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Implementation of the k_0-NAA method in the NAA#3 irradiation hole of the HANARO research reactor

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Abstract

The NAA#3 irradiation hole in the 30 MW HANARO research reactor of the Korea Atomic Energy Research Institute (KAERI), with a thermal neutron flux of $1.26 \times 10^{14}\,cm^{-2}\,s^{-1}$, has been regarded as suitable for the application of k_0-based neutron activation analysis (k_0-NAA). The objectives of the present work were: (a) to characterize the NAA#3 irradiation hole via the determination of the neutron spectrum parameters required for the method, (b) to calibrate the HPGe gamma-ray spectrometer system via establishing the detection efficiency curves and (c) to assess the quality of the k_0-NAA method by the analysis of six certified reference materials, three of which were of biological nature and three of environmental origin. The results obtained indicated that, by using the k_0-NAA method, approximately 25 or 35 elements could be quantitatively determined in the biological and environmental samples, respectively. The deviations between the experimental and the certified values for the determined elements were generally within 12% with *u*-scores mostly below 2. The results prove that the k_0-NAA method, implemented in the HANARO research reactor, is applicable for multi-element analysis in biological and environmental samples with a rather high analytical performance and that the method is available for further practical applications.

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Keywords: Neutron activation analysis; k_0 standardization method; Reactor neutron spectrum; Gamma-ray spectrometer; Standard reference materials

1. Introduction

In k_0-based neutron activation analysis (k_0-NAA), the evaluation of the analytical result is based on the so-called k_0-factors that are associated with each gamma line in the gamma-spectrum of the activated sample. These factors replace nuclear constants, such as cross-sections and gamma-emission probabilities, and are determined in specialized NAA laboratories. The method enhances the accuracy of the analytical results by avoiding the unnecessary accumulation of uncertainties on the underlying physical constants. The k_0-factors are reactor and detector independent, and their values are agreed upon and used by an increasing number of k_0-users all over the world. The k_0-NAA method is at present capable of tackling a large variety of analytical problems when it comes to the multi-element determination in many practical samples. The objective of the present work was to assess the applicability of the k_0-NAA method using the experimental system and irradiation protocol in the NAA#3 hole of the HANARO research reactor.

2. Experimental

2.1. Characterization of the NAA#3 irradiation hole

The reactor neutron spectrum parameters considered for the corrections in the k_0-NAA method are the deviation of the epithermal neutron flux distribution from the ideal $1/E$

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law approximated by a $1/E^{1+\alpha}$ shape (α), the ratio of the thermal-to-epithermal neutron flux (f), the ratio of the thermal-to-fast neutron flux (f_F) to evaluate interferences by threshold reactions [1,2], and the neutron temperature (T_n) used for nuclides with a Westcott g-factor different from unity [3]. These parameters change according to the reactor configuration and the irradiation hole position. Therefore, their determination is required when applying the k_0-NAA method. The arrangement of the irradiation holes in the HANARO reactor is shown in Fig. 1.

Monitors of Al-0.1% Au and Al-0.1% Lu in wire shape with weights of about 5 mg along with Zr and Ni foils with weights of about 10 mg were irradiated in the NAA#3 irradiation hole for 1 min. The Ni and Lu monitors were measured after a decay time of 3–5 h, the Au and Zr monitors after a decay time of 1 day, and the Zr, Ni and Lu monitors were re-measured after a decay time of 2–3 days. All the measurements were performed on a calibrated gamma-ray spectrometer (see below) with counting times ranging from 30 min to several hours.

