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Implementation of the k_0-standardization method for analysis of geological samples at the Neutron Activation Analysis Laboratory, São Paulo, Brazil

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Abstract The Neutron Activation Analysis Laboratory (LAN-IPEN) has been analysing geological samples for many years with the INAA comparative method, for geochemical and environmental research. This study presents the results obtained in the implementation of the k_0-standardization method at LAN-IPEN, for geological samples analysis, by using the program k_0-IAEA, provided by the International Atomic Energy Agency (IAEA). The thermal to epithermal flux ratio f and the shape factor α of the epithermal flux distribution of the IEA-R1 nuclear reactor of IPEN were determined for the pneumatic irradiation facility and one selected irradiation position, for short and long irradiations, respectively. To obtain these factors, the "bare triple-monitor" method with ^{197}Au–^{96}Zr–^{94}Zr was used. In order to validate the methodology, the geological reference materials basalts JB-1 (GSJ) and BE-N (IWG-GIT), andesite AGV-1 (USGS), granite GS-N (ANRT), SOIL-7 (IAEA) and sediment Buffalo River Sediment (NIST–BRS-8704), which represent different geological matrices, were analysed. The concentration results obtained agreed with assigned, with bias <10 % except for Zn in AGV-1. The *U-score* test showed that all results, except Mg in JB-1, are within 95 % confidence interval. These results indicate excellent possibilities of using this parametric method at the LAN-IPEN for geochemical and environmental studies.

Keywords Instrumental neutron activation analysis · k_0-INAA · Neutron flux parameters · Geological samples

Introduction

The Neutron Activation Analysis Laboratory at IPEN (LAN-IPEN) has been analyzing geological samples for decades, using the comparative method of analysis by neutron activation at the IEA-R1 nuclear research reactor. By using this analytical technique, rare earths and trace elements were analysed in rocks [1–5], for geochemical and petrogenetic studies, and heavy metals and other elements of interest in sediments [6, 7] and soils [8, 9], for environmental geochemical studies. In this method, samples and standards are irradiated simultaneously with neutrons under the same conditions. Elemental concentrations are calculated by comparison of the activities of the gamma-rays from the sample and standard. This procedure requires the preparation of element standards, which is very laborious and time consuming. Furthermore, some elements present in the sample can not be analyzed due to the absence of a corresponding element standard. The k_0-NAA method, developed by the Institute of Nuclear Sciences, Gent, Belgium [10], has been increasingly used, as it requires only a single comparator such as ^{197}Au for multielement determination instead of the multielement standards required in the relative method. Some attempts to introduce the k_0-NAA at LAN-IPEN have been made [11] but, actually, the INAA comparative method is still used at LAN-IPEN. Although this standardization method is considered to be one of the most accurate methods of INAA, more and more neutron activation laboratories in Brazil and in other countries all over the world are using the

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k_0-NAA method, due to the improvement of the analytical procedure and analytical time while still providing quite accurate analytical results [12–17].

Many INAA laboratories developed k_0 software using different approaches. The k_0-IAEA program was developed to be distributed free of charge by the IAEA, in order to assist users of the k_0-approach in NAA to harmonize their results, and to encourage NAA laboratories to adopt the k_0-standardization method. The mathematical approach used and how k_0 data catalogue along with additional information on coincidence and sum peaks are incorporated in the program are described by Rossbach et al. [18]. The k_0-IAEA program has been used successfully worldwide [18, 19].

The IEA-reactor has a pneumatic station facility adequate for short time irradiations, providing accurate irradiation and decay times. Samples are sent to irradiation pneumatically and, after irradiation, are sent back automatically to the station. Samples can then be measured in gamma-ray spectrometers located in counting rooms next to the pneumatic station which allow immediate measurement of the induced activity.

The objective of the present study was to assess the applicability of the k_0-INAA method with the k_0-IAEA software using the pneumatic station facility and a selected irradiation position in the IEA-R1 research nuclear reactor, to analyse trace elements in geological matrices. For this purpose, the characterization of the neutron flux parameters in the IEA-R1 nuclear reactor was performed. The methodology accuracy and precision were evaluated by analyzing the certified geological reference materials basalt BE-N (IWG-GIT), basalt JB-1 (USGS), andesite AGV-1 (USGS), granite GS-N (IWG-GIT), soil SOIL-7 (IAEA) and sediment Buffalo River Sediment (NIST–BRS-8704), which represent different geological matrices.

