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A preliminary test for the application of the k_0 standardization method of neutron activation analysis at the Radiochemistry and Spectroscopy Laboratory of the Istituto Nazionale di Ricerca Metrologica

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Abstract

The application of the k_0-standardization method of neutron activation analysis offers the great advantage to perform multi-elemental analyses using a single element as a comparator. On the other hand, some knowledge of the neutron flux energy distribution is mandatory. Recently, neutron flux parameters f and α in three irradiation channels of the TRIGA Mark II nuclear reactor in Pavia were measured for the first time to allow the application of the k_0-standardization method at the Radiochemistry and Spectroscopy Laboratory of the Istituto Nazionale di Ricerca Metrologica. In this framework, a preliminary test was performed by analyzing a set of samples irradiated in Central Channel, including a number of elements prepared with mono-elemental solutions and a reference material. The results of all the selected elements were in agreement with the expected value and confirmed the applicability of the k_0-standardization method in Central Channel.

Keywords k_0-method · TRIGA Mark II · Instrumental neutron activation analysis

Introduction

Since its definition [1], the k_0-standardization method of neutron activation analysis (NAA), hereafter called k_0-NAA, introduced a much faster and routinely used version of multi-elemental analysis compared to the relative standardization method. The possibility to quantify sixty-seven elements present in the k_0-database [2] using a unique comparator replaced the cumbersome preparation of multi-standards mixtures.

The application of k_0-NAA requires the knowledge of the ratio of the thermal to epithermal neutron flux, f, and of the parameter α, describing the epithermal neutron flux

$1/E^{1+\alpha}$, where E is the neutron energy. Moreover, several additional parameters are included in the measurement model, e.g. the irradiation time, the detection efficiency and the k_0-factor. Although the added parameters might reflect in a generally higher uncertainty compared to the results obtained with the relative standardization method, the advantages of application of k_0-NAA are consistent.

Recently, aiming at the application of the k_0-NAA at the Radiochemistry and Spectroscopy Laboratory of the Istituto Nazionale di Ricerca Metrologica (INRIM), the f and α values in three irradiation channels of the 250 kW TRIGA Mark II reactor operated by the Laboratorio Energia Nucleare Applicata (LENA) of the University of Pavia were obtained through neutron irradiations and γ-countings of a defined monitor set with and without a Cd-cover [3]. In this framework, additional neutron activation and γ-counting experiments were performed using known amounts of selected elements to check the agreement of the results obtained with the k_0-NAA with the expected values. A description of the adopted measurement model, details of the experiment and the obtained results are reported in this study together with a preliminary evaluation of the measurement uncertainty.

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Theory

A comprehensive description of the k_0-standardization method can be found in [1], including several references to published papers. Here, for convenience of the reader, the basic equations used to build the measurement model are recalled.

The key parameter for the application of k_0-NAA is the k_0 factor of an element, i, with respect to Au:

$$k_{0,\mathrm{Au}}(i) = \frac{M_{\mathrm{Au}}\theta_i\gamma_i\sigma_{0,i}}{M_i\theta_{\mathrm{Au}}\gamma_{\mathrm{Au}}\sigma_{0,\mathrm{Au}}}, \tag{1}$$

where M is the molar mass, θ is the isotopic fraction, γ is the absolute gamma intensity and σ_0 is the 2200 m s^{-1} (n,γ) reaction cross-section; subscripts i and Au refer to the element i and Au, respectively.

