Characterization of dry tube location in KAMINI reactor towards the development of k_0 -based IM-NAA

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Abstract

Characterization studies were carried out for the freshly installed dry tubes (DT-1 and DT-2) in KAMINI reactor. The maximum gold equivalent neutron flux at 20 kW for DT-1 and DT-2 were found to be $(1.02 \pm 0.02)E+10$ cm⁻² s⁻¹ at 368 and $(4.90 \pm 0.30)E+7$ cm⁻² s⁻¹ at 300 mm respectively. The sub-cadmium to epithermal neutron flux ratio (*f*) and epithermal neutron flux shape factor (α) were found to be 144 \pm 7 and -0.289 ± 0.010 respectively at this position for DT-1 alone. These parameters were validated by analyzing the IAEA standard (SL-1) and applied to analyze jarosite samples using k_0 -based internal monostandard neutron activation analysis.

Keywords KAMINI reactor · Sub-cadmium to epithermal neutron fux ratio · Epithermal neutron fux shape factor · IM-NAA · In-situ relative detection efficiency

Introduction

Elemental analysis of small to large samples [\[1](#page-7-0)–[4\]](#page-8-0) with irregular geometry is always a challenging task in comparison with the conventional samples and often questionable in terms of true analytical representativeness. Routine analysis of such samples demands sub-sampling and other laborious sampling methodologies. Samples which are precious in nature or difficult to dissolve or with irregular size and shape require special analytical methods. Development of non-destructive analytical techniques is preferable for both analytical as well as economical reasons [[4–](#page-8-0)[7\]](#page-8-1) for the above referred samples. NAA is one of the non-destructive analytical techniques used to establish the elemental profle of samples in a wide varieties of matrices.

There are several standardization methods such as absolute, relative and k_0 -method [\[8](#page-8-2)] that exist in NAA with their associated merits and limitations. The most frequently used method is the relative NAA, which demands a priori knowledge of the analytes present in the sample to be analyzed.

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This necessitates either individual elemental standards or suitable multi-elemental standards like certifed reference materials (CRMs) that are to be irradiated simultaneously along with the sample towards the estimation of its elemental composition. Sometimes, CRMs may not have included our element of interest and also irradiating multi standards may encounter some practical difficulties. In such cases, use of a single comparator like gold (Au) instead of multi standards is preferable for assaying the spectrum of elements present in the samples, which is known as k_0 -NAA method [[9–](#page-8-3)[12\]](#page-8-4). In this case, the knowledge of a composite nuclear constant (k_0) , absolute efficiency of the detector and parameters based on the irradiation position in a reactor such as sub-cadmium to epithermal neutron fux ratio (*f*) and epithermal neutron flux shape factor (α) are required. However, the major challenge involved in this method for the analysis of large or irregular samples is to address the challenges maintaining the consistency of the efficiency which is otherwise disturbed by $[13]$ $[13]$ (1) The variation of neutron fux across the sample during the irradiation (2) The selfattenuation of gamma rays within the sample volume and (3) The practical limitations in preparing standard with identical geometry of the sample.

A k_0 -based internal monostandard neutron activation analysis (IM-NAA) method $[5, 6]$ $[5, 6]$ $[5, 6]$ $[5, 6]$ for non-destructive analysis of elements in irregular samples is an attractive option for many applications such as the analysis of

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precious archaeological, geological, biological, environmental, alloy and special nuclear samples [[7,](#page-8-1) [14](#page-8-8)]. This is an efficient method, developed by Nair et al. [\[6,](#page-8-7) [15](#page-8-9)] in 2003 for the analysis of large and non-standard geometry samples and provides relative concentration of an element with respect to the monostandard that is present within the sample itself. The absolute elemental concentration can be obtained from these ratios for the samples such as metals and alloys by using the mass balance, if all the major and minor elements are established by NAA. Otherwise, knowledge on the concentration of the monostandard or any other element in the sample is needed to establish the absolute concentrations of the other elements. The neutron flux perturbation within the sample, if any, during the irradiation would be mitigated by considering the internal monostandard. Further, gamma rays from various activated products of each sample would be used to obtain the in situ relative detection efficiency [[14](#page-8-8)[–16\]](#page-8-10) that would also mitigate the perturbation in the detection efficiency due to γ-ray self-attenuation and geometrical effects of the sample. The above mentioned advantages make IM-NAA as a unique and geometry independent method and thus enhancing the applicability of the k_0 -NAA method. The concentration ratio of an element (*x*) with respect to the internal monostandard (y) in k_0 -based IM-NAA is calculated using the following Eq. ([1\)](#page-1-0)

