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k_0-INAA of biological matrices at IPEN neutron activation analysis laboratory, São Paulo, using the k0_IAEA software

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Abstract This study presents the results obtained in the application of the k_0-standardization method at the Neutron Activation Analysis Laboratory at IPEN (LAN-IPEN), for biological sample analysis, by using the k0_IAEA software, 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. To evaluate the accuracy of the results, bias (%) and E_n-number test were applied to the results obtained in the analysis of the biological reference materials NIST SRM 1547 peach leaves, INCT-MPH-2 mixed polish herbs and NIST SRM 1573a tomato leaves. Bias (%), for most elements, ranged from 0 to 30 %, in relation to certified values. E_n-number values showed that, with few exceptions (Na in NIST SRM 1547 and NIST SRM 1573a, and Al, Cr, Sc and Zn in INCT-MPH-2), the results were within a 95 % CI. These results pointed to the possibility of using the k_0-INAA method with the k0_IAEA software for analysis of biological samples at LAN-IPEN.

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Introduction

Comparative neutron activation analysis at the IEA-R1 nuclear research reactor has been used by the Neutron Activation Analysis Laboratory of IPEN (LAN-IPEN) for studies in different fields of research [1–4]. Due to the advantages of the k_0-INAA method in relation to the comparative method, such as the use of a single comparator (usually ^{197}Au) for multielemental determination instead of the multi-standards required in the comparative method, and to the improvement of the k_0-INAA parameters and software, this parametric method has been increasingly used in neutron activation laboratories in Brazil and in other countries [5–9].

The k0_IAEA software was provided free of charge by the IAEA, in order to assist and encourage k_0-NAA users. The mathematical approach used in the program is described by Rossbach et al. [10]. The k0_IAEA program has been used successfully worldwide [11]. As such, the k_0-INAA method with the k0_IAEA software has also been successfully applied at LAN-IPEN to analyze geological samples [12]. Furthermore, studies involving plants as biomonitors in metal atmospheric monitoring have also been important in research projects developed by LAN-IPEN [3, 13].

With this in mind, the objective of the present study was to assess the applicability of the k_0-INAA method with the k0_IAEA software (version 5.22), using the pneumatic station facility and a selected irradiation position at the IEA-R1 research nuclear reactor, to analyze trace elements in plant matrices.

For this purpose, the characterization of the neutron flux parameters of the IEA-R1 nuclear reactor was performed. Methodology trueness was evaluated by comparing the results with certified values (bias, %) and applying the E_n-number test to the results obtained in the analysis of the biological reference materials NIST SRM 1547 peach leaves, INCT-MPH-2 mixed polish herbs and SRM 1573a tomato leaves.

Experimental

Irradiation facilities

The IEA-R1 is a nuclear research reactor (5 MW) immersed in a 273 m^3 demineralized water pool. For sample irradiation purposes, IEA-R1 has 7 manually loaded irradiation positions (out-core), which can be used for long irradiations. 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 approximately 12 s, short irradiations up to 5 min. Samples are accurately weighed in polyethylene bags, 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, the samples are accurately weighed in polyethylene bags, enclosed in aluminum capsules (rabbits) and irradiated at the IEA-R1 reactor. The selected irradiation position at the IEA-R1 reactor for long irradiations in this study was the 24B/2.

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 40 mg of a Zr foil (Aldrich Chemical Company, 0.25 mm thick, purity 99.8 %) together with 8 mg of Al-0.1 %Au wire (Certified Reference Material IRMM-530R). The irradiation time for short irradiation at the pneumatic station was of 2 min and for the long irradiation at the 24B/2 position was of 4 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 1332 keV gamma-ray of ^{60}Co.

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 1 shows the full-energy peak efficiency curve for the coaxial HPGe detector fitted using the k0_IAEA software, at a sample-detector distance of 94.8 mm.

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

Reference materials analysis

Aliquots of 150 mg of the reference materials NIST SRM 1547 peach leaves, INCT-MPH-2 mixed polish herbs and NIST SRM 1573a tomato leaves and standards (Al-0.1 %Au alloy) sealed in plastic bags were irradiated at the pneumatic station and at the selected irradiation position of the IEA-R1 reactor, and the induced gamma-activities were measured using the calibrated gamma-spectrometer. Tables 3 and 4 show the decay time, counting time and distance between sample-detector of the samples.

