



# Combination of $k_0$ -IAEA and KayWin for the characterization of neutron flux parameters at Triga Mark II research reactor, Morocco

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## Abstract

The aim of this work was the characterization of the neutron flux parameters in two irradiation facilities; pneumatic tube system (PTS) and rotary specimen rack (RSR); at the Moroccan Triga Marck II research reactor using  $k_0$ -IAEA and KayWin softwares. The analysis of several combinations of flux monitor sets is the basis for this study. The efficiency calibrations of the detector used have been carried out using the  $k_0$ -IAEA software and then exported to the KayWin software for the determination of the  $f$  (thermal to epithermal neutron flux ratio) and  $\alpha$  (deviation from the  $1/E$  distribution) parameters.

**Keywords** Neutron activation analysis ·  $k_0$ -IAEA · KayWin ·  $f$  (thermal-to-epithermal ratio) ·  $\alpha$  (deviation from the  $1/E$  distribution)

## Introduction

According to the accepted convention, the energy distribution of the neutrons generated in nuclear research reactors is described by flux parameters. Accurate knowledge of these parameters is crucial when using neutron activation analysis (NAA) to simulate the activation of target nuclides in samples.

There are several methods available for quantitative calibration in neutron activation studies, including relative method using single or multiple standards, comparative method, and absolute method. An example of a single comparator method is the  $k_0$  method, proposed by De Corte et al. [1].

The epithermal neutrons flux is assumed to be inversely proportional to the energy of the neutrons in "ideal" reactors and isotopic neutron sources [2]. However, one can expect epithermal neutron spectra at actual irradiation sites to deviate from this ideal  $1/E$  form [1–3]. It has been shown that

these deviant spectra follow approximately a  $1/E^{1+\alpha}$  ( $\alpha > 0$  or  $\alpha < 0$ ) [4, 5]. In other hand, epithermal neutrons contribute significantly to, or perhaps entirely, the activity generated in many analytical activation procedures. When this is the case, and when using resonance integrals, in addition to absolute or single comparator approaches, the impact of non-ideality of the epithermal spectrum should not be undervalued [6].

The determination of the alpha neutron flux parameter (epithermal flux distribution parameter), which takes into account the epithermal neutron flux deviation from the ideal  $1/E$  law by a  $1/E^{(1+\alpha)}$  form, as well the parameter  $f$  (thermal to epithermal neutron flux ratio) should be used in the  $k_0$  standardization method of the neutron activation analysis ( $k_0$ -NAA).

During the development of the  $k_0$  standardization method, different approaches for determining the mentioned parameters ( $\alpha$  and  $f$ ) have been suggested [7, 8]. The "Cd-ratio for multi-monitor" approach, which yields the most trustworthy results if a good choice of monitors is chosen, is advised to be used when the reactor's neutron spectrum is steady and the irradiation of a sheet of cadmium covering is permitted. The "bare multi-monitor" method, which is suggested for various multifunctional reactors with frequently changing neutron flux parameters since these monitors can be co-irradiated with the samples to be studied, is a method for calculating the neutron flux parameters. The single comparator method, the  $k_0$  method, as well as newer technique that have developed through

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time, like the Au-Zr method or the triple and multi-monitor approach, have all been applied in several laboratories throughout the world. Zinc has been employed as a comparator since the early 1970s, and over the past years, a hybrid of the  $k_0$  approach with the  $k_{Zn}$  catalog of calibration factors has been established together with the reactor core fuel enrichment [8, 9]. The neutron spectrum's parameters were determined using the  $^{95-97}\text{Zr}$ - $^{198}\text{Au}$  method in order to convert the  $k_{Zn}$  calibration factors.

Since then, in particular for long-term irradiations, the Au-Zr monitor approach has been advised for the determination of neutron flux parameters [10, 11].

