

# CHARACTERISTICS OF THE NEW PROMPT GAMMA NEUTRON ACTIVATION ANALYSIS FACILITY AT THE DALAT RESEARCH REACTOR.

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**Abstract:** A new PGNAA facility has been installed at horizontal channel No.4 of the Dalat research reactor. The facility consists of the following parts: collimators, beam stopper, sample holder table, beam catcher, and biological shielding. The construction takes into account the basic parameters associated with the facility, such as adequate thermal neutron flux and cadmium ratio, good signal-to-background condition, small gamma-ray contamination in the beam and appropriately low dose-rate to the operating personnel. The materials used in the construction are lead, lithium florin, cadmium, Teflon, and ion. The characteristics of the new PGNAA facility, such as neutron flux, gamma dose and cadmium ratio at sample position, absolute efficiency curve, background gamma-ray spectrum, analytical sensitivities and detection limits for some elements were determined, and proven to be much better than those of the old one.

**Keyword:** PGNAA facility, Dalat research reactor, characteristics of the PGNAA facility

## Introduction

Prompt Gamma-ray Activation Analysis (PGAA) is a nuclear analytical technique that based on the  $(n,\gamma)$  reaction. Upon neutron capture, the resulting compound nucleus is raised to an excited energy state and gives off the excess energy in the form of one or more gamma rays to return to a stable state. The gamma rays are measured by semi-conductor gamma spectrometry. The energies of the gamma rays are characteristic of the elements present in the sample, and intensities of the gamma rays are proportion of their concentrations. The PGAA technique can simultaneously determine trace and other elements in solid, liquid or gaseous samples by measuring the gamma-rays emitted by the sample during neutron irradiation. The PGAA method has been applied to materials science[1,2], chemistry[3,4], geology[5,6,7], mining[8,9], archaeology[10,11], environment[12,13], food analysis[14,15], medicine[16,17], forensic science[18,19,20,21] and other areas. PGAA is a complementary method to instrumental neutron activation analysis (INAA) and highly sensitive for the elements: H, B, C, N, P, S, Cd, Sm, Gd and Pb [22,23].

The characteristics of some PGNAA facilities in over the world can be seen at literatures[24-30]

The prompt gamma neutron activation analysis facility had been constructed and installed in 1985 at the radial beam port of the 500 kW Dalat Research Reactor which was reconstructed and upgraded from 250 kW TRIGA Mark II reactor [24]. The configuration shielding of the facility is shown in Fig. 1. Since its design and construction were intentionally kept simple, in addition with the use of materials such as borated paraffin and lead for shielding, the gamma background of the high purity germany (HPGe) spectrometer was high. There were two sources that contributed to the gamma background:

- The old facility lacked a proper sample box, which was to shield gamma and neutron radiations scattering from the sample into the surrounding environment. These scattered neutrons can activate materials around to induce both delay and prompt gamma radiations. A part of these gamma radiations could enter the HPGe gamma detector and then contributed to the gamma background.
- The material used to shield the detector was borated paraffin. Boron and paraffin have large neutron capture cross sections and high gamma emission intensities in reactions with neutrons. So after reacting with neutrons, these materials emitted gamma lines with high intensities. In the gamma background of the spectrometer, the fairly high peaks of gamma rays and Compton flats of boron and hydrogen could be seen.

