DETERMINATION OF THERMAL NEUTRON FLUX DISTRIBUTION AT ROTARY RACK SERVED FOR ELEMENTAL CONCENTRATION ANALYSIS USING THE K0-INAA METHOD

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Abstract: The accuracy of elements concentration determination using the k0-standardization method directly depends on irradiation and measurement parameters including Non-1/E epithermal neutron flux distribution shape α ($\varphi_{epi} \approx 1/E^{1+\alpha}$), thermal-to-epithermal neutron flux ratio *f*, efficiency ε, peak area…. In the case of the irradiation position at the rotary rack of the Dalat Nuclear Research Reactor (DNRR), the difference of thermal neutron flux between the bottom (3.54×10^{12} $n.cm^{-2}.s^{-1}$) and the top (1.93×10^{12} $n.cm^{-2}.s^{-1}$) of the 15 cm aluminum container is up to 45%. Therefore, it is necessary to accurately determine above-mentioned parameters in the sample irradiation position. The present paper deals with the determination of the distribution of thermal neutron flux along with sample irradiation container by using 0.1% Au–Al wire activation technique. The thermal neutron flux was then used to calculate the concentration of elements in the Standard Reference Material 2711a and SMELS type III using k0-INAA method at different positions in the container. The obtained results with the neutron flux correction were found to be in good agreement with the certified values. In conclusion, the proposed technique can be applied for activation analyses without sandwiching flux monitors between samples during irradiations.

Keywords: *k0-standardization method, Dalat nuclear research reactor, neutron spectrum parameters.*

Introduction

Nuclear analytical techniques have been developed for decades. It has been used to solve environmental problems, legal investigations…. Since March 2012, the DNRR has been continuously operated about 100÷130 hours per month at a nominal power of 500 kW for radioisotopes production, activation analysis and other researches. The k0-standardisation method (k_0-NAA) has been applied and developed at the DNRR over 17 years. Its main applications include the studies in geology, bio-medicine, material, petroleum, archaeology and environment among others. The advantages of k0-NAA in the applications are a capability of the determination of multi-element with high precision and accuracy as well as a minimized sample preparation [1].

At the DNRR, there are three irradiated channels used for NAA (Fig.1): (1) The fast pneumatic transfer system for very short irradiation at the channel 13-2 and thermal column (T_{irr}<45 sec); (2) The pneumatic transfer system for short and medium irradiations at the 7-1 channel (T_{irr} = 45÷1200 sec); (3) The rotary rack with 40 irradiated holes placed inside the graphite reflector for long irradiation (T_{irr}>20 min). The experiments for determination of thermal flux were carried out at the 6th hole of the rotary

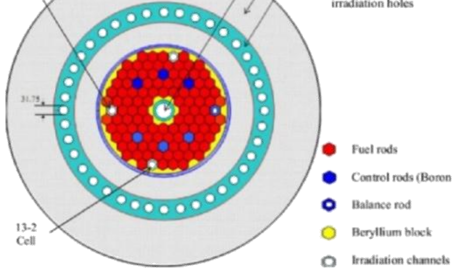

Fig. 1. *Dalat research reactor cross-section*

rack of the DNRR.

The neutron spectrum parameters of the DNRR were reported to be very stable which permit the use of k_0-NAA method [2]. Instrumental neutron activation analysis with research reactors has some special characteristics which makes it more attractive to use for routine analysis. These include multi-element capability, reproducibility of the results and independence of the chemical state of the element [3,4] Thermal, epithermal and fast neutron fluxes determination are useful when characterizing the activation site in instrumental neutron activation analysis. In this perspective NAA using reactor neutrons plays a vital role due to its high sensitivity and detection limits for many elements in a variety of matrices [5], but these could not be achieved without proper knowledge of the neutron flux [6].

It is necessary to measure thermal neutron flux distribution at various points in sample irradiation container and neutron flux monitoring is, therefore, required to be carried out regularly for any reactor for analytical quality control. This is to guarantee continues application of neutron activation analysis since the main sources of measurement uncertainty in an NAA are parameters such as flux variation within a sample and irradiation geometry in the container. When the sample is irradiated with neutrons, the activation rates depend on the geometry effect due to the irradiation position within the container, the variation and the differences within the irradiation site [4].

