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Determination of α in the Cadmium Lined Irradiation Channel of the NIRR-1 for Use in k_0 -ENAA

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Abstract

The epithermal neutron shape factor (α) was determined in the permanent cadmium lined irradiation channel installed in the large outer irradiation site of the Nigeria Research Reactor-1 (NIRR-1) by the cadmium covered multimonitor method using the monitors Al-0.1% Au, Zr and Zn thin foils and Mo thin wire and was found to be -0.137 ± 0.018 . The high negative value of α indicates a hardened epithermal neutron spectrum in the cadmium lined irradiation channel. The α value obtained was used in the single comparator method of ENAA (k_o -ENAA) for the determination of the concentrations of elements in the standard reference material NIST 1515 Apple leaves using Al-0.1% Au thin foil as the single comparator. The concentrations of the elements Sm and Br were determined in the NIST 1515 Apple leaves because of their high Q_o values and are in good agreement with the certified values.

Keywords

 α Value, Cadmium Covered Multimonitor Method, Cadmium Lined Irradiation Channel, k_0 -ENAA

1. Introduction

The 30 kW Nigeria Research Reactor-1 (NIRR-1) is located at the Centre for Energy Research and Training, Ahmadu Bello University, Zaria, Nigeria. It is an undermoderated tank-in-pool commercial Miniature Neutron Source Reactor (MNSR) that is used for neutron activation analysis (NAA). The reactor core has highly enriched uranium (90.2%) as fuel, light water as coolant and moderator and metallic beryllium as reflector. The NIRR-1 has 10 irradiation sites (5 small inner, 3 small outer and 2 large outer). However, only six irradiation

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sites are connected to the pneumatic transfer systems types A and B control desks: 4 small inner (B1, B2, B3 and A1), 1 small outer (B4) and 1 large outer (A3). The rabbit system type A is used for pneumatic transfer of samples into and out of the small inner irradiation sites A1 and the large outer irradiation site A3 with a permanent cadmium (Cd) lining of thickness 1.00 mm, length 250.00 mm and cut-off energy of 0.55 eV. The rabbit system type B is used for the pneumatic transfer of samples into and out of the irradiation sites B1, B2, B3 and B4. At full operating power, the maximum thermal neutron fluxes in the inner and outer irradiation sites (excluding the Cd lined irradiation channel A3) are approximately $1 \times 10^{12} \text{ n·cm}^{-2} \cdot \text{s}^{-1}$ and $5 \times 10^{11} \text{ n·cm}^{-2} \cdot \text{s}^{-1}$ respectively. The schematic diagram of the top view of the reactor complex of the NIRR-1 is shown in Figure 1 [1].

The cadmium (Cd)-lined irradiation channel A3 was recently installed to include the capabilities of the NIRR-1 facilities for epithermal neutron activation analysis (ENAA) protocols. Consequently, there is a need to characterize the Cd-lined irradiation channel. In this work, the epithermal neutron shape factor (α) was determined in the Cd-lined irradiation channel for implementation of the single comparator method of epithermal neutron activation analysis (k_o -ENAA). The cadmium covered multimonitor method was used for the determination of α in the irradiation channel.

In the cadmium covered multimonitor method, a set of N suitable α monitors are coirradiated under cadmium cover and the induced activities measured on an efficiency calibrated high purity germanium (HPGe) detector system. For elements having a cross section $\sigma(v) \propto 1/v$ in the range up to ≈ 1.5 eV, α can be found as the slope $(-\alpha)$ of the straight line when plotting:

$$\log \frac{\overline{E}_{r,i}^{-\alpha} \cdot \left(A_{sp,i}\right)_{\text{Cd}}}{k_{o,\text{Au}(i)} F_{\text{Cd},i} Q_o\left(\alpha\right)_i \varepsilon_{d,i}} \quad \text{versus} \quad \log \overline{E}_{r,i}$$
 (1)

where

 $k_{o,Au(i)} = k_o$ -factors for the *i*th monitor with respect to Au,

 $Q_o(\alpha)_i$ = resonance integral to thermal neutron cross section ratio in the actual irradiation site,

$$(A_{sp,i})_{Cd} = \left[\frac{N_p/t_c}{SDCw}\right]_{Cd}$$
 is the specific activity of the *i*th monitor the Cd index denotes Cd cover irradiation,

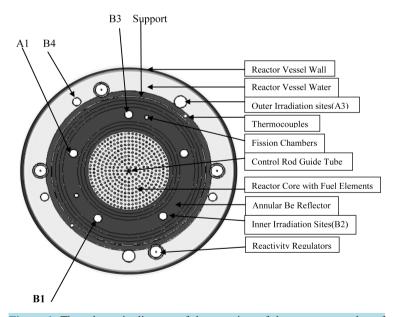


Figure 1. The schematic diagram of the top view of the reactor complex of the NIRR-1. A3—cadmium lined irradiation channel, A1—small inner irradiation channel, B1—small inner irradiation channel, B2—small inner irradiation channel, B3—small inner irradiation channel, B4-small outer irradiation channel.

