

Development of the k_0 -standardized cyclic neutron activation analysis using short-lived radionuclides at the Dalat research reactor

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Abstract

An optimized k_0 -standardized neutron activation analysis method incorporating cyclic irradiations (k_0 -CNAA) for short-lived radionuclides (SLRNs) has been developed at the Dalat research reactor. This paper highlights precise reactor parameter characterization using a cyclic irradiation system, simple sample preparation, and advanced calibration of HPGe detector-based gamma-ray spectrometry for SLRNs' rapid multielement determination. By targeting SLRNs such as ^{77m}Se , ^{110}Ag , ^{20}F , ^{179m}Hf , ^{52}V , and ^{46m}Sc , with half-lives from seconds to minutes, the method enables quantification of elements essential for biological and environmental research. The in-house developed “ k_0 -Dalat” software, featuring high automation, supports complete analysis. Method accuracy was validated using certified reference materials (SMELS-I, NIST-SRM-1566b, NIST-SRM-2711a), achieving deviations under 8% from certified values. Detection limits ranged from 0.1 to 1.9 mg/kg for target elements in biological samples, confirming the method's high sensitivity and suitability for similar matrices.

Keywords: neutron activation analysis, cyclic NAA, short-lived radionuclides, k_0 -standardization, Dalat research reactor

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1. Introduction

Neutron activation analysis (NAA) is a robust analytical method valued for its high sensitivity and nondestructive or minimally invasive nature. It enables precise, simultaneous multielement quantification across various sample types, including environmental, geological, and biological matrices. Key advantages of NAA include minimal sample preparation, excellent trace element precision, and the ability to detect multiple elements at once.

Radionuclides produced in NAA are generally grouped by half-life: short-lived (seconds to minutes), medium-lived (minutes to hours), and long-lived (days to years). Traditional NAA

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typically relies on medium- or long-lived radionuclides like ^{24}Na , ^{42}K , ^{60}Co , or ^{152}Eu , often resulting in turnaround times of days to weeks. In contrast, this study focuses on NAA using SLRNs such as $^{77\text{m}}\text{Se}$, ^{110}Ag , ^{20}F , $^{179\text{m}}\text{Hf}$, ^{52}V , and $^{46\text{m}}\text{Sc}$, whose short half-lives allow rapid results — often within an hour or a single day [1–5].

However, analyzing SLRNs poses unique challenges due to their rapid decay. For example, ^{20}F has a half-life of just 11 s, requiring precise timing for irradiation, transfer, and counting. To meet these demands, specialized setups such as high-speed pneumatic transfer systems (with transfer times around one second) and automated cyclic neutron activation analysis (CNAA) are essential to accumulate enough radioactivity through repeated cycles [6–9].

The Dalat research reactor (DRR), originally a 250-kW TRIGA Mark-II from the 1960s, was rebuilt in 1984 as a 500-kW pool-type reactor using 36% HEU fuel and converted to 19.75% LEU in 2011 [10]. While DRR has supported diverse NAA applications for decades, its potential for SLRN analysis was largely unexploited. With growing need for rapid, multielement analysis in environmental monitoring and public health, the Dalat research reactor has developed an optimized k_0 -based CNAA (k_0 -CNAA) approach. This combines cyclic activation with the k_0 -standardization method [11–15], which uses nuclear constants and reactor flux parameters instead of comparator standards to calculate elemental concentrations.

Significant upgrades support these advanced methods, including a fast pneumatic transfer system (PTS) with a transit time of about 0.65 s and an advanced DSPEC Pro spectrometer with zero dead time (ZDT) correction for high-activity samples [16, 17]. The k_0 -CNAA protocols at DRR have been successfully applied to SLRNs like $^{77\text{m}}\text{Se}$, ^{110}Ag , ^{20}F , $^{179\text{m}}\text{Hf}$, ^{52}V , and $^{46\text{m}}\text{Sc}$, and validated using certified reference materials from environmental, biological, and geological origins [18, 19].

This study's core innovation is the successful combination and optimization of k_0 -CNAA with SLRNs at DRR. This, coupled with advanced instrumentation, significantly advances the DRR's capabilities for rapid trace element analysis and precise multielement quantification, overcoming previous challenges in SLRN analysis.

