

Validation of the full spectrum analysis technique for determining radionuclide activity in environmental samples using HPGe spectrometry

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ABSTRACT

Introduction: Analytical method validation is crucial for ensuring data quality in environmental radioactivity monitoring. This study presents a systematic validation and comparative assessment of two prominent techniques for high-purity germanium (HPGe) gamma-ray spectrometry: full spectrum analysis (FSA) and the traditional energy window analysis (EWA).

Methods: FSA models a measured spectrum as a linear combination of basis spectra, while EWA relies on individual peak-area analysis. The accuracy and robustness of both methods were rigorously evaluated by analyzing a diverse set of IAEA reference materials with varying matrices and activity levels.

Results: Both methods demonstrated high accuracy, with most results for both FSA (71%) and the corrected EWA (86%) falling within a $\pm 15\%$ relative deviation from the certified values. EWA showed exceptional accuracy for nuclides with clear, high-intensity peaks. FSA provided comparable or slightly better results for certain complex spectra and low-activity nuclides. Additionally, a strong physical correlation ($R^2 = 0.89$) between the FSA background-scaling factor and sample density was confirmed, highlighting FSA's capability to partially compensate for matrix effects.

Conclusion: This validation study confirms that both the corrected EWA and FSA are highly accurate, precise, and reliable methods for environmental radioactivity analysis. EWA exhibited slightly more consistent accuracy across this dataset; however, FSA can serve as a powerful complementary tool, excelling in cases with complex interferences. The choice of method is application-dependent, but both are suitable for routine use in accredited laboratories.

Key words: Full spectrum analysis (FSA), Energy window analysis (EWA), Method validation, HPGe spectrometry, Environmental radioactivity, IAEA reference materials, Radionuclide activity.

INTRODUCTION

Monitoring environmental radioactivity is paramount for public health, radiation protection, and understanding geochemical processes. Environmental matrices, such as soil and sediment, contain naturally occurring radioactive materials (NORM), primarily from the ^{23}U and ^{232}Th decay series and the primordial radionuclide $^{\circ}\text{K}$. Additionally, anthropogenic radionuclides, most notably ^{137}Cs from nuclear weapons testing and accidents like Chernobyl, persist in the environment and require careful surveillance.

High-purity germanium (HPGe) gamma-ray spectrometry is the gold standard for identifying and quantifying these radionuclides because its unparalleled energy resolution allows for the clear separation of complex gamma-ray lines from multiple isotopes within a sample. Traditionally, the quantification of radionuclide activity from a gamma-ray spectrum relies on energy window analysis (EWA), also known

as the peak-area method. This approach involves defining a region of interest around a specific full-energy peak and calculating its net area after subtracting the underlying background continuum. Although EWA is a straightforward and widely used method, it has limitations. For instance, statistical precision for low-activity peaks may be insufficient, often requiring long measurement times to achieve acceptable uncertainty. Furthermore, the accurate subtraction of the Compton background, especially in complex spectra with multiple overlapping peaks, is challenging and can be an important source of systematic error.

Full-spectrum analysis (FSA) has emerged as a powerful alternative to overcome these challenges. Instead of focusing on isolated photopeaks—like the traditional EWA method—FSA uses the information contained within the entire gamma-ray spectrum. Its core principle is to model the measured spectrum as a linear combination of standard “basis” spectra from each radionuclide in the sample. This holistic approach offers several key advantages: It im-

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proves counting statistics by incorporating the Compton continuum and smaller peaks, which can lead to lower detection limits or reduced measurement times; it simplifies background handling by treating the background as a component to be fitted simultaneously, eliminating the need for manual subtraction procedures; and it inherently mitigates spectral interferences by fitting the combined shape of overlapping peaks, reducing errors that can arise from deconvolution algorithms.

FSA's application has evolved since its inception and remains an active area of research. Foundational work by Hendriks et al. (2001)¹ established the method's viability, whereas recent studies have focused on enhancing its robustness and expanding its applications. For instance, advancements in computational power and machine learning have led to more sophisticated FSA algorithms. Hanka L. (2020)² demonstrated the use of a deep neural network to deconvolve complex spectra, improving the accuracy of FSA when heavily overlapping peaks are present. Concurrently, FSA has become indispensable for emerging mobile survey platforms where short measurement times are required. Von Frankenberg et al. (2021)³ integrated FSA into an uncrewed aerial vehicle (UAV) system, enabling the rapid and efficient radiological mapping of large areas. The technique is also being applied to more complex and challenging matrices beyond standard environmental samples. Ali et al. (2021)⁴ explored the application of FSA for the characterization of NORM-contaminated industrial residues, where the sample composition and density can differ substantially from calibration standards, highlighting the need for robust matrix effect corrections. Naeem et al. (2025)⁵ showcased the advantages of FSA in real-time, in-situ soil analysis, demonstrating its enhanced precision and speed, compared to traditional methods.