The co-irradiation of a sample and a single comparator, yields the mass fraction of investigated analyte, ρ_a, in the sample, thanks to the introduction of $k_{0,\mathrm{Au}}$ factors. Specifically, the k_0-NAA measurement model is:

$$\rho_a = \frac{\left.\frac{\lambda n_p t_r/t_l}{(1-e^{-\lambda t_i})e^{-\lambda t_d}(1-e^{-\lambda t_r})}\right|_a}{\left.\frac{\lambda n_p t_r/t_l}{(1-e^{-\lambda t_i})e^{-\lambda t_d}(1-e^{-\lambda t_r})}\right|_c} \frac{w_c}{w} \frac{k_{0,\mathrm{Au}}(c)}{k_{0,\mathrm{Au}}(a)} \frac{G_{\mathrm{th,c}}+G_{\mathrm{e,c}}\frac{Q_{0,c}(\alpha)}{f}}{G_{\mathrm{th,a}}+G_{\mathrm{e,a}}\frac{Q_{0,a}(\alpha)}{f}} \frac{\varepsilon_{\mathrm{p,c}}}{\varepsilon_{\mathrm{p,a}}}, \tag{2}$$

where $\lambda = \frac{\ln 2}{t_{1/2}}$ is the decay constant (with $t_{1/2}$ being the half-life of the activated radionuclide), n_p is the number of counts in the full-energy γ-peak corrected for true coincidences, t_l and t_r are the live and real times of the detection system, t_i and t_d are the irradiation and decay times, w_c is the mass of the comparator element and w is the mass of the sample containing the investigating analyte, $Q_0(\alpha) = \frac{Q_{0-0.429}}{\bar{E}_r^\alpha} + \frac{0.429}{(2\alpha+1)0.55^\alpha}$ is the resonance integral to 2200 m s^{-1} cross section ratio in a $1/E^{1+}$ neutron spectrum, f is the thermal to epithermal neutron flux ratio, ε_p is the full-energy γ-peak detection efficiency, and G_{th} and G_e are the thermal and epithermal self-shielding factors; subscripts a and c refer to the investigated analyte and the comparator, respectively.

In details, Q_0 is the resonance integral to 2200 m s^{-1} cross section ratio in $1/E$ neutron spectrum, $\bar{E}_r$ is the effective resonance energy. Moreover, the ratio between efficiencies is approximated, under the assumption that sample and comparator are acquired at the same distance and far from the detector, with $\frac{\varepsilon_{\mathrm{p,c}}}{\varepsilon_{\mathrm{p,a}}} = \frac{\varepsilon_{\mathrm{pref,c}}}{\varepsilon_{\mathrm{pref,a}}}\frac{F_{\mathrm{att,c}}^{self}}{F_{\mathrm{att,a}}^{self}}\frac{F_{\mathrm{geo,c}}}{F_{\mathrm{geo,a}}}$, where $\frac{\varepsilon_{\mathrm{pref,c}}}{\varepsilon_{\mathrm{pref,a}}}$ is the ratio of detection efficiencies of comparator and analytes at reference position, F_{att}^{self} is the self-

attenuation correction factor [4] and F_{geo} is the geometrical correction factor.

In this study the mass of the sample, w, in Eq. (2) is replaced with the mass of the investigated analyte, w_a. Accordingly, ρ_a expresses the ratio, hereafter called r_a, of the quantified to the expected mass of the analyte; in the case of an unbiased result, $r_a = 1$.

Experimental

Samples of the measured analytes were prepared starting from mono-elemental standard solutions (VWR Chemicals, 1000 µg mL^{-1}) and IAEA-SOIL-7 reference material.

Known amounts of fifteen selected elements (Ag, Au, Ce, Cr, Cs, Hf, Ir, La, Re, Sb, Sc, Se, Ta, Tb, Tm) from standard solutions were pipetted in seven 1 mL polyethylene (PE) vials, 5 mm internal diameter and cut to 4 mm internal height. Specifically, a small volume of each elemental standard solution (in the range from 10 to 30 µL) was dropped on an absorbent paper disc situated at the bottom of the corresponding vial.

Twelve elements (Au, Ce, Cs, Hf, Ir, Re, Sb, Sc, Se, Ta, Tb, Tm) were distributed among four vials while three elements (Ag, Cr, La) were separately distributed in three vials together with aliquots of a Co standard solution used as comparator. Co was used as comparator because of its long half-life, comparable with that of the most part of the investigated analytes. An additional vial was irradiated as a blank. The elements were distributed among the vials with respect of the similar half-lives of the produced radioisotopes and in order to avoid interferences among the analytical γ-peaks.