Results and discussion

For a statistical accuracy evaluation, the E_n-number [14] was used. The E_n-number is defined by the following equation:

$$E_n = \frac{X_{Lab} - X_{Cert}}{\sqrt{U_{Lab}^2 + U_{Cert}^2}}$$

where the numerator gives the absolute difference between the experimental result (X_{Lab}) and the assigned value (X_{Cert}) of elemental concentration, and U_{Cert} and U_{Lab} are the expanded uncertainties ($k = 2$) of the recommended and experimental mass fraction, respectively. Expanded Laboratory uncertainty with a coverage factor of $k = 2$ is calculated as follows:

$$U_{Lab} = 2 \cdot U_{Lab\_Comb} = 2 \cdot \sqrt{(SD)^2 + u_{method}^2}$$

where SD is the standard deviation of independent measurements and u_{method} is the estimated uncertainty of the k0_IAEA software (3.5 % with a coverage factor $k = 1$).

In k0_IAEA software, uncertainty is calculated by considering uncertainty sources such as literature values for $T_{1/2}$, $\bar{E}_r$, Q_0 and k_0, the irradiation, decay and measuring times, true-coincidence factor (COI), Au composition in Al-0.1 %Au alloy, masses of sample and standard, neutron flux parameters (f, α, fast flux and neutron temperature) and detection efficiency.

Tables 5, 6 and 7 present the analytical results of six replicates (if not stated otherwise) as well as assigned values with confidence level of 95 %. Experimental values and assigned values were statistically compared (bias, %, and E_n-numbers). Figures 2, 3 and 4 show the ratios to

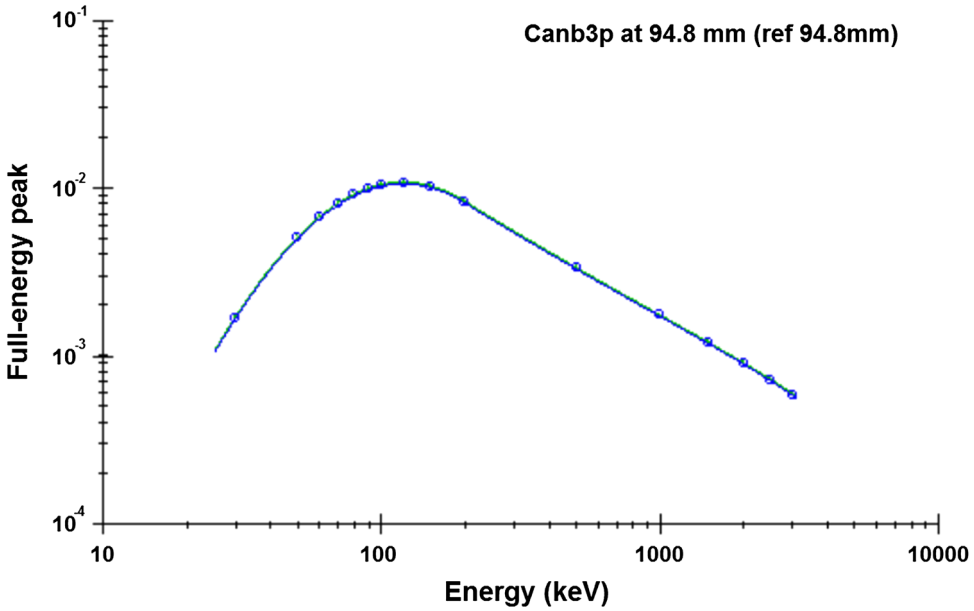

Fig. 1 Efficiency curve of the Ge detector at a sample-detector distance of 94.8 mm. Fitting coefficients: $a1 = -1.007264$; $a2 = -6.331193$; $a3 = -0.9314355$; $a4 = 7.651488$; $a5 = -1.160507E-05$; $a6 = -2.25336$

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

Parameters	Values
Thermal neutron flux, Φ_{th} (m^{-2} s^{-1})	$(1.82 \pm 0.05) \times 10^{16}$
Fast neutron flux, Φ_{fast} (m^{-2} s^{-1})	$(3.66 \pm 0.37) \times 10^{15}$
Neutron temperature, T_n (K)	310 ± 5[a]
Thermal to epithermal flux ratio, f	35.6 ± 1.07
Deviation of the epithermal neutron flux distribution from the ideal $1/E$ law, α	0.0288 ± 0.0058