$$
\frac{m_x}{m_y} = \frac{((SDC)(f + Q_0(\alpha)))_y}{((SDC)(f + Q_0(\alpha)))_x} \cdot \frac{PA_x}{PA_y} \cdot \frac{(\epsilon_y)_y}{(\epsilon_y)_x} \cdot \frac{k_{0, \text{Au}}(y)}{k_{0, \text{Au}}(x)}
$$
(1)

where *S* is the saturation factor, *D* is the decay correction factor, *C* is the counting correction factor, $Q_0(\alpha)$ is the ratio of the resonance integral (I_0) to thermal neutron cross section (σ_0) corrected for α , PA is the net peak area of the interested gamma lines from the same spectrum, $k_{0, Au}$ is the literature reported $k_{0,\text{Au}}$ -factors [[12\]](#page-8-4) and ε_{γ} is the in situ detection efficiency $[16]$ $[16]$.

The reliability of the method depends on the accurate knowledge of the reactor based parameters f and α as they vary with the irradiation position in the nuclear reactor [[17](#page-8-11)]. The *f* value determines the thermal and epithermal flux components present at the irradiation locations, whereas, α represents the shape of the non-ideal epithermal neutron spectrum. In an ideal case, neutron flux spectrum in epithermal region follows 1/E law, but in practical cases, it invariably deviates from the ideality and rather follows $1/E^{1+\alpha}$ [[18](#page-8-12)–[22](#page-8-13)]. The α value can be either positive or negative depending upon the irradiation position with respect to the reactor core, which may indicate ''softened'' or 'hardened'' epithermal neutron spectrum, respectively. The *α* value is used to calculate the $Q_0(\alpha)$ [[18](#page-8-12)–[22](#page-8-13)].

KAMINI reactor: a neutron source for NAA

KAMINI (KAlpakkam MINI) is a unique research reactor (30 kW power) which is operational at Indira Gandhi Centre for Atomic Research, Kalpakkam with 233U as the fuel. The fuel is in the form of plates of an alloy of 233 U and aluminium which are assembled in an aluminium casing to constitute the fuel subassembly. The reactor has three beam tubes, two thimble irradiation locations and one pneumatic fast transfer system (PFTS) (Fig. [1](#page-2-0)), which are used to carryout irradiation experiments towards radiography, shielding experiments, activation analysis, radiation physics research and testing of neutron detectors.

Recently, two dry tubes were installed in the KAMINI reactor, namely DT-1 and DT-2. These dry tubes are made up of aluminium and positioned just outside the BeO refector blocks in the west and south side of the reactor as shown in Fig. [1.](#page-2-0) These dry tubes are installed for the neutron activation analysis studies and for testing the neutron detectors developed by Bhabha Atomic Research Centre (BARC), Mumbai as well as Electronics Corporation of India Limited (ECIL), Hyderabad. The DT-1 is positioned at a distance of 350 mm from the centre of the reactor core and placed on the base plate with the height, inner and outer diameter of the tube being 3750, 75 and 80 mm respectively. Similarly, the DT-2 is positioned on ring collar at a distance of 900 mm away from the centre of the reactor core with height, inner and outer diameter of the tube being 3750, 103 and 109 mm respectively. These dry tubes are sufficiently large and characterization of these positions towards the determination of neutron flux, f and α is a mandatory requirement to carryout the *k*0-based IM-NAA experiments.