Drop deposition was performed on an analytic balance calibrated with SI-traceable weights. The vial was then placed under an IR lamp until the solution was completely dried. The procedure was repeated for each standard in the same vial, for every vial with exception of the blank. When all the liquid lying on the filter papers was dried, the vials were sealed.

On the other hand, 153.84(1) mg of the IAEA-SOIL-7 reference material was weighed in a 5 mL PE vial, in this case without the filter paper at the bottom, on the same analytical balance and sealed. Here and hereafter, values in parenthesis indicate the standard uncertainty and refer to the last digits if not stated otherwise. Relative moisture of the reference material was evaluated and corrected accordingly.

The vials were placed in three vertical levels starting from the floor of the PE irradiation container; the eight 1 mL vials occupied the lower levels grouped by four while the reference material was placed on the higher level.

The whole structure was then fixed by a dummy plastic vial placed at the top. Figure 1 shows the composition scheme within the irradiation container while Table 1 indicates the composition of each vial.

The neutron irradiation lasted 60 min and was performed at the Central Channel of the TRIGA Mark II reactor. The Central Channel was chosen for the experiment because it is the most widely used among the Pavia's TRIGA Mark II irradiation channels due to its high conventional thermal flux, $6.11(12) \times 10^{12}$ cm^{-2} s^{-1} [3]. The container was placed closest to the equator of the reactor core, at position 1. After 3 days from the end of the irradiation, the PE vials were unpacked and externally rinsed with a diluted HNO_3 solution.

The detection system used for the counting of γ-emissions consisted of a 35% relative efficiency coaxial Ge detector manufactured by CANBERRA, model GC3518, connected to a digital signal processor ORTEC DSPEC 502 and a personal computer running GammaVision [5] software. During counting, the end-cap of the detector was placed inside a low-background graded lead shield located in an underground laboratory with controlled temperature of 23 °C. The PE vials were put separately in different plastic counting containers and centered with a rubber filler (i.e. a rubber disc placed in the counting container; the disc had a hole in the center in which the vial was inserted).
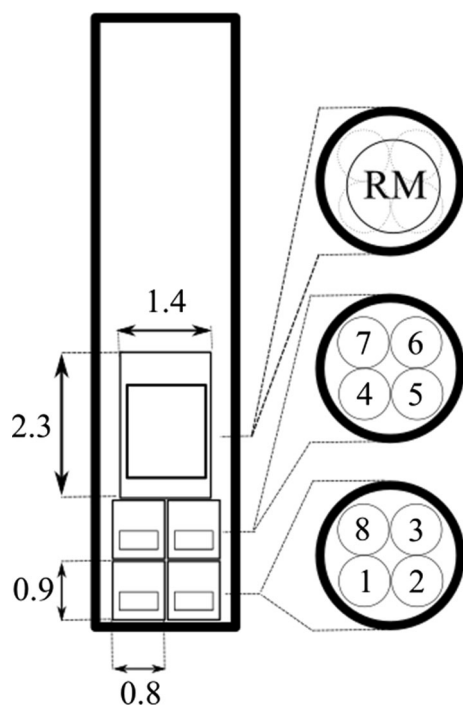
Table 1 Identification code of each vial and the respective elemental content are listed

Sample code	Content	Main γ (keV)
1	Co	1173.2
	Cr	320.1
2	Au	411.8
	Sb	564.2
	Re	137.2
3	Sc	889.3
	Ir	316.5
	Tb	879.4
4	Co	1173.2
	La	1596.2
5	Ce	145.4
	Tm	84.3
	Hf	482.2
6	Cs	604.7
	Se	136.0
	Ta	1121.3
7	Co	1173.2
	Ag	657.8
8	Blank	–
RM	IAEA-SOIL-7	Multiple

The energy of the γ-photons [2] used for quantification is also given

Fig. 1 The position of the samples within the irradiation container. An empty 8 mL PE vial was used to fix underneath vials in their position. Distances are in cm. Identifying codes visible in the figure are explained in Table 1

Each container was placed on a sample holder at 10 cm from the detector end-cap. The number of counts in the full energy γ-peaks were corrected for coincidences; the correction was always below 1% with the exception of ^{110m}Ag where it reached 2%.

