

The development of the k_0 method in Peru: Past, present, and future perspectives

Eduardo Montoya^{1,*}, Patricia Bedregal¹, Pablo Mendoza¹, Marco Ubillús¹, Blanca Torres¹,
Isac Cohen²

¹ Instituto Peruano de Energía Nuclear, Av. Canadá 1470 Lima 41, Perú

² Universidad Tecnológica Nacional, Facultad Regional Avellaneda, Secretaría de Ciencia,
Tecnología e Innovación Productiva. Av. Mitre 750 (1870) Avellaneda, Argentina

Resumen

El método del k_0 se implementó en el Instituto Peruano de Energía Nuclear (IPEN) hacia 1993. Desde mediados de 1994, casi todo el trabajo de análisis por activación neutrónica instrumental en el IPEN se viene realizando mediante el método del k_0 . El establecimiento, aplicaciones, desarrollo y futuro del método en el Perú, se presentan desde una perspectiva dual, retrospectiva / prospectiva. Se presta especial atención sobre algunos aspectos difíciles, tales como eficiencia de conteo, caracterización de la facilidad de irradiación, comportamiento no ideal del espectro neutrónico epitérmico y consistencia de los datos nucleares de entrada.

Abstract

The k_0 method was implemented at the Peruvian Institute of Nuclear Energy (IPEN), by 1993. Since the middle of 1994, almost all INAA work at IPEN is performed by the k_0 method. The establishment, applications, development and future of the method in Peru are presented from a retrospective and prospective view. Attention is paid to difficult aspects, e. g. counting efficiency, characterization of irradiation facility; non ideal behaviour of the epithermal neutron spectrum and consistency of nuclear input data.

1. The antecedents

A relevant reason for the success of the k_0 -method [1] can be found by inspection of the equation used for the calculation of the concentration Q_a for an analyte "a" through a comparator "c" (All symbols are explained in [2]):

$$Q_a = \frac{(N_p / W t_m SDC)_a k_{o,Au}(c) \epsilon_c f(\alpha) + Q_{o,c}(\alpha)}{(N_p / w t_m SDC)_c k_{o,Au}(a) \epsilon_a f(\alpha) + Q_{o,a}(\alpha)} \quad (1)$$

The last factor of the right side in (1) is very important, because has a buffer action against the uncertainties in the thermal and the non-ideal epithermal neutron fluxes, as well as the thermal (n, γ) cross sections and the non-ideal resonance integrals. In addition, since only one comparator has to be measured (provided that f and α are known), it has intrinsically better performance than the traditional method.

A primary version of the k_0 method of INAA was implemented at the laboratories of IPEN, by 1993. The system was conceived on the basis of a pragmatic treatment of relevant correction factors, like those related to the

non ideal behaviour of the epithermal neutron spectrum [3], neutron self-absorption, neutron temperature, geometric counting efficiency, gamma self shielding and self-coincidences. Since the mid of 1994 nearly all of the INAA activities at the laboratory, which cover different materials: minerals [4], geological and environmental samples (water and biological materials [5]), archaeological ceramics [6-8], are performed by the k_0 method.

2. The starting version

The implementation of a basic version of the k_0 method implied to accept some diminution of its versatility and therefore a reduced initial scope of application. The adopted operating procedures and policies included restricted irradiation and counting conditions, nominally identical size, shape and source-detector distance for both comparators and samples, rigorous control of irradiation and decay times, and use of a well thermalized neutron flux. Two comparators (^{197}Au and ^{23}Na) were chosen, so that to minimize the errors caused by the uncertainties in the f and

* Correspondencia autor: emontoya@ipen.gob.pe

α values. The accuracy and precision were assessed as proposed by Heydorn [9] and through the routine use of reference materials.

The experimental details have been published elsewhere [10].

3. The determination of gamma counting efficiency

The efficiency curve of the detector at 210.7 mm was determined using standard sources of ^{133}Ba , ^{109}Cd , ^{57}Co , ^{137}Cs , ^{54}Mn , ^{65}Zn and ^{60}Co , from Canberra. The activities of ^{165}Dy , ^{203}Hg , ^{51}Cr and ^{198}Au sources, prepared at the laboratory, were measured and used as secondary standards, together with the standard sources of ^{109}Cd , ^{57}Co , ^{137}Cs , ^{54}Mn and ^{65}Zn , for determination of the efficiency curve at 58.2 mm (^{133}Ba was not employed in this case, in order to avoid coincidence summing effects). Further details, including an example of efficiency curve, have been published elsewhere [11]. All used sources (samples, comparators and secondary standards for efficiency calibration) were well-defined cylindrical disks, of 9.0 mm diameter and 2 mm height [10].