However, some researches have revealed that the Zr-Au method is not particularly acceptable for daily application as a monitor between typical samples under routine circumstances [12]. It is difficult to integrate the measurement of the induced radioactivity of  $^{95-97}\text{Zr}$  and  $^{198}\text{Au}$  into a routine analysis protocol (e.g., irradiation for 1–4 h, decay for 3–5 days, and measurement for 1–4 h) where real samples are measured. The  $^{97}\text{Zr}$  at 743 keV peak's statistics will not be satisfactory. To comply with these restrictions, studies on new modified flux monitors have to be created [12]. This can be achieved by using the reaction  $^{50}\text{Cr}(n,\gamma)^{51}\text{Cr}$  to measure the thermal neutron flux rate directly and once it has been determined, it is considerably simpler to use two other acceptable nuclear reactions to determine the epithermal neutron flux rate and the epithermal spectrum parameter by selecting of Au and Mo for monitoring the rate of epithermal flux using the reactions  $^{197}\text{Au}(n,\gamma)^{198}\text{Au}$  and  $^{98}\text{Mo}(n,\gamma)^{99}\text{Mo}$  [12].

Similar to  $^{97}\text{Zr}$ , epithermal activation can be detected, but  $^{99}\text{Mo}$ 's substantially longer half-life (2.7 days when compared to 16.9 h) makes it more appropriate for usage following a normal decay time check of three to five days.

In fact, CNESTEN has implemented a variety of neutron activation analysis techniques since the installation of the 2 MW TRIGA Mark II reactor in Morocco in 2008. These techniques include the relative method using NADA software and the  $k_0$  method using  $k_0$ -IAEA software. The research lab is currently attempting to introduce and assess the performance of KayWin software in order to compare it with the prior software utilized within the laboratory [13]. For the most common irradiation channels of the Triga Mark II research reactor, neutron flux characterization is performed using the KayWin program.

Therefore, the current study's goal was to calculate the neutron flux's parameters using both  $k_0$ -IAEA and KayWin software and compare them to those of earlier research done in this reactor in 2014 [14]. The determination of  $\alpha$

and  $f$  parameters was performed using different monitor sets (Zr-Au, Au-Zr-Zn and a combination of standard liquids (JML code) used as flux monitor at two typical irradiation sites of the Moroccan TRIGA Mark II research reactor, mainly the pneumatic transfer system (PTS) and the rotary specimen rack (RSR) using the KayWin program.

## Experimental

NAA laboratory at CNESTEN is connected with the Triga Mark II research reactor via two facilities for the irradiation of samples: the first one is used to irradiate samples with the pneumatic transfer system (PTS) from a station located at the NAA laboratory and the second one to irradiate samples at the rotary specimen rack (RSR) which present 40 irradiation positions. The pneumatic tube system (PTS) is usually used for the determination of short half-lived elements, and the rotary specimen rack (RSR) is used for the determination of medium- and long-lived elements.

A complete calibration of the detector to be used was carried out. This was performed using HPGe coaxial detector and calibration using  $k_0$ -IAEA; the with specifications such as a relative efficiency of 30%, a crystal length of 76.3 mm, a diameter of 58.8 mm, and a resolution of 1.95 keV at 1333.5 keV for gamma rays from  $^{60}\text{Co}$ . The peak/total ratio was assessed using a single energy peak source of  $^{137}\text{Cs}$  point source and peak energy efficiency calibration was made using a point source of  $^{152}\text{Eu}$ . Areva-certified  $^{152}\text{Eu}$  source of 43 kBq was counted at the nearest distance to induce 19% downtime (DT). At all distances from the end cap used in practice and on the end cap itself, a  $^{137}\text{Cs}$  source of 40 kBq was measured. For all measurements, peak/total ratio and efficiency calibration were determined using  $k_0$ -IAEA software. Then, all the data were exported to the KayWin software, in a test file format, to be used in the determination of  $\alpha$  and  $f$  parameters.

Irradiation without Cd covers was mainly applied for prior monitoring of  $\alpha$  and  $f$  in irradiation positions, which includes the triple and multi-monitor approach and the Zirconium method. For the Zirconium Method, a set of Al (99.9%)–Au (0.1%) alloys and Zr (99.8%) was used. Zr's foil had been folded twice and Al-Au wire had been pelletized so they could all be irradiated in the same capsule. These foils were placed on the detector for counting.

For the multi-monitor method, NIST-certified standards liquids solutions (JML code) pipetted on filters containing Mo, Au, Cr, and Mn were also used. These solutions were made by taking 50  $\mu\text{l}$  of 1000 ppm Mo, Cr, and Mn

solutions for each. For the triple monitor method, a combination of neutron spectrum monitors containing Au, Mo, Zn were used which having been prepared in the same way as described previously. All of these monitor combinations were irradiated into the pneumatic transfer system (PTS) and the rotary specimen rack (RSR).