In the determination of elemental concentration in unknown samples using k0-method, samples and flux monitors were simultaneously irradiated together. The flux monitors were usually positioned at the top, middle and bottom of the containers. Therefore, we proposed the technique that can be applied for k0-method with suitable accuracy without sandwiching flux monitors between samples during irradiations. An experimental determination of the thermal neutron flux in the inner sample irradiation container at the rotary rack of the DNRR using foil activation technique was undertaken in this work.

Theory of method

In the absolute method, the thermal neutron flux is given as [7]:

$$\Phi_{th} = \frac{A_{sp}\left(\frac{M}{N_A \sigma_0 \theta \gamma}\right) f}{[f + Q_0(\alpha)]\varepsilon_p} \quad (1)$$

$$A_{sp} = \frac{N_p}{wSDCt_m} \quad (2)$$

where A_{sp} is the specific activity, M is the atomic mass, θ is the isotopic abundance, σ_0 is the 2200 $m.s^{-1}$ (n, γ) cross-section, γ is the absolute gamma-intensity, N_p is the number of counts under the full-energy peak during the counting time t_m, w is the sample weight in gram, S = $1\text{-}\exp(-\lambda t_{irr})$ is the saturation factor with t_{irr} being the irradiation time, $D = e^{-\lambda t_d}$ is the decay factor with t_d being decay time, C = $[1\text{-}\exp(-\lambda t_m)]/\lambda t_m$ is the measurement factor correcting for decay during the measurement time t_m, λ is the decay constant, N_A is the Avogadro's number, ε_p is the full energy peak detection efficiency, f is the thermal to epithermal neutron flux ratio; α is the epithermal neutron flux shape factor.

For ideal situation $Q_0 = I_0/\sigma_0$; I_0 - resonance integral for an ideal (assumed 1/E) epithermal neutron flux distribution.

For a non-ideal situation, the Q_0 (I_0) need to be modified with an α-dependent term. The conversions from the tabulated Q_0 (I_0) values to $Q_0(\alpha)$ (or ($I_0(\alpha)$)) are given by:

$$Q_0(\alpha) = \left[\frac{Q_0 - 0.429}{(E_r)^\alpha} + \frac{0.429}{(2\alpha+1)(E_{Cd})^\alpha}\right](1eV)^\alpha \qquad (3)$$

where E_{Cd} is the effective Cd cut-off energy (E_{Cd}=0.55 eV in standard conditions) and E_r is the effective resonance energy, defined by Ryves [8]. The $(1eV)^\alpha$ a term (numerically unity) originates from the definition of the epithermal neutron flux in a $1/E^{1+\alpha}$ distribution [9,10].

For an ideal reactor flux, the slowing down neutrons after collision with the moderator atoms, show an energy distribution $\Phi_e(E)$ which varies as E^{-1}. This means that the epithermal neutron flux integrated over one logarithmic energy interval can be represented by a constant Φ_e since:

$$\Phi_e = \frac{\Phi_e}{E} \qquad (4)$$

However, it was found that applying a single comparator method (k0-standardization) with reactor neutrons, using Eq. (4) is unacceptable from the standpoint of accuracy [11]. This is due to the fact that the epithermal flux is shown to deviate from the ideal situation with a factor α. Eq. (4) should, therefore, be modified to take care of the flux-shaping factor and thus we have a semi-empirical relationship given as:

$$\Phi_e = \frac{\Phi_e}{E^{1+\alpha}} \qquad (5)$$

where α is the characteristic of the reactor irradiation position and was shown [11] to be positive (softened) or negative (hardened) depending on the reactor epithermal spectrum. Eq.(5) was proved to be satisfactory for instrumental neutron activation analysis [6,11] and it enables the correction of the resonance integral to the deviating spectrum. Thus to preserve accuracy in the k0-method, α should be known when calculating the concentration of an element in a sample [6,12].

The k0-standardization method was introduced in NAA [13]. In terms of the k_0-methodology, adopting the Høgdahl convention [14], the concentration calculations are based on the fundamental equation:

$$\rho(ppm) = \frac{\frac{N_p/t_m}{SDCW}}{\left(\frac{N_p/t_m}{SDCw}\right)^*}\frac{1}{k_0}\frac{f+Q_0^*(\alpha)\varepsilon_p^*}{f+Q_0(\alpha)\varepsilon_p} \qquad (6)$$

with k_0 defined as:

$$k_0 = \frac{M^*\theta\sigma_0\gamma}{M\theta^*\sigma_0^*\gamma^*} \qquad (7)$$

SMELS type III [13] (a multi-element synthetic material producing the long-lived radionuclides when irradiated with neutrons) and SRM-2711a (the certified reference material from National Institute for Standard and Technology (NIST) were used to calculate the concentration of the elements at different positions in the container using k0-INAA.