2. Theory and methodology

2.1. Theoretical basis of k_0 standardization

The k_0 -standardization method in NAA is based on the k_0 factor, defined as the ratio of specific count rates for the analyte and a comparator element (typically gold) irradiated under identical conditions [11–13]:

$$k_{0,\text{Au}}(a) = \frac{M_{\text{Au}} \cdot \theta_a \cdot \sigma_{0,a} \cdot \gamma_a}{M_a \cdot \theta_{\text{Au}} \cdot \sigma_{0,\text{Au}} \cdot \gamma_{\text{Au}}}, \quad (1)$$

where M represents the atomic mass, θ is the isotopic abundance, σ_0 is the thermal neutron cross section, and γ is the gamma-ray abundance.

For an actual analysis, the mass fraction of an element in a sample is determined using the equation

$$\rho_a = \frac{N_{p,a} \cdot t_{m,\text{Au}} \cdot W_{\text{Au}}}{N_{p,\text{Au}} \cdot t_{m,a} \cdot W_a} \cdot \frac{1}{k_{0,\text{Au}}(a)} \cdot \frac{G_{\text{th},\text{Au}} \cdot f + G_{e,\text{Au}} \cdot Q_{0,\text{Au}}(\alpha)}{G_{\text{th},a} \cdot f + G_{e,a} \cdot Q_{0,a}(\alpha)} \cdot \frac{\varepsilon_{p,\text{Au}}}{\varepsilon_{p,a}}, \quad (2)$$

where N_p is the measured net peak area, t_m is the measuring time, W_a and W_{Au} are the weight of the sample and Au comparator, G_{th} and G_e are the thermal and epithermal neutron

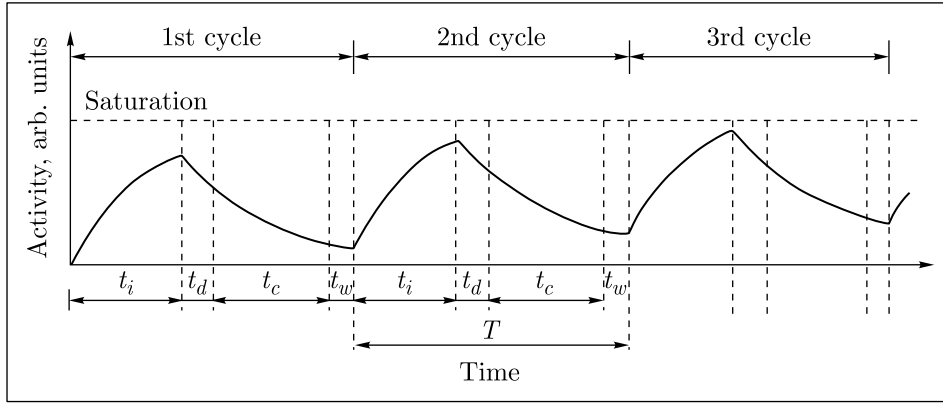


Figure 1. Time parameters of CNA and the variation of the activity of nuclides with time and number of cycles.

self-shielding correction factors, f is the thermal to epithermal neutron flux ratio, $Q_0(\alpha)$ is the resonance integral to thermal cross-section ratio corrected for the epithermal neutron flux distribution, and ε_p is the full-energy peak detection efficiency.

The principle of cyclic NAA has been described by Spyrou [7]. In this method, the sample is irradiated for a short period of time, and after a delay period from the end of irradiation, the radiation emitted is counted for a short period of time, then the sample is irradiated again and the entire process is repeated for a number of cycles (Figure 1). The detected radiations at each counting period are summed, and finally, a total cumulative detector response is obtained.