To study the accuracy of the results in this work, SRM 2709a-San Joaquin Soil and SRM 1547- peach leaves from NIST were irradiated in both irradiation channels.

Samples and flux monitors made of zinc wire were prepared in small polyethylene capsules before being transferred to a polyethylene rabbit for irradiations. Rabbits were irradiated for 30 s to 1 min for short irradiation, and for 5 h for long irradiations. Measurements were performed at such distances that the dead time was kept below 10% with negligible random coincidences. The detector used was connected to DSPEC Jr 2.0 Digital Signal Processing Gamma Ray Spectrometer from ORTEC company. The acquisition of spectra was done using maestro multichannel analyzer emulation software. The hyper lab program was used for the fit of peak areas and the adjustment of spectrum used by the KayWin software.

## Results and discussion

Before the characterization of the reactor can be done, the used detector need to be calibrated. Detector was calibrated in accordance with the  $k_0$  approach using the  $k_0$ -IAEA

software. The determinations of the peak to total ratio and the efficiency at different geometries from sources to detector are the two key steps in the characterization process.

A mono-energetic source which emits only one gamma ray and a multi-energetic source that produces multi-gamma rays simultaneously are both required. Both sources must be measured with the detector long enough time so that the peak statistics in each major peak of each radionuclide are better than 1%. The single radionuclide source was measured in different positions. The multi-energetic source was counted at a precisely known distance from the detector.

The peak detection efficiency ( $\epsilon_p$ ) is used for the determination of concentration and the parameters  $f$  and  $\alpha$ . The energy range of the gamma sources used extends from 45 to 1408 keV. A better fit is obtained for experimental points in regions distinct by polynomials.

$$\text{Log}\epsilon_p = a_1 + a_2\text{Log}E_\gamma + \dots + a_n(\text{Log}E_\gamma)^n.$$

In order to calculate the correction factor, the peak detection efficiency at total energy ( $\epsilon_p$ ) and the total detection efficiency for the gamma lines of interest must be known. Both are characteristics of the detector.

Therefore, the total detection efficiency is obtained by determining the  $P/T$  ratio, which is an experimentally measurable amount and can be determined via the  $P/T$  ratio:

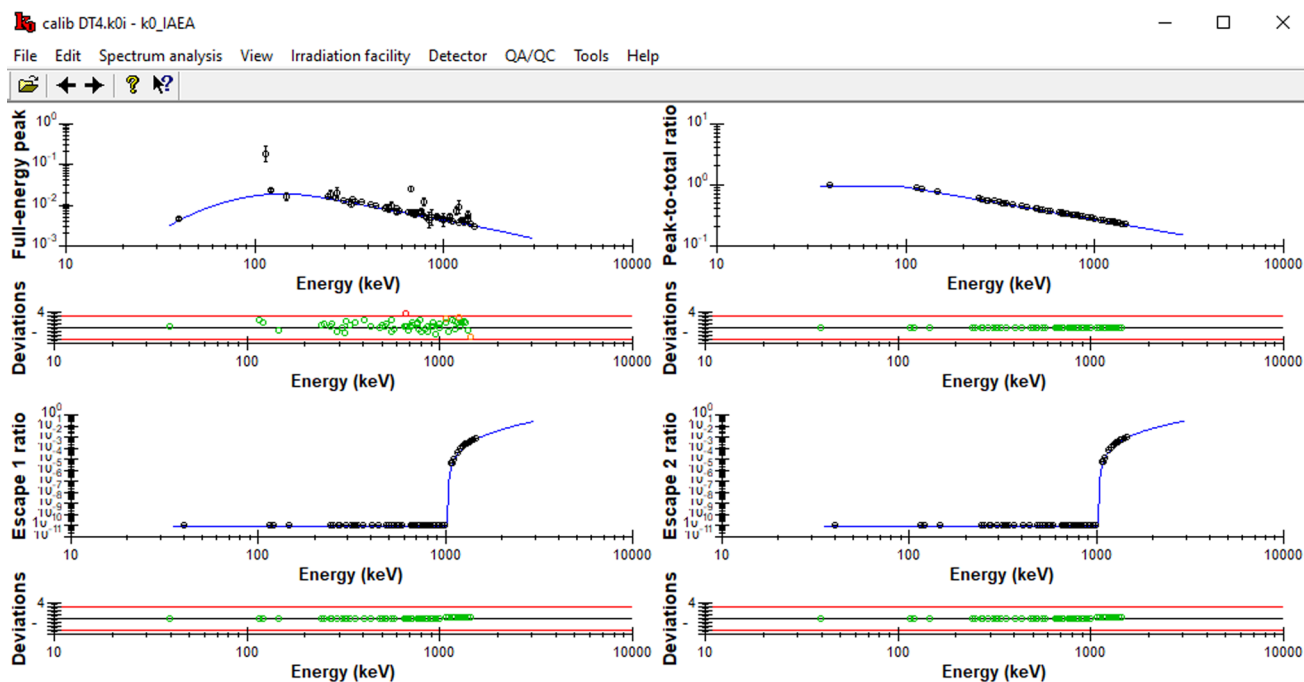


Fig. 1 Example of efficiency calibration and peak/total ratio curves obtained by  $k_0$ -IAEA

$$\frac{\epsilon_p}{\epsilon_t} = \frac{P}{T}$$

The latter depends on the following parameters: the energy of the photon; the source-detector distance; the composition and geometry of the source and the presence of absorbent and diffusing materials.

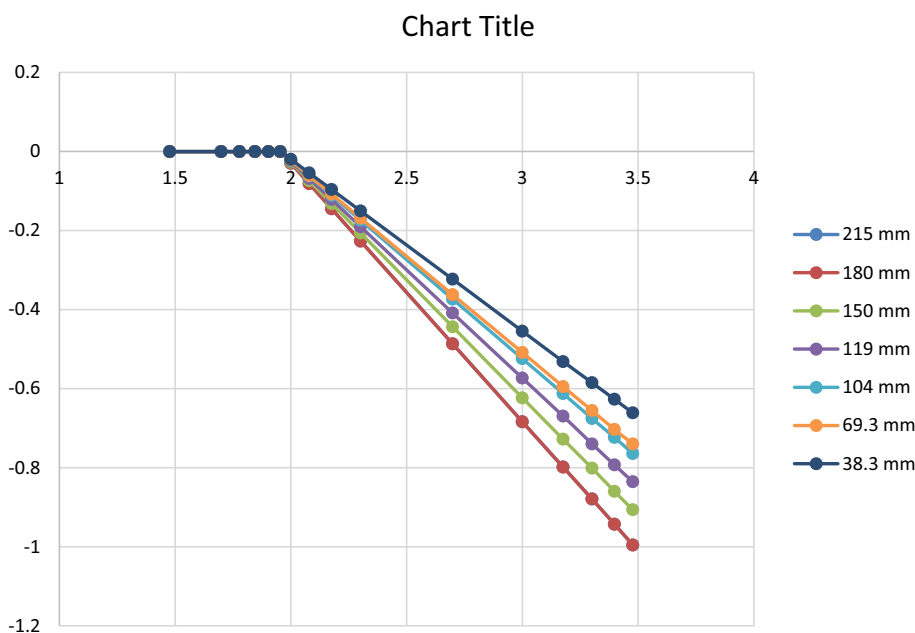
Following the procedure suggested by De Corte [15], we determined  $P/T=f(E\gamma)$ . Figure 1 displays an illustration of the efficiency results using the  $^{152}\text{Eu}$  source with an example of a  $P/T=f(E\gamma)$  curve obtained by the  $k_0$ -IAEA software.

For peak-to-total calibration a  $^{37}\text{Cs}$  source was counted at reference position (215 mm). Then, it was also counted at 215 mm, with the  $^{152}\text{Eu}$  source at the closest distance with

20% dead time (DT). In order to correct for the dead time, the ratio of the count rates in the 662 keV peak at 20% and 0% DT gives an excellent result, proving that the detector is capable of being operated dependably with dead time less than 20%. Although in our laboratory (10% DT) is the most that can be used [16].