 N_p = Net photopeak area (counts) of each monitor irradiated in Cd lined channel,

 $S = (1 - e^{-\lambda t_i})$ = saturation factor for each monitor,

 $D = e^{-\lambda t_d} = \text{decay factor for each monitor.}$

$$C = \left(\frac{1 - e^{-\lambda t_c}}{\lambda t_c}\right) = \text{ correction factor for decay during counting for each monitor,}$$

 $\lambda = \text{decay constant}, \ t_i = \text{irradiation time}, \ t_d = \text{decay time}, \ t_c = \text{counting time}, \ w = \text{mass of element in sample of thin foil or wire monitors irradiated (g),}$

 $F_{Cd,i}$ = cadmium epithermal neutron transmission factors for the *i*th nuclide,

 ε_{di} = full energy peak detector efficiency of the *i*th nuclide,

 $\varepsilon_{\nu} = \gamma$ -ray intensity,

 $i = 1, 2, \dots, N = \text{number of nuclides},$

 $\overline{E}_{r,i}$ = average resonance energy of the *i*th monitor.

The left hand term of Equation (1) is itself a function of α , and thus an iterative procedure can be applied with square regression analysis to fit the experimental data to the straight lines for every iterative step [2].

Mathematically, the final α result of this iteration procedure is identical with solving for the value of α from the equation

$$\alpha + \frac{\sum_{i=1}^{N} \left\{ \left[\log \overline{E}_{r,i} - \frac{\sum_{i=1}^{N} \log \overline{E}_{r,i}}{N} \right] \left[\log T_{i} - \frac{\sum_{i=1}^{N} \log T_{i}}{N} \right] \right\}}{\sum_{i=1}^{N} \left[\log \overline{E}_{r,i} - \frac{\sum_{i=1}^{N} \log \overline{E}_{r,i}}{N} \right]^{2}} = 0$$
(2)

where
$$T_i = \frac{\overline{E}_{r,i}^{-\alpha} \cdot \left(A_{sp,i}\right)_{\mathrm{Cd}}}{k_{o,\mathrm{Au}(i)} F_{\mathrm{Cd},i} Q_o\left(\alpha\right)_i \varepsilon_{d,i}}$$
, $\frac{k_{o,\mathrm{Au}(i)}}{E_{r,i}} = k_o$ factors for the i th monitor with respect to Au, $\overline{E}_{r,i}$ average resonance energy of the i th monitor,

 $i = 1, 2, \dots, N = \text{number of nuclides}.$

2. Materials and Methods

The cadmium covered multimonitor method was use for the determination of α in the cadmium lined irradiation channel of the NIRR-1. One set of four monitors comprising of high purity Al-0.1% Au, Zr and Zn thin foils and Mo thin wire were weighed, arranged in a small polyethylene thin film bag and then put inside a 25 cm³ polyethylene vial. They were covered with cotton wool and the top of the polyethylene vial properly sealed with a celotape. The characteristics of the detector thin foils and wire used are shown in Table 1.

The set of monitors Al-0.1% Au, Zr and Zn thin foils and the Mo thin wire in the 25 cm³ polyethylene vial were irradiated simultaneously in the Cd lined irradiation channel of the NIRR-1 at a preset thermal neutron flux of $5 \times 10^{11} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$ for 3 hours. The induced activities on the irradiated set of monitors were measured using the full energy peak efficiency calibrated P-type GEM 30195 HPGe coaxial detector system at the distance of 2 cm from the detector. The energy resolution of the detector system is 1.95 keV for the 1332 keV peak of ⁶⁰Co and the relative detector efficiency is 30%. The full energy peak efficiency of the P-type GEM 30195 HPGe coaxial detector was measured at the distances 2 cm and 15 cm from the detector over the energy range 59.54 -1408 keV using the set of IAEA standard sources ²⁴¹Am, ¹⁵²Eu ²²⁶Ra, ¹³⁷Cs, ⁶⁰Co and ²²Na. A detailed description of the measured full energy peak efficiency curves and the theoretical fitting function to the experimental efficiency curves is given elsewhere [3].