A spectrum was acquired for each vial of standard solution by adjusting the counting time to limit the contribution to the uncertainty due to counting statistics below the 0.5%. The acquisitions lasted in a range between a few hours to 2 days and never overcame the half-life of the evaluated radionuclides. For what concerns the IAEA-SOIL-7 sample, a spectrum was acquired in a single acquisition lasting 6 days with the counting container located at 10 cm from detector end-cap.

The values of the parameters included in the measurement model were obtained both from the experimental and literature data, including a preliminary evaluation of the uncertainties.

The net areas of the γ-peaks and the relative uncertainties due to counting statistics were obtained using the gaussian fit implemented in the algorithm WAN32 of GammaVision software. Relative uncertainties ranged from 0.06% (Au and Ir) to 0.6% (Se) for the acquisition of the analytes prepared with the mono-elemental standards

solutions and from 0.9% (La) to 8.9% (Nd) for the acquisition of the analytes quantified in the IAEA-SOIL-7 reference material. Co comparators were acquired with statistical uncertainty within 0.3 and 0.5%.

Live and real times were measured and recorded by the acquisition software with negligible uncertainty. The relative dead time, $\frac{t_r - t_l}{t_r}$, was always lower than 1% except in the case of acquisition of samples containing Au and Ir, vials 2 and 3, when it raised up to 9 and 8%, respectively; in all cases, t_r was sufficiently smaller than $t_{1/2}$ of the acquired radioisotopes. In addition, the pile up rejection circuit was active during acquisition.

The 3600 s irradiation time was measured from the attainment of criticality at 250 kW power to the shut down. The reactor was powered on with the irradiation container already inside the channel. Uncertainty on t_i was evaluated assuming a uniform probability distribution within the interval $\pm$ 30 s of the measured irradiation time. [6]

In addition, a uniform probability distribution within the interval $\pm$ 60 s was conservatively assumed for t_d, as clocks used to calculate the time difference from the irradiation end to start counting, were not automatically synchronized to each other. The standard uncertainty of t_d resulted to be 34 s. Since the decay times ranged from 93.5 to 281.0 h, the contribution to the combined uncertainty was negligible.

Masses of elements contained in the samples prepared with the standard solutions (comparators and analytes) were obtained from the weighed pipetted solutions using the certified density and elemental concentration values. The certificate of analytical balance reported 0.008 mg ($k = 2$) calibration uncertainty in the range between 10 and 100 mg. Water evaporation played an important role during mass measurement of the solutions. An approximated time of 15 s was waited after the liquid deposition until the reading. During this period, a previously estimated 6 μg s^{-1} evaporation rate was considered to correct for the evaporated mass. The resulting 90 μg mass was conservatively considered as the half-width of a uniform probability distribution assigned to the weighted mass. The corresponding relative standard uncertainty on mass ranged from 0.17 to 0.49%, depending on the pipetted volume. Density and mass concentration were retrieved from the certificates with uncertainties 0.001 g cm^{-3} and 0.001 μg mL^{-1}, respectively [7]. Accordingly, in the case of standard solutions, the uncertainty of the w_a in Eq. (2) is not significant.

For what concerns the reference material, the weighed mass of sample was corrected for moisture content. The moisture was measured by weighing about 500 mg of a representative subsample on a Mettler Toledo HC103 thermal balance. The resulting moisture content was 1.51%

of the weighed mass and a value of 0.1515(13) g dry mass was assigned to the reference material. The expected mass values for each investigated analyte in IAEA-SOIL-7 were calculated from the respective recommended mass fraction values stated in the reference sheet [8]. It is worth to note that the 95% confidence interval reported in the reference sheet are significant, i.e. between a few and tens of percent.