The parameters α and f were calculated by the "bare" monitor method using the ^{198}Au, ^{95}Zr and ^{97}Zr nuclides [4]. The parameter f_F was calculated via the ^{198}Au and ^{58}Co/^{65}Ni nuclides in which ^{58}Co is induced by the ^{58}Ni(n, p) reaction. T_n was calculated using the ^{198}Au and ^{177}Lu/^{176m}Lu nuclides via the Westcott $g(T_n)$ factor for Lu, the modified spectral index $r(\alpha)\sqrt{T_n/T_0}$ and the ratio of the modified reduced resonance integral to the 2200 m s^{-1} cross section [$s_0(\alpha)$]. The results are shown in Table 1. The neutron spectrum parameters were determined several times to calculate their average values, after which each sample irradiation just required the co-irradiation of one single flux monitor. This condition is valid until a significant change occurs in the configuration of the irradiation hole [5].

2.2. Calibration of the gamma-ray spectrometer

The characterization procedure of the gamma-ray spectrometer includes the calibration of the energy, the peak width and the detection efficiency. The detection efficiency that has to be applied to the actual samples is one of the key parameters in the k_0-NAA method. In the present work, experimental efficiencies were obtained via measurement of a multi-nuclide standard source produced by Isotope Products Laboratories that included ^{241}Am (59.5 keV), ^{109}Cd (88.0 keV), ^{57}Co (122.1 keV), ^{123m}Te (159.0 keV), ^{113}Sn (391.7 keV), ^{85}Sr (514.0 keV), ^{137}Cs (661.7 keV), ^{88}Y (898.0 and 1836.0 keV), ^{60}Co (1173.2 and 1332.5 keV). The experimental efficiencies (in a log–log plot) were fitted with a 4th degree polynomial, i.e. the calculation of the efficiency values involved the use of five coefficients of the polynomial [2].

Fig. 2 shows the efficiency curves of the gamma-ray spectrometer for neutron activation analysis using an Ortec GEM20180-P detector coupled to an Ortec Spectrum MASTER 919 and an automatic sample changer. The three positions of the sample shelf that were chosen for the

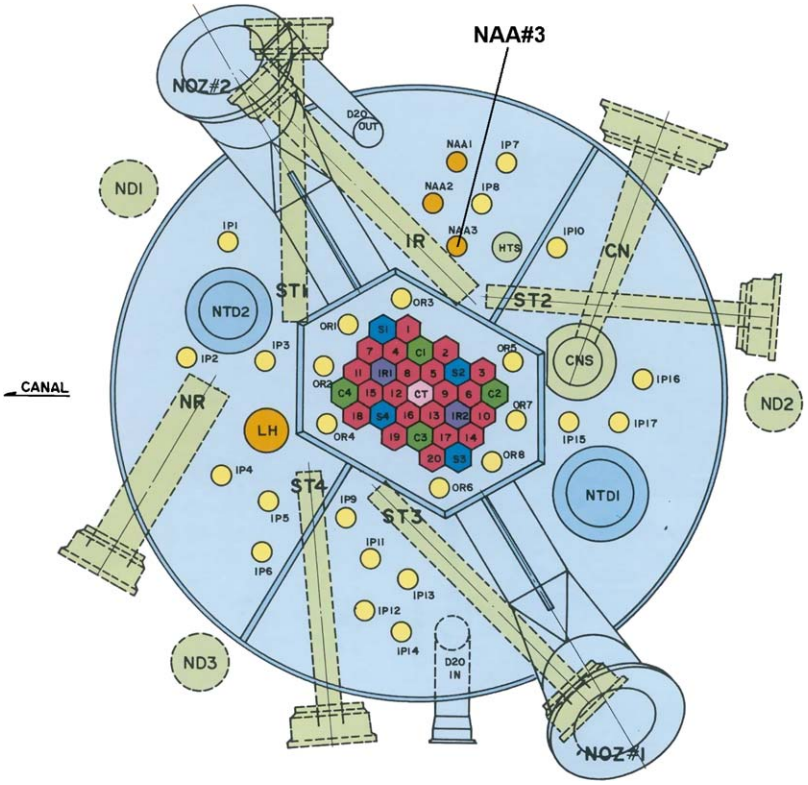

Fig. 1. Arrangement of the NAA#3 irradiation hole in the HANARO research reactor.