[a] Not determined in this experiment, taken as default value in the k0_IAEA software

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})	$(8.51 \pm 0.09) \times 10^{16}$
Fast neutron flux, Φ_{fast} (m^{-2} s^{-1})	$(4.00 \pm 0.20) \times 10^{15}$
Neutron temperature, T_n (K)	310 ± 5[a]
Thermal to epithermal flux ratio, f	62.75 ± 6.03
Deviation of the epithermal neutron flux distribution from the ideal $1/E$ law, α	0.034 ± 0.010

[a] Not determined in this experiment, taken as default value in the k0_IAEA software

certified and information values for the reference materials analyzed.

If only the certified values are considered, all elements, except Na in NIST SRM 1547, showed bias within 0–30 %. It is important to note that the results obtained were randomly above and below the assigned values, showing that there is no systematic error. For the sake of

Table 3 Decay time, counting time and distance between sample-detector in the short irradiations

	Decay time	Counting time (s)	Distance from HPGe detector (mm)
Reference material	5 min	240	94.8
Reference material	15 min	600	94.8
Reference material	30 min	900	94.8
Al-0.1 %Au	2 h	7,200	35.4
Reference material	12 h	10,800	35.4

Table 4 Decay time, counting time and distance between sample-detector in the long irradiations

	Decay time (days)	Counting time (s)	Distance from HPGe detector (mm)
Reference material	5–7	7,200	35.4
Al-0.1 %Au	5–7	3,600	35.4
Reference material	10–15	7,200	35.4

comparison, bias (%) was also calculated in relation to information values. It should be noted, nevertheless, that these values were considered as a guide, since they are not certified values. With few exceptions, the results obtained agreed with information values. The greatest discrepancies were Ce, Cr, Eu, Nd, Sc and Sm (long irradiation) in SRM 1547 and Th in SRM 1573a. It is important to observe, on the other hand, that the same elements that presented significant deviations from information values in SRM 1547 and SRM 1573a, showed better agreement in INCT-MTH-2, where these elements have certified values, although Cr and Sc still presented E_n values slightly above 1.

Table 5 Results obtained by k0_IAEA software for 6 replicates in mg kg^{-1} (dry mass basis), if not stated otherwise

NIST SRM 1547 peach leaves											
Element	X_{cert}	X^{a}_{Lab}	Bias (%)	CV (%)	E_n	Element	X_{cert}	X^{a}_{Lab}	Bias (%)	CV (%)	E_n
Al	249 ± 8	258 ± 17[b]	3.7	5.4	0.27	Mn	98 ± 3	95 ± 9[b]	−3.1	8.9	0.16
Ba	124 ± 4	127 ± 7[b]	2.2	4.6	0.18	Na	24 ± 2	45 ± 5[b]	85.4	11.0	1.96
Ba	124 ± 4	131 ± 14[c]	5.6	9.9	0.25	Nd	7[d]	9.7 ± 0.6[c]	38.1	5.3	*
Br	11[d]	10.8 ± 0.6[b]	−1.5	3.8	*	Rb	19.7 ± 1.2	20.7 ± 1.2[c]	5.0	4.6	0.37
Ca (%)	1.56 ± 0.02	1.52 ± 0.19[c]	−2.8	12.3	0.11	Sb	0.02[d]	0.022 ± 0.010[c]	8.3	45.4	*
Ce	10[d]	13.3 ± 0.7[c]	33.3	3.9	*	Sc	0.04[d]	0.055 ± 0.006[c]	37.5	10.0	*
Cl	360 ± 19	368 ± 31[b]	2.1	7.6	0.12	Sm	1[d]	1.1 ± 0.1[b]	10.0	10.0	*
Cr	1[d]	1.8 ± 0.2[c]	82.7	13.0	*	Sm	1[d]	1.45 ± 0.14[c]	45.2	8.8	*
Eu	0.17[d]	0.18 ± 0.01[b]	7.8	6.6	*	Sr	53 ± 4	55 ± 3[b,e]	3.4	5.2	0.23
Eu	0.17[d]	0.24 ± 0.02[c]	38.2	7.5	*	Tb	0.1[d]	0.11 ± 0.01[c]	11.7	10.5	*
Fe	218 ± 14	240 ± 18[c,f]	10.2	6.9	0.56	Th	0.05[d]	0.063 ± 0.011[c]	26.7	16.3	*
K (%)	2.43 ± 0.03	2.45 ± 0.12[b]	0.7	3.2	0.07	Yb	0.2[d]	0.20 ± 0.01[c]	0.8	5.8	*
La	9[d]	8.8 ± 1.0[b]	−1.9	11.1	*	Zn	17.9 ± 0.4	23.2 ± 7.2[c]	29.4	30.7	0.37
Mg (%)	0.432 ± 0.008	0.425 ± 0.035[b]	−1.5	7.4	0.10						

