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Measurement of k_0 and Q_0 values for lutetium and europium

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Abstract It was shown that the k_0-method using the recently developed extended Hogdahl formalism for non-1/v nuclides and neutron temperatures from thermocouple readings can give k_0-NAA results for Eu and Lu accurate to about 1 %. The necessary Q_0 and k_0 values for ^{151}Eu(n,γ)^{152}Eu, ^{151}Eu(n,γ)^{152m}Eu and ^{176}Lu(n,γ)^{177}Lu were measured by reactor irradiation of certified standards and gamma-ray counting. The present measurements of k_0-values did not confirm the previously published values; they were higher by 6 % for ^{152}Eu and by 16 % for ^{152m}Eu.

Keywords Neutron activation analysis · Europium · Lutetium · Non-1/v · k_0-method · k_0-values · Q_0-values

Introduction

Neutron activation analysis is an excellent technique for the analysis of a wide variety of materials because it is applied directly to solid materials without the need for complex sample preparation. For multi-element analysis, k_0-NAA eliminates the tedious task of preparing standards of all elements to be determined: only one standard is needed and the relative sensitivities for all other elements are obtained from their k_0-factors. The k_0-method can be applied with high accuracy using any research reactor because the neutron capture reaction rates with different neutron spectra are well-determined by knowing the thermal and epithermal components and because the thermal neutron (n,γ) reaction rates all vary in the same fashion with changing reactor neutron temperature, the 1/v law. This is important because the reaction rate for each element is compared with the reaction rate for a monitor element and the ratio of the two should be independent of neutron temperature.

However, there are two notable exceptions: the ^{151}Eu(n,γ)^{152}Eu and ^{176}Lu(n,γ)^{177}Lu reactions are highly non-1/v. For a 10 K increase in thermal neutron temperature, the ^{151}Eu(n,γ)^{152}Eu reaction rate decreases by about 1 % and the ^{176}Lu(n,γ)^{177}Lu reaction rate increases by about 4 % relative to all the 1/v reaction rates [1]. To permit accurate k_0-NAA for Eu and Lu and for a few other slightly non-1/v nuclides, a more elaborate set of k_0 equations, using a modified Westcott formalism, was developed [2], replacing the commonly used and simpler modified Hogdahl formalism [3]. The use of the Westcott formalism required a slightly different neutron spectrum characterization, measuring the spectral index rather than the thermal/epithermal flux ratio of the Hogdahl formalism. Also, for each irradiation to determine Eu or Lu it was necessary to irradiate a Lu standard as a neutron temperature monitor. This requirement of preparing and irradiating a Lu standard in order to determine only one or two more elements, Eu and Lu, went against the aim of the k_0-method: more convenient routine multi-element analysis.

A recent paper [4] proposed a simplification of the equations needed for k_0-NAA of Eu and Lu, retaining the thermal/epithermal flux ratio, f, of the Hogdahl formalism and the familiar ratio, Q_0, of resonance integral to thermal activation cross-section; it was called the extended Hogdahl method. The equations of the extended Hogdahl method included the variation of thermal neutron activation with neutron temperature, the Westcott $g(T_n)$ factor [1]. That paper [4] also proposed, as did a previous paper [5],

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estimating the neutron temperature from reactor moderator temperature readings, thus eliminating the need to irradiate Lu temperature monitors.

In [4], neglecting for the moment neutron self-shielding, Eu and Lu mass fractions are calculated by the formula

$$\rho_a(\mu g/g) = \frac{\left(\frac{N_p/t_c}{SDCW}\right)_a}{\left(\frac{N_p/t_c}{SDC_w}\right)_m} \cdot \frac{1}{k_{0,m}(a)} \cdot \frac{1 + Q_{0,m}(\alpha)/f}{g_a(T_n) + Q_{0,m}(\alpha)/f} \cdot \frac{\varepsilon_{p,m}}{\varepsilon_{p,a}} \cdot 10^6 \quad (1)$$

which is the same formula as is used in the modified Hogdahl formalism [3] except for the $g(T_n)$ factor. If we multiply top and bottom by f and introduce the thermal and epithermal self-shielding factors G_{th} and G_e, we have Eq. (11) of [4]:

$$\rho_a(\mu g/g) = \frac{\left(\frac{N_p/t_c}{SDCW}\right)_a}{\left(\frac{N_p/t_c}{SDC_w}\right)_m} \cdot \frac{1}{k_{0,m}(a)} \cdot \frac{G_{th,m} f + G_{e,m}\, Q_{0,m}(\alpha)}{G_{th,a} f\, g_a(T_n) + G_{e,a}\, Q_{0,a}(\alpha)} \cdot \frac{\varepsilon_{p,m}}{\varepsilon_{p,a}} \cdot 10^6 \quad (2)$$