The elemental concentration in the sample is calculated by the k_0 -CNA equation as follows [15]:

$$\rho = \frac{N_{pc}/t_m}{S \cdot D \cdot C \cdot W \cdot k_0 F_c \cdot [G_{th} \varphi_{th} \sigma_0 + G_e \varphi_e I_0(\alpha)] \cdot \varepsilon_p},$$

where the cumulated net peak area is $N_{pc} = k \varphi_n \sum_{n'=1}^n \frac{1 - e^{-n' \lambda T}}{1 - e^{-\lambda T}} \cdot \frac{\varphi_{n+1-n'}}{\varphi_n}$ and the dead time

$$\text{and pile-up correction} - F_c = \left[\frac{n}{(1 - e^{-\lambda T})} - \frac{e^{-\lambda T}(1 - e^{-n \lambda T})}{(1 - e^{-\lambda T})^2} \right].$$

2.2. Specific considerations for short-lived radionuclides

For short-lived radionuclides, additional corrections must be applied to account for decay during irradiation, transfer, and counting periods due to their short half-lives, while the high activity generated by cyclic irradiation requires separate dead time and pile-up corrections [17, 26]:

$$C_{\text{decay}} = (1 - e^{-\lambda t_i}) \cdot e^{-\lambda t_d} \cdot \frac{(1 - e^{-\lambda t_m})}{\lambda t_m}, \quad (3)$$

where λ is the decay constant, t_d is the decay time (from the end of irradiation to the start of counting), t_m is the measurement time, and t_i is the irradiation time.

Additionally, for cyclic irradiations with n cycles, a saturation factor must be applied:

$$S_n = \frac{1 - e^{-\lambda n(t_i + t_w)}}{1 - e^{-\lambda(t_i + t_w)}}, \quad (4)$$

where t_w is the waiting time between successive irradiations.

3. Experimental setup

The automated cyclic activation system managed by a programmable logic controller (PLC) ensures the precise timing for irradiation, transfer, and counting, which is critical for SLRNs and enhances reproducibility and throughput. The setup features the fast PTS with its extremely short transit times of (0.63 ± 0.02) s for Channel 13-2 and (0.25 ± 0.02) s for the thermal column (TC). This is a technical specification that enables the analysis of SLRNs. The experimental setup and procedures employed for k_0 -CNA A using SLRNs at DRR is detailed as follows.

3.1. Irradiation facility

Samples for analysis were irradiated at specific channels of DRR providing distinct neutron flux characteristics. The thermal neutron fluxes observed were approximately $4.09 \times 10^{12} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$ in Channel 13-2 and about $1.10 \times 10^{11} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$ in TC. Throughout the irradiation process, important flux parameters, including α , f , and T_n , were consistently monitored to ensure precise control over the irradiation environment. A diagram of the fast PTS for cyclic irradiations in Channel 13-2/TC is displayed in Figure 2, and the detailed parameters are presented in Table 1 in the next section.

3.2. Cyclic activation system

A central component of this study is the fast PTS, which is essential for handling short-lived radionuclides due to their rapid decay. This system enabled rapid sample transfer with