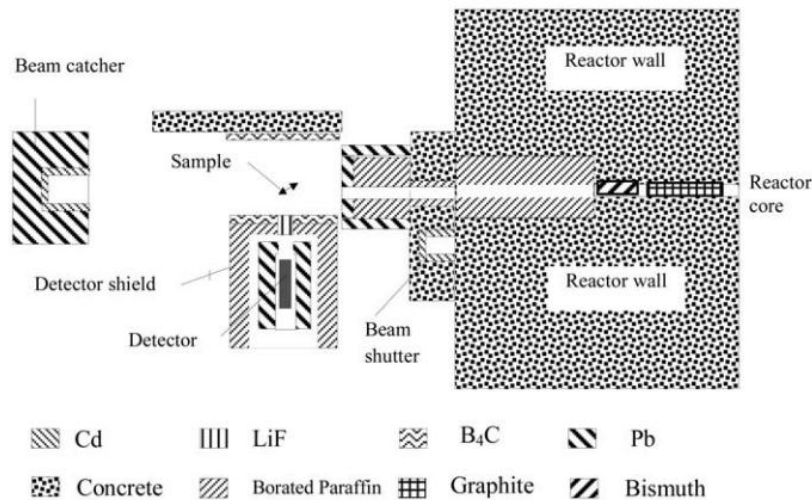


Fig.1 Configuration of the old PGAA at Dalat research reactor (reproduction from reference 24 with a permission)

The high gamma ray background of the gamma spectrometer constrained the detector to achieve the detection limits at ppm concentration levels, therefore the gamma ray background should had been reduced by the construction of a new neutron beam filters and neutron and gamma shielding for detector and a sample box using Teflon, LiF, which emits low gamma rays when react with neutrons. The new facility consists of collimators, beam shutter, biological shielding, sample box, beam stopper. These additions ensure that its characteristics are better than those of the old one.

## Experimental configuration

### Neutron beam

Radial beam port No. 4 was selected for the installation of the PGNAA facility. The

average neutron flux inside the reactor core is of the order of  $10^{13}$  n.cm<sup>-2</sup>.s<sup>-1</sup> from which a neutron flux level of  $10^6$  n.cm<sup>-2</sup>.s<sup>-1</sup> is required at the sample position of the collimator for PGNAA. The beam emerging from the reactor beam port consists mainly of fast and thermal neutrons and high-energy gamma rays. Peak to background ratio of the gamma-ray spectrum depends upon the background gamma radiation within the thermal neutron beam and scattered neutron into gamma detector. Thermal neutrons are extracted from the beam port for PGNAA by slowing down the fast neutrons to thermal energy and filtering out the high-energy gamma rays. Single crystal silicon was selected as the moderator because of its availability and large diffusion length (40-cm thick, and placed 85 cm from the end side wall of the reactor). A 2-cm thick block of titanium was used as beam filter to minimize the high-energy gamma radiation at the sample position and to reduce the need for additional shielding outside the biological shielding. The beam aperture consists of two boron carbide sheets (3-mm thick each) to give an aperture diameter of 25 mm. A hollow graphite block of 15-cm thick separates the aperture from the moderator block in order to obtain a uniform neutron beam, and the outer diameter of the divergent beam collimator is 30 mm. Streaming of the radiation is eliminated by using titanium and lead as beam stoppers that intercept all the radiation coming from the core of the reactor, gamma rays that arise from radiative capture of the neutrons, and scattered radiation from the sample and sample holder. The beam position was determined by neutron radiography, and the Fig. 2 is the image of the neutron beam taken by neutron radiography.