Experimental

Irradiation and counting

The IEA-R1 is a nuclear research reactor (5 MW) immersed in a pool containing 273 m^3 of demineralized water. The pool is about 9 m deep by 3 m wide and 11 m long. The reactor core is located 6.9 m from the surface of the pool, and has the form of a cobble stone composed by 20 standard fuel elements, four control fuel elements and about 25 reflectors. For sample irradiation purposes, IEA-R1 has seven manually loaded irradiation positions (out-core), which can be used for long irradiations (Fig. 1).

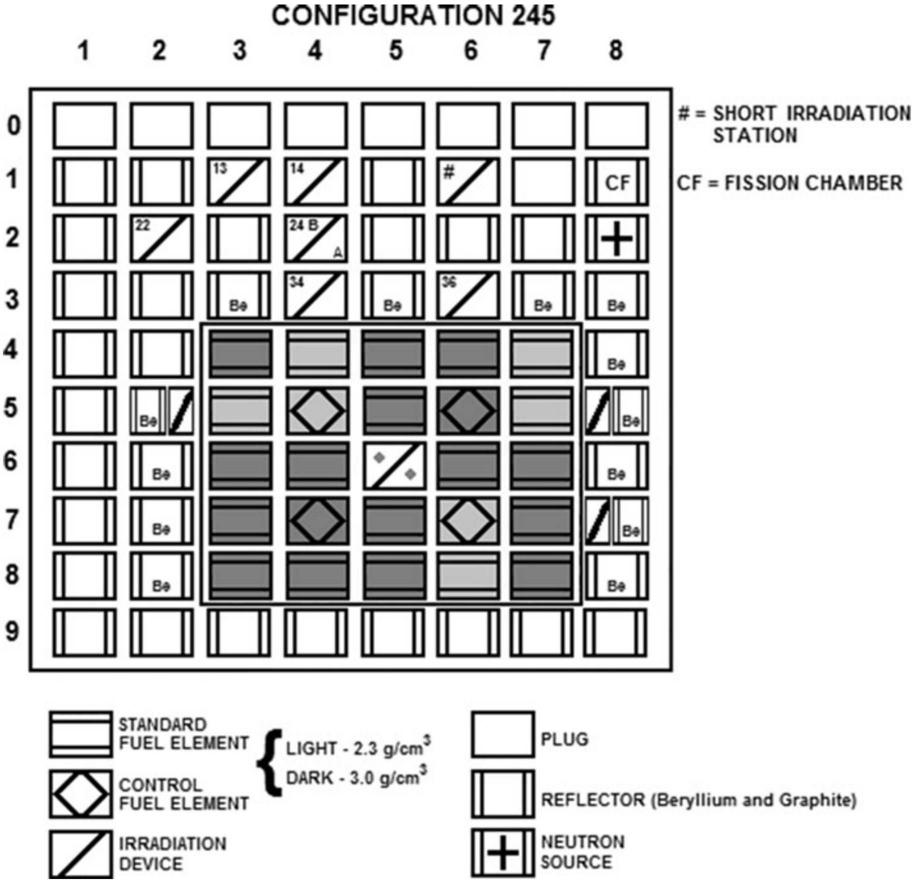

Fig. 1 IEA-R1 nuclear reactor configuration

Fig. 2 Efficiency curve of the Ge detector at a sample-detector distance of 9 cm

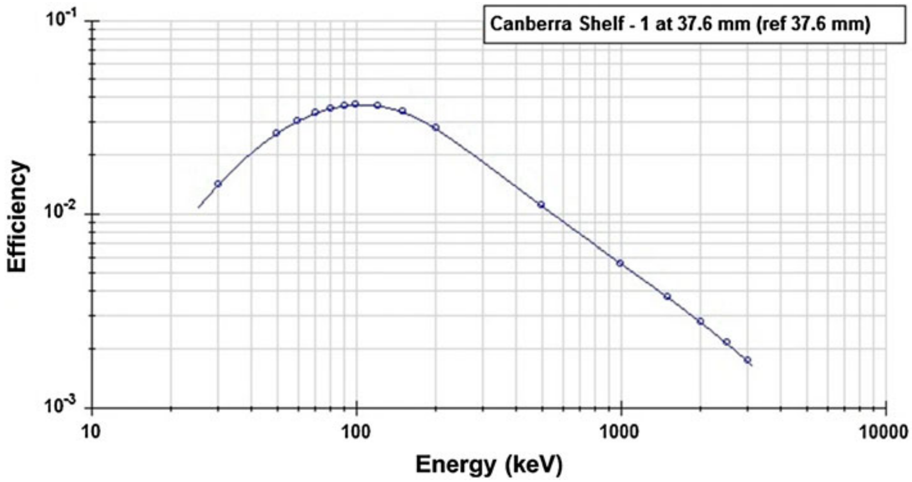

Table 1 Flux parameters for irradiation in the Pneumatic Station of IEA-R1

Parameters	Values
Thermal neutron flux, Φ_{th} (m^{-2} s^{-1})	(8.6 ± 0.2) × 10^{16}
Neutron temperature, T_n (K)	310.00 ± 0.01
Thermal to epithermal flux ratio, *f*	44 ± 6
Deviation of the epithermal neutron flux distribution from the ideal 1/E law, α	−0.08 ± 0.02

Table 2 Flux parameters for irradiation in the 24B/2 irradiation position of IEA-R1

Parameters	Values
Thermal neutron flux, Φ_{th} (m^{-2} s^{-1})	(6.3 ± 0.09) × 10^{16}
Neutron temperature, T_n (K)	310 ± 5
Thermal to epithermal flux ratio, *f*	35 ± 1
Deviation of the epithermal neutron flux distribution from the ideal 1/E law, α	−0.049 ± 0.006

A fast pneumatic system station specifically designed for INAA of short-lived and medium-lived nuclides was installed and is used to perform, with a transfer time of ~12 s, short irradiations up to 30 min. Samples (up to 1 g) are accurately weighed in polyethylene bags, are sealed in polyethylene capsules (rabbits) and fed into the loading/reception station connected to a terminal station by an air supply line.