At all distances from the end cap used in practice, as well as directly on the end cap,  $^{152}\text{Eu}$  source was counted with a statistical error less than 1%. The 1529 keV sum peak requires a precision better than 1% for the end-cap measurement. By using the  $k_0$ -IAEA software for fitting, the peak areas were identified. Figure 2 represents  $\text{Log}(P/T)=f(\text{Log } E\gamma)$  for the different positions used in this work.

**Fig. 2** Experimental  $P/T$  curve for the different positions



**Table 1**  $\alpha$  &  $f$  parameters obtained for the PTS and RSR facility

| Monitor  | PTS              |                       | RSR              |                       |
|----------|------------------|-----------------------|------------------|-----------------------|
|          | $f \pm U_f$      | $\alpha \pm U_\alpha$ | $f \pm U_f$      | $\alpha \pm U_\alpha$ |
| Au-Zr    | $19.68 \pm 0.65$ | $-0.031 \pm 0.001$    | $37.87 \pm 1.73$ | $-0.034 \pm 0.005$    |
| Au-Zr-Zn | $18.05 \pm 0.99$ | $-0.042 \pm 0.0012$   | $35.6 \pm 0.45$  | $-0.043 \pm 0.02$     |
| JML      | $19.74 \pm 0.49$ | $-0.023 \pm 0.006$    | $38.4 \pm 2.8$   | $-0.047 \pm 0.06$     |

**Table 2** Comparison of  $\alpha, f, \emptyset_{th}, \emptyset_e$  in present work with previous work done in TRIGA Mark II reactor

| Study        | Experiment date | Channels | $\alpha \pm U_\alpha$ | $f \pm U_f$      | $\emptyset_{th} \pm U_{\emptyset_{th}}$<br>$10^{12}\text{cm}^{-2}\text{s}^{-1}$ | $\emptyset_e \pm U_{\emptyset_e}$ |
|--------------|-----------------|----------|-----------------------|------------------|---|-----------------------------------|
| Last work    | 2013            | PTS      | $-0.02 \pm 0.003$     | $22.82 \pm 0.95$ | $6.20 \pm 0.21$   | $2.28 \pm 0.18$                   |
|              |                 | RSR      | $-0.04 \pm 0.013$     | $37.33 \pm 0.82$ | $2.09 \pm 0.16$   | $0.27 \pm 0.05$                   |
| Present work | 2020            | PTS      | $-0.028 \pm 0.34$     | $19.46 \pm 0.06$ | $4.86 \pm 0.23$   | $2.09 \pm 0.01$                   |
|              |                 | RSR      | $-0.038 \pm 1.13$     | $37.29 \pm 0.12$ | $1.85 \pm 0.11$   | $0.18 \pm 0.07$                   |

The Kayzero for Windows was used to calculate  $f$  and  $\alpha$  using the results of peak to total ratio and efficiency calibration obtained by  $k_0$ -IAEA software. For this reason, different monitor sets that were previously prepared were irradiated in the typical facilities (PTS and RSR).

The results obtained for  $\Phi_{th}$  (thermal flux),  $\Phi_e$  (epithermal flux),  $f$  and  $\alpha$  in the two irradiation facilities of CNESTEN's TRIGA Mark II research reactor are presented in Tables 1 and 2. In Table 2 we used the average  $f$  and  $\alpha$  from the results obtained from the different flux monitor used (Table 1).

It is noted that the  $\alpha$  parameter in the PTS and RSR irradiation channels have a negative value, which is very small and cannot significantly influence the final result obtained by the  $k_0$  method. This negative value can be explained by a lower thermalisation as described in [16]. It is also noted that the  $f$  parameter in the RSR is higher than in the PTS since the RSR channel is located in a graphite reflector and is relatively far from the reactor core, while the PTS is in the core. The measured  $f$  values show that the neutrons at PTS are well thermalized than at the RSR.

In the PTS channel the  $f$  values vary between 18.95 and 19.74 as long as the  $\alpha$  values vary between  $-0.042$  and  $-0.023$ . We notice that there is a great consistency between the  $f$  parameter measured using the "Au-Zr" flux monitor and the one measured by the "JML" monitor: on the other hand, the  $f$  parameter found by the "Au-Zr-Zn" monitors is slightly smaller than the others.