Nonetheless, rigorous validation against certified standards is necessary to establish the credibility of any analytical method for routine, accredited use. Although the power of FSA is well-recognized, its adoption by accredited laboratories often requires comprehensive evidence of its accuracy, precision, and traceability. Initial work in Vietnam has focused on comparing the performance of FSA against the conventional EWA, confirming its potential for environmental analysis (Hồng et al., 2021)⁶. However, a systematic validation of the method against internationally recognized reference materials is necessary to establish its metrological credentials.

Therefore, this study aims to perform a rigorous validation of FSA for determining the ^{23}U , ^{232}Th , $^{\circ}\text{K}$, and

^{13}Cs activities in various environmental matrices. By analyzing a wide range of IAEA reference materials, this work critically assesses the accuracy and precision of FSA. Furthermore, a direct comparative analysis between FSA and EWA is conducted on the same set of samples to quantitatively evaluate the performance of FSA vs. the traditional approach, especially in challenging scenarios, such as low-level activity and complex sample matrices. The objective is to provide robust evidence that FSA is not only a faster alternative but also a more accurate and reliable method suitable for routine use in environmental monitoring and accredited laboratories.

MATERIALS AND METHODS

Gamma-Ray Spectrometry System

All gamma-ray measurements were performed using a p-type coaxial HPGe detector system (Canberra, model GC3520), located at the Nuclear Engineering Laboratory. The detector has a relative efficiency of 35% and an energy resolution (FWHM) of 1.85 keV at the 1332.5 keV gamma-ray peak of ^{60}Co . The detector is housed within a 10-cm-thick lead shield with a graded inner lining of copper and tin to absorb X-rays and thus minimize the contribution from environmental background radiation. Spectral data were acquired and processed using a Lynx digital signal processing unit in conjunction with the GENIE 2000 data acquisition and analysis software. The system's energy calibration and peak shape parameters were regularly monitored using a multi-nuclide standard source to ensure stability throughout the measurement campaign.

Reference Materials and Sample Preparation

A suite of certified reference materials (CRMs), provided by the International Atomic Energy Agency (IAEA), was used as both calibration standards and validation samples. All materials were first dried at 105 °C to a constant weight to remove moisture, thoroughly homogenized to ensure uniformity, and packed into identical 250 mL cylindrical containers to maintain a consistent and reproducible measurement geometry. After packing, the containers were hermetically sealed and stored for at least three weeks to allow for secular equilibrium to be established between ^{226}Ra and its short-lived, gamma-emitting decay products (primarily ^{21}Pb and ^{21}Bi)⁷.

A set of high-purity, single-radionuclide-dominant CRMs from the IAEA was used for calibration, which involved establishing the energy-dependent efficiency

curve for EWA and generating the basis spectra for FSA. Specifically, IAEA-RGU-1, a uranium ore standard with a certified ^{226}Ra activity of 4940 Bq/kg, was used for the ^{23}U decay series. IAEA-RGTh-1, a thorium ore standard with a certified ^{232}Th activity of 3250 Bq/kg, was used for the ^{232}Th decay series. The potassium sulfate standard IAEA-RGK-1, with a high activity of approximately 13,500 Bq/kg, was employed for ^{40}K . Following the calibration, a separate and diverse set of multi-radionuclide reference and intercomparison samples was analyzed as unknowns to rigorously validate and compare the performance of the analytical methods. This validation set was chosen to represent a wide range of environmental matrices and activity concentrations, including IAEA-312 (soil), IAEA-375 (soil with elevated ^{13}Cs), IAEA-434 (a challenging phosphogypsum matrix), IAEA-444 (Chinese soil), IAEA-447 (moss-soil), SL-2 (lake sediment), and TEL-2011-03 (soil). Table 1 presents the official certified activity concentrations for all samples in this validation set, serving as the benchmark for accuracy assessment.