Characterization of the various irradiation positions i.e. PFTS, thimble location and beam tubes were already accomplished and reported elsewhere i.e. Ashok et al. [[23](#page-8-14)] characterized the PFTS position and Mohapatra et al. [\[24,](#page-8-15) [25\]](#page-8-16) characterized thimble and beam tube locations of KAMINI reactor. Brahmaji Rao et al. [[17\]](#page-8-11) validated *k*0- NAA method at PFTS position for the reference materials (1) NIST SRM 1646a (Estuarine Sediment) and (2) BCS Nb-stabilized Stainless Steel (BCS/SS No.261/1). Swain et al. [[26](#page-8-17)] analyzed Synthetic MultiELement Standards (SMELS) by k_0 -based IM-NAA using PFTS of KAMINI reactor for an exercise towards its quality control and reported relative concentration ratios of 22 elements with respect to gold as the internal monostandard.

The present work established the neutron fux profles inside the dry tubes i.e. DT-1 and DT-2 and the fux determination was carried out from the bottom of the tubes. However, the characterization towards the determination of f and α has been carried out for DT-1 only initially.

The *α* values were measured using cadmium-ratio and cover methods, whereas, the *f* values were measured using cadmium-ratio method. The *f* and *α* values so obtained in DT-1 were validated using CRM IAEA Lake Sediment (SL-1) by k_0 -based IM-NAA.

Jarosites are produced in high quantities as solid residues during the extraction of zinc from zinc sulphide ores. About 13 Mt of zinc is produced annually worldwide and India is the second largest and also one of the cheapest Zinc–Lead producers [\[27\]](#page-8-18). Hindustan Zinc Limited (HZL) is India's only integrated Zinc–Lead–Silver producer located in the states of Rajasthan, Andhra Pradesh, Bihar and Odisha as a multi-unit mining and smelting organization with a capacity of 3.5 Mt of Zinc per year. In India, about 0.25 Mt of jarosite per annum is being produced [[28\]](#page-8-19). The jarosite mainly constitutes of toxic elements like chromium, cadmium, arsenic, lead, aluminium etc. Due to their toxic nature, disposal and storage of these jarosite residues without further treatment becomes a major environmental and ecological concern. The disposal and the subsequent impact on the environment with the evaluation of its recycling (or) utilization potential demand the characterization of the jarosite waste with its corresponding elemental composition [[29](#page-8-20)]. However,

conventional analytical techniques would generally demand the destruction of the sample followed by separation of specifc analytes of interest, for the estimation of its elemental composition. The validity of the method demands further a quantitative dissolution of the sample besides demanding a cumbersome separation procedure. Non-destructive assay using NAA is an attractive alternate analytical tool for the determination of elemental composition of such samples. The k_0 -based IM-NAA method was subsequently applied to two jarosite samples received from National Metallurgical Laboratory, Council of Scientifc and Industrial Research (CSIR), Jamshedpur, India to obtain their elemental profle and reported using IM-NAA method.

Experimental

Materials and equipment

The activation foils such as Au and Zr of purity $>99.9\%$ used in this study were procured from Good-fellow foils, U.K. The assay of the irradiated activation foils was accomplished by high resolution gamma spectrometry using a 30% co-axial High Purity Germanium (HPGe) detector procured from M/s Eurisys Mesures. The detector has a resolution of 1.85 keV at 1332 keV of ${}^{60}Co$ and coupled with an associated PC based 8 k multichannel analyzer (MCA) ISA card of Aptec along with acquisition software. Energy calibration of the detector was established using the standard sources of 241 Am, 137 Cs, 133 Ba and 152 Eu, which were procured from M/s Amersham, Inc.

Characterization of dry tube

Axial fux profle

Typically the fux along the axial direction of DT-1 was monitored by irradiating multiple standard sources of gold solution. These standard sources secured at predetermined heights using a thread hanging axially along the tube from the top that extended up to the bottom of the tube covering a total height of a meter. However, the gold standard sources fastened at the bottom of the thread was only accommodated in a polypropylene (PP) container of 100 mL capacity, the weight of which was expected to stabilize the hanging position of the thread as well as to ensure the container just touching the bottom position to maintain the height of the source as accurately as possible (Fig. [2](#page-3-0)). The size of the PP container was chosen based on the inner diameter of the tube such that it can facilitate the irradiation of diferent sizes of samples. Typically, the gold standard source was prepared by taking a known amount of Au standard solution on a flter paper on weight basis, dried under the IR lamp and doubly sealed. The frst Au standard in the PP container was positioned at 37 mm of height from the bottom of DT-1. The height measurement refers to the height from the bottom of the tube. A total of 17 Au fux monitors with the amounts ranging from 100 to 250 μg were fxed axially to the thread inside the DT-1. The fux monitors were all irradiated simultaneously for a period of 2 h at 20 kW power. Experiments with similar design philosophy were carried out to obtain the neutron fux profle inside the DT-2 also.