In the RSR channel the  $f$  values vary between 35.68 and 38.45, and the  $\alpha$  value varies between  $-0.047$  and  $-0.034$ . We also notice that the  $f$  setting found by the monitor "Au-Zr-Zn" is slightly smaller than the settings found by the monitors "Au-Zr" and "JML", while there is a good consistency between these two sets.

As far as the flux is concerned, the  $\Phi_{th}$  in the PTS channel is at least 10 times larger than that of the RSR channel, which also verifies the difference in the previously explained  $f$  setting between these two channels.

Table 2 includes all the results obtained by the previous study carried out in 2014 using the  $k_0$ -IAEA software and the present study carried out by the KayWin software. It is noted that there is a slight deviation between the results obtained by the previous study compared to the results found in this study but in general, it can be assumed that the deviation of the  $f$  and  $\alpha$  parameters between the two studies is acceptable and therefore it can be concluded that all reported results have an acceptable level of consistency with those of previous study.

In order to verify the experimental parameters of this study, irradiations were carried out in the irradiation positions under study using the certified reference materials: San Joaquin Soil (SRM 2709a) and SRM 1547- peach leaves from NIST. The accuracy of the samples in terms of concentration was statistically evaluated using the En factor as described in [12, 16] for comparison between experimental results and certified values.

Using En number, performance levels are normally determined as follows:  $|En| \leq 1$  satisfactory performance,  $|En| \geq 1$  unsatisfactory performance. Tables 3 and 4 show that results obtained in this work agree with the assigned values within the uncertainty margins, resulting in  $|En| \leq 1$ , thus proving the correct performance of  $k_0$  standardization method using KayWin software.

The expanded uncertainties of the certified values ( $k=2$ ) were taken from the certificates, but for those that lacked certified uncertainties, they were set at 10% in relation to the indicated values in the certificates. This was done so we could use the same criterion as for certified values. This can also be considered a reasonable estimate of uncertainty, because the relative uncertainties of certified values are frequently between 1 and 7%, and it seems reasonable to assume that the uncertified uncertainties may be somewhat larger than their certified equivalents. Therefore, the results are consistent with the certified values except for Ce, Cs, Hf and Nd in the case of the San Joaquin Soil (SRM 2709a) and for Sc in the case of SRM 1547- peach leaves from NIST. This good consistency indicates that the parameters of the reactor's neutron flux were well determined.

## Conclusion

The combination of the  $k_0$ -IAEA and KayWin used for the characterization of neutron flux parameters at the Triga Mark II research reactor was the most significant accomplishments of this work.

To achieve this, the coaxial HPGe detector used in our laboratory had to be calibrated. This calibration included the absolute efficiency calibration, the peak-to-total calibration in the range where the samples being analyzed would be counted, the FWHM and energy calibrations using point radioactive sources ( $^{137}\text{Cs}$  and  $^{152}\text{Eu}$ ) with  $k_0$ -IAEA software and then exported to the KayWin in order to characterize the neutron flux parameters, mainly  $f$  and  $\alpha$  in the PTS and RSR using the different flux monitor sets.

In general, it can be said that there has been a great deal of agreement between the element contents determined in this work and the certified and non-certified values for the standard reference materials used in this work (San Joaquin Soil (SRM 2709a) and SRM 1547- peach leaves from NIST).