Energy Window Analysis (EWA)

For the conventional EWA method, the activity concentration A (in Bq/kg) for each radionuclide was determined based on the net count rate in its characteristic full-energy peak, according to the following equation⁸:

$$A = \frac{C}{\epsilon_p \cdot I_\gamma \cdot m \cdot t}$$

where C is the net peak area (total counts minus the background continuum) of the selected gamma-ray line, ϵ_p represents the full-energy peak detection efficiency at that specific energy, I_γ symbolizes the gamma-ray emission probability (yield) for the transition⁹, m stands for the dry mass of the sample (in kg), and t is the live counting time (in s).

The full-energy peak efficiency calibration curve (ϵ p vs. energy) was established by measuring the IAEA calibration standards (RGU-1, RGTh-1, and RGK-1) in the same geometry as the samples. The characteristic gamma-ray lines used for the quantification were: 609.3 keV (from ^{21}Bi) for the ^{23}U series, 583.2 keV (from ^{208}Tl) and 911.2 keV (from ^{22}Ac) for the ^{232}Th series, 1460.8 keV for ^{40}K , and 661.7 keV for ^{13}Cs ⁷.

Several corrections were applied using the GENIE 2000 software to ensure the accuracy of the EWA results. First, a density correction was performed to account for the differences in self-absorption between the calibration standards and the validation samples. This was achieved using the density correction feature within GENIE 2000, which models the sample matrix.

Second, corrections for coincidence-summing effects were applied, particularly for nuclides like ^{226}Ra and ^{22}Ac , which have complex decay schemes. The software's algorithm was used to calculate and apply these correction factors. Finally, the net peak area was determined using a linear background subtraction model, which was consistently applied across all spectra to ensure reproducibility.

Full Spectrum Analysis (FSA)

The fundamental principle of the FSA method is to model the measured gamma-ray spectrum of a sample, $Y(i)$, as a linear superposition of the basis spectra of its constituent radionuclides and a background spectrum^{1,10}. This relationship is expressed mathematically as:

$$Y(i) = \sum [C_j \cdot X_j(i)]_{j=1}^n + b \cdot B(i)$$

where i represents the channel number, $Y(i)$ is the count rate (counts per second) in channel i of the sample spectrum, $X_j(i)$ symbolizes the normalized basis spectrum for the j -th radionuclide (in units of counts per second per Bq/kg), C_j indicates the unknown activity concentration (in Bq/kg) of the j -th radionuclide, which is the parameter to be determined, $B(i)$ stands for the experimentally measured spectrum of the empty container, representing the instrumental and environmental background, and b is a dimensionless background scaling factor that accounts for differences in radiation scattering between the sample matrix and the empty container.

The basis spectra, $X_j(i)$, were constructed by measuring the calibration standards (RGU-1, RGTh-1, RGK-1, and IAEA-375 for ^{13}Cs) under the same geometry and normalizing the resulting net spectra by their certified activity and measurement time. The unknown activity concentrations, C_j , and the background factor, b , were subsequently determined for each validation sample, using a weighted least-squares fitting algorithm. This algorithm minimizes the chi-squared (χ^2) statistic between the measured spectrum and the fitted model spectrum, revealing the optimal set of parameters¹¹.

Method Validation and Data Analysis

The accuracy of both methods was assessed by comparing the determined activity concentrations with their corresponding certified values. The agreement was quantified using the relative deviation (RD), calculated as:

$$RD(\%) = \left[\frac{C_{\text{measured}} - C_{\text{certified}}}{C_{\text{certified}}} \right] \times 100$$

A relative deviation within $\pm 15\%$ represents good agreement, in line with common practice for interlaboratory comparisons in environmental radioactivity measurements¹².

RESULTS AND DISCUSSION

Comparative Analysis of FSA and EWA Performance

This study primarily aimed to validate the FSA method through a direct comparison of its performance against both certified reference values and the results from the traditional EWA method. Table 1 presents the results obtained for a suite of IAEA reference materials, selected to cover a wide range of matrices and activity levels. An analysis of these results reveals a nuanced performance, where both methods demonstrate high accuracy, often with relative deviations (RD) well within the acceptable $\pm 15\%$ range, but with distinct performance characteristics under different analytical conditions.

Overall, EWA, when corrected, is a highly robust and accurate technique. Among the 14 EWA measurements, 12 (86%) fell within the $\pm 15\%$ RD threshold, with many showing excellent agreement. For instance, the analysis of ^{23}U in IAEA-312 (+1.12%), $^{\circ}\text{K}$ in IAEA-375 (+2.00%), and ^{13}Cs in IAEA-447 (-0.12%) yielded deviations below 6%, highlighting EWA's capability for high precision under favorable conditions. Even in the case of the challenging phosphogypsum matrix (IAEA-434), the corrected EWA result for ^{23}U showed a remarkably small deviation of -3.19% , a notable improvement that underscores the effectiveness of proper matrix corrections.