The use of these formulas requires k_0 and Q_0 values; they are measured in this work. Previously, k_0 values have been measured and compiled for the gamma-rays emitted by ^{152m}Eu and ^{152}Eu [6, 7] where the Westcott convention was used. They should be quite comparable to those of this work because the modified Westcott formalism of [2, 6] and Eq. (1) differ mainly in the treatment of epithermal neutron activation and, as was noted in [4], epithermal neutrons produce only a small fraction of the total ^{152m}Eu or ^{152}Eu activity in all research reactor neutron spectra. No k_0 value has been measured for ^{177}Lu; the value given in [6, 7] was calculated from the 2200 m/s neutron activation cross-section and the gamma-ray intensity.

No Q_0 values have previously been measured for ^{151}Eu$(n,\gamma)^{152}$Eu and ^{176}Lu$(n,\gamma)^{177}$Lu since the use of Q_0 values for these non-1/v reactions was proposed for the first time in [4]. For 1/v reactions, the Q_0 values can be determined using activation measurements with and without Cd cover, but the Q_0 values proposed in [4] for these non-1/v reactions are not easily related to activities measured with and without Cd cover. Rather, they should be thought of as the values needed to give consistent results with Eq. (1) when using neutron spectra with different f. Therefore, in this work the Q_0 values are measured by the 2-channel method [8] where activities produced in two irradiation channels with different f are compared.

Table 1 shows the parameters associated with the neutron activation of Eu and Lu taken from [7]. In the modified Westcott formalism [2], s_0 is a measure of the ratio of the epithermal integral to thermal (n,γ) cross-section and $g(20\ ^\circ C)$ and $g(100\ ^\circ C)$, from [1], are the Westcott g factors at these neutron temperatures. E_r are the effective resonance energies. For the reaction ^{153}Eu$(n,\gamma)^{154}$Eu, which is close to 1/v, Q_0 is the ratio of resonance integral to thermal (n,γ) cross-section. The k_0 and Q_0 values of ^{153}Eu$(n,\gamma)^{154}$Eu are measured in this work for completeness and to compare with the literature values.

Besides the highly non-1/v nuclides ^{151}Eu and ^{176}Lu, there are also a number of other non-1/v nuclides used in k_0-NAA [2]: ^{103}Ru, ^{115}In, ^{164}Dy, ^{168}Yb, ^{175}Lu, ^{191}Ir, ^{193}Ir, and ^{197}Au. For all of these, except ^{168}Yb, the Westcott $g(T_n)$ factor varies by less than 0.9 % [1, 9] as the neutron temperature varies from 20 to 60 °C, the range observed in commonly used reactor irradiation channels. There is thus almost no loss of accuracy if this variation is completely ignored and the Hogdahl formalism is used. Indeed, this is what has been done in the past and k_0 values for these slightly non-1/v nuclides were measured [3, 10, 11] assuming $g(T_n) = 1$. Therefore these k_0 values must continue to be used with the Hogdahl formalism and $g(T_n) = 1$. For ^{168}Yb, the Westcott $g(T_n)$ factor varies by 2.4 % [2, 9] as the neutron temperature varies from 20 °C to 60 °C and the k_0 values for ^{168}Yb were measured [6] using the Westcott formalism and taking $g(T_n)$ into account. To use ^{168}Yb with the extended Hogdahl method, new measurements of the k_0 values and Q_0 would be desirable.

Experimental

Neutron spectrum and gamma-ray detector characterization

Irradiation sites 1 and 8 of the Ecole Polytechnique Montreal SLOWPOKE reactor were used. They were previously characterized [8] for k_0 measurements. Site 1 was found to have $f = 18.0 \pm 0.2$ and $\alpha = -0.051$ while site 8 was found to have $f = 52.7 \pm 1.0$ and $\alpha = +0.018$. The f-values were verified in 2013 using Cd-ratio measurements with Au monitors and they were found not to have changed. A HPGe detector with relative efficiency 33 % was used and relative efficiencies were calculated as described in [8, 12–14].