In order to evaluate the laboratory performance, the u-score test was used in which the u-score is calculated according to the following equation:

$$u - score = \frac{x_{lab} - x_{ref}}{\sqrt{u_{lab}^2 + u_{ref}^2}} \quad (8)$$

where x_{lab}, u_{lab}, x_{ref}, and u_{ref} are the experimental and reference values and uncertainties, respectively [2].

The relative bias between the experiment result and the reference value is calculated and expressed as a percentage:

$$RB = \frac{x_{lab} - x_{ref}}{x_{ref}}.100\% \quad (9)$$

Experiment

For determination of the axial flux distribution, the gold wire (Al-0.1%Au, ϕ=0.6mm) was used. The 12cm long Au wire was placed at the center of the aluminum container. After irradiation and suitable decay, it was cut in pieces of 5 mm with the weight of about 2 - 6 mg. The gold foils (Al-0.1%Au, d=0.1mm) and zirconium foils (Zr-99.98%) were also placed at some positions 1, 2, 6, 7, 12, 14 cm in order to determine the thermal neutron flux and α, f values.

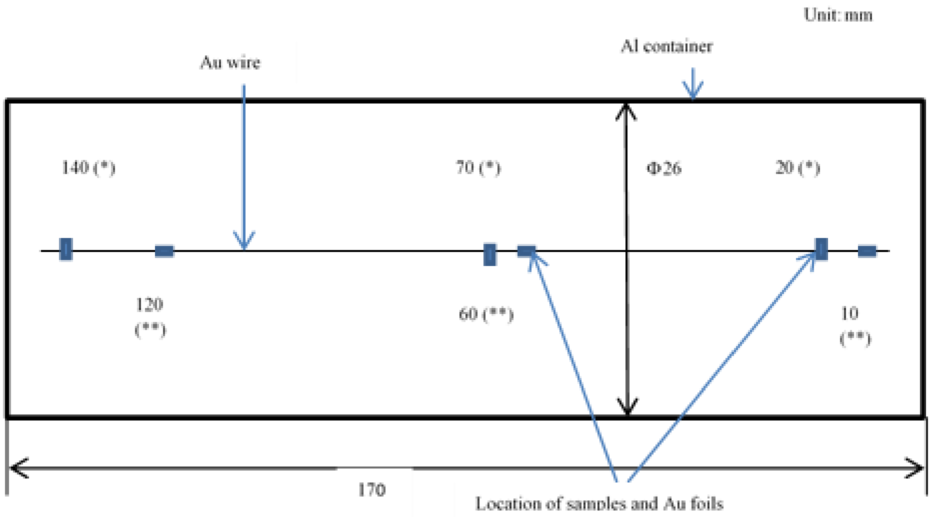

Fig.2. *Typical aluminum sample irradiation container usually used at the rotary rack of the DNRR.*

The information of the irradiation, decay, counting times of samples was shown in Table 1.

Table 1. *The irradiation, decay, counting times for Au wire/foil, Zr monitor, NIST2711a, SMELS III.*

SRM/Monitor	Irradiation time (t_{irr})	Decay time (t_d)	Counting time (t_m)
Au wire	1h	≈12d	17÷39h
Au foil	1h/10h	3÷4 d	10 ÷ 15m
Zr monitor	1h	3d	120m
NIST2711a	1h/10h	9÷12d	2÷15h
SMELS III	10h	10÷18d	2÷23h

After an appropriate decay time, the Au wire was cut into sixteen pieces of 5mm and measured on the HPGe coaxial detector (GMX30190), which has a relative efficiency of 30% and energy resolution of 1.9 keV at 1332.5 keV. The full-peak energy efficiency of the detector was determined using standard gamma-ray sources of ^{241}Am, ^{133}Ba, ^{109}Cd, ^{137}Cs, ^{60}Co, ^{57}Co and ^{152}Eu.

Results and discussion

The thermal neutron flux distribution in the sample irradiation container of the DNRR were shown in Table 2 and Fig. 2. The difference of thermal neutron flux between the bottom (3.54×10^{12} n.cm^{-2}.s^{-1}) of the 0.75 cm and the top (1.93×10^{12} n.cm^{-2}.s^{-1}) of the 15 cm aluminum container is up to 45%. The measured results of α and f at the rotary rack of the DNRR were found of 0.088 and 39.5, respectively. The values are in good agreement with the previous measurements [16].