For long irradiations (more than 30 min), the samples (up to 1 g) are accurately weighed in polyethylene bags, enclosed in aluminum capsules (rabbits) and irradiated at the 24B/2 irradiation position in the IEA-R1 reactor.

The irradiation time for short irradiation at the pneumatic station was of 10 s and for the long irradiation of 8 h.

The measurements of the induced gamma-ray activity were carried out using a GX20190 hyperpure Ge detector. The multichannel analyzer was a 8192 channel Canberra S-100 plug-in-card in a PC computer. The resolution (FWHM) of the system was 1.90 keV for the 1,332 keV gamma-ray of ^{60}Co. Decay and counting times were 5 min for the 10 s irradiation. Another measurement was taken after 1 h. Two series of measurements were performed after the 8 h irradiation: the first from 5 to 7 days after irradiation and the second after 15–20 days of decay.

For calibration (energy and efficiency) of the HPGe detector, standard radioactive point sources of ^{137}Cs and ^{152}Eu, provided by Nuclear Metrology Laboratory, IPEN-CNEN/SP, were used. Figure 2 shows the full-energy peak efficiency curve for the coaxial HPGe detector fitted using the k_0-IAEA software, at a sample-detector distance of 9 cm.

Flux parameters

The parameters *f* and α for the short and long irradiation facilities of the IEA-R1 nuclear reactor were determined by using the “bare triple-monitor” method with ^{197}Au–^{96}Zr–^{94}Zr and irradiating a set consisting of ~20 mg of a 0.127 mm thick Zr foil (purity 99.5 %) together with 3 mg of Al-0, 1 % Au wire (Certified Reference Material IRMM-530R) for 2 min. The neutron temperature was evaluated by irradiating ~3 mg Lu foil (purity 99.963 %).

The flux parameters determined for the pneumatic station of IEA-R1 and for the 24B/2 irradiation position at the IEA-R1 reactor are in Tables 1 and 2, respectively.