FSA also performed well, with 10 out of 14 results (71%) falling within the $\pm 15\%$ RD threshold. This method demonstrated particular strengths in certain scenarios. For example, in the analysis of ^{232}Th and $^{\circ}\text{K}$ in the multi-nuclide sample IAEA-447, FSA exhibited excellent accuracy (+7.02% and +0.61%, respectively), outperforming EWA in these specific instances. Thus, FSA's ability to handle spectral interferences and utilize information from multiple peaks can be advantageous for complex spectra.

However, the data reveal an interesting behavior regarding the performance of the methods with low-activity nuclides. Both methods performed comparably well for the measurement of ^{13}Cs in sample SL-2, which exhibited minimal certified activity (1.2 ± 0.3 Bq/kg): EWA showed a deviation of -5.00% and FSA indicated -3.33% . This result implies that with modern analytical software, EWA can still be effective for low-count statistics, although FSA remains slightly more accurate. The most significant discrepancy for FSA was observed in the analysis of $^{\circ}\text{K}$ in SL-2 (-17.61%), where EWA (+3.79%) was clearly superior. This observation suggests potential issues in the FSA model's basis spectra or fitting algorithm for

certain combinations of low-activity nuclides and specific matrices.

In conclusion, although both methods are highly capable, the corrected EWA demonstrates a slightly more consistent overall accuracy across this diverse dataset. Nonetheless, FSA serves as a powerful complementary tool, excelling in specific cases with complex interferences but also showing potential for large deviations in others. Hence, the choice of method is application-dependent, but a corrected EWA remains a formidable benchmark for routine environmental analysis.

Statistical Evaluation of Method Accuracy and Precision Using the z-Score

z-Scores were calculated for each measurement to provide a more rigorous statistical comparison of the performance of both methods (Table 1). The z-score evaluates the agreement between the measured and certified values while considering the combined uncertainties of both. A result is considered statistically acceptable if its absolute value, $|z\text{-score}|$, is less than 2. The analysis provides a clear quantitative overview of method performance. For the corrected EWA method, 12 out of 14 measurements (86%) yielded acceptable z-scores, demonstrating its high and consistent statistical accuracy across the diverse set of samples. By contrast, the FSA method yielded 7 out of 14 measurements (50%) with a $|z\text{-score}|$ greater than 2, suggesting that FSA is more sensitive to certain matrix compositions.

Nonetheless, the z-score analysis highlights the superior precision of FSA in low-activity scenarios. Both methods produced excellent z-scores ($z_{\text{EWA}} = -0.19$; $z_{\text{FSA}} = -0.13$) for the measurement of ^{13}Cs in the low-activity SL-2 sample. However, the measurement uncertainty (SD) for the FSA result (0.02 Bq/kg) was six times smaller than that for the EWA result (0.12 Bq/kg). This confirms that FSA delivers a smaller combined uncertainty and higher precision for this low-count scenario, reinforcing its advantage in improving counting statistics.

The cases where FSA showed significant statistical deviation, such as for ^{13}Cs in IAEA-444 ($z = -4.32$) and IAEA-447 ($z = 4.38$), are informative. These samples (Chinese soil and moss-soil) may possess mineralogical or organic compositions that are not well-represented by the basis spectra used for the FSA model. This suggests that although FSA's background scaling factor partially compensates for bulk density, its accuracy depends on the similarity between the calibration standards and the unknown samples, a key consideration for its practical application.

Table 1: Comparison between activity concentrations (Bq/kg) determined by full spectrum analysis (FSA) and IAEA certified values.