The obtained neutron flux was then used to calculate concentrations of the elements in the Standard Reference Material 2711a and SMELS III using k0-INAA method at different positions in the container. The x_{lab}/x_{ref} ratios, RB values and u-scores were used to evaluate the precision of data. The neutron flux distribution in the container were shown in Table 2 and Fig 3. The concentrations of elements Fe, Cr, Co, Sc in NIST-2711a were compared with the reference values in which the RB values are less than 5% in both cases of sandwiching and linear interpolation at difference position in the container, except for Cr were about 12% at the position of 14 cm. The u-score values were within ±1.64 for all elements (see Table 3-5).

Table 2. *The axial thermal neutron flux profile in the sample irradiation container.*

Axial position in the container (cm)	**Thermal neutron flux** (n.cm^{-2}s^{-1})	**Uncertainty** (n.cm^{-2}s^{-1})	**Axial position in the container** (cm)	**Thermal neutron flux** (n.cm^{-2}s^{-1})	**Uncertainty** (n.cm^{-2}s^{-1})
0.75	3.51E+12	1.43E+11	2(*)	3.35E+12	1.32E+11
1.25	3.43E+12	1.39E+11	7(*)	2.84E+12	1.13E+11
2.25	3.48E+12	1.43E+11	14(*)	2.06E+12	8.31E+10
3.25	3.28E+12	1.32E+11	1(**)	3.51E+12	1.38E+11
4.25	3.16E+12	1.29E+11	6(**)	3.07E+12	1.22E+11
5.25	3.08E+12	1.25E+11	12(**)	2.27E+12	9.11E+10
6.25	2.90E+12	1.18E+11			
6.75	2.86E+12	1.17E+11			
7.25	2.77E+12	1.13E+11			
8.25	2.71E+12	1.15E+11			
9.25	2.68E+12	1.09E+11			
10.25	2.55E+12	1.04E+11			
12.25	2.26E+12	9.25E+10			
13.25	2.13E+12	8.63E+10			
13.75	2.09E+12	8.67E+10			
14.25	1.98E+12	8.16E+10			

(*) Neutron flux in January 2019. (**) Neutron flux in February 2019.

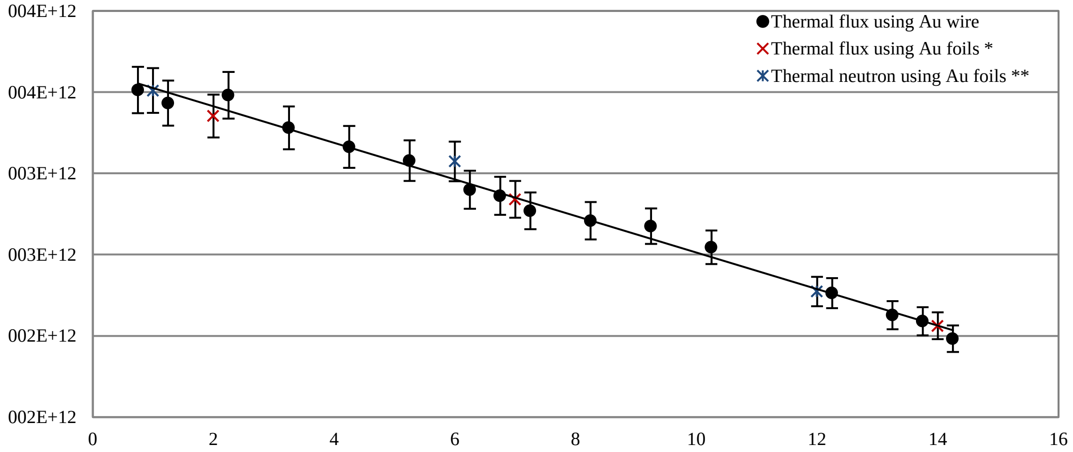

Fig.3. *Thermal neutron flux distribution from the bottom to top of the container*

Table 3. *The concentrations of elements and uncertainties in mg/kg, x_{lab}/x_{ref} ratios, RB and u-score values for NIST 2711a at position 2cm in the container.*