'*' Not calculated

[a] Laboratory combined standard uncertainty U_{Lab_Comb}

[b] Values obtained in short irradiation

[c] Values obtained in long irradiation

[d] Information values

[e] $n = 5$

[f] $n = 4$

The variation coefficient (CV) presented a deviation between replicates below 30 % except for Sb in SRM 1547 and Nd in INCT-MPH-2 (45.4 and 31.3 %, respectively, see Tables 5 and 6). As most of the elements analyzed are in the concentration range of mg kg^{-1} and ng kg^{-1}, and considering the criteria described by Wood [15], these values are still acceptable, since they are below 45 %, acceptable for elements in the ng kg^{-1} concentration range. These results show that the reproducibility obtained was satisfactory.

The E_n-number (Figs. 5, 6, 7) showed that all the results, except Na in NIST SRM 1547 and NIST SRM 1573a, and Al, Cr, Sc and Zn in INCT-MPH-2, are within 95 % CI. Values above the certified values for Na in NIST SRM 1547 had been observed also in our previous studies [16, 17], as well as in published literature [18]. This seems to indicate that Na is unstable in this SRM. The high values of the E_n-number for Na in NIST SRM 1573a may possibly be associated with relatively small uncertainty of the certified value (136 ± 4 mg kg^{-1}, 2.94 % for the 95 % CI). The high value of the E_n-number for Al in INCT-MPH-2 ($|E_n| = 1.33$) can possibly be associated with problems regarding the accuracy of irradiation times at the pneumatic station of the IEA-R1 reactor for short radionuclides (^{28}Al, $T_{1/2} = 2.241$ min, $E_\gamma = 1{,}778.9$ keV). For Cr, Sc and Zn in INCT-MPH-2, the high E_n values (1.12, 1.16 and 1.56, respectively) may be related to insufficient counting statistics in the experimental conditions (see Table 4 of experimental set-up) or net peak area evaluation by k0_IAEA software for a multiplet (i.e. 1,112.1, 1,115.5 and 1,120.5 keV for ^{152}Eu, ^{65}Zn and ^{46}Sc, respectively) and partly due to underestimation of the U_{Lab}. Some possible blank contribution from plastic bags, in which the samples were packed, cannot be excluded.

In the SRM 1573a, Br, K and Na were determined both in short and long irradiations, with quite similar results. For Na, even though the results obtained were the same, the uncertainty of the laboratory was lower in the results obtained in the long irradiation, and therefore, the E_n value for long irradiation was higher than the E_n value for short irradiation, but both values showed that the result was unsatisfactory. In the SRM 1547, Ba and Sm were determined by short and long irradiation. The results obtained for Sm in the short irradiation were in better agreement with the information value than the results obtained in the long irradiation. Nevertheless, since there is no certified value, this information is not conclusive. For Ba, the results showed that the use of the radioisotopes ^{139}Ba (for short irradiation) as well as ^{131}Ba (for long irradiation) provides reliable results.

Table 6 Results obtained by k0_IAEA software for 6 replicates in mg kg^{-1} (dry mass basis), if not stated otherwise