Sample	Isotope	IAEA Certified Value (Mean ± SD)	EWA Result (Mean ± SD)	EWA Rel. Dev. (%)	EWA z-score	FSA Result (Mean ± SD)	FSA Rel. Dev. (%)	FSA z-score
IAEA-312	²³ U	269.0 ± 18.5	272.0 ± 1.5	+1.12	0.16	274.56 ± 1.06	+2.07	0.30
IAEA-375	²³ U	24.4 ± 2.0	26.0 ± 0.6	+6.56	0.77	22.03 ± 0.17	-9.71	-1.18
	²³² Th	20.50 ± 1.35	19.3 ± 0.8	-5.85	-0.76	21.03 ± 0.15	+2.59	0.39
	⁴⁰ K	424 ± 9	432.5 ± 5.5	+2.00	0.81	451.50 ± 2.62	+6.49	2.92
IAEA-434	¹³ Cs	28.9 ± 0.9	30.44 ± 0.4	+5.33	1.57	30.91 ± 0.05	+6.96	2.23
	²³ U	780 ± 11	755.1 ± 3.1	-3.19	-2.17	750.07 ± 3.26	-3.84	-2.62
	¹³ Cs	68.5 ± 1.4	65.8 ± 0.6	-3.94	-1.78	62.05 ± 0.53	-9.42	-4.32
	²³ U	22.2 ± 0.8	21.6 ± 0.5	-2.70	-0.64	25.45 ± 0.20	+14.64	3.95
IAEA-447	²³² Th	37.3 ± 2.0	35.2 ± 0.8	-5.63	-0.97	39.92 ± 0.24	+7.02	1.30
	⁴⁰ K	550 ± 18	582.1 ± 7.2	+5.84	1.66	553.38 ± 4.04	+0.61	0.18
	¹³ Cs	32.8 ± 0.8	31.76 ± 0.2 ¹	-3.17	-1.26	36.39 ± 0.15*	+10.95	4.38
SL-2	⁴⁰ K	240 ± 24	249.1 ± 4.3	+3.79	0.37	197.74 ± 1.55	-17.61	-1.76
	¹³ Cs	1.2 ± 0.3	1.14 ± 0.12	-5.00	-0.19	1.16 ± 0.02	-3.33	-0.13
TEL-2011-03	⁴⁰ K	529.2 ± 23.9	515.6 ± 6.5	-2.57	-0.55	537.33 ± 3.10	+1.54	0.34

Quality of the Spectral Fit

The quantitative accuracy of FSA is underpinned by the excellent qualitative agreement between the measured experimental spectra and the spectra reconstructed by the FSA model. Figure 1 presents a representative example of this spectral fit for the IAEA-312 soil sample, illustrating the model’s performance in a typical environmental matrix containing multiple natural radionuclides.

As illustrated in Figure 1, the fitted spectrum (red line) almost perfectly overlays the experimental data points (blue line) across the entire energy range, from the complex low-energy region to the highest-energy gamma rays. This excellent match demonstrates the model’s high fidelity. The model accurately reproduces not only the prominent photopeaks, such as the 1460.8 keV peak of ⁴⁰K, but also the numerous smaller

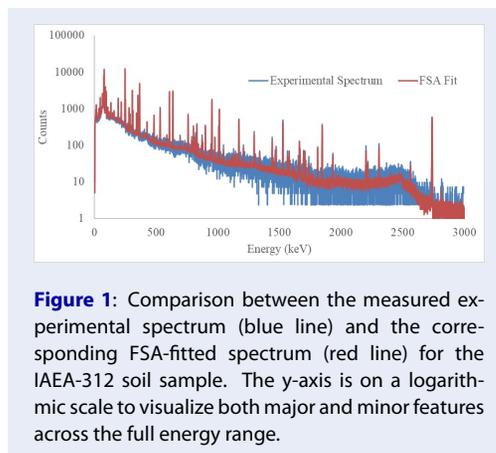


Figure 1: Comparison between the measured experimental spectrum (blue line) and the corresponding FSA-fitted spectrum (red line) for the IAEA-312 soil sample. The y-axis is on a logarithmic scale to visualize both major and minor features across the full energy range.

peaks associated with the ^{23}U and ^{232}Th decay series, which are characteristic of this soil sample.

A key strength of the FSA method, highlighted by this fit, is its ability to accurately model the entire Compton continuum as an integral part of the fitting process. The smooth shape of the fitted spectrum correctly captures the broad, underlying structure of the experimental data, eliminating the need for manual (and often subjective) background subtraction procedures, which are a major source of uncertainty in the traditional EWA method. This integrated approach ensures that the contributions of all radionuclides are accounted for simultaneously, resolving spectral interferences from overlapping peaks (e.g., the 609.3 keV peak of ^{21}Bi and the 583.2 keV peak of ^{208}Tl). The quality of the fit, even in regions with low counts and complex features, validates the robustness of the FSA model and demonstrates the reliability of the activity concentrations of IAEA-312 and similar samples.