Element	$x_{lab}\pm u_{lab}$[1]	$x_{lab}\pm u_{lab}$[2]	$x_{ref}\pm u_{ref}$	x_{lab}/x_{ref}[1]	x_{lab}/x_{ref}[2]	RB[1](%)	RB[2](%)	u-score[1]	u-score[2]
Fe	29621±1235	29258±1223	28200±400	1.05	1.04	5.04	3.75	1.09	0.82
Cr	54.2±3.2	53.5±3.2	52.3±2.9	1.04	1.02	3.63	2.29	0.44	0.29
Co	10.31±0.55	10.19±0.54	9.89±0.18	1.04	1.03	4.25	3.03	0.73	0.52
Sc	8.6±0.4	8.5±0.3	8.5±0.1	1.01	1.00	1.18	0.00	0.24	-0.05

[1] using sandwiching flux monitor

[2] using linear interpolation

Table 4. *The concentrations of elements and uncertainties in mg/kg, x_{lab}/x_{ref} ratios, RB and u-score values for NIST 2711a at position 7 cm in the container.*

Element	$x_{lab}\pm u_{lab}$[1]	$x_{lab}\pm u_{lab}$[2]	$x_{ref}\pm u_{ref}$	x_{lab}/x_{ref}[1]	x_{lab}/x_{ref}[2]	RB[1](%)	RB[2](%)	u-score[1]	u-score[2]
Fe	28786±1197	28854±1180	28200±400	1.02	1.02	2.08	2.32	0.46	0.53
Cr	54.1±2.6	52.0±2.5	54.2±2.6	1.00	1.00	-0.18	-4.06	0.47	0.5
Co	10.25±0.47	9.85±0.44	9.89±0.18	1.04	1.00	3.64	-0.40	0.72	0.78
Sc	8.6±0.4	8.3±0.3	8.6±0.4	1.00	0.97	0.00	-3.49	0.32	0.38

Table 5. *The concentrations of elements and uncertainties in mg/kg, x_{lab}/x_{ref} ratios, RB and u-score values for NIST 2711a at position 14 cm in the container.*

Element	$x_{lab}\pm u_{lab}$[1]	$x_{lab}\pm u_{lab}$[2]	$x_{ref}\pm u_{ref}$	x_{lab}/x_{ref}[1]	x_{lab}/x_{ref}[2]	RB[1](%)	RB[2](%)	u-score[1]	u-score[2]
Fe	28726±1234	28985±1223	28200±400	1.02	1.03	1.87	2.78	0.41	0.61
Cr	58.2±3.2	58.7±3.2	52.3±2.9	1.11	1.12	11.28	12.24	1.37	1.49
Co	10.16±0.51	10.25±0.51	9.89±0.18	1.03	1.04	2.73	3.64	0.49	0.67
Sc	8.8±04	8.9±0.4	8.5±0.1	1.04	1.05	3.53	4.71	0.73	0.95

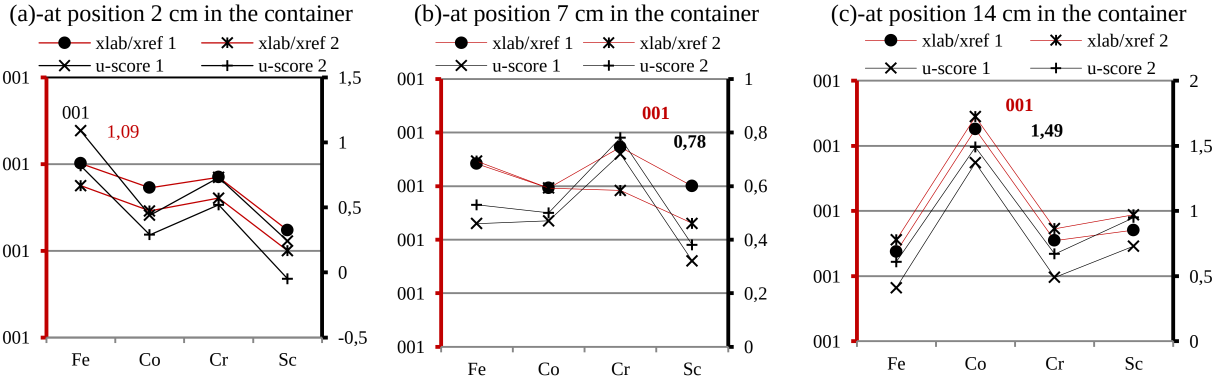

Fig. 4. *The x_{lab}/x_{ref} ratio (left Y-axis) and the u-score (right Y-axis) for NIST 2711a.*