**Table 3** Results obtained for the standard reference materials: San Joaquin Soil (SRM 2709a)

| Elements | Certified values<br>Xref ± Uref (mg kg <sup>-1</sup> ) | K0 for windows values<br>Xlab ± Ulab (mg kg <sup>-1</sup> ) | Relative bias (%) | En   |
|----------|--|---|-------------------|------|
| Al       | 73,700 ± 1600  | 72,042 ± 10,549   | 2.25              | 0.16 |
| As       | 10.5 ± 0.3   | 10.4 ± 0.8  | 0.99              | 0.13 |
| Ba       | 979 ± 28   | 1074 ± 98   | 9.72              | 0.93 |
| Ca       | 19,100 ± 900   | 18,259 ± 1377   | 4.40              | 0.51 |
| Ce       | 42 ± 1   | 49 ± 5  | 18.29             | 1.57 |
| Co       | 12.8 ± 0.2   | 13.7 ± 1.0  | 7.37              | 0.92 |
| Cr       | 130 ± 9  | 132 ± 14  | 1.93              | 0.15 |
| Cs       | 5.0 ± 0.1  | 5.6 ± 0.4   | 12.99             | 1.37 |
| Dy       | 3.0 ± 0.3  | 2.83 ± 0.2  | 5.59              | 0.46 |
| Eu       | 0.83 ± 0.02  | 0.82 ± 0.10   | 1.61              | 0.13 |
| Fe       | 33,600 ± 700   | 34,314 ± 2668   | 2.13              | 0.26 |
| Hf       | 4.0 ± 0.4  | 5.7 ± 0.4   | 42.66             | 2.87 |
| K        | 21,100 ± 600   | 20,070 ± 3085   | 4.88              | 0.33 |
| La       | 21.7 ± 0.4   | 20.6 ± 1.5  | 4.78              | 0.65 |
| Mn       | 529 ± 18   | 552 ± 45  | 4.45              | 0.48 |
| Na       | 12,200 ± 300   | 12,579 ± 935  | 3.11              | 0.39 |
| Nd       | 17.0 ± 1.7   | 26.0 ± 3.2  | 52.63             | 2.48 |
| Rb       | 99 ± 3   | 105 ± 19  | 6.42              | 0.32 |
| Sb       | 1.55 ± 0.06  | 1.63 ± 0.13   | 5.07              | 0.55 |
| Sc       | 11.1 ± 0.1   | 11.7 ± 0.8  | 5.35              | 0.70 |
| Sm       | 4.0 ± 0.4  | 4.0 ± 0.5   | 1.51              | 0.09 |
| Sr       | 239 ± 6  | 239 ± 16  | 0.19              | 0.03 |
| Ta       | 0.70 ± 0.07  | 0.76 ± 0.13   | 8.98              | 0.43 |
| Ti       | 3360 ± 70  | 3389 ± 308  | 0.88              | 0.09 |
| V        | 110 ± 11   | 117 ± 9   | 7.06              | 0.53 |
| Yb       | 2.0 ± 0.2  | 2.1 ± 0.2   | 5.87              | 0.42 |
| Zn       | 103 ± 4  | 114 ± 10  | 10.71             | 0.99 |
| Zr       | 195 ± 46   | 255 ± 34  | 31.27             | 1.06 |

**Table 4** Results obtained for the standard reference materials: Peach leaves (SRM 1547)

| Elements | Certified values<br>Xref ± Uref (mg kg <sup>-1</sup> ) | K0 for windows values<br>Xlab ± Ulab (mg kg <sup>-1</sup> ) | Relative bias (%) | En   |
|----------|--|---|-------------------|------|
| Al       | 248.9 ± 6.5  | 243.8 ± 23.8  | 2.05              | 0.21 |
| Ba       | 123.7 ± 5.5  | 121.0 ± 8.4   | 2.13              | 0.26 |
| Br       | 11 ± 1   | 12 ± 1  | 14.86             | 1.05 |
| Ca       | 15,590 ± 160   | 15,967 ± 1230   | 2.42              | 0.30 |
| Ce       | 10 ± 1   | 10.0 ± 0.7  | 5.96              | 0.48 |
| Fe       | 219.8 ± 6.8  | 227.3 ± 16.7  | 3.44              | 0.42 |
| K        | 24,330 ± 380   | 24,401 ± 2110   | 0.29              | 0.03 |
| La       | 9.0 ± 0.9  | 9.0 ± 0.7   | 7.39              | 0.58 |
| Mg       | 4320 ± 150   | 4564 ± 460  | 5.67              | 0.51 |
| Mn       | 97.8 ± 1.8   | 100.4 ± 7.8   | 2.65              | 0.32 |
| Na       | 23.8 ± 1.6   | 27.2 ± 4.0  | 14.50             | 0.80 |
| Rb       | 19.65 ± 0.89   | 19.68 ± 1.57  | 0.16              | 0.02 |
| Sc       | 0.04 ± 0.002   | 0.05 ± 0.002  | 22.74             | 1.46 |
| Sm       | 1.0 ± 0.1  | 1.07 ± 0.08   | 6.98              | 0.54 |
| V        | 0.37 ± 0.04  | 0.34 ± 0.02   | 8.33              | 0.67 |

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## Declarations

**Conflict of interest** No potential conflict of interest was reported by the authors.

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