Standards used for k_0-measurements

Eight Eu standards of approximate mass 100 µg were prepared using a 1000 ± 3 µg/mL Eu certified Specpure Plasma Standard Solution, density 1.020 ± 0.002 g/mL, purchased from Alfa Aesar, USA. Approximately 100 µL was pipetted onto a 16 mm × 80 mm strip of Whatman 50 filter paper with polyethylene backing and weighed

Table 1 Parameters of neutron activation of Eu and Lu

Reaction	Half-life	s_0	g (20 °C)	g (100 °C)	E_r (eV)	Q_0
^{151}Eu(n,γ)^{152m}Eu	9.312 h	1.20	0.901	0.831	0.448	
^{151}Eu(n,γ)^{152}Eu	13.54 years	1.25	0.901	0.831	0.448	
^{153}Eu(n,γ)^{154}Eu	8.593 years		0.974	0.952	5.80	5.66
^{176}Lu(n,γ)^{177}Lu	6.73 days	1.67	1.746	2.344	0.158	

immediately, using a stopwatch to correct for evaporation between the beginning of pipetting and weighing. After drying, the filter paper was rolled into a 6 mm diameter, 16 mm long cylinder and fixed with adhesive tape. Eight Lu standards of approximate mass 100 μg were prepared in a similar manner using a 1000 ± 3 μg/mL Lu certified Specpure Plasma Standard Solution, density 1.022 ± 0.002 g/mL, purchased from Alfa Aesar, USA. The Au reference standard was Al–Au wire, 1 mm diameter, containing 0.100 % Au (IRMM-530, Belgium); 10 mm lengths were used.

Measurement of Q_0-values

The two-channel method was used to determine Q_0-values for the ^{151}Eu(n,γ)^{152}Eu, ^{151}Eu(n,γ)^{152m}Eu, ^{153}Eu(n,γ)^{154}Eu and ^{176}Lu(n,γ)^{177}Lu reactions. The Q_0-values for ^{151}Eu(n,γ)^{152}Eu and ^{151}Eu(n,γ)^{152m}Eu should be similar because the neutron capture is to the same compound nucleus states which then de-excite by prompt γ emission to ^{152}Eu or ^{152m}Eu. The two-channel method involves irradiating a standard of the element, along with Au monitor, in two reactor irradiation channels with very different f, to determine the k_0-value of one of the gamma-rays emitted by the nuclide produced. For each k_0 determination, a Q_0-value is needed to correct for epithermal activation. The best Q_0-value is the one which gives equal k_0-values with the two channels. The equation needed to calculate the k_0-value can be derived from Eq. (1); it is:

$$k_0 = \frac{A_{sp}}{A_{sp,Au}} \frac{1 + Q_{0,Au}(\alpha)/f}{g(T_n) + Q_0(\alpha)/f} \frac{\varepsilon_{p,Au}}{\varepsilon_p} \quad (3)$$

where A_{sp} is the measured specific activity, ε_p is the gamma-ray detection efficiency and the subscript Au refers to the Au comparator. If we write similar equations for channel 1 and channel 2 and equate $k_{0,1}$ and $k_{0,2}$, we obtain the following:

$$\frac{A_{sp,1}}{A_{sp,Au,1}} \frac{1 + Q_{0,Au}(\alpha_1)/f_1}{g(T_{n,1}) + Q_0(\alpha_1)/f_1} = \frac{A_{sp,2}}{A_{sp,Au,2}} \frac{1 + Q_{0,Au}(\alpha_2)/f_2}{g(T_{n,2}) + Q_0(\alpha_2)/f_2} \quad (4)$$

The new Q_0-value is obtained by solving Eq. (4) for Q_0. It should be pointed out that when the Q_0-value is determined using Eq. (4), which stems from Eq. (1), it is therefore defined by Eq. (1). With this definition, the meaning of the Q_0-value is as follows: it is the value that needs to be used with Eq. (1) to give consistent results when using irradiation channels with different f.