Tables 6, 7 and Fig. 5 show elements concentration and uncertainties in mg/kg for SMELS III sample along with x_{lab}/x_{ref} ratios, RB values and u-scores at different placed sample positions in the container with sandwiching flux monitors and with linear interpolation of thermal neutron flux. These results were compared with the obtained results by the other authors [18] in which the RB values for all elements were within ±5%. Generally, most u-score values were within ±1.96 except for Th, Tm, Yb (the second method was marked 2, at position 6 cm in the container) were bigger than this value (Table 7 and Fig. 5b). If we increase the limiting value for the u-score to 2.58 for a level of probability at 99%, all our analytical results will pass.

Table 6*. The concentrations of elements and uncertainties in mg/kg, x_{lab}/x_{ref} ratios, and u-scores for SMELS III at position 1 cm in the container.*

Element	$x_{lab}\pm u_{lab}^1$	$x_{lab}\pm u_{lab}^2$	$x_{ref}\pm u_{ref}$	x_{lab}/x_{ref}^1	x_{lab}/x_{ref}^2	RB^1(%)	RB^2(%)	U-score^1	U-score^2
Fe	8672±358	8655±357	8200±190	1.06	1.06	5.76	5.55	1.16	1.12
Co	25.50±1.04	25.45±1.04	24.3±0.33	1.05	1.05	4.94	4.73	1.09	1.05
Cr	90.2±3.79	90.0±3.8	86.7±2.6	1.04	1.04	4.04	3.81	0.76	0.72
Sc	1.21±0.05	1.21±0.01	1.140±0.031	1.06	1.06	6.14	6.14	1.16	1.12
Cs	22.58±0.92	22.53±0.92	20.80±0.34	1.09	1.08	8.56	8.32	1.80	1.76
In	510±21	509±21	462±19	1.10	1.10	10.28	10.17	1.69	1.66
Sb	54.8±2.2	54.6±2.2	51.2±1.3	1.07	1.07	7.03	6.64	1.37	1.33
Se	145±6	144±6	131±6	1.11	1.10	10.69	9.92	1.61	1.57
Sr	8909±375	8891±374	8150±200	1.09	1.09	9.31	9.09	1.79	1.75
Th	29±1	29±1	26.2±0.9	1.11	1.11	10.69	10.69	1.88	1.84
Tm	25±1	25±1	23.3±0.7	1.07	1.07	7.30	7.30	1.40	1.36
Yb	22.6±0.9	22.5±0.9	20.7±0.5	1.09	1.09	9.18	8.70	1.76	1.72
Zn	661±27	660±27	618±11	1.07	1.07	6.96	6.80	1.49	1.44

Table 7*. The concentrations of elements and uncertainties in mg/kg, x_{lab}/x_{ref} ratios, and u-scores for SMELS III at position 6 cm in the container*

Element	$x_{lab}\pm u_{lab}^1$	$x_{lab}\pm u_{lab}^2$	$x_{ref}\pm u_{ref}$	x_{lab}/x_{ref}^1	x_{lab}/x_{ref}^2	RB^1(%)	RB^2(%)	U-score^1	U-score^2
Fe	8329±485	8697±505	8200±190	1.02	1.06	1.57	6.06	0.25	0.92
Co	24.4±1.2	25.5±1.3	24.3±0.33	0.96	1.05	0.41	4.94	0.07	0.90
Cr	91±5	95±5	86.7±2.6	1.05	1.10	4.96	9.57	0.82	1.49
Sc	1.15±0.06	1.20±0.06	1.140±0.031	1.01	1.05	0.88	5.26	0.20	0.95
Cs	21±1	22±1	20.80±0.34	1.01	1.06	0.96	5.77	0.19	1.02
In	488±22	510±23	462±19	1.06	1.10	5.63	10.39	0.91	1.62
Sb	52±3	55±3	51.2±1.3	1.02	1.07	1.56	7.42	0.42	1.17
Se	138±7	144±7	131±6	1.05	1.10	5.34	9.92	0.77	1.42
Sr	8454±433	8829±453	8150±200	1.04	1.08	3.73	8.33	0.64	1.37
Th	28.1±1.2	29.4±1.3	26.2±0.9	1.07	1.12	7.25	12.21	1.29	2.06
Tm	25.6±1.3	26.8±11.3	23.3±0.7	1.10	1.15	9.87	15.02	1.62	2.32
Yb	22.6±1.2	23.6±1.2	20.7±0.5	1.09	1.14	9.18	14.01	1.53	2.24
Zn	623±32	650±34	618±11	1.01	1.05	0.81	5.18	0.14	0.91