Although the routine analytical work is based on identical shapes, sizes and source-detector distance for samples and comparator, it is worthwhile mentioning that no relevant geometric effects between point and our samples, comparators or secondary standard sources, were found with the relatively small detector, even at the minimum distance of 58.2mm. Because of the low efficiency of the detector (15% relative) approximate coincidence corrections factors (COI) can be safely used. COI factors extracted from the De Corte's thesis work [12] were initially used, except for ^{76}As and ^{82}Br , for which experimental values were determined. Since 2008, the k0-IAEA software [13] is being used for estimation of COI factors (crystal: 48 mm diameter, 40 mm length, ~ 0.5 mm dead layer, 5 mm end cap to crystal; top cover: 76 mm diameter, 1.5 mm thickness, metallic aluminium; core: 7 mm diameter, 22 mm height), in good agreement with the formerly used values. Gamma spectra were processed with the Canberra Genie v2.1 software and calculations were made by an in house developed, MS Excel application.

4. The determination of the f and α parameters

The relevant data for the monitors used in the successive determinations are presented in Table 1. The literature data were extracted from [2,14-15]. A multi element solution, containing known concentrations of Co(II), Cr(III), Mn(II), Mo(VI) and Sc(III), was prepared by mixing and dilution of gravimetrically determined aliquots of the corresponding standard solutions (See Table 1). The eventual effects of cross contamination were negligible, because of the high purity of the starting standard solutions: for example, the concentrations of Cr, Mn, Mo and Sc in the Co(II) solution were as low as $\leq 0.02 \mu\text{g/mL}$.

Mixed monitors were synthesised by slowly evaporating to dryness 350.0 mg of the multi element solution on 30 mg of high purity cellulose contained in small polyethylene capsules [10]. A calibrated analytical balance was used and the liquid aliquots were weighed in covered capsules, in order to avoid any uncertainty due to the evaporation of liquid. Since the media of the primary standard solutions were nitric acid or water, no losses of the monitor elements as volatile species were expected.

The disk-shaped mixed monitors, together with the metal monitors, were irradiated by (939 ± 1) seconds at a thermal neutron flux of $1.93 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$. After appropriate decay times, they were counted with a Canberra GC1518 HPGe detector. All measurements were performed at a crystal – source distance of 58.2 mm, except for the case of ^{56}Mn (see asterisk in Table 1), which was measured at 210.7 mm. Neutron self shielding factors for the metallic foil monitors were calculated as described by Chilian *et al.* [16].

Because a pneumatic rabbit system was used for irradiations at a thermal power of 10 MW, irradiations under cadmium cover could not be considered. Thus the method of triple bare monitors for the determination of f and α was initially adopted [17]. The results showed a significant degree of variability, as others have observed [18-20].

Afterwards, it was shown that by irradiation in positions having f >15, with gold and sodium as comparators for determining

different groups of elements (classified by the values of their $Q_0(0)$ factors and effective resonance energies) the k_0 method could tolerate considerable uncertainties in the

values of f and α , without introducing unacceptable errors in the analytical results [3].

Table 1. Relevant data of the used monitors (M). Concentrations in mg/L refer to standard solutions.

M	Description / source / etc.	w (μg)	u_w (μg) (k=1)	Nuclide	$E\gamma$ (keV)	Dead time %	u_{peak} (%) (k=1)	counting efficiency	COI
Au	IRMM-530R A $G_{\text{th}}=1.0000, G_{\text{epi}}=0.9961$	14.724	0.088	Au-198	411.8	4.2	0.13	0.00584	1
Co	1000 mg/L, Merck CertiPUR.	114.40	0.23	Co-60	1173.2	0.8	0.15	0.00202	0.9852
Co	1000 mg/L, Merck CertiPUR.	114.40	0.23	Co-60	1332.5	0.8	0.16	0.00179	0.9822
Cr	1000 mg/L, Merck CertiPUR.	173.30	0.87	Cr-51	320.1	0.8	0.26	0.00776	1
Fe	Foil, 20 μm thick, 99.9 % pure. $G_{\text{th}}=0.9960, G_{\text{epi}}=0.9997$	20060	20	Fe-59	1099.2	0.2	0.27	0.00215	0.9983
Fe	Foil, 20 μm thick, 99.9 % pure.	20060	20	Fe-59	1291.6	0.2	0.30	0.00185	1.0005
Lu	0.1 % Lu-Al wire, 250 μm ϕ .	7.340	0.010	Lu-177	208.4	0.5	0.18	0.0121	0.9951
Mn	1010 mg/L, Accu Trace.	5.787	0.029	Mn-56*	846.8	0.9	0.08	0.000330*	0.9992*
Mn	1010 mg/L, Accu Trace.	5.787	0.029	Mn-56*	1810.7	0.9	0.24	0.000162*	0.9976*
Mo	1000 mg/L, Merck CertiPUR.	350.4	1.8	Mo-99	181.1	0.8	0.45	0.0136	0.9805
Mo	1000 mg/L, Merck CertiPUR.	350.4	1.8	Mo-99	739.5	0.8	0.42	0.00314	0.9816
Sc	1010 mg/L, Aldrich.	5.840	0.029	Sc-46	889.3	0.8	0.13	0.00263	0.982
Sc	1010 mg/L, Aldrich.	5.840	0.029	Sc-46	1120.5	0.8	0.14	0.00211	0.9807
Zn	Foil, 100 μm thick, 99.9 % pure. Merck. $G_{\text{th}}=0.9976, G_{\text{epi}}=0.9752$	16823	17	Zn-65	1115.5	0.6	0.19	0.00212	1
Zr	Foil, 127 μm thick, 99.8 % pure, Johnson Matthey. ^{94}Zr : $G_{\text{th}}=0.9996, G_{\text{epi}}=0.9875$ ^{96}Zr : $G_{\text{th}}=0.9996, G_{\text{epi}}=0.9665$	42430	85	Zr-95	742.2	2.8	0.29	0.00313	1
Zr	Foil, 127 μm thick, 99.8 % pure, Johnson Matthey	42430	85	Nb-97m	743.4	2.8	0.11	0.00312	1