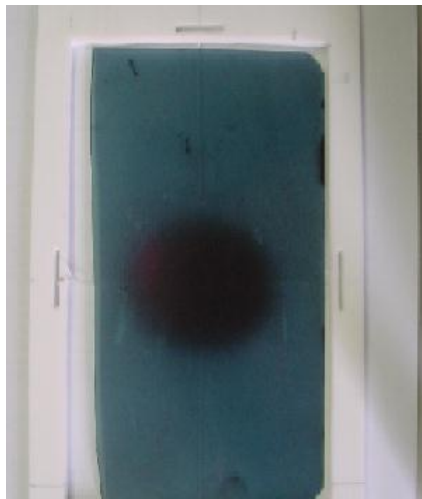


Fig. 2 Neutron beam position was determined by neutron radiography

The beam shutter ensures the safe operation of the facility while positioning the sample. This shutter system consists of two parts: beam shutter and trap. Beam shutter plays a key role in controlling the safe operation of the facility and avoiding radiation exposure to workers while positioning the samples, etc. Beam trap is used to intercept all the radiation coming from the reactor core, gamma radiation resulted from radiative capture of neutrons and scattered radiation from the object and the object holder etc. The first part was made from borated paraffin and boron carbide and cadmium sheets, and it was lined by LiF rubber. It was enclosed in an aluminum casing. The slowed down and thermalized neutrons were absorbed by borated paraffin, cadmium and boron carbide sheets. The second part of the shutter was made from lead bricks and boron carbide sheets and enclosed in a steel casing. The shutter was mounted on a trolley and was operated manually. The beam trap was

fabricated from borated paraffin, lead, boron carbide and steel. Additional shielding from scattered gamma and neutron radiation is provided by erecting a radiation enclosure of ordinary concrete blocks. The layout of the facility shown in Fig.3 describes the details of the beam port, single crystal silicon block, source aperture, titanium block, collimators, beam shutter, biological shielding, sample box, beam stopper, etc.

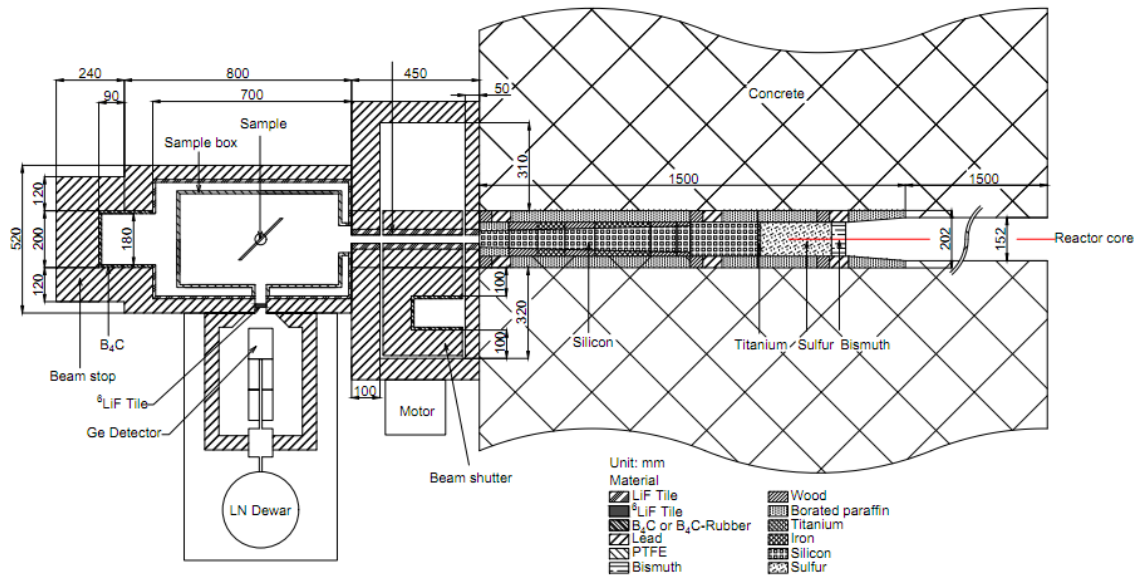


Fig. 3 The layout of the new PGNAA facility at Dalat research reactor

The neutron beam at the channel No. 4 is applied not only for PGNAA facility but also for other experiments based on filtered neutron beams, such as total and capture neutron cross section measurements[25, 26], and other experiments. For this reason, the facility was designed and constructed such that the shielding part of the PGNAA can be moved backward and forward manually on a rail to has a space for setting up the experiments and to change neutron filter compositions. Fig.4 shows the layout of the position of the shielding part when the neutron beam is used for the total neutron cross section measurement experiment.