INCT-MPH-2 mixed polish herbs											
Element	X_{cert}	X^{a}_{Lab}	Bias (%)	CV (%)	E_n	Element	X_{cert}	X^{a}_{Lab}	Bias (%)	CV (%)	E_n
Al	670 ± 111	854 ± 42[b]	27.5	3.4	1.33	Mn	191 ± 12	197 ± 11[b]	3.0	4.3	0.23
As	0.191 ± 0.023	0.169 ± 0.027[c]	−11.3	15.4	0.37	Mo	0.520[d]	0.531 ± 0.030[c,f]	2.2	4.5	*
Ba	32.5 ± 2.5	40.9 ± 5.8[b]	25.8	13.8	0.71	Na	350[d]	411 ± 31[b]	17.5	6.7	*
Br	7.71 ± 0.61	8.31 ± 0.61[b]	7.8	6.4	0.44	Nd	0.457 ± 0.091	0.320 ± 0.101[c,e]	−30.0	31.3	0.62
Ca (%)	1.08 ± 0.07	0.99 ± 0.10[c]	−8.5	9.1	0.45	Rb	10.7 ± 0.7	11.9 ± 0.6[c,e]	10.8	3.8	0.82
Ce	1.12 ± 0.10	1.17 ± 0.06[c,e]	4.1	3.2	0.31	Sb	0.066 ± 0.009	0.069 ± 0.008[c,f]	3.8	10.8	0.14
Cl (%)	0.284 ± 0.020	0.293 ± 0.015[b]	3.2	3.7	0.25	Sc	0.123 ± 0.009	0.141 ± 0.006[c]	14.4	2.6	1.16
Co	0.210 ± 0.025	0.226 ± 0.042[c]	7.8	18.1	0.19	Sm	0.094 ± 0.008	0.091 ± 0.014[c]	−3.2	14.5	0.11
Cr	1.69 ± 0.13	2.04 ± 0.14[c]	20.9	6.1	1.12	Ta	0.019 ± 0.002	0.014 ± 0.004[c,e]	−28.4	29.7	0.64
Cs	0.076 ± 0.007	0.067 ± 0.006[c]	−12.3	7.6	0.71	Tb	0.014 ± 0.001	0.013 ± 0.001[c,e]	−7.1	5.4	0.51
Eu	0.016 ± 0.002	0.018 ± 0.002[c]	12.5	9.3	0.49	Th	0.154 ± 0.013	0.149 ± 0.007[c,f]	−3.4	3.1	0.28
Fe	460[d]	605 ± 49[c]	31.5	7.3	*	U	0.049[d]	0.050 ± 0.012[c,e]	1.2	24.8	*
Hf	0.236 ± 0.020	0.232 ± 0.009[c]	−1.8	1.5	0.16	V	0.952 ± 0.163	0.994 ± 0.251[b,e]	4.5	25.0	0.08
K (%)	1.91 ± 0.12	1.96 ± 0.11[b]	2.4	4.7	0.17	Yb	0.053 ± 0.007	0.049 ± 0.004[c]	−6.9	7.9	0.33
La	0.571 ± 0.046	0.604 ± 0.044[c,e]	5.7	6.5	0.33	Zn	33.5 ± 2.1	42.2 ± 2.6[c]	26.1	5.1	1.56
Mg (%)	0.292 ± 0.018	0.290 ± 0.017[b]	−0.8	4.7	0.06	–	–	–	–	–	–

'*' Not calculated

[a] Laboratory combined standard uncertainty U_{Lab_Comb}

[b] Values obtained in short irradiation

[c] Values obtained in long irradiation

[d] Information values

[e] $n = 5$

[f] $n = 4$

Table 7 Results obtained by k0_IAEA software for 6 replicates in mg kg^{-1} (dry mass basis), if not stated otherwise