The product nuclides ^{69m}Zn, ¹⁹⁸Au, ⁹⁷Zr and ⁹⁹Mo were counted for 3600 seconds within one day after irradiation of the α monitors. The product nuclides ⁶⁵Zn and ⁹⁵Zr were counted for 72000 seconds after 14.91 - 14.99 days. The photopeak areas of the gamma-ray energies for the product nuclides of the irradiated α monitors were obtained from the gamma-ray acquisition system of the P-type GEM 30195 HPGe coaxial detector system that consists of the Maestro Multicannel Annalyser (MCA) emulation software card coupled to the detector via electronic nuclear Instrumentation modules manufactured by Ortec.

Table 2 shows the nuclear data for the product nuclides of the four monitors irradiated for α determination in the Cd lined irradiation channel of the NIRR-1. The efficiencies ε of the gamma-ray energies for the product nuclides of the four monitors were calculated by the authors using mathematical fitting functions described elsewhere [3]. The specific activities $A_{\rm sp\ (Cd)}$ for each of the product nuclides of the four monitors were also calculated by the authors. The nuclear data $\overline{E}_{\rm r}$, Q_o , $F_{\rm Cd}$ and $k_{o, \rm Au}$ -factors were obtained from the literature [4]-[8].

3. Results and Discussions

The α value was determined by solving Equation (1) by the iterative linear regression method based on MS Excel spreadsheet. Starting with step 1 by setting $\alpha = 0$ resulted in the value of $\alpha_1 = -0.123 \pm 0.020$. The final value of α was obtained after four iterative procedures. The third and fourth iterations both led to α_3 and α_4 equal to -0.137 ± 0.018 . Thus, the final value of α by the iterative procedure was -0.137 ± 0.018 .

Furthermore, the epithermal neutron flux value in the Cd lined irradiation channel at the preset power of the NIRR-1 was determined using the peak area of the 411.8 keV gamma-ray line from ¹⁹⁸Au.

Table 3 shows the values of the epithermal neutron shape factor (α) and the epithermal neutron flux determined in the Cadmium lined irradiation channel of the NIRR-1. From **Table 3**, the value of α is negative. The negative value of α indicates a hardened (poorly thermalized) epithermal neutron spectrum in the Cd lined irradiation channel of the NIRR-1.

Also from **Table 3**, the value of epithermal neutron flux calculated using the activity of 198 Au in Al-0.1% Au thin foil in the monitor set irradiated for α determination in the cadmium lined irradiation channel of the NIRR-1 was found to be $(4.51 \pm 0.09) \times 10^9 \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$. This value is comparable to a theoretical value of $5.05 \times 10^9 \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$ in the outer irradiation site A2 of the NIRR-1 based on the assumption that the thermal neutron flux values in the outer irradiation sites are 50% of the values in the inner irradiation sites [9]. The thermal to epithermal neutron flux ratio in the outer irradiation site A2 of the NIRR-1 has been found to be approximately 49.5 ± 0.96 [9] as shown in **Table 4**. It should be noted that the channel A2 is now the channel A3 that has been lined with the 1.00 mm thick Cd-liner.

Table 4 shows a comparison of the α values in the Cd lined irradiation channel with results of other irradiation sites of the NIRR-1 and other miniature neutron source reactors [9]-[12]. From **Table 4**, it can be observed that due to the installation of the Cd liner in one of the outer irradiation channels, the α value is different from the old data in similar outer irradiation sites of the NIRR-1. The high negative value of α in the cadmium lined

Table 1. Description of the activation detector foils and wire for α determination.

Element	Material description	Diameter (cm)	Mass (g)
Au	Al-0.1% Au thin foil, 0.1mm thick, IRMM-530	0.9	0.0132
Zn	99.95% Zn foil, 0.025 mm thick, Goodfellow	0.8 0.8	0.0083
Zr	99.8% Zr foil, 0.125 mm thick, Goodfellow	0.8	0.0446
Mo	Thin wire	0.8	0.0168

Table 2. Nuclear data for the product nuclides of 4 monitors irradiated for α determination in the Cd lined irradiation channel.