Determination of *f* **and** *α*

The activation foils such as Au and Zr with and without cadmium cover of 1 mm thickness and width to length ratio of 1:2 were irradiated for 6 h and 2 h respectively. The Au and Zr foils were positioned at a height of 37 mm which is the centre of the PP container and 368 mm which is the position of maximum fux from the bottom which are the positions expected to be used for NAA. All the irradiated

Fig. 2 Irradiation of fux monitors (Au) in dry tubes of KAMINI reactor

foils were assayed by HPGe detector at a distance of 20 cm and the spectra were analyzed for the peak areas using PHAST peak fitting software.

Validation of *f* **and** *α* **and their application towards the sample analysis**

To validate the freshly characterized reactor parameters of *f* and α at the maximum flux position of 368 mm from the bottom of DT-1, an SL-1 standard was analyzed for the elemental concentrations by k_0 -based IM-NAA. Approximately 100 mg of the SL-1 was doubly sealed in a polythene cover and irradiated at the peak fux position of DT-1 for 6 h at 20 kW power.

Similarly, two jarosite samples of 5 g each were irradiated at the same position of DT-1 for 6 h at 20 kW power. The packaging materials were also irradiated along with SL-1 and jarosite samples for the sake of blank correction. The irradiated SL-1 standard and two jarosite samples were sufficiently cooled to bring down the corresponding radiation dose and their subsequent radiometric assay was carried out using HPGe detector.

Results and discussion

Characterization of dry tube

Axial fux profle

The neutron fux profle was established for DT-1 using the measured activity of the activated product of gold standard i.e. 198Au from the peak reports of the spectra of the irradiated Au targets. The gold equivalent neutron fux i.e. the neutron fux obtained using the thermal neutron capture cross section for the reaction $197Au(n,\gamma)^{198}Au$, at the different positions were calculated using following Eq. [\(2\)](#page-4-0)

$$
Neutron flux (\phi) = \frac{A}{N\sigma SDC}
$$
 (2)

where *A* is the activity of the activated product (dps), *N* is the number of target nuclides and σ is the cross-section for the ¹⁹⁷Au(*n*, γ ¹⁹⁸Au nuclear reaction i.e. 98.65E–24 cm².

The calculated neutron fux was plotted as a function of the axial distance from the bottom of the tube DT-1 (Fig. [3](#page-4-1)). From the fgure, it was observed that the fux was increasing with the axial height from the bottom of the tube and peaked at a height of 368 mm which is just parallel and located about the nearest point on the thread in the tube to the core centre point and started decreasing from there on. The maximum neutron flux was observed to be (1.02 ± 0.02) E+10 cm⁻² s⁻¹ at 368 mm. Similarly, the neutron flux at diferent positions starting from the distance of 67 mm to 1000 mm of the DT-2 was also determined. The neutron fux profle of DT-2 (Fig. [4](#page-4-2)) was found to be similar to DT-1 and the maximum neutron flux was observed to be (4.90 ± 0.30) E+7 cm⁻² s⁻¹ at 300 mm from the bottom of the tube. The maximum neutron fux at DT-1 was three order higher than

Fig. 4 Variation of neutron fux along the vertical axis of DT-2

that of the DT-2 owing to its closer vicinity to the core compared to the DT-2. The present study focuses on the complete fux profle of DT-1 due to the presence of higher neutron fux for IM-NAA studies and further study towards the augmentation of complete fux profle would be extended to DT-2 also in future.