Neutron flux parameters f and α for Central Channel were previously measured with irradiations of a monitor set with and without Cd cover [3]. Relative uncertainties were assigned from the average of the values obtained from each monitor for f, and according to the formula described by De Corte, for α [9]. The resulting values are $f = 15.6(3)$ and $\alpha = -0.036(6)$. Although the Co comparators were located at a different height (see Fig. 1), the measured specific count rates were in agreement to each other, confirming that no visible differences in neutron flux intensity were appreciable in such positions. Therefore, the neutron flux was considered constant in the zone occupied by irradiation vials and the Co comparator in vial 1 was used to quantify all the analytes.

The values of k_0, Q_0, $\bar{E}_r$ and $t_{1/2}$ were retrieved, together with their respective standard uncertainties, for each nuclide of interest from the k_0-database [2]. Relative standard uncertainties of k_0, Q_0 and $\bar{E}_r$, when not explicitly reported, were assigned to 5, 20 and 50%, respectively, as indicated in k_0-database guidelines.

The detection efficiency curve was obtained from a spectrum collected with a multi-γ calibration point source, 3 mm diameter, (12ML01EGMA15) placed at 10 cm from the end-cap. A polynomial curve was fitted to the data. The main contribution to the uncertainty of the detection efficiency was due to sample positioning. In fact, albeit vials containing the samples were accurately fixed at the center of their respective counting container, the vertical position represented an issue due to a non-perfect alignment of filter paper at the bottom of sample vials. A uniform probability distribution within the interval $\pm$ 0.2 cm was assigned to the counting distance. At the 10 cm reference distance, the corresponding standard uncertainty was 0.1 cm. Consequently, 1% relative standard uncertainty due to positioning contributed to the detection efficiency.

Geometric and self-absorption factors were only considered in the evaluation of reference material as its shape strongly differed from that of the point-like multi-γ source used for calibration. Value of F_{geo} was analytically evaluated from geometrical features of the IAEA-SOIL-7 within its irradiation vial and corresponded to a correction factor of 0.985 (a decrease in efficiency in the order of 1.5% with respect to the reference point source) with negligible uncertainty, while values of F_{att}^{self} were

calculated using energy dependent mass attenuation coefficients retrieved from literature [10].

Values of r_a were obtained according to Eq. (2) while combined uncertainties were evaluated with quadratic propagation of standard uncertainty for each input quantity using its respective sensitivity coefficient. The sensitivity coefficient was calculated by averaging the coefficients obtained after perturbation of the input quantity by + 1 and − 1 standard uncertainty. The calculations were performed using a Microsoft Excel spreadsheet.

Results and discussion

The results obtained with samples prepared from mono-elemental solutions and from the reference material are reported in Figs. 2 and 3, respectively.

The u-score statistical test [11] was performed using the quantified and expected values by considering the respective expanded uncertainties at 95% confidence level; the obtained u-scores were always lower than 0.9 confirming the good agreement with the expected values.

In the case of the standard solutions, a weighted average of the obtained results was calculated using the standard uncertainty of each result as a weight and plotted in Fig. 2 (horizontal solid line) to compare the overall results with the expected unity value. It is worth to note that, since the uncertainties of the expected values are not significant, the uncertainty of the expected unity values can be neglected.

Instead, in the case of the IAEA-SOIL-7, the 95% confidence intervals associated to the recommended values and reported in the reference sheet, are significant. The corresponding intervals were calculated and plotted in
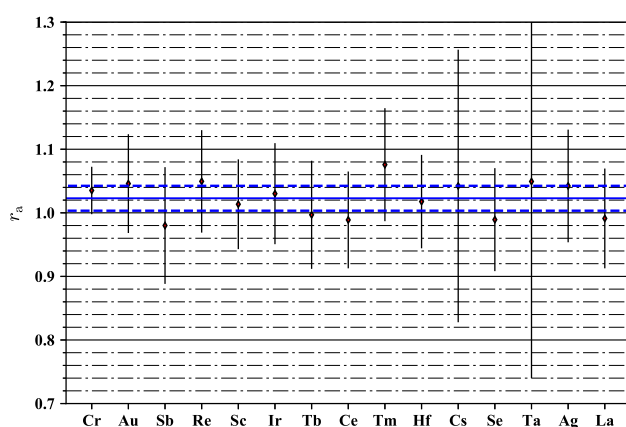