NIST SRM 1573a tomato leaves											
Element	X_{cert}	X^{a}_{Lab}	Bias (%)	CV (%)	E_n	Element	X_{cert}	X^{a}_{Lab}	Bias (%)	CV (%)	E_n
Al	598 ± 12	643 ± 42[b]	7.5	5.5	0.53	La	2.3[d]	2.4 ± 0.4[c]	5.1	18.2	*
Br	1,300[d]	1,278 ± 57[b]	−1.7	2.7	*	Mg (%)	1.2[d]	1.14 ± 0.08[b]	−5.4	6.5	*
Br	1,300[d]	1,261 ± 73[c]	−3.0	4.6	*	Mn	246 ± 8	251 ± 11[b]	2.0	2.7	0.21
Ca (%)	5.05 ± 0.09	5.05 ± 0.42[c]	0.0	7.5	0.00	Na	136 ± 4	179 ± 16[b]	31.4	8.1	1.34
Ce	2[d]	2.0 ± 0.1[c]	0.0	0.0	*	Na	136 ± 4	178 ± 10[c]	30.5	4.6	1.98
Cl	6,600[d]	7,130 ± 384[b]	8.0	4.1	*	Rb	14.89 ± 0.27	16.50 ± 1.70[c]	10.8	9.7	0.47
Cr	1.99 ± 0.06	2.27 ± 0.46[c]	14.1	20.1	0.30	Sc	0.10[d]	0.11 ± 0.02[c]	6.7	14.1	*
Fe	368 ± 7	428 ± 100[c]	16.3	23.1	0.30	Sm	0.19[d]	0.16 ± 0.03[c]	−16.7	21.7	*
Hf	0.14[d]	0.13 ± 0.01[c]	−9.5	9.6	*	Th	0.12[d]	0.062 ± 0.012[c]	−48.6	19.0	*
K (%)	2.70 ± 0.05	2.78 ± 0.15[b]	2.8	4.0	0.26	Zn	30.9 ± 0.7	30.9 ± 9.5[c]	0.1	30.5	0.00
K (%)	2.70 ± 0.05	2.84 ± 0.18[c,e]	5.3	5.4	0.39	–	–	–	–	–	–

'*' Not calculated

[a] Laboratory combined standard uncertainty U_{Lab_Comb}

[b] Values obtained in short irradiation

[c] Values obtained in long irradiation

[d] Information values

[e] $n = 5$

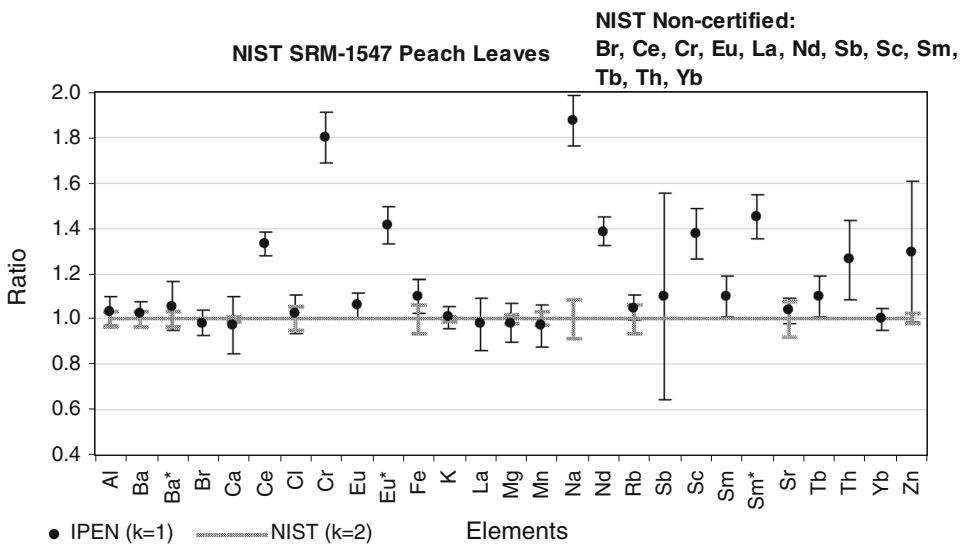

Fig. 2 Ratios to certified and information values for NIST SRM-1547 peach leaves. Element marked with *asterisk* means value obtained in long irradiation