Target nucleus	Product nucleus	Half life	E _γ (keV)	$\bar{E}_{r}\left(eV\right)$	Q_o	$k_{o,\mathrm{Au}}$ factors	A _{sp(Cd)} (C/s.g)	ε	F_{Cd}
¹⁹⁷ Au	¹⁹⁸ Au	2.70 d	411.8	5.65	15.71	1	5.43×10^{8}	0.0212	0.991
64 Zn	65 Zn	243.9 d	1115.5	2560	1.908	5.72×10^{-3}	5.02×10^{5}	0.00784	1
68 Zn	69m Zn	13.76 h	438.6	590	3.19	3.98×10^{-4}	8.49×10^4	0.0197	1
94 Zr	95 Zr	64.02 d	724.2,	6260	5.05	9.32×10^{-5}	1.27×10^4	0.0110	1
			756.7			1.15×10^{-4}	1.53×10^{4}	0.0105	1
96 Zr	97 Zr	16.74 h	743.4	338	248	1.30×10^{-5}	4.06×10^4	0.0107	1
⁹⁸ Mo	⁹⁹ Mo	65.94 h	140.51	241	53.1	5.27×10^{-4}	1.73×10^6	0.0336	1

irradiation channel indicates a hardened (poorly thermalized) epithermal neutron spectrum in the cadmium lined irradiation channel (A3) of the NIRR-1. This is to be expected because the thermal neutron component of the reactor neutron spectrum has been highly cut-off by the 1.00 mm thick cadmium of the cadmium lined irradiation channel causing the neutron spectrum to be shifted to higher energies in the epithermal region.

In order to test the α value obtained, the concentrations of Sm and Br in the NIST 1515 Apple leaves were determined by the single comparator method of epithermal neutron activation analysis (k_o -ENAA) in the cadmium lined irradiation channel of the NIRR-1. The standard reference materials NIST 1515 Apple leaves was irradiated together with the Al-0.1% Au thin foil as the single comparator in the Cd lined irradiation channel at a preset thermal neutron flux of $5 \times 10^{11} \, \text{n·cm}^{-2} \cdot \text{s}^{-1}$ for 6 hours. The induced activities on the elements of interest in the samples of NIST 1515 Apple leaves were measured under the same counting conditions at the distance of 2 cm from the efficiency calibrated P-type GEM 30195 HPGe coaxial detector system after appropriate decay and counting periods.

The concentrations of the elements determined in the NIST 1515 Apple leaves were calculated by the k_o -ENAA method with the MS Excel spreadsheet using the equation [13]

$$C_{s(Cd)} = \left[\frac{\left(\frac{N_{p,s}/t_c}{SDCW} \right)_s}{\left(\frac{N_{p,Au}/t_c}{SDCW_{Au}} \right)_{Au}} \right]_{Cd} \cdot \left[\frac{F_{Cd,Au} \cdot Q_o(\alpha)_{Au} \cdot \varepsilon_{d,Au}}{k_{o(Au)} F_{Cd,s} Q_o(\alpha)_s \varepsilon_{d,s}} \right]$$
(3)

where the $k_{o,Au}$ factor is a compound nuclear constant of the nuclide in the sample of material with respect to Au [14]

 M_{Au} = atomic mass of gold,

 M_s = atomic mass of the element of interest in the sample,

 θ_s = isotopic abundance of the element of interest in the sample,

 θ_{An} = isotopic abundance of gold,

 $\varepsilon_{r,s} = \gamma$ -ray intensity of the element of interest in the sample,

Table 3. Results of neutron spectrum parameters in the Cd lined irradiation channel.

Irradiation channel	α	$\Phi_e (ext{n} \cdot ext{cm}^{-2} \cdot ext{s}^{-1})$
Cd lined (A3)	-0.137 ± 0.018	$(4.51 \pm 0.09) \times 10^9$

Table 4. Comparison of the α values in the Cd lined irradiation channel with results in other irradiation sites of the NIRR-1 and other MNSRs.

Facility	Inner	Channel	Outer Channel		
	f ratio	α	f ratio	α	
Prototype MNSR, Beijing, China, 27 kW, 90.2% ²³⁵ U(Sites: Inner and oute r)	19.8 ± 0.4	-0.009 ± 0.004	58.5 ± 3	$+0.023 \pm 0.010$	
GHARR-1 MNSR, Accra, Ghana, 30 kW, 90.2% ²³⁵ U(Sites:1—inner, 10—outer)	18.8	-0.104	49.0	-0.0261	
GHARR-1 MNSR, Accra Ghana, 30kW, 90.2% 235U(Sites: 5—inner, 7—outer)	17.28	-0.107 ± 0.001	40.38	-0.173 ± 0.0001	
NIRR-1 MNSR, Zaria, Nigeria, 90.2% ²³⁵ U(Sites: B2—inner, B4—outer)	19.2 ± 0.5	-0.052 ± 0.002	48.3 ± 3.3	$+0.029 \pm 0.003$	
NIRR-1 MNSR, Zaria, Nigeria, 30kW, 90.2% ²³⁵ U(Sites: A1—inner, A2—outer)	18.4 ± 0.34	-0.046 ± 0.005	49.5 ± 0.96	$+0.024 \pm 0.002$	
NIRR-1 MNSR, Zaria, Nigeria, 30kW, 90.2% ²³⁵ U(Sites: A3(Cd Lined Channel))				-0.137 ± 0.018	