Interpretation of the Background Scaling Factor

This study revealed a strong correlation between the fitted background scaling factor, b , and the bulk density of the measured samples (Table 2 and Figure 2). The scatter plot reveals a clear and strong positive linear trend, described by the linear regression equation $y = 0.4945x + 0.7878$, where y symbolizes the background scaling factor and x represents the density. The high coefficient of determination ($R^2 = 0.8932$) indicates that this linear model explains nearly 90% of the variation in the scaling factor, confirming a robust physical relationship.

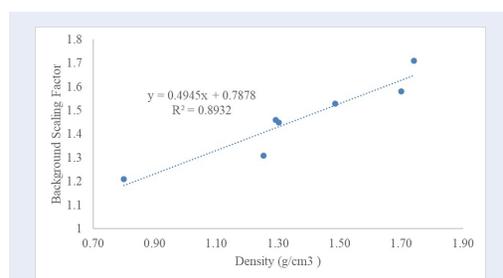


Figure 2: Correlation between the fitted background scaling factor (b) and the bulk density of the analyzed samples.

The positive slope (0.4945) confirms that the background-scaling factor increases with density. This slope represents the average rise in the scaling factor for each unit (g/cm^3) increase in sample density, reflecting the greater probability of Compton

scattering in denser materials. The y -intercept (0.7878) represents the theoretical scaling factor for a sample with zero density ($x = 0$). Although a zero-density sample is physically impossible, this value being close to 1.0 is mathematically consistent with the model. This value suggests that, even without a scattering medium, the model accounts for a baseline background contribution that is slightly different from the measured empty-beaker spectrum, possibly due to minor geometric or model-fitting effects.

This strong, quantifiable relationship proves that the b parameter is not only a mathematical free parameter but also serves as a quantitative measure of a real physical effect: the modification of the background radiation field by the sample matrix. When a sample is introduced, its matrix acts as an additional scattering medium, redirecting ambient gamma rays into the detector. The b factor, consistently greater than 1 and increasing linearly with density, demonstrates this enhancement.

The ability of the FSA model to automatically quantify this effect during the fitting process is a key advantage. This ability provides an inherent, data-driven correction for density-related matrix effects on the background component, enhancing the method's robustness and accuracy across samples of varying densities. This self-correcting feature distinguishes FSA from the traditional EWA method, which relies on a static background subtraction and can be more susceptible to errors when the sample matrix alters the background shape and intensity.

CONCLUSIONS

We performed a systematic and rigorous validation of full-spectrum analysis (FSA) against the traditional, corrected energy window analysis (EWA). The validation confirms that both are powerful and reliable methods for environmental radioactivity analysis, each with distinct strengths. EWA, when fully corrected for matrix and spectral effects, demonstrates superior statistical accuracy, with 86% of its results showing statistical agreement with certified values based on z -score analysis. This finding confirms its status as a highly robust and reliable benchmark for routine analysis across various sample types. By contrast, FSA exhibits advantages in terms of precision, delivering substantially smaller measurement uncertainties, particularly for low-activity nuclides. This makes FSA an invaluable tool for applications requiring low detection limits or shorter counting times. However, this study also highlights

Table 2: Bulk density and corresponding background scaling factor for the IAEA reference samples.

Sample ID	Density (g/cm ³)	Background Scaling Factor (b)
IAEA-434	0.80	1.21
IAEA-447	1.25	1.31
IAEA-312	1.29	1.46
IAEA-444	1.30	1.45
TEL-2011-03	1.49	1.53
IAEA-375	1.70	1.58
SL-2	1.74	1.71

that FSA's accuracy can be more sensitive to complex matrix compositions that deviate from the calibration standards. Therefore, a corrected EWA remains the recommended primary method for accredited laboratories seeking consistent accuracy. Nevertheless, FSA serves as an excellent complementary technique, indispensable for high-precision measurements of low-level samples and for cross-checking results. The choice of method is application-dependent, but a combined approach leveraging the strengths of both is expected to yield the most comprehensive and reliable environmental monitoring results.

LIST OF ABBREVIATIONS

CRM: Certified Reference Material
 EWA: Energy Window Analysis
 FSA: Full Spectrum Analysis
 FWHM: Full Width at Half Maximum
 HPGe: High-Purity Germanium
 IAEA: International Atomic Energy Agency
 NORM: Naturally Occurring Radioactive Material
 RD: Relative Deviation

COMPETING INTERESTS

The authors declare that they have no competing interests.

AUTHORS' CONTRIBUTIONS

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 Formal Analysis and Writing – Original Draft: Huynh Thi Yen Hong.
 Writing – Review & Editing: All authors contributed to the review and editing of the final manuscript.
 Supervision: Huynh Thi Yen Hong.

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