Fig. 3 Values for r_a of fifteen elements obtained with the sample of the IAEA-SOIL-7 reference material. The solid error bars indicate the 95% confidence interval of the measured values while the dashed error bars indicate the 95% confidence interval of the expected values

Fig. 3 (vertical dashed lines) to compare each result with the expected values.

The quantified and expected masses of for the elements from standard solutions and from IAEA-SOIL-7 material are reported in Tables 2 and 3, respectively.

The relative standard uncertainty reached in the analysis of the mono-elemental standard solutions was about 4%, with the exception of Cs and Ta, when it reached about 15% due to the non-recommended Q_0 values. In general, the main contributor to the combined uncertainty, evaluated via Eq. (2), was the detection efficiency. In the case of

Fig. 2 Values for r_a of fifteen elements obtained with samples prepared from mono-elemental standard solutions. The error bars indicate a 95% confidence interval. The horizontal solid line indicates the weighted average of results and the dashed lines the associated 95% confidence interval
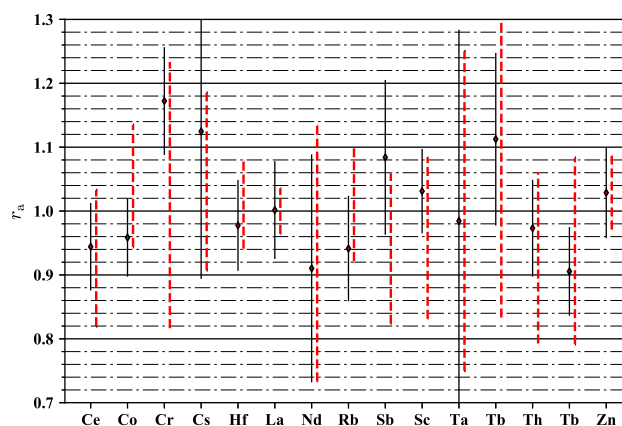
Table 2 Comparison between quantified mass for elements from standard solutions and respective expected values

Element	Quantified mass (g)	Expected mass (g)
Cr	$2.125(38) \times 10^{-5}$	2.053×10^{-5}
Au	$1.092(40) \times 10^{-5}$	1.044×10^{-5}
Sb	$2.016(94) \times 10^{-5}$	2.057×10^{-5}
Re	$2.165(83) \times 10^{-5}$	2.063×10^{-5}
Sc	$2.084(72) \times 10^{-5}$	2.056×10^{-5}
Ir	$2.109(81) \times 10^{-5}$	2.047×10^{-5}
Tb	$2.027(86) \times 10^{-5}$	2.034×10^{-5}
Ce	$2.94(11) \times 10^{-5}$	2.972×10^{-5}
Tm	$2.204(91) \times 10^{-5}$	2.049×10^{-5}
Hf	$2.092(75) \times 10^{-5}$	2.055×10^{-5}
Cs	$2.11(22) \times 10^{-5}$	2.029×10^{-5}
Se	$3.03(12) \times 10^{-5}$	3.065×10^{-5}
Ta	$2.16(32) \times 10^{-5}$	2.058×10^{-5}
Ag	$2.112(90) \times 10^{-5}$	2.027×10^{-5}
La	$2.98(12) \times 10^{-5}$	3.011×10^{-5}

The standard uncertainty of the quantified masses is indicated within parenthesis and refers to the last digits; the (negligible) standard uncertainty of the expected masses is omitted

Table 3 Comparison between quantified mass for elements from IAEA-SOIL-7 and respective expected values