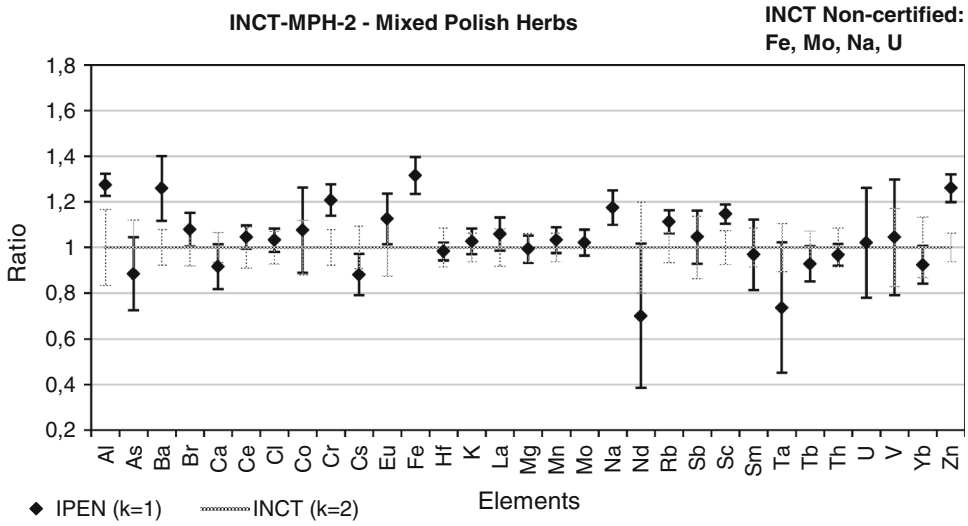

Fig. 3 Ratios to certified values for INCT-MPH-2 mixed polish herbs

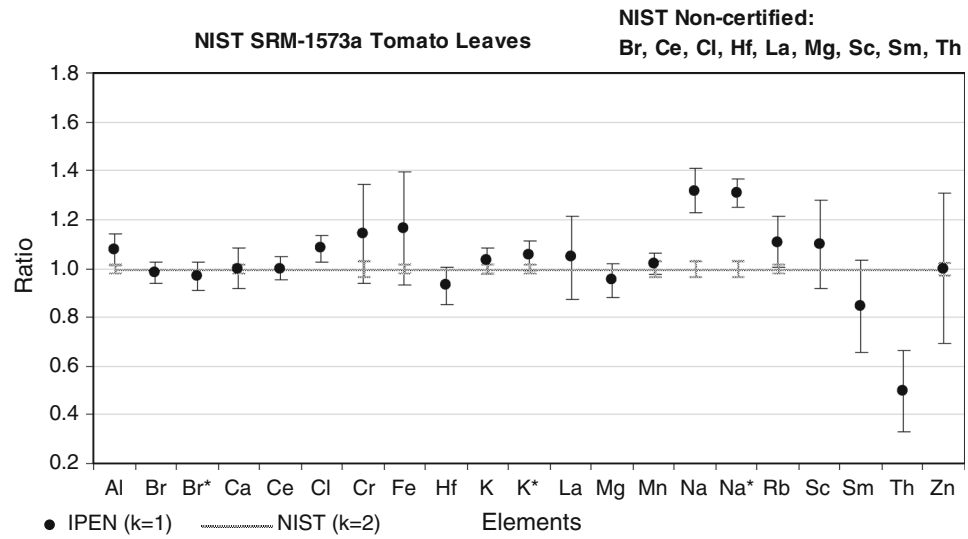

Fig. 4 Ratios to certified and information values for NIST SRM-1573a tomato leaves. Element marked with *asterisk* means value obtained in long irradiation

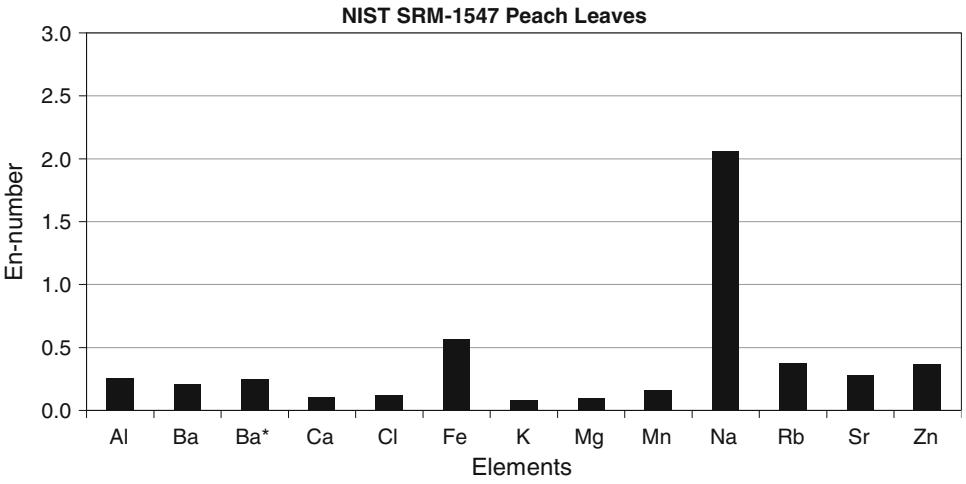

Fig. 5 E_n numbers obtained for NIST SRM-1547 peach leaves. Element marked with *asterisk* means value obtained in long irradiation