Reference materials analysis

Aliquots of 50–100 mg of the reference materials basalt JB-1 (GSJ) and BE-N (IWG-GIT), granite GS-N (IWG-GIT), andesite AGV-1 (USGS), soil SOIL-7 (IAEA) and sediment Buffalo River Sediment (NIST–BRS-8704) were irradiated at the pneumatic station and at the selected

Table 3 Results obtained for the reference materials AGV-1 (USGS), BE-N (IWG-GIT) and GS-N (ANRT)

	AGV-1 (mg/kg)				BE-N (mg/kg)				GS-N (mg/kg)			
	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*
Al (%)	9.08 ± 0.68	9.18 ± 0.25	1.1	0.14	5.33 ± 0.04	5.42 ± 0.18	1.7	0.49	7.76 ± 0.05	7.83 ± 0.27	0.9	0.25
Ca (%)	–	–	–	–	9.92 ± 0.16	9.61 ± 1.27	−3.1	0.24	–	–	–	–
Fe (%)	4.73 ± 0.38	4.74 ± 0.08	0.2	0.03	8.98 ± 0.04	9.04 ± 0.19	0.7	0.31	2.62 ± 0.03	2.68 ± 0.05	2.3	1.03
K (%)	2.42 ± 0.74	2.45 ± 0.13	1.2	0.04	1.15 ± 0.02	1.21 ± 0.07	5.2	0.82	3.84 ± 0.05	3.82 ± 0.16	−0.5	0.12
Mg (%)	0.92 ± 0.19	0.93 ± 0.15	1.1	0.04	7.93 ± 0.05	7.59 ± 0.25	−4.3	1.33	1.39 ± 0.05	1.58 ± 0.10	13.7	1.70
Mn	710 ± 100	731 ± 29	3.0	0.20	1,550 ± 30	1,470 ± 39	−5.2	1.63	430 ± 30	387 ± 20	−10.0	1.19
Na (%)	3.16 ± 0.24	3.09 ± 0.52	−2.2	0.12	2.36 ± 0.03	2.35 ± 0.07	−0.4	0.13	2.8 ± 0.04	2.75 ± 0.07	−1.8	0.62
Ti (%)	0.63 ± 0.10	0.59 ± 0.03	−6.3	0.38	1.56 ± 0.02	1.54 ± 0.06	−1.3	0.32	0.41 ± 0.03	0.37 ± 0.02	−9.8	1.11
As	–	–	–	–	1.8 ± 0.03	1.7 ± 0.4	−5.6	0.25	–	–	–	–
Ba	1,230 ± 32	1,221 ± 72	−0.7	0.11	–	–	–	–	–	–	–	–
Co	15 ± 2.4	15.2 ± 0.3	1.3	0.08	60 ± 2	60.4 ± 2.2	0.7	0.13	65 ± 4	67.7 ± 1.2	4.2	0.65
Cr	10 ± 6	10.4 ± 1.6	4.0	0.06	360 ± 12	362 ± 7	0.6	0.14	55 ± 4	51 ± 2	−7.3	0.89
Cs	1.3 ± 0.2	1.26 ± 0.23	−3.1	0.13	–	–	–	–	5.4 ± 0.3	5.5 ± 0.3	1.9	0.24
Ga	20 ± 6	21.6 ± 1.0	8.0	0.26	–	–	–	–	–	–	–	–