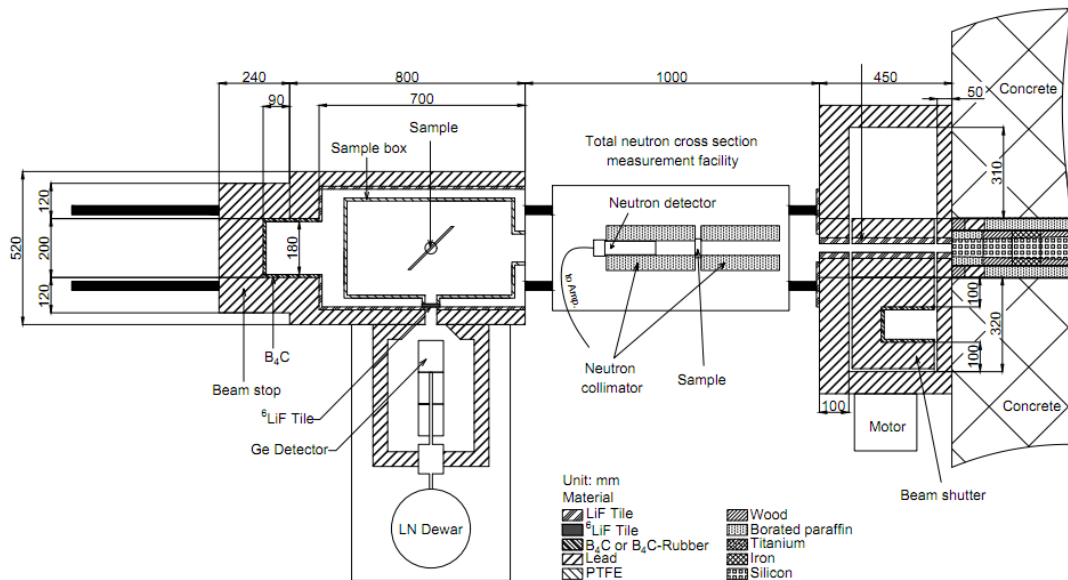


Fig. 4 The layout of the new PGNAA facility at Dalat research reactor

### ***Gamma spectrometer and sample arrangement***

A 16k ADC was used to collect the detector signal and the ORTEC Gamma-Vision was employed to display and conduct on-line analyses of the accumulating spectrum on the connected PC desktop. GEM series HPGe-254 cc detector manufactured by Ortec is used to count the prompt gamma rays (resolution of 1,9 keV and relative efficiency of 58% at 1332 keV of  $^{60}\text{Co}$ ).

The detector is shielded from the gamma background by a cylinder of 20-cm thick lead. The lead cylinder is lined by 3-mm thick of LiF rubber to reduce neutron absorption and scattering which would likely cause formation of background prompt gamma rays. The prompt gamma rays are detected through a window of LiF rubber (32-mm diameter) located in the upper lead layer. A PTFE sample box is placed behind the beam collimator. Samples are sealed in a film of 25- $\mu\text{m}$  thick fluorinated ethylenepropylene resin (FEP), and placed on the sample holder. In order to reduce the  $\gamma$ -ray background that originates from neutrons scattered by construction materials and by the sample itself, all walls facing the neutron beam are covered with LiF rubber and  $\gamma$ -ray are shielded with 10 cm of Pb.

## **Characteristics of the system**

### **1. Energy calibration**

Since both delay gamma rays and prompt gamma rays are detected by the gamma detector, leading to the gamma spectrum are very complicated, then to identify the photo peak correspond to isotope, energy calibration procedure must be taken care in detail. For energy calibration, the prompt gamma-rays from the  $^{14}\text{N}(n,\gamma)\text{N}^{15}$  reaction were chosen because they have high precision and they essentially cover the entire range of interest, which are from 121.3 keV to 10829.4 keV. Moreover, most strong lines have their intensities measured quite recently [27] with high precision with respect to the  $^{14}\text{N}(n,\gamma)\text{N}^{15}$  capture lines.

