref}(E_\gamma)$$

Variation of the obtained $T_z(E_\gamma)$ with bulk sample density at a selected sample height h is represented by the function [2–4, 18, 25]:

$$(25) \quad T_z(E_\gamma) = Ke^{-L\rho},$$

where K and L are energy-dependent parameters [3].

Comparing the values of $T_z(E_\gamma)$, calculated from Eqs. (14) and (24), it can be noticed that the values obtained from Eq. (14) are higher for $\rho_s < \rho_{ref}$ and lower for $\rho_s > \rho_{ref}$ than from Eq. (24) at selected energy and geometry. The difference increases with increasing sample height, but decreases with increasing gamma-ray energy. Galloway [8] also demonstrated that the real self-absorption is bigger than the Cutshall one, especially below 300 keV. McMahon *et al.* [18] were examined the differences between the measured self-absorption correction factors based on spiked matrix-matched samples and those obtained using the direct transmission method (Cutshall approximation). The authors showed that the value of deviation between the two methods ranged from 0 to 15% and is the greatest at lower energies and for samples with higher density.

Validation tests

Validity of the proposed comprehensive calibration in this work has been tested for the three spectrometers by performing some measurements of radioactivity of reference materials for their different height in a polyethylene container. IAEA-373 and IAEA-375 used as calibrands were chosen. The obtained results are presented in Tables 2 and 3.

The differences (in %) between the measured and reference value, shown in Tables 2 and 3, are given in brackets. Obtained results show that the total error of calibration method is $\pm 5\%$ for the determined radioactivity of ^{137}Cs and about $\pm 10\%$ for ^{40}K . The greater measurement error for ^{40}K is related to its background in our measurement conditions, especially for small mass or low radioactivity of the sample. It is obvious that for the same sample weight and counting time, the relative efficiency of detector play a role in the statistical error of measurement.

Conclusions

A general method for γ -ray efficiency calibration of HPGe coaxial detectors is presented. The direct transmission method was used. The empirical functions were found, which relate the full-energy peak efficiency with sample height, energy, matrix composition and bulk density for voluminal samples in a cylindrical counting geometry.

Table 2. The radioactivity of ^{137}Cs and ^{40}K measured for IAEA-373 (grass, $\rho = 0.38 \text{ g/cm}^3$, Kiev region, Ukraine, reference value: $^{137}\text{Cs} - 10\,118 \text{ Bq/kg}$ and $^{40}\text{K} - 432 \text{ Bq/kg}$). Measured and reference radioactivities were counted to the date 01.09.2000

h (cm)/ m_s (g)	Spectrometer no. 1		Spectrometer no. 2		Spectrometer no. 3	
	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)
0.8/3.4	9 805.3 (-3.1%)	541.5 (+25.3%)	10 354.4 (+2.3%)	504.2 (+16.7%)	10 321.3 (+2.0%)	473.1 (+9.5%)
1.7/8.95	10 389.4 (+2.7%)	498.7 (+15.4%)	9 917.9 (-2.0%)	394.3 (-8.7%)	9 964.7 (-1.5%)	409.2 (-5.3%)
2.3/13.2	10 207.8 (+0.9%)	428.2 (-0.9%)	9 958.4 (-1.6%)	406.4 (-5.9%)	10 243.6 (+1.2%)	405.1 (-6.2%)
3.4/22.4	9 743.0 (-3.7%)	440.7 (+2.0%)	9 673.8 (-4.4%)	435.9 (+0.9%)	9 688.3 (-4.3%)	431.6 (-0.1%)
4.8/31.2	10 068.5 (-0.5%)	459.3 (+6.3%)	10 295.4 (+1.8%)	442.6 (+2.5%)	10 209.8 (+0.9%)	438.7 (+1.6%)

Table 3. The radioactivity of ^{137}Cs and ^{40}K measured for IAEA-375 (soil, $\rho = 1.43 \text{ g/cm}^3$, Bryansk region, Russia, reference value: $^{137}\text{Cs} - 4326 \text{ Bq/kg}$ and $^{40}\text{K} - 424 \text{ Bq/kg}$). Measured and reference radioactivities were counted to the date 01.09.2000

h (cm)/ m_s (g)	Spectrometer no. 1		Spectrometer no. 2		Spectrometer no. 3	
	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)
1.0/21.25	4108.3 (-5.0%)	390.1 (-8.0%)	4486.2 (+3.7%)	436.2 (+2.9%)	4494.2 (+3.9%)	437.7 (+3.2%)
1.9/41.25	4267.3 (-1.4%)	412.4 (-2.7%)	4232.0 (-2.2%)	425.2 (+0.3%)	4483.6 (+3.6%)	435.4 (+2.7%)
2.9/72.05	4287.5 (-0.9%)	407.2 (-4.0%)	4313.2 (-0.3%)	412.7 (-2.7%)	4475.3 (+3.4%)	446.9 (+5.4%)
4.5/119.8	4393.1 (+1.6%)	420.1 (-0.9%)	4381.7 (+1.3%)	417.8 (-1.5%)	4398.5 (+1.7%)	421.9 (-0.5%)

In this paper a simple correlation between the self-absorption correction factor $T_z(E_\gamma)$ and the bulk density and height of the measured sample (Eqs. (15) and (16)) was proposed.

The direct transmission coefficients T_γ of different environmental samples (moss, bilberry, grass, soil and sediment) in the range of bulk density from 0.15 to 1.8 g/cm^3 and height of sample from 10 to 70 mm was determined. The obtained values of T_γ form a database consisting of three parameters: E_γ , ρ and h . The database can be used for activity measurement of other radionuclides in environmental samples emitting gamma-ray in the energy range from 100 to 1500 keV as well as after a repair of gamma spectrometers.

The test involving the validation of the comprehensive calibration with the sample bulk density and height has shown good agreement of the measured radioactivity and the values recommended by the IAEA to the IAEA-373 and IAEA-375 calibrands.

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