 $\varepsilon_{\gamma,Au} = \gamma$ -ray intensity of gold,

 $\sigma_{o.s}$ = thermal neutron cross section of elements of interest in the sample,

 $\sigma_{a \text{ Au}}$ = thermal neutron cross section for Au,

 $F_{\text{Cd Au}}$ = the cadmium epithermal neutron transmission factor for gold,

 $F_{\text{Cd.}s}$ = the cadmium epithermal neutron transmission factor for elements in sample,

 $Q_o(\alpha)_{Au}$ = corrected resonance integral to the thermal neutron cross sections for gold in the Cd lined irradiation channel,

 $Q_o(\alpha)_s$ = corrected resonance integral to the thermal neutron cross sections for the elements in the sample in the Cd lined irradiation channel,

 α = measured epithermal neutron shape factor in the Cd lined irradiation channel,

 $\varepsilon_{d,s}$ = detector efficiency for γ -ray energy of nuclide in sample,

 $\varepsilon_{d,Au}$ = detector efficiency for γ -ray energy of ¹⁹⁸Au,

 $W_s = \text{total amount of sample irradiated,}$

 C_s = concentrations of the unknown element in samples irradiated in the Cd lined irradiation channel (mg·kg⁻¹),

 $w_{\rm s}$ = calculated unknown weight of element in sample,

 w_{Au} = weight of the sample of gold foil,

 $N_{n,s}$ = Net photopeak area of radionuclide of interest in sample,

 $N_{p,\text{Au}} = \text{Net photopeak area of }^{198}\text{Au},$

 $D = e^{-\lambda t_d} = \text{decay factor for gold or elements of interest in sample,}$

 $S = (1 - e^{-\lambda t_1})$ = saturation factor for gold or elements of interest in sample,

$$C = \frac{\left(1 - e^{-\lambda t_c}\right)}{\lambda t_c} = \text{ correction factor for decay during counting for gold or elements in sample,}$$

 $\lambda = \text{decay constant}, t_i = \text{irradiation time}, t_d = \text{decay time}, t_c = \text{counting time}.$

The results of the concentrations of the elements determined in the NIST 1515 Apple leaves by k_o -ENAA are shown in **Table 5**.

From Table 5, the elements Sm and Br with high Q_o values were determined in the NIST 1515 Apple leaves by the k_o -ENAA method. The elements Sm and Br strongly absorb neutrons of specific resonance energies in the epithermal neutron region. These elements have high resonance activation integrals compared to their thermal neutron cross sections in the epithermal neutron region. The concentrations of Sm and Br in the NIST 1515 Apple leaves are in good agreement with the certified values. The k_o -ENAA method in the cadmium lined irradiation can be regarded as a reliable method of neutron activation analysis (NAA) in the NIRR-1. It can be used for the determination of bromine in bread to control the illegal use of potassium bromate (KBrO₃) in bread making in Nigeria.

4. Conclusions

The value of the epithermal neutron shape factor (α) and epithermal neutron flux (φ_e) in the permanent cadmium lined irradiation channel installed in the large outer irradiation site of the NIRR-1 have been determine for the purpose of adopting the k_o -ENAA method in the NIRR-1 NAA laboratory. The value of α was determined by the cadmium covered multimonitor method using the monitors Al-0.1% Au, Zr and Zn thin foils and Mo thin

Table 5. Results of concentrations of elements determined in NIST 1515 Apple leaves by k_o -ENAA in mg·kg⁻¹.

Element	Certified (mg·kg ⁻¹)	This work (mg·kg ⁻¹)
Sm	3.00	2.44 ± 0.04
Br	1.80	1.80 ± 0.27

wire and was found to be -0.137 ± 0.018 . The epithermal neutron flux was found to be $(4.51 \pm 0.09) \times 10^9$ n·cm⁻²·s⁻¹. The high negative value of α indicates a hardened epithermal neutron spectrum in the cadmium lined irradiation channel.

The α value obtained was used in the single comparator method of ENAA (k_o -ENAA) for the determination of the concentrations of elements in the SRM NIST 1515 Apple leaves using Al-0.1% Au thin foil as the single comparator. The concentrations of the elements Sm and Br with high Q_o values determined in the NIST 1515 Apple leaves are in good agreement with the certified values. This is an important evidence of the reliability of the result of the α value obtained.

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