Determination of *f* **and** *α*

The values of f and α were determined using the specific activities of the activated products of the fux monitors Au and Zr i.e. 198 Au, ^{95}Zr and ^{97}Zr . In DT-1, the monitors (Au and Zr) were irradiated at two diferent positions i.e. at 37 and 368 mm from the bottom of the tube. The *α* values for the DT-1 was estimated by Cd-ratio method using dual monitors as well as Cd-cover triple monitor $(^{197}Au, ^{96}Zr, ^{94}Zr)$ method [\[20](#page-8-21)]. The relevant nuclear data of the fux monitors used to determine the *f* and α are given in Table [1](#page-5-0) [\[12](#page-8-4)].

In the triple monitor method, the plot of $Log(T_i)$ versus Log($\bar{E}_{r,i}$) was used to obtain the slope ($-\alpha$) as explained in the references [[18,](#page-8-12) [19](#page-8-22)]. The α value was obtained by iteration procedure [\[21](#page-8-23), [22\]](#page-8-13) using Microsoft Excel sheet. The plot obtained from the fnal iteration was shown in Fig. [5](#page-5-1). The average α value was found to be -0.273 ± 0.044 at 37 mm and -0.289 ± 0.010 at 368 mm from the bottom of DT-1. The negative sign of the α values indicates that the neutron spectra at these positions were relatively hard compared to the ideal epithermal neutron fux distribution.

The *f* values were calculated in the cadmium ratio method [[19\]](#page-8-22).

The average *f* values were found to be 347 ± 18 and 144 ± 7 at 37 mm and 368 mm from the bottom of DT-1 respectively indicating the corresponding presence of 99.7% and 99.3% thermal neutron components respectively. The calculated *f* and α values are presented in the Table [2.](#page-5-2) From **Fig. 3** Variation of neutron flux along the vertical axis of DT-1 the table, it can be inferred that the *f* values increase with

Table 1 The nuclear data of flux monitors

Table 1 The nuclear data of flux monitors	Target	Q_0	Effective resonance Corresponding energy E_r (eV)	activated product	$t_{1/2}$	E_{ν} (keV)	$k_{0,\text{Au}}$ (% uncertainty)
	197 Au	15.71	5.65	198Au	2.69d	411.8	
	96Zr	251.6	338	^{97}Zr	16.74h	743	$1.24E - 5(0.3)$
	^{94}Zr	5.31	6260	^{95}Zr	64.02 d	724.2 756.7	$8.90E - 5(1.3)$ $1.10E-4(1.3)$

Fig. 5 Log (T_i) versus Log $(\overline{E}_{r,i})$ in Cd-cover method

the decrease in the neutron fux. This may be attributed to the increased moderation of neutrons with distance from the core.

In-situ relative detection efficiency

The activated products of SL-1 and jarosite samples associated with at least one pair of gamma rays with an accurately known intensity and their peak areas were used for the calibration of in situ relative detection efficiency. The activated products of SL-1 i.e. 24 Na, 56 Mn, 140 La and 152 mEu and of jarosite samples i.e. 24 Na, 46 Sc, 59 Fe, 124 Sb and 140 La were used for the in situ relative efficiency calibration. The whole efficiency curve was obtained by the least square fitting

method. The fitted efficiency ratios were obtained using the following Eq. ([3\)](#page-5-3) [\[14](#page-8-8)[–16](#page-8-10)].

$$
\ln(\varepsilon_{\gamma}) = \sum_{i=0}^{2} A_i \left[\ln(E_{\gamma}) \right]^i \tag{3}
$$

where ε_{γ} is the in situ detection efficiency and E_{γ} is the gamma ray energy. A_i 's are the coefficients of the second order polynomial. For both SL-1 and jarosite samples, a set of nine such linear equations were iterated for the determination of the coefficients. A typical plot of in situ relative detection efficiency for $SL-1$ is shown in Fig. [6.](#page-6-0)

Validation of *f* **and** *α* **and its application towards the sample analysis**

The elemental concentration ratio of 11 elements present in the SL-1 were obtained with respect to Sc by k_0 -based IM-NAA using the above established values of f and α . The relative concentrations were converted into the absolute concentrations by using certifed value of the Sc (Table [3](#page-6-1)). The comparison of the calculated values with that of the certifcate values was accomplished by calculating the % deviation and *Z*-score value. The following Eq. ([4\)](#page-5-4) was used to calculate the *Z*-score.