Hf	5.1 ± 0.8	4.9 ± 0.5	−3.9	0.21	5.6 ± 0.16	5.5 ± 0.2	−1.8	0.39	6.2 ± 0.3	6.0 ± 0.5	−3.2	0.34
Rb	67 ± 2	68 ± 3	1.5	0.28	47 ± 2	50 ± 3	6.4	0.83	185 ± 5	189 ± 11	2.2	0.33
Sb	4.3 ± 0.8	4.1 ± 0.2	−4.7	0.24	–	–	–	–	–	–	–	–
Sc	12 ± 2	12.1 ± 0.2	0.8	0.05	22 ± 1.5	22.5 ± 1.1	2.3	0.27	7.3 ± 0.4	7.25 ± 0.42	−0.7	0.09
Sr	660 ± 18	625 ± 58	−5.3	0.58	1,370 ± 25	1,355 ± 38	−1.1	0.33	570 ± 19	553 ± 101	−3.0	0.17
Ta	0.90 ± 0.18	0.82 ± 0.10	−8.9	0.39	5.7 ± 0.4	5.9 ± 0.3	3.5	0.40	2.6 ± 0.2	2.7 ± 0.2	3.8	0.35
Th	6.5 ± 1.0	6.3 ± 0.5	−3.1	0.18	10.4 ± 0.65	10.3 ± 0.4	−1.0	0.13	41 ± 3.5	37.9 ± 1.0	−7.6	0.85
U	1.92 ± 0.30	1.82 ± 0.61	−5.2	0.15	2.4 ± 0.18	2.37 ± 0.94	−1.2	0.03	7.5 ± 0.8	7.8 ± 1.2	4.0	0.21
V	120 ± 22	121 ± 4	0.8	0.04	235 ± 10	230 ± 6	−2.1	0.43	65 ± 8	64.9 ± 3.8	−0.2	0.01
W	–	–	–	–	–	–	–	–	450 ± 63	490 ± 12	8.9	0.62
Zn	88 ± 18	98 ± 7	11.4	0.52	120 ± 13	126 ± 9	5.0	0.38	–	–	–	–
Zr	227 ± 36	239 ± 74	5.3	0.15	–	–	–	–	–	–	–	–
La	38 ± 4	37.8 ± 3.1	−0.5	0.04	82 ± 1.5	80 ± 4	−2.4	0.47	75 ± 2.7	69.5 ± 1.5	−7.3	1.78
Ce	67 ± 12	69 ± 5	3.0	0.15	152 ± 4	151.7 ± 4.2	−0.2	0.05	135 ± 7	122 ± 3	−9.6	1.71
Nd	33 ± 6	33.5 ± 5.1	1.5	0.06	67 ± 1.5	69 ± 5	3.0	0.38	49 ± 1.5	46 ± 4	−6.1	0.70
Sm	5.9 ± 0.8	6.0 ± 0.3	1.7	0.12	12.2 ± 0.3	12.9 ± 0.7	5.7	0.92	7.5 ± 0.22	6.9 ± 0.5	−8.0	1.10
Eu	1.6 ± 0.2	1.52 ± 0.06	−5.0	0.38	3.6 ± 0.18	3.42 ± 0.11	−5.0	0.85	1.7 ± 0.06	1.61 ± 0.09	−5.3	0.83
Tb	0.70 ± 0.2	0.63 ± 0.05	−10.0	0.34	1.3 ± 0.1	1.24 ± 0.10	−4.6	0.42	–	–	–	–
Dy	3.6 ± 0.8	3.73 ± 0.19	3.6	0.16	6.4 ± 0.2	6.5 ± 1.1	1.6	0.09	3.1 ± 0.3	3.2 ± 0.3	3.2	0.24
Yb	1.72 ± 0.4	1.70 ± 0.13	−1.2	0.05	1.8 ± 0.2	1.79 ± 0.13	−0.6	0.04	1.4 ± 0.15	1.32 ± 0.13	−5.7	0.40