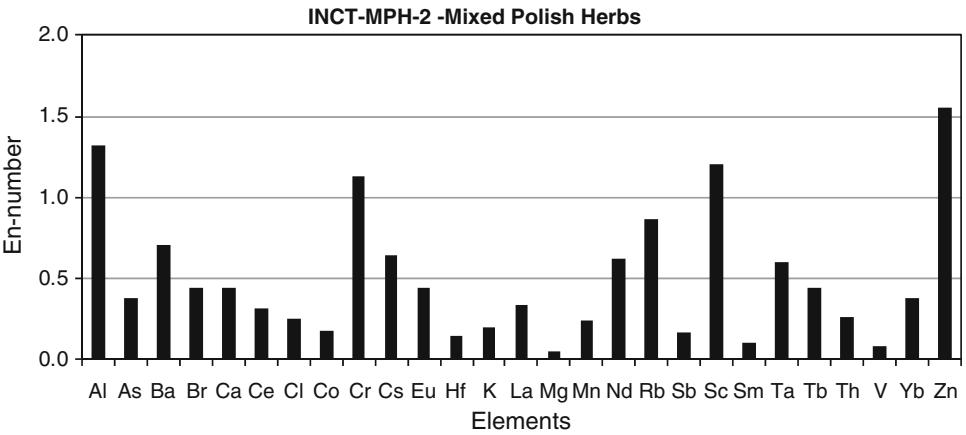

Fig. 6 E_n numbers obtained for INCT-MPH-2 mixed polish herbs

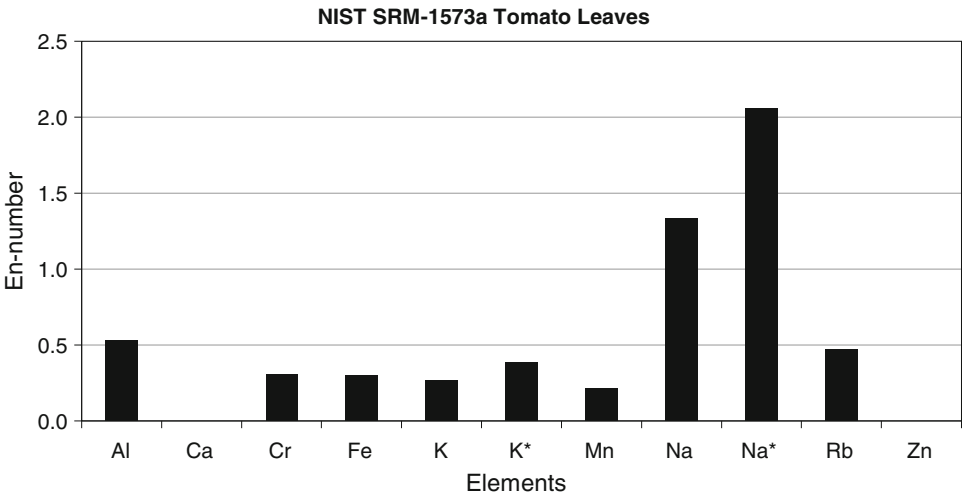

Fig. 7 E_n numbers obtained for NIST SRM-1573a tomato leaves. Element marked with *asterisk* means value obtained in long irradiation

Conclusions

The k_0-INAA method with the k0_IAEA software provided reliable results for several elements in the biological reference materials analyzed. The present study showed also that some changes in experimental set-up of irradiation and measurement conditions are needed including more efforts of the sample preparation in plastic bags.

In the case of Na, higher values than expected were obtained. This could be explained by a possible instability

of Na in a specific SRM. In the case of Al, more investigation is needed to obtain more accurate irradiation times for short-lived radionuclides using the pneumatic system facility of the IEA-R1 reactor. $E_n > 1$ was obtained for Cr, Sc and Zn in the INCT-MPH-2 indicating insufficient counting statistic and partly underestimation of the U_{Lab} when applying only the additional 3.5 % systematic error of the k_0-method.

The results obtained indicate the potential of the k_0-INAA method with the k0_IAEA software for biological sample analysis. This, in turn, should improve the efficiency of LAN-IPEN for the analysis of biological matrices.

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