The filter paper Eu and Lu standards were irradiated one at a time in sites 1 and 8 of the SLOWPOKE reactor along with Au monitors. The thermal neutron fluence rate in site 1 was approximately 5×10^{11} cm^{-2} s^{-1}. The irradiation time was 10 min to produce ^{152m}Eu, 6 h to produce ^{152}Eu and ^{154}Eu, and 1 h to produce ^{177}Lu. The neutron temperatures for these irradiations were estimated from the nearby moderator temperature by the method of Ref. [5] which uses available reactor thermocouple measurements. After decay times of approximately 1 day for ^{152m}Eu, ^{177}Lu and ^{198}Au and 8 days for ^{152}Eu and ^{154}Eu, the specific activities of these nuclides were measured by counting the samples at a distance of 10 cm from a 33 % efficiency HPGe detector. A duplicate set was irradiated and counted 1 week later. The gamma-ray peak areas and specific activities were calculated using the EPAA software [15].

Measurement of k_0-values

The same measurements that were used to determine the Q_0-values were also used to determine the k_0-values. The values were calculated from the measured specific activities using Eq. (3). For each calculation of a k_0-value, the Q_0-value used in Eq. (3) was the mean of all the measured values. The relative detection efficiencies were calculated as described previously [8, 12–14]. These calculations included corrections for coincidence summing, which were up to 3.1 % for the samples counted 10 cm from the detector. Neutron self-shielding was negligible in these standards; using Sigmoid [16] the thermal neutron self-shielding correction was calculated to be 0.2 % for ^{151}Eu(n,γ)^{152}Eu and 0.004 % for ^{176}Lu(n,γ)^{177}Lu.

Variation of results with neutron temperature

The reactor was operated with and without cooling of the pool water to give temperature variations up to 25 K; these variations are greater than those normally encountered in routine NAA work. The temperature of the water moderator entering and exiting the reactor core was measured

with thermocouples and these readings were converted to estimated neutron temperatures at the irradiation sites by the method of Ref. [5]. A number of 100 μg samples were irradiated in sites 1 and 8 to verify the accuracy of the method for neutron temperature variations up to 25 K. The Eu and Lu concentrations were calculated by Eq. (1).

Results and discussion

Q_0-values

To convert $Q_0(\alpha)$ to $Q_0(\alpha = 0)$ and vice versa, the following relation from [7] was used:

$$Q_0(\alpha) = \frac{Q_0 - 0.429}{E_r^{\alpha}} + \frac{0.429}{(2\alpha + 1)E_{cd}^{\alpha}} \quad (5)$$

with $E_{Cd} = 0.55$ eV. This relation is physically justified only for 1/v reactions. However, for the non-1/v reactions ^{151}Eu(n,γ)^{152}Eu and ^{176}Lu(n,γ)^{177}Lu, because of the low Q_0-values and E_r-values, using Eq. (5) or just using $Q_0(\alpha) = Q_0(\alpha = 0)$ makes only a 0.2 % difference in calculated k_0-values or analysis results even for an irradiation channel with $f = 18.0$ and $\alpha = -0.051$. As in [4], it was decided to use Eq. (5) so that the same relation could be used for 1/v and non-1/v reactions.

Table 2 shows the Q_0-values measured by the two-channel method and solving Eq. (4) for Q_0. Each value is the mean of two measurements. The uncertainties (combined standard uncertainties) include a contribution of 0.2 from the uncertainties in the measured specific activities (0.3 for ^{154}Eu), 0.2 from the uncertainties of the estimated neutron temperatures (0.0 for ^{154}Eu) and 0.1 from the uncertainties of f and α of the two irradiation channels used.

The measured Q_0-value for ^{153}Eu(n,γ)^{154}Eu, 5.1, compares fairly well with the literature value of 5.66. The Q_0-values for ^{151}Eu(n,γ)^{152}Eu and ^{151}Eu(n,γ)^{152m}Eu should be similar since they are essentially the same reaction. The observed difference, between 0.1 and 0.5, must be due to experimental error and we suggest that the mean value, 0.3 ± 0.2, should be used for both reactions. The uncertainty of this value may appear large but in fact it is not that important because even in poorly thermalized site 1 of this reactor, which has $f = 18.0$, only 1.7 % of the total ^{152}Eu activity is produced by epithermal neutrons. The Q_0-value 0.3 ± 0.2 cannot be directly compared with the literature s_0-values of 1.20 and 1.25 because the s_0-values are used with the Westcott convention. For ^{176}Lu(n,γ)^{177}Lu, the measured Q_0-value of 3.2 cannot be directly compared with the literature s_0-value, but again it is fairly small and indicates that even in poorly thermalized irradiation channels activation by epithermal neutrons is not very important.