X_{Ass} assigned value, X_{Exp} experimental result, (–) not determined

Table 4 Results obtained for the reference materials JB-1(GSJ), BR (NIST) and Soil-7 (IAEA)

	JB-1 (mg/kg)				BR (mg/kg)				Soil-7 (mg/kg)			
	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*
Al	7.69 ± 0.20[b]	7.67 ± 0.16	−0.3	0.08	6.10 ± 0.18[b]	6.11 ± 0.21	0.2	0.04	47,000 ± 8289[a]	47,732 ± 1,991	1.6	0.09
Ca	6.61 ± 0.12[b]	6.25 ± 0.28	−5.4	1.18	2.641 ± 0.083[b]	2.55 ± 0.09	−3.4	0.74	163,000 ± 23576[a]	156,224 ± 10,558	−4.2	0.26
Fe	1.63 ± 0.13[b]	1.63 ± 0.23	0.1	0.01	3.97 ± 0.10[b]	3.97 ± 0.49	−0.01	0.001	25,700 ± 1852[a]	25,661 ± 625	−0.2	0.02
K	1.19 ± 0.07[b]	1.17 ± 0.08	−1.7	0.19	2.001 ± 0.041[b]	2.02 ± 0.06	0.9	0.26	12,100 ± 1770[a]	11,940 ± 777	−1.3	0.08
Mg	4.65 ± 0.16[b]	4.20 ± 0.14	−9.7	2.12	1.200 ± 0.020[b]	1.18 ± 0.08	−1.7	0.24	11,300 ± 947[a]	12,194 ± 893	7.9	0.69
Mn	0.118 ± 0.16[b]	0.116 ± 0.027	−1.7	0.01	544 ± 21	555 ± 35	2.0	0.27	631 ± 68[a]	638 ± 23	1.1	0.10
Na	2.05 ± 0.12[b]	2.17 ± 0.39	5.9	0.29	0.553 ± 0.015[b]	0.55 ± 0.02	−0.5	0.12	2,400 ± 282[a]	2,396 ± 419	−0.2	0.01
Ti	0.79 ± 0.06[b]	0.78 ± 0.08	−1.3	0.10	0.457 ± 0.020[b]	0.45 ± 0.02	−1.5	0.25	3,000 ± 1141[a]	3,022 ± 223	0.7	0.02
As	2.33 ± 0.31	2.45 ± 0.32	5.2	0.27	–	–	–	–	13.4 ± 2.1	14.11 ± 0.09	5.3	0.34
Ba	493 ± 46	475 ± 73	−3.7	0.21	413 ± 13	407 ± 49	−1.5	0.12	159 ± 73[a]	158 ± 6	−0.6	0.01
Co	38.2 ± 5.2	39 ± 4	2.1	0.12	13.57 ± 0.43	13.67 ± 0.28	0.7	0.19	8.9 ± 2.4	9.0 ± 0.4	1.1	0.04
Cr	425 ± 63	428 ± 7	0.7	0.05	121.9 ± 3.8	121 ± 3	−0.7	0.19	60 ± 40	63 ± 3	5.0	0.07
Cs	1.23 ± 0.19	1.27 ± 0.11	3.3	0.18	5.83 ± 0.12	5.85 ± 0.26	0.3	0.07	5.4 ± 1.4	5.0 ± 0.4	−7.4	0.27
Hf	3.31 ± 0.56	3.30 ± 0.19	−0.3	0.02	8.4 ± 1.5	8.2 ± 0.9	−2.4	0.11	5.1 ± 0.5	5.0 ± 0.4	−2.0	0.16
Mo	27.4 ± 10.2	29.2 ± 3.6	6.6	0.17	–	–	–	–	–	–	–	–
Rb	41.3 ± 5.1	44.4 ± 3.4	7.5	0.51	–	–	–	–	51 ± 11	50.7 ± 3.2	−0.6	0.03
Sb	0.28 ± 0.065	0.295 ± 0.024	5.4	0.22	3.07 ± 0.32	2.97 ± 0.16	−3.3	0.28	1.7 ± 0.4	1.65 ± 0.13	−2.9	0.13
Sc	27.5 ± 1.95	27.8 ± 0.7	1.1	0.14	11.26 ± 0.19	11.80 ± 0.34	4.8	1.39	8.3 ± 2.4	8.5 ± 0.9	2.4	0.08
Sr	444 ± 29	448 ± 87	0.9	0.04	–	–	–	–	–	–	–	–
Ta	2.93 ± 0.79	2.80 ± 0.18	−4.4	0.16	–	–	–	–	0.8 ± 0.3	0.72 ± 0.10	−10.0	0.25
Th	9.3 ± 0.71	9.10 ± 0.24	−2.2	0.27	9.07 ± 0.16	8.62 ± 0.21	−5.0	1.70	8.2 ± 2.2	7.7 ± 0.2	−6.1	0.23
U	1.67 ± 0.28	1.7 ± 0.4	1.8	0.06	3.09 ± 0.13	3.13 ± 0.29	1.3	0.13	2.6 ± 1.0	2.8 ± 0.3	7.7	0.19
V	211 ± 20	208 ± 6	−1.4	0.14	94.6 ± 4	100.6 ± 4.7	6.3	0.97	66 ± 14	67 ± 4	1.5	0.07
W	17.1 ± 3.5	16.1 ± 1.2	−5.8	0.27	–	–	–	–	–	–	–	–
Zn	85.2 ± 9.9	90 ± 5	5.6	0.43	408 ± 15	419 ± 12	2.7	0.57	104 ± 20	107 ± 6	2.9	0.14
Zr	–	–	–	–	–	–	–	–	185 ± 19	199 ± 6	7.6	0.70
La	38.6 ± 4.3	37.5 ± 0.6	−2.8	0.25	–	–	–	–	28 ± 2	28.0 ± 0.7	0.1	0.01
Ce	67.8 ± 6.8	69 ± 3	1.8	0.16	66.5 ± 2	64.4 ± 2.5	−3.2	0.66	61 ± 12	59 ± 3	−3.3	0.16
Nd	26.8 ± 2.3	28 ± 3	4.5	0.32	–	–	–	–	30 ± 6	28.4 ± 2.5	−5.3	0.25
Sm	5.13 ± 0.50	5.60 ± 0.34	9.2	0.78	–	–	–	–	5.1 ± 0.6	5.4 ± 0.4	5.9	0.42
Eu	1.49 ± 0.15	1.47 ± 0.13	−1.3	0.10	1.31 ± 0.038	1.21 ± 0.06	−7.6	1.41	1 ± 0.3	0.95 ± 0.11	−5.0	0.16
Tb	–	–	–	–	–	–	–	–	0.6 ± 0.3	0.62 ± 0.08	3.3	0.06
Dy	4.14 ± 0.38	4.25 ± 0.55	2.7	0.16	–	–	–		3.9 ± 0.4	3.8 ± 0.8	−2.6	0.11