A sample of about 2.10 gram  $\text{NH}_4\text{Cl}$  was irradiated for 6000 seconds, and at the same time the capture gamma-rays spectra was accumulated. The channel number in the measured spectrum, in respect of the energies of prompt gamma-rays, and the uncertainties of these channel numbers can be observed from the analyzed spectrum. The second order polynomial in energy was used for energy calibration. Non-linearity over the above energy range was not significant.

## 2. Neutron flux, cadmium ratio and gamma dose rate

The neutron flux was measured by means of activated Au foils and cadmium ratio was also determined by the activation of Au foils with and without a cadmium cover. Neutron flux and cadmium ratio are  $(2.10 \pm 0.11) \times 10^7 \text{ n.cm}^{-2}.\text{s}^{-1}$  and  $110 \pm 6$ , respectively. Flux variations at the sample position during one reactor operation cycle of 100 hours were measured every 5 hours by using 0.025-mm thick Au foils, and found to be  $(1.2 \pm 0.02)\%$ . The gamma dose rate at the sample position was determined by TLD to be  $198 \pm 12 \text{ mR/h}$ .

## 3. Gamma-ray background

The distance from detector to sample was determined by observing the total background counting rate for neutron beam on blank target, keeping the dead time of the ADC less than a few %. Hence the normal position of the detector is 25.5 cm distance from the target and the observed total background rate is about 3.4k cps.

The prompt gamma-ray background spectrum was collected in 60000 seconds and was processed. Information on capture gamma-ray is taken from reference[27] to identify the prompt gamma-ray lines of different background elements. The sensitivity of gamma-ray lines of the background of the old and the new facility are shown in Table 3.

Table 3. Background peak count rate with beam incident on an empty Teflon bag.

Energy [keV]	Element or Nuclide	Count rate [cps] New facility	Count rate [cps] Old facility	Count rate ratio
75	Pb $K_\alpha$	54.2	155.2	2.9
86	Pb $K_\beta$	17.8	58.4	3.3
112	Unknown	2.2	10.5	4.8
140	Unknown	17.9	56.7	3.2
159	$^{76}\text{Ge}$	24.1	78.4	3.3
175	$^{70}\text{Ge}$	4.9	12.1	2.5
198	Unknown	16.8	60.3	3.6
254	$^{74}\text{Ge}$	2.9	8.6	3.0
500	$^{72}\text{Ge}$ capture + $^{73}\text{Ge}$ inelastic	2.9	7.9	2.7
511	Annihilation	23.6	77.5	3.3
570	$^{207}\text{Bi}$ + $^{70}\text{Ge}$	0.05	0.3	6.0
596	$^{73}\text{Ge}$ capture + $^{74}\text{Ge}$ inelastic	10.6	40.2	3.8
708	$^{70}\text{Ge}$	1.22	5.4	4.4
870	Unknown	2.68	6.4	2.4
1063	$^{207}\text{Bi}$	0.06	0.21	3.5
1096	$^{70}\text{Ge}$	1.30	4.5	3.5

1294	Unknown	4.03	8.5	2.1
1460	<sup>40</sup> K	1.31	6.4	4.9
1632	<sup>70</sup> Ge, <sup>20</sup> F, <sup>207</sup> Bi	0.27	3.4	12.6
1779	<sup>28</sup> Al	0.46	2.1	4.6
1885	N	0.41	3.4	8.3
2032	Li	0.09	0.3	3.3
2223	H	0.80	2.8	3.5
7368	Pb	0.36	2.1	5.8
10829	N	0.03	0.1	3.3