Table 2 Measured Q_0-values

Reaction	Half-life	This work		Literature [7]		
		Q_0	Unc.	Q_0	s_0	Unc.
^{151}Eu(n,γ)^{152m}Eu	9.312 h	0.1	0.3		1.20	–
^{151}Eu(n,γ)^{152}Eu	13.54 years	0.5	0.3		1.25	–
^{153}Eu(n,γ)^{154}Eu	8.593 years	5.1	0.4	5.66		–
^{176}Lu(n,γ)^{177}Lu	6.73 days	3.2	0.3		1.67	–

k_0-values

Table 3 shows the k_0-values measured in this work. The uncertainties include the contributions from the estimated uncertainties in the amount of the element in the standard, the measured specific activities, the relative gamma-ray detection efficiencies, the estimated neutron temperatures, the Q_0-values, and f and α. A 5 K uncertainty in the neutron temperature was assumed; this contribution would lead to a 0.5 % uncertainty in the k_0-values for ^{152}Eu and a 2 % uncertainty in the k_0-values for ^{177}Lu.

Looking first at the k_0-values for ^{154}Eu, which is produced by a nearly 1/v reaction and which were all calculated by the equation of the Hogdahl formalism using $g(T_n) = 1$, we see that for the more intense gamma-rays the k_0-values of this work are generally 6–8 % higher than the literature values. It is speculated that these differences may be due to the Eu standards used, one of them being inaccurate by 6 %. The literature values are the means of k_0 measurements [3, 17] performed in two different laboratories using different types of Eu standards. The k_0-value for the 123.1 keV gamma-ray of ^{154}Eu is difficult to measure because the peak is generally not well resolved from the more intense 121.8 keV peak of ^{152}Eu. In this work the peaks were resolved by counting the standards on a germanium low-energy photon spectrometer which had 0.7 keV resolution FWHM at 122 keV.

For the non-1/v nuclides ^{152}Eu and ^{152m}Eu, the k_0-values determined in this work with the extended Hogdahl formalism and Eq. (3) are not directly comparable with those determined previously using the Westcott formalism even if epithermal activation is negligible, because the equation for k_0 of the Westcott formalism has $g(T_n)$ of the Au monitor in the numerator [2]. In this work the typical neutron temperature was 30 °C, where $g(T_n)$ for ^{197}Au(n,γ)^{198}Au is 1.007 [1]. This implies that, everything else being equal, the literature k_0-values for ^{152}Eu and ^{152m}Eu should be 0.7 % higher than those of the present work. In fact, the opposite was observed. The k_0-values measured here for ^{152}Eu are generally 5–8 % higher than the literature values. It is speculated that the reason for

Table 3 Measured k_0-values compared to literature values

Nuclide	Gamma energy (keV)	This work		Literature [7]	
		k_0	Unc %	k_0	Unc %
^{152m}Eu	121.8	1.84	3	1.48	–
	344.2	0.59	2	0.498	–
	841.6	3.52	2	3.02	–
	963.3	2.89	2	2.49	–
^{152}Eu	121.8	13.7	3	12.8	0.8
	244.7	3.57	2	3.44	0.3
	344.3	12.5	2	11.9	0.9
	444.0	1.51	2	1.39	1.2
	778.9	6.16	2	5.70	0.8
	867.4	2.02	2	1.88	0.9
	963.4	7.00	2	6.46	0.4
	1084.0	4.80	2	4.57	0.4
	1112.0	6.49	2	6.07	0.8
	1408.0	9.80	2	9.36	0.6
^{154}Eu	123.1	1.49	4	–	–
	248.0	0.155	2	0.155	–
	591.8	0.117	2	0.108	1.5
	723.3	0.479	2	0.446	1.5
	756.9	0.121	2	0.108	–
	873.2	0.298	2	0.272	1.4
	996.4	0.230	2	0.230	–
	1004.8	0.584	2	–	–
	1274.4	0.826	2	0.777	1.1
^{177}Lu	112.9	0.0432	4	0.0415*	–
	208.4	0.0730	3	0.0714*	–

The literature values for ^{177}Lu were calculated from the 2200 m/s neutron activation cross-section and the gamma-ray intensities

these differences may be the same as for the case of ^{154}Eu since they were measured at the same time using the same standards. The uncertainties given in the literature [6, 7] are based mainly on the observed differences between independent measurements carried out in two different laboratories. For ^{152m}Eu, the disagreement is much worse: the k_0-values measured here for the 841.6 and 963.3 keV gamma-rays are 16–17 % higher than the literature values. Here the literature values [6] are only tentative; they are the means of two measurements carried out in one laboratory using Eu_2O_3 heated to remove the H_2O, dissolved in nitric acid, and pipetted onto filter paper. The reason for the 16 % discrepancy between the measurements of this work and the literature values is not known.