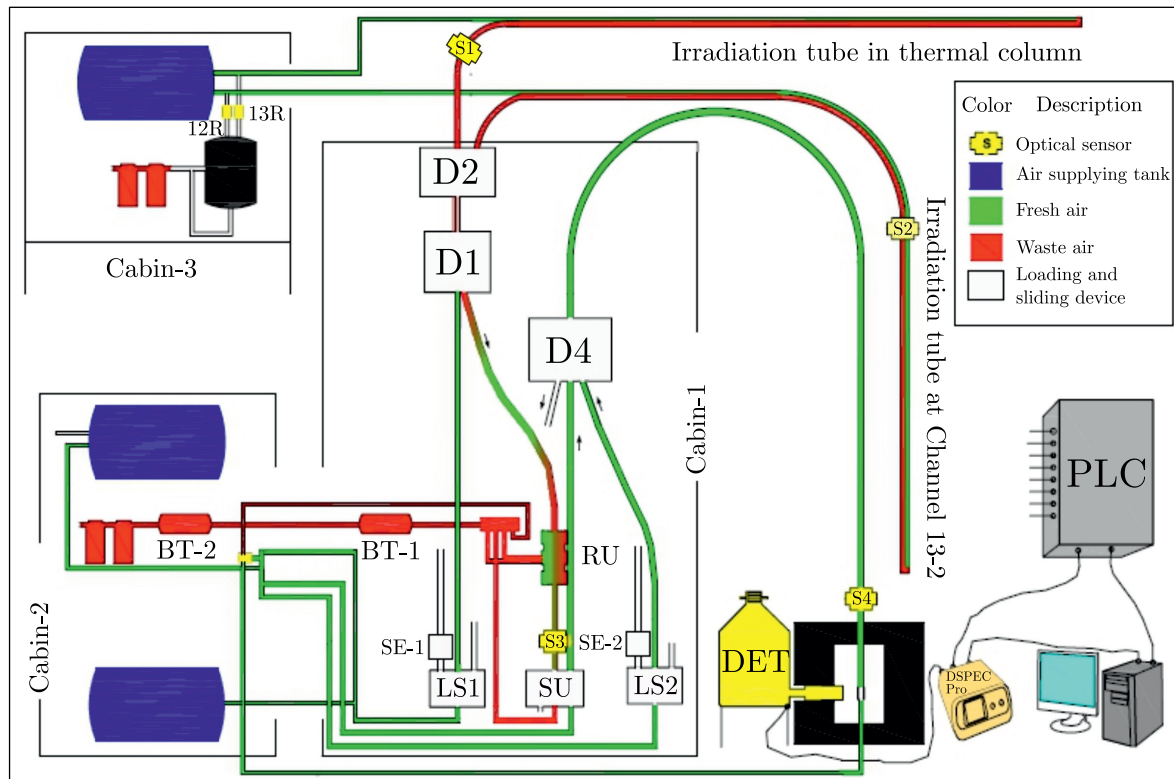


Figure 2. A schematic diagram of the automated pneumatic transfer system for CNA A. The system transports samples between three cabins: two irradiation tubes at Channel 13-2 or in TC, and a detector (DET), all controlled by PLC.

measured transit times of less than a second for Channel 13-2 and even faster speeds for TC. The CNAA process is automated, involving a precisely controlled sequence of irradiation (t_i), decay (t_d), counting (t_c), and waiting (t_w) periods, with the total period of a cycle defined as $T = t_i + t_d + t_c + t_w$. The system's design incorporates components for air supply, waste air management, loading/unloading devices, optical sensors (S1, S2, S3, S4), and various cabins (Cabin-1, Cabin-2, Cabin-3) for operational control and sample handling. A PLC manages the automated cycles, interfacing with the detection system (DET).

3.3. Gamma-ray spectrometry

The detection system utilized for gamma-ray spectrometry in the CNAA setup at DRR comprises an ORTEC GMX-4076 HPGe detector with 40% efficiency housed within an open-sided lead (Pb) shield. This detector is coupled to a DSPEC Pro spectrometer, which is equipped with ZDT correction capabilities. This feature is particularly crucial for accurately measuring high-activity samples by recovering a significant percentage of counts lost due to dead time and pile-up effects. The spectrometer and associated computer system allow for precise control over counting parameters and data acquisition. An ORTEC GMX-4076 HPGe detector-based gamma-ray spectrometry system for CNAA at the Dalat research reactor is presented in Figure 3.