Element	Quantified mass (g)	Expected mass (g)	Expected mass 95% confidence interval (g)
Ce	$8.73(31) \times 10^{-6}$	9.24×10^{-6}	7.58×10^{-6}–9.55×10^{-6}
Co	$1.293(40) \times 10^{-6}$	1.35×10^{-6}	1.27×10^{-6}–1.53×10^{-6}
Cr	$1.066(37) \times 10^{-5}$	9.09×10^{-6}	7.42×10^{-6}–1.12×10^{-5}
Cs	$9.20(94) \times 10^{-7}$	8.18×10^{-7}	7.42×10^{-7}–9.70×10^{-7}
Hf	$7.55(27) \times 10^{-7}$	7.73×10^{-7}	7.27×10^{-7}–8.33×10^{-7}
La	$4.25(16) \times 10^{-6}$	4.24×10^{-6}	4.09×10^{-6}–4.39×10^{-6}
Nd	$4.14(40) \times 10^{-6}$	4.55×10^{-6}	3.33×10^{-6}–5.15×10^{-6}
Rb	$7.27(31) \times 10^{-6}$	7.73×10^{-6}	7.12×10^{-6}–8.48×10^{-6}
Sb	$2.79(15) \times 10^{-7}$	2.58×10^{-7}	2.12×10^{-7}–2.73×10^{-7}
Sc	$1.297(40) \times 10^{-6}$	1.26×10^{-6}	1.05×10^{-6}–1.36×10^{-6}
Ta	$1.19(18) \times 10^{-7}$	1.21×10^{-7}	9.09×10^{-8}–1.52×10^{-7}
Tb	$1.011(61) \times 10^{-7}$	9.09×10^{-8}	7.58×10^{-8}–1.36×10^{-7}
Th	$1.209(46) \times 10^{-6}$	1.24×10^{-6}	9.85×10^{-7}–1.32×10^{-6}
Tb	$3.29(12) \times 10^{-7}$	3.64×10^{-7}	2.88×10^{-7}–3.94×10^{-7}
Zn	$1.621(54) \times 10^{-5}$	1.58×10^{-5}	1.53×10^{-5}–1.71×10^{-5}

For the quantified masses the standard uncertainty, referring to the last digits, is indicated within parenthesis while for expected masses, the intervals at 95% confidence level are given

Cs and Ta, the major contribution came from the uncertainty assigned to non-recommended Q_0 values, i.e. $Q_{0,Cs} = 12.7(25)$ and $Q_{0,Ta} = 33.3(66)$.

The uncertainty of the quantified elements in the IAEA-SOIL-7 reference material showed an analogue situation. In general, 5% relative uncertainty was achieved for the best results. The main contributors to the combined uncertainty were the detection efficiency and the Q_0 values. In the case of Nd, also the poor counting statistics had a significant effect.

Conclusions

The application of the k_0-NAA at the Radiochemistry and Spectroscopy Laboratory of the Istituto Nazionale di Ricerca Metrologica using the LENA 250 kW TRIGA Mark II reactor was preliminary tested. Results obtained from the analysis of fifteen elements in seven samples prepared from mono-elemental standard solutions and fifteen elements in one sample of a reference material were in good agreement with the expected values. This outcome is an incentive to adopt, in a near future, the routinely use of the k_0-NAA standardization method in addition to the presently adopted relative standardization method.

However, the weighted average of the results obtained from standard solutions yielded a value that was 2% higher than expected with an expanded uncertainty that didn't cover the bias. This highlights the possible presence of some uncorrected systematic effect or an underestimation of the evaluated uncertainty that deserves further investigations.

Moreover, albeit the uncertainty evaluation anticipated that results were mainly affected by the detection efficiency, the Q_0 values, especially when marked as non-recommended in the k_0-database, can have a dramatic impact on the overall uncertainty when evaluated in poorly-thermalized facilities like the TRIGA Mark II reactor. Consequently, efforts dedicated to additional measurements of the non-recommended Q_0 values are required.

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