(a)-at position 1 cm in the container

(b)-at position 6 cm in the container

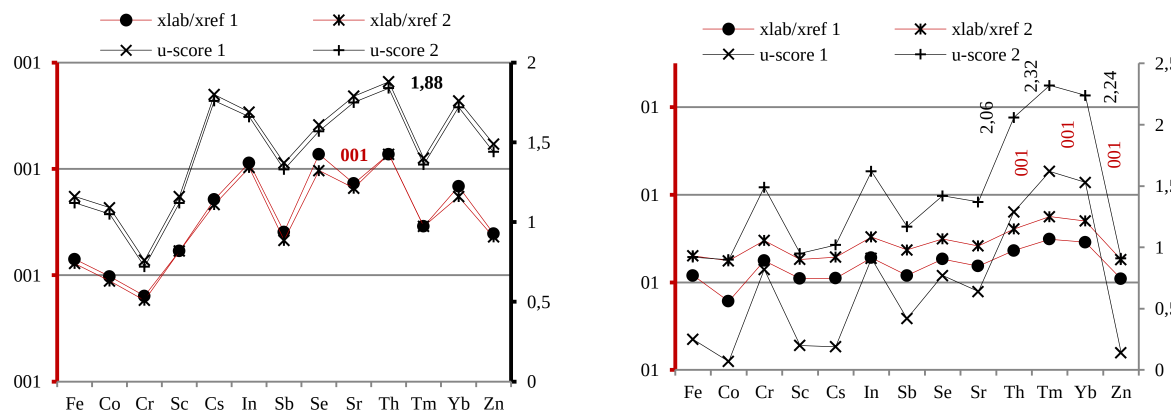

Fig. 5. *The x_{lab}/x_{ref} ratio (left Y-axis) and the u-score (right Y-axis) for SMELS III*

Fig. 6 and Fig. 7 show that determination of concentrations of elements using k0-INAA method with or without sandwiching flux monitors are acceptable. It means that we can get the precise neutron flux at each position of the sample from the linear interpolation instead of sandwiching the monitor as usual.

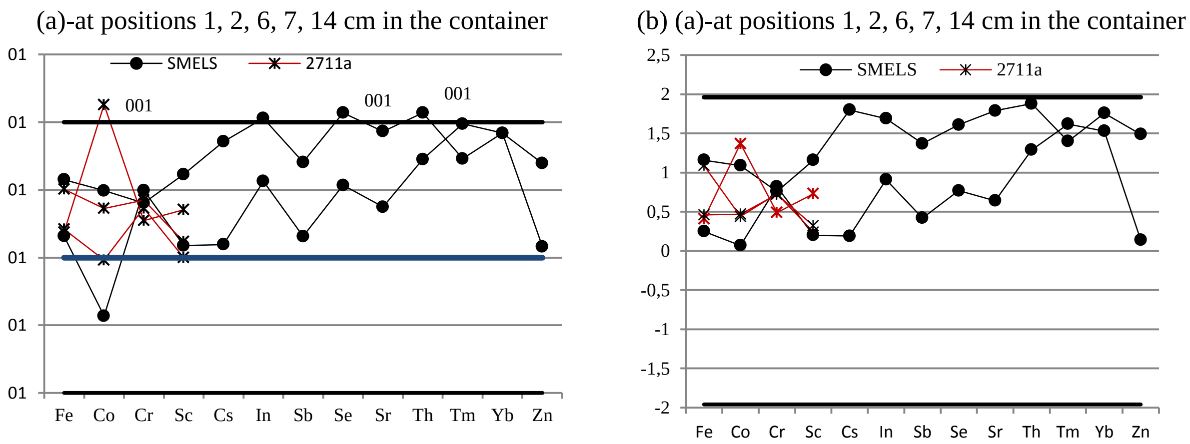

Fig. 6. *The x_{lab}/x_{ref} (a) and u-score (b) for NIST-2711a and SMELS III with sandwiching flux monitors (Au-Al foils).*