$$
Z\text{-score} = \frac{x_i - x_R}{\sigma_R} \tag{4}
$$

where x_i is the measured elemental concentration in the present work, x_R is the certified elemental concentration and σ_R is the uncertainty in the elemental concentration of the certifcate values of SL-1 at 95% confdence level. The % deviations for the elements determined in SL-1 with respect

a Cd-ratio method using dual monitors

^bCd-cover triple monitor method

 $DT-1$

Fig. 6 The variation of in situ relative detection efficiency with the gamma energy for SL-1

to certified values were within $\pm 5\%$ for most of the elements except for Na (11.8%), Th (8.6%) and As (6.5%) and *Z*-score values at the 95% confidence level were within ± 1 except for Na (2.00), Th (1.20) and Fe (1.18). The accuracy in the elemental concentrations in the SL-1 validates the above characterized irradiation input parameters by using IM-NAA method. The possible interference from the fssion product of the Th towards the calculation of the amount of La and Eu in the jarosite samples is negligible owing to the too low fast neutron fux at the irradiation location and Th content in the samples.

The relative elemental concentration of 14 elements present in the jarosite samples were obtained with respect

Fig. 7 The variation of in situ relative detection efficiency with the gamma energy for the jarosite sample

to Sc using in situ relative detection efficiency calibration of the sample as shown in Fig. [7.](#page-6-2) The relative concentrations were converted into their absolute concentrations by using the concentration of Sc in the same samples obtained by the relative NAA method (Table [4](#page-7-1)).

The elemental concentrations in jarosite samples vary from ppm to percentage levels. The major elements were found to be iron (15 and 26%), zinc (2.9 and 4.8%) and Na (1 and 2.2%) as shown in the Fig. [8](#page-7-2) whereas, the toxic and heavy elements such as arsenic, cadmium, antimony, copper, silver, tungsten etc. were present in trace to minor levels varying from 4 ppm to 0.4%. The presence of these toxic elements makes these jarosites hazardous thus demanding an appropriate disposal protocol.

Table 3 Measured elemental concentration in SL-1 by k_0 based IM-NAA method

a Certifed value

Table 4 Elemental concentrations in jarosite samples obtained using k_0 based IM-NAA method

^aObtained using the absolute concentration of Sc which was determined by relative method

Fig. 8 Concentration (%) of major elements in jarosite samples

Conclusions

The dry tube positions of KAMINI reactor was characterized towards the development of k_0 -based IM-NAA. The axial fux profle was obtained for the DT-1 and DT-2 and observed that the maximum neutron fux in DT-1 was $(1.02 \pm 0.02)E+10$ cm⁻² s⁻¹ at 368 mm and (4.90 ± 0.30) E+7 cm⁻² s⁻¹ at 300 mm in DT-2, the position measured from its bottom. The ƒ value for DT-1 at this location of maximum neutron-flux was found to be 144 ± 7 indicating the presence of more than 99% of thermal neutrons component. The α value of DT-1 was found to be -0.289 ± 0.010 indicating the existence of a hard epithermal neutron spectrum. The size of the DT-1 is sufficiently large and can therefore accommodate samples of varying sizes from mg to kg. An IAEA CRM SL-1 was analyzed by IM-NAA method in DT-1 and the elemental concentrations established using this method were found to be in good agreement with the certifed values. The deviations were within \pm 5% while the *Z*-score was found to be \pm 1 for most of the elements. The jarosite samples were analyzed and the elemental profle consisting of 14 elements was reported. The elemental profle in jarosite samples obtained using IM-NAA indicates that these residues contain signifcant quantity of compounds of iron, zinc, cadmium, arsenic etc. The present DT-1 position of KAMINI reactor would be used in the future for the assay of irregular geometry samples in the felds of archaeological, forensic, nuclear, metallurgical etc. using k_0 -based IM-NAA method. Similar study for the characterization of the other dry tube facility (DT-2) is in progress.

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