The discrepancies pointed out here between k_0-values measured with different Eu standards should not be considered as a weakness of the k_0-method. On the contrary, if the relative method had been used, with the same standards, then many inaccurate results would have been reported and the errors would never have been discovered. With the k_0-method, since several standards are used to verify the same k_0-values, poor standards will eventually be discovered and weeded out. This should be considered as an advantage of the k_0-method.

For ^{177}Lu the difference between the k_0-value measured here for the 208 keV gamma-ray and the literature value calculated from the 2200 m/s neutron activation cross-section and the gamma-ray intensity is 2.2 %. It was calculated that a 6 °C change in the neutron temperatures estimated in this work would account for this 2.2 % difference. Stated differently, if the Lu standard had been used as a neutron temperature monitor it would have given neutron temperatures 6 °C higher than those deduced from the thermocouple readings. This is reasonable because it is estimated that the neutron temperatures of this work have an uncertainty of 5 °C. Recall that the present method proposes to move away from using a Lu standard, the 2200 m/s neutron activation cross-section, the 208 keV gamma-ray intensity and Holden's $g(T_n)$ calculations [1] to determine the neutron temperature and to use thermocouple readings instead.

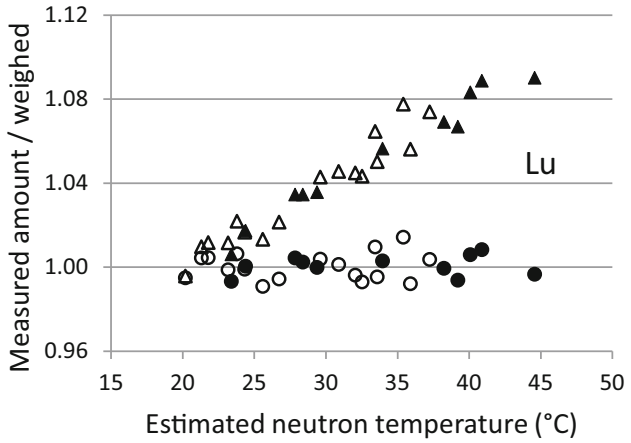

Fig. 1 Lu measured using the method proposed here, *triangles* not corrected for temperature, *closed symbols* irradiation site 1, *open symbols* irradiation site 8

Accuracy of the method for varying neutron temperatures

With the reactor operating at various temperatures, the amounts of Eu and Lu were measured in 100 μg prepared standards by the method proposed here and calculated by Eq. (1). For Lu the 208.4 keV gamma-ray was used with Q_0 and k_0 values from Tables 2 and 3. The results are shown by the circles in Fig. 1. The triangles were calculated without temperature correction, i.e., using a constant $g(20\ °C)$ in Eq. (1). The results for Eu (using the ^{152m}Eu 842 keV gamma-ray) were similar although the variation of the uncorrected data with neutron temperature was about 4 times less, decreasing by about 2.5 % over the 25 °C temperature range.

These results confirm that the proposed method gives accurate results over a neutron temperature range of 25 °C. The consistency of the results for irradiation sites 1 and 8 is not a confirmation that the method gives consistent results for different irradiation channels because the Q_0 values measured here were adjusted to give consistent results for the two irradiation sites.

Conclusions

The measurements of this work confirm that the proposed simplified method using the Hogdahl formalism extended with a $g(T_n)$ factor and neutron temperatures from thermocouple readings can give k_0-NAA results for Eu and Lu accurate to about 1 %. However, accurate k_0-values are needed. The present k_0 measurements did not confirm the previously published values; they were higher by 6 % for ^{154}Eu and ^{152}Eu and by 16 % for ^{152m}Eu. Does this mean that all Eu determinations up to now using the k_0-method are in error by 6–16 %? Further k_0 measurements with different standards are needed to answer this question.

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