#### 4. Efficiency calibration

Efficiency calibration of the detector has been performed by using a set of radioisotope sources and (n,γ) reactions. In this particular case, delay gamma-rays from isotope <sup>152</sup>Eu were used to determine the absolute efficiency curve in the lower energy range of 121.3–1408.4 keV by fixing the scale. The gamma emission probability of <sup>152</sup>Eu was taken from IAEA-TECDOC-619[28]. After the absolute efficiency curve had been completed with the assistance of the standard radioisotope, the measurement of prompt gamma-rays from the (n,γ) reactions of Cl and N was performed to extend the energy range to 10,829 keV. The NH<sub>4</sub>Cl sample and the melamine sample were prepared and irradiated for a sufficient period of time to obtain the statistical uncertainty below 1% for the weakest peak from each set. The absolute gamma intensities for (n,γ) reactions of Cl and N were taken from the evaluated dataset [27]. The corroborated datasets were combined with the normalization determined through a fitting process by the application of the 5<sup>th</sup> order polynomial form for the whole energy range of 60 – 10,829 keV. In Fig. 4, the full energy peak efficiency of the PGNAA spectrometer is shown with the polynomial fitting line. The relative standard uncertainty is less than 1.5% for the lower energy range and less than 2.6% for the whole energy range.

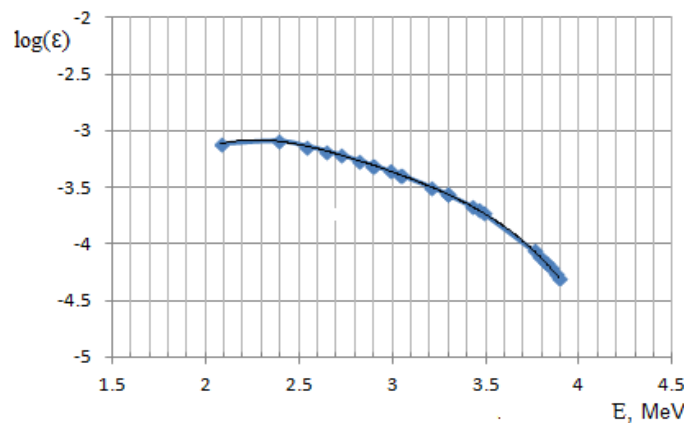


Fig. 4 The absolute efficiency curve of the new PGNAA facility at Dalat research reactor

#### 5. Examination of element sensitivities and detection limits

Sensitivity of each element at specify energy can be derived as follow:

$$S = \frac{\rho_{\gamma}}{m} = \frac{N_A}{M} \sigma_{\gamma,0} \phi_0 \varepsilon(E) \quad (1)$$

The prompt gamma spectrum of NBS-1633a standard was irradiated and collected in 51000 seconds. The spectrum was processed using Gamma-Vision software, and the sensitivities of some elements in the standard set are shown in Table 3.

Table 3 Sensitivities of the old and the new PGNA facilities

Element	Energy (keV)	Sensitivity (c/s/mg) Old facility	Sensitivity (c/s/mg) New facility	Ratio of sensitivity (New facility to old one)
K	771	0.00042	0.017	40.5
Ca	1942	0.000112	0.003	26.8
	4419		0.006	
Si	3539	0.000059	0.001	16.9
Al	1778	0.000244	0.004	16.4
Fe	352	0.000112	0.012	89.3
	1725		0.010	
B	478		84.86	----
Cl	786+788	0.00401	0.237	59.1
	1165		0.159	
H	2223	0.00561	0.211	37.6
Ti	342	0.0082	0.979	119.4
	1381		0.084	

Bảng 5.4. Giá trị độ nhạy đối với một số nguyên tố của hệ cũ, mới tại Đà Lạt và của JAERI

Nguyên tố	Năng lượng (keV)	Độ nhạy Hệ cũ (cps)	Độ nhạy Hệ mới (cps)	Tỷ số độ nhạy
K	771	0.00042	0.017	40.5
Ca	1942	0.000112	0.003	26.8
	4419		0.006	
Si	3539	0.000059	0.001	16.9
Al	1778	0.000244	0.004	16.4
Fe	352	0.000112	0.032	21.8
	1725		0.010	
B	478	0.423	84.86	20.1
Cl	786+788	0.00401	0.237	59.1
	1165		0.159	
H	2223	0.00561	0.211	37.6