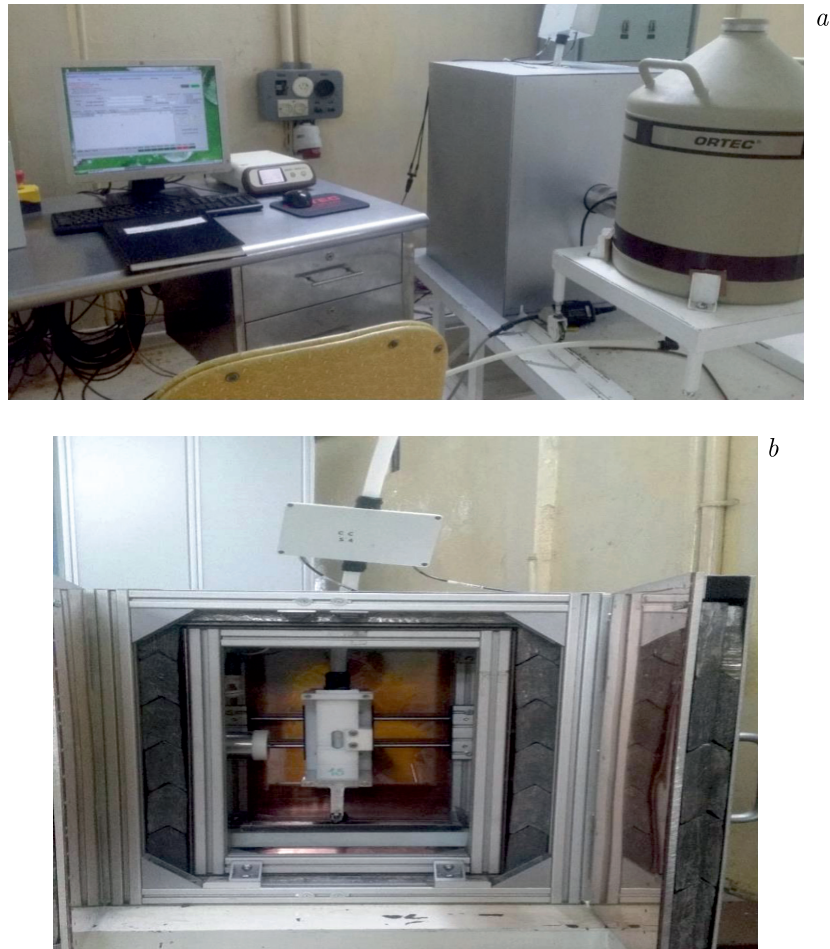


Figure 3. The gamma-ray spectrometry system used for CNAA at the Dalat research reactor: an ORTEC GMX-4076 HPGe detector setup (a); an open-sided Pb shield (a closer view) with sample holder inside (b).

Figure 3, *a* views from left to right, includes a personal computer for data acquisition, a digital signal processor (DSPEC Pro), and a Pb shield housing the detector. A dewar containing liquid nitrogen is attached to the cryostat to cool the detector, which is essential for its operation. Figure 3, *b* displays the interior of an open-sided Pb shield designed for the CNAA system. The image shows a sealed sample measurement shelf, designed to hold the activated samples for gamma-ray measurement after each irradiation–transfer–counting cycle.

3.4. The k_0 -CNAA software

The elemental concentration in samples is calculated using the k_0 -CNAA method, which relies on nuclear constants and reactor flux parameters, thus eliminating the need for comparator standards. The “ k_0 -Dalat” software [24, 25] has been developed and improved for the processing of k_0 -standardized data from instrumental, epithermal and cyclic NAA. This software incorporates dead time and pile-up corrections through novel techniques like ZDT and loss-free counting (LFC), which have been shown to recover up to 95% of counts lost at $\leq 50\%$ dead time, thereby significantly enhancing data reliability. The software provides a user interface for various functions, including viewing results, reformatting spectra, and managing irradiation parameters.

4. Results and discussion

4.1. Characterization of neutron parameters [22]

A characterization of the neutron parameters at DRR irradiation channels was performed to establish the foundational data for k_0 -CNAA. These characterized parameters are important inputs for the k_0 -CNAA calculations, allowing for accurate quantification of elements without external standards. The measured neutron parameters for both the Channel 13-2 and TC are presented in Table 1.

Table 1. Neutron parameters needed for k_0 -CNAA at DRR irradiation facilities.

Neutron parameters	Channel 13-2	Thermal column
Thermal flux ($\times 10^{12} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$)	4.09 ± 0.09	0.11 ± 0.01
Epithermal flux ($\times 10^9 \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$)	3.78 ± 0.20	0.56 ± 0.01
Fast flux ($\times 10^{11} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$)	6.60 ± 0.22	0.84 ± 0.06
α	-0.038 ± 0.006	0.092 ± 0.035
f	10.9 ± 0.6	203 ± 25
$T_n(K)$	312 ± 31	298 ± 28

4.2. Validation with certified reference materials

The developed k_0 -CNAA methodology was validated using three certified reference materials with diverse matrices: SMELS-I (Synthetic Multi-Element Standard) [20, 23] for method linearity and assessment, NIST-SRM-1566b (Oyster Tissue) representing biological matrices, and NIST-SRM-2711a (Montana Soil) representing environmental matrices.

Each reference material was analyzed in triplicate following the optimized protocol. Table 2 presents the comparison between measured and certified values for elements determined via k_0 -CNAA (5 cycles) using short-lived radionuclides.