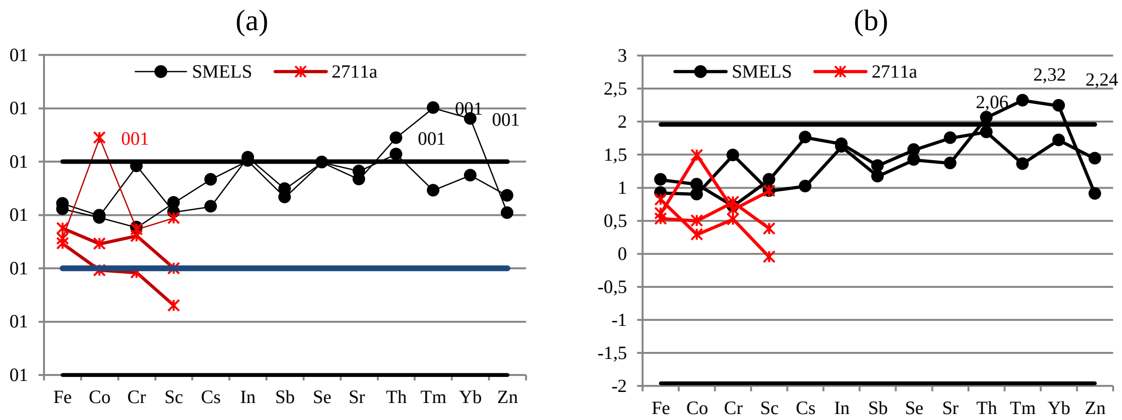

Fig. 7. *The x_{lab}/x_{ref} ratio (a) and u-score (b) for NIST-2711a and SMELS III with linear interpolation of the neutron flux.*

Conclusion

The axial thermal neutron flux distribution in the sample irradiation container at the rotary rack of the DNRR was determined by gold wire activation analysis. The difference of thermal neutron flux between the bottom and the top of the aluminum container is up to 45%. The accuracy of element analysis using k0-INAA method strongly depends on the thermal neutron flux. Hence, the proposed technique can be applied for the determination of concentrations of elements without sandwiching flux monitors.

Acknowledgments

The authors wish to thank the DNRR's operators for their kind supporting the implementation of this work.

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XÁC ĐỊNH PHÂN BỐ THÔNG LƯỢNG NƠTRON NHIỆT TẠI VỊ TRÍ CHIẾU MẪU Ở MÂM QUAY PHỤC VỤ CHO PHÂN TÍCH HÀM LƯỢNG NGUYÊN TỐ SỬ DỤNG PHƯƠNG PHÁP K0-INAA

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Tóm tắt

Độ chính xác của việc xác định hàm lượng nguyên tố bằng phương pháp chuẩn hóa k0 phụ thuộc trực tiếp vào những thông số chiếu và đo bao gồm: Độ lệch khỏi qui luật 1/E của phân bố thông lượng nơtron trên nhiệt $\alpha\ (\varphi_{epi} \approx 1/E^{1+\alpha})$, tỷ số thông lượng nơtron nhiệt trên thông lượng nơtron trên nhiệt f, hiệu suất ghi ε, diện tích đỉnh…. Trong trường hợp vị trí chiếu mẫu tại mâm quay của lò phản ứng hạt nhân Đà Lạt, sự khác nhau của thông lượng nơtron nhiệt giữa phần đáy (3.54×10^{12} $n.cm^{-2}.s^{-1}$) tại 0.75 cm và phần đầu (1.93×10^{12} $n.cm^{-2}.s^{-1}$) tại 15 cm của container nhôm lên đến 45%. Do vậy, cần xác định chính xác những thông số được đề cập ở trên tại vị trí chiếu mẫu. Bài báo đề cập đến việc xác định phân bố thông lượng nơtron nhiệt dọc theo container chiếu mẫu sử dụng kỹ thuật kích hoạt dây vàng (0.1%). Thông lượng nơtron nhiệt sau đó được sử dụng để tính toán hàm lượng nguyên tố trong mẫu tham khảo SRM 2711a và mẫu SMELS III bằng phương pháp chuẩn hóa k0-INAA tại một vài vị trí mẫu trong container. Những kết quả đạt được phù hợp tốt với những giá trị phê chuẩn. Kết luận, kỹ thuật được đề suất có thể được áp dụng cho phân tích kích hoạt với độ chính xác chấp nhận được mà không cần những monitor thông lượng kèm theo mẫu trong khi chiếu.

Từ khóa: *Phương pháp chuẩn hóa k0, lò phản ứng nghiên cứu Đà Lạt, thông số phổ nơtron.*