Table 4 continued

	JB-1 (mg/kg)				BR (mg/kg)				Soil-7 (mg/kg)			
	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*	$X_{Ass} \pm U_{Ass}$	$X_{Exp} \pm U_{Exp}$	Bias (%)	*U-score*
Yb	2.13 ± 0.26	2.08 ± 0.13	−2.3	0.17	–	–	–	–	2.4 ± 0.6	2.28 ± 0.12	−5.0	0.20
Lu	0.31 ± 0.029	0.30 ± 0.03	−3.2	0.24	–	–	–	–	0.3 ± 0.2[a]	0.29 ± 0.05	−3.3	0.05

[a] Information values

[b] Reference concentration values in mass fraction (%)

X_{Ass} assigned value, X_{Exp} experimental result, (–) not determined

irradiation position of the IEA-R1 reactor, and the induced gamma-activities were measured using the calibrated gamma-spectrometer. The elements Al, Dy, Mg, Mn, V, Ti and W were determined in the short-irradiation and the other elements in the long irradiation.

Results and discussion

For a statistical accuracy evaluation, the U-*score* parameter [20] was used. The *U-score* is defined by the following equation:

$$U_{score} = \left|X_{Ass} - X_{Exp}\right| / \left\{ (U_{Ass})^2 + (U_{Exp})^2 \right\} \quad (1)$$

where the numerator gives the absolute difference between the experimental result (X_{Exp}) and the assigned value (X_{Ass}) of elemental concentration, and U_{Ass} and U_{Exp} are the uncertainties of the recommended and experimental mass fraction, respectively, within an expanding 95 % confidence interval. The experimental uncertainties used in the calculation were the reported uncertainties by the k_0-IAEA program exclusively, that is the accuracies of the measured concentrations. In k_0-IAEA software, uncertainty is calculated by considering uncertainty sources such as peak area, k_0 constant, neutron flux, *f* and alpha.

Tables 3 and 4 present the analytical results of six replicates as well as assigned values with confidence level of 95 %. Experimental values and assigned values were statistically compared by Relative Bias (in %) and *U-score* test. For most of the elements, the Relative Bias was within 0–10 % (except for Zn in AGV-1 and Mg in GS-N). It is important to note that the results obtained were randomly above and below the assigned values, showing that there is no systematic error. The *U-score* showed that all the results, except the value for Mg in JB-1, are within 95 % of confidence interval (if the limiting value for the *U* test parameter is 1.96 for a level of probability at 95 %). The result of Mg in JB-1 may be accepted in a confidence interval of 99.5 % (*U-score* < 2.58).

Conclusions

Six different geological reference materials were irradiated and analyzed by k_0-INAA method by using the k$_0$-IAEA software, in order to validate the k_0-IAEA method at IPEN for geological sample analysis. The statistical tests applied were Relative Bias (in %) and *U-score*. The results showed, with exception of Mg in JB-1, Relative Bias within 10 % and *U-scores* < 1.96 ($P = 95$ % or $\pm 2\sigma$), demonstrating that the results are within 95 % confidence interval.

The results obtained show the good performance of the k_0-NAA method with k_0-IAEA software used at LAN-IPEN to analyse different types of geological matrices with a very wide range concentration, providing reliable results for up to 30 elements. The results indicate that the implementation of the k_0-NAA at the Neutron Activation Laboratory LAN-IPEN should increase the analytical potential of the laboratory for geochemical and environmental studies, while maintaining the quality of the data.

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