Ti	342	0.0082	0.979	119.4
	1381	0.00838	0.084	10.2

Bảng 5.5: Giới hạn ghi nhận của một số nguyên tố trong mẫu chuẩn NBS 1577a

Mẫu		Đà Lạt	JAERI[77]
Khối lượng mẫu (g)		3.5031	0.17213
Thời gian đo (s)		42644	25 000
N	%	2.8	1.0
K	%	0.098	0.021
Cl	%	0.011	0.0024
Fe	ppm	1200	690
Ca	ppm	1860	1400
Mn	ppm	210	74
B	ppm	0.11	0.089
H	%	0.020	0.0035
S	%	1.3	0.044
C	%	12.9	5.6
P	%	4.8	0.35

Bảng 5.6: Giới hạn ghi nhận của một số nguyên tố trong mẫu chuẩn NBS 1633a

Mẫu		Đà Lạt	JAERI[77]
Khối lượng mẫu (g)		1.4024	0.14848
Thời gian đo (s)		50248	10000
Ti	%	0.05	0.009
K	%	0.23	0.036
Al	%	1.34	0.13
Si	%	3.25	0.27

Fe	%	3.08	0.11
Ca	%	10.9	0.13
Gd	ppm	0.15	0.09
Sm	ppm	1.28	0.25
B	ppm	1.08	0.17
H	%	0.03	0.0036

## Conclusion

The new PGNAA facility installed at the radial channel No. 4 of the Dalat research reactor and its components is summarized and presented in this paper. The characteristics of the facility are also presented. The PGNAA facility was designed not only for PGNAA technique but also for other purposes of using neutron beam at this channel. From our investigation, gamma-ray background at typical peaks of the new facility is reduced from several times to more than ten times when comparing with those of the old one. Furthermore, analytical sensitivity of some elements increased by some tens to more than hundred times.

In the next stage, the BGO detectors and their suitable electronic modules should be used for setting up the gamma spectrometer to install Compton suppression mode in order to reduce gamma background. As a result, analytical sensitivities and detection limits would be significantly improved.

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CÁC ĐẶC TÍNH CỦA THIẾT BỊ PHÂN TÍCH KÍCH HOẠT NƠTRON GAMMA TỨC THỜI MỚI TẠI Lò PHẢN ỨNG HẠT NHÂN ĐÀ LẠT

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Tóm tắt: Thiết bị phân tích kích hoạt neutron gamma tức thời đã được xây dựng tại kênh ngang số 4 của lò phản ứng hạt nhân Đà Lạt. Thiết bị gồm các phần: ống chuẩn trực neutron, chặn dòng, bàn đặt mẫu, bắt dòng, và che chắn bức xạ. Cấu trúc của thiết bị đã tính đến các tham số cơ bản liên quan như thông lượng neutron nhiệt và tỷ số cadmi thích hợp, tỷ số tín hiệu trên nhiễu tốt, bức xạ gamma lẫn trong dòng neutron nhỏ, và suất liều bức xạ ảnh hưởng tới nhân viên làm việc thấp ở mức hợp lý. Vật liệu sử dụng để xây dựng gồm chì, lithium florin (LiF), cadmium, Teflon, sắt. Các đặc trưng của thiết bị PGNAA như thông lượng neutron, tỷ số cadmi, suất liều gamma tại vị trí chiếu mẫu, đường cong hiệu suất tuyệt đối, phổ phân tích bức xạ gamma, độ nhạy phân tích và giới hạn ghi nhận của vài nguyên tố đã được xác định, và cho thấy chúng tốt hơn nhiều so với các tham số của thiết bị cũ.