* Mn-56 counted at a crystal source distance of 210.7 mm

By that time, the configuration of the core of the 10 MW research reactor changed frequently. Therefore, the need of alternative methods for the characterisation of the irradiation sites was apparent. Besides the conditions of simplicity, reliability, accuracy and robustness, an additional requirement was that these alternative methods could retrieve direct information about the consistency of the neutron flux monitors. A first method for simultaneous determination of f and α was developed [21]. Briefly, the method consists of the simultaneous irradiation of a set of four or more bare monitors, and the determination of the normalised specific experimental activities for each one. Then, for the most convenient pairs of monitors, a whole set of apparent values of $f(\alpha)$ as a function of α is generated, by application of equation (2). Finally, the curves ($\alpha, f(\alpha)$) are plotted, and the best values can be found by averaging of the

different f values defined by the intersections of the curves (which implies the determination of α).

$$f(\alpha) = \frac{\frac{A_{\text{esp},2} Q_{o,1}(\alpha) - A_{\text{esp},1} Q_{o,2}(\alpha)}{k_{o,2} \epsilon_2} - \frac{A_{\text{esp},1} Q_{o,1}(\alpha) - A_{\text{esp},2} Q_{o,2}(\alpha)}{k_{o,1} \epsilon_1}}{\frac{A_{\text{esp},1}}{k_{o,1} \epsilon_1} - \frac{A_{\text{esp},2}}{k_{o,2} \epsilon_2}} \quad (2)$$

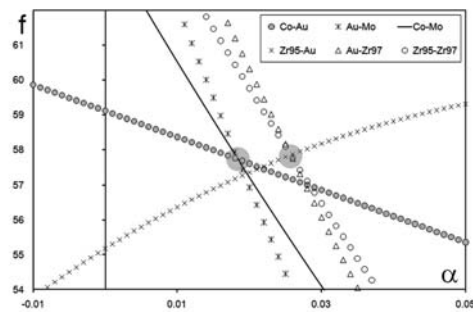


Figure 1. Simultaneous determination of f and α , as described in [21]. Crossing points for the ^{60}Co - ^{99}Mo - ^{198}Au ; ^{95}Zr - ^{97}Zr - ^{198}Au indicated by grey circles.

Figure 1 shows a recent example of application of the method, which corresponds to a simultaneous irradiation of Co, Zr, Mo and Au monitors. The sets ^{94}Zr - ^{96}Zr - ^{197}Au and ^{59}Co - ^{98}Mo - ^{197}Au noticeably behave as two different systems (grey circles). Following Koster-Ammerlaan *et al* [20], a Monte Carlo simulation of 1000 trials were performed, randomly varying the activities of all monitors by $\pm 0.5\%$. The values obtained for f and α were respectively 57.2 ± 1.2 and 0.0279 ± 0.0037 (1σ), which demonstrates that, whenever the counting statistics is good, the method provides reliable values. A comparison between the distributions and the data shown in [20] and our results, indicates that the use of six monitors lead to a considerable better precision in the characterization of the reactor facility.

The problem inherent to this method is that, like the three bare monitors method, it is based on equation (2) so that it does not allow a direct assessment of the performance of each individual monitor. Thus, a second method was developed, which is a modification of that developed by Arribé and Kestelman [22] for the simultaneous determination of the thermal and epithermal fluxes. In that method, the specific activity of a monitor i , evaluated at infinite irradiation and zero decay times, is expressed as

$$g(T)\sigma_i G_{\text{th}} \phi_t + [I(\alpha)_i G_{\text{epi}} + \Delta I_i G_{\text{th}}] \phi_e = A_i \quad (3)$$

where $g(T)$ is the Westcott's factor and $\Delta I_i = 0.56\sigma_0$ is the resonance integral between $E = \mu kT$ and the cadmium cut-off energy, provided that no resonance peaks exist in this interval. The equation (3) is used to calculate, for each monitor, a series of values of the thermal neutron flux as a function of a series of arbitrarily given values for the epithermal one. The values of the thermal and epithermal fluxes are calculated from the average of the values obtained at all the crossing points.

The modification proposed is as follows: provided that the input data of cross sections, resonance integrals and Westcott's factors are accurate, a right value of the α parameter should lead to a single crossing point for the curves corresponding to all the (n,γ) reactions, thus implying that all the monitors should render the same values for the thermal

and the epithermal neutron fluxes. If, in addition, the value of the Westcott's factor for ^{176}Lu is correct, the curve for the reaction $^