Table 2. Comparison of measured and certified values for elements determined via k_0 -CNAA (5 cycles) using short-lived radionuclides in certified reference materials (uncertainties at $k = 1$).

Element	Nuclide	Half-life	SMELLS-I		NIST-SRM-1566b		NIST-SRM-2711a	
			Measured (mg/kg)	Certified (mg/kg)	Measured (mg/kg)	Certified (mg/kg)	Measured (mg/kg)	Certified (mg/kg)
Se	^{77m}Se	17.45 s	9.8 ± 0.6	10.0 ± 0.2	2.1 ± 0.2	2.06 ± 0.15	1.9 ± 0.2	2.0 ± 0.3
Ag	^{110}Ag	24.6 s	1.05 ± 0.09	1.00 ± 0.03	0.67 ± 0.07	0.666 ± 0.009	4.4 ± 0.4	4.63 ± 0.39
F	^{20}F	11.02 s	—	—	5.8 ± 0.5	5.5*	610 ± 50	590*
Hf	^{179m}Hf	18.68 s	1.02 ± 0.08	1.00 ± 0.02	0.009 ± 0.001	0.01*	8.6 ± 0.7	9.2 ± 0.9
V	^{52}V	3.93 min	9.7 ± 0.5	10.0 ± 0.2	0.58 ± 0.04	0.577 ± 0.023	79 ± 5	80.7 ± 5.7
Sc	^{46m}Sc	18.75 s	1.03 ± 0.07	1.00 ± 0.01	0.015 ± 0.002	0.01*	8.9 ± 0.6	8.5 ± 0.3

*Information value (not certified).

In Table 2, based on the results for the analysis of NIST-SRM-1566b for ^{77m}Se , the analyzed concentration was (2.1 ± 0.2) mg/kg, which compares well with the certified value of (2.06 ± 0.15) mg/kg, yielding an analyzed/certified ratio of 0.98 ± 0.13 and a u -score of -0.15 . Similarly, for ^{110}Ag , the analyzed value was (0.67 ± 0.07) mg/kg against a certified value of (0.666 ± 0.009) mg/kg, resulting in a ratio of 0.95 ± 0.12 and a u -score of -0.39 . Radionuclides ^{20}F , ^{179m}Hf , ^{52}V , and ^{46m}Sc were also analyzed, with most showing good agreement with certified values where available. Notably, the method was able to determine ^{20}F ($T_{1/2} = 11.02$ s) at (5.8 ± 0.5) mg/kg, ^{179m}Hf ($T_{1/2} = 18.68$ s) at (0.009 ± 0.001) mg/kg, ^{52}V ($T_{1/2} = 3.93$ min) at (0.58 ± 0.04) mg/kg, and ^{46m}Sc ($T_{1/2} = 18.75$ s) at (0.015 ± 0.002) mg/kg, despite no certified values being available for comparison for these specific elements (F, Hf, and Sc) in this SRM.

Based on the results for NIST-SRM-2711a for ^{77m}Se , the analyzed concentration was (1.9 ± 0.2) mg/kg against a certified value of (2.0 ± 0.3) mg/kg. ^{179m}Hf showed an analyzed value of (8.6 ± 0.7) mg/kg (certified (9.2 ± 0.9) mg/kg), yielding a ratio of 0.87 and a u -score of -1.20 . ^{110}Ag , ^{20}F , and ^{52}V also demonstrated good agreement as displayed in Table 2. ^{46m}Sc was analyzed at (8.9 ± 0.6) mg/kg (certified (8.5 ± 0.3) mg/kg), with a ratio of 0.96 and a u -score of -0.14 .

The results demonstrate excellent agreement between measured and certified values, with relative deviations typically less than 8%. The validation confirms the accuracy and reliability of the developed methodology across different matrix types and concentration ranges.

4.3. Detection limits

The method detection limits (MDLs) were calculated based on Currie's criterion [27]:

$$\text{MDL} = \frac{3\sqrt{N_B}}{S \cdot w}, \quad (5)$$

where N_B is the background counts under the peak region, S is the sensitivity (counts per unit mass), and w is the sample mass.

Table 3 presents the detection limits with 5 cycles achieved for the elements of interest in different matrices of biological samples.

The achieved detection limits with 5 cycles are sufficiently low for the determination of these elements in most biological and environmental samples, demonstrating the high sensitivity of the developed method despite the challenges associated with short-lived radionuclides. Compared

Table 3. Method detection limits with 5 cycles for elements determined via SLRNs [21].

Element	Nuclide	Half-life	γ -energy (keV)	Detection limit (mg/kg)
Se	^{77m}Se	17.4 s	161.9	0.3
Ag	^{110}Ag	24.6 s	657.8	0.2
F	^{20}F	11.0 s	1633.6	1.9
Hf	^{179m}Hf	18.7 s	214.3	0.4
V	^{52}V	3.93 min	1434.1	0.2
Sc	^{46m}Sc	18.7 s	142.5	0.1

to conventional single-shot NAA at DRR, the cyclic approach improved detection limits by factors of 3–7 for the target elements: Se (factor of 5), Ag (factor of 4), V (factor of 7), F (factor of 3), Hf (factor of 4), and Sc (factor of 6). These limits are superior to traditional k_0 -NAA for these specific elements in biological and environmental matrices.

4.4. Calibration of detector

An ORTEC GMX-4076 HPGe detector with 40% efficiency was used for the gamma-ray spectrometry system. To calibrate efficiency, a suite of point sources, including single-peak emitters (^{241}Am , ^{109}Cd , ^{57}Co , and ^{137}Cs) and multipeak emitters (^{133}Ba , ^{60}Co , and ^{152}Eu) were measured, at three source-detector distances (50, 100, and 150 mm) to minimize the effects of true-coincidence summing. The explicit true-coincidence summing corrections to the multipeak sources were applied using the “TrueCoinc” code [28]. After these corrections, the “ANGLE – Ver 3” code [29] was used to convert the reference efficiency to the specific counting geometries of the samples. The final analysis was performed using the “k0-Dalat” software [24, 25]. This software employs a semiempirical method to convert point source efficiency to sample efficiency by calculating the ratio of their solid angles, which inherently accounts for geometric effects and gamma-ray self-absorption within the sample matrix.

4.5. Method optimization

Optimization of the k_0 -CNAA method is focused on important parameters to enhance sensitivity and lower detection limits for SLRNs. It was determined that saturated activity for SLRNs could be effectively achieved within 4–9 cyclic activation cycles. The optimal irradiation time (t_i) was found to be in the range of 5–10 s. As an example of the improved detection capabilities, the limit of detection (LOD) for ^{77m}Se in NIST-SRM-1566b was determined to be 0.06 μg . The accumulation of counts over multiple cycles, demonstrating increased signal intensity, is illustrated in the provided spectra for different numbers of cycles (N1 to N7), where counts significantly increase with more cycles [18, 19]. Our detection limits for short-lived radionuclides in biological samples are competitive with or superior to those achieved at other research reactors [6–9]. The effectiveness of our method is entirely due to an optimized cyclic activation approach and a fast pneumatic transfer system, with these results obtained using just 5 cycles.

5. Conclusion

This study significantly enhances the analytical capabilities of the Dalat research reactor for short-lived radionuclides using k_0 -based cyclic neutron activation analysis (k_0 -CNAA).

By integrating the fast PTS with advanced processing techniques, including ZDT and LFC, the facility's performance was markedly improved. Our upgraded "k0-Dalat" software successfully recovered up to 95% of lost counts, ensuring high data reliability. The method was validated with certified reference materials, demonstrating deviations below 8% and achieving LOD of 0.1–1.9 mg/kg for biological samples. This approach offers a 3–7 fold improvement in detection limits for key elements compared to conventional neutron activation analysis. Ultimately, the combination of these advanced systems establishes DRR as a more robust, highly automated, and efficient facility for rapid and precise trace element analysis in both research and routine applications.

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Author contributions

Ho Van Doanh: Data curation, Writing, Original draft preparation; Tran Quang Thien: Visualization, Investigation, Validation; Ho Manh Dung: Conceptualization, Methodology, Software; Nguyen Nhi Dien: Conceptualization, Methodology review; Hoang Sy Minh Tuan: Software, Data curation, Validation.

Conflicts of interest

The authors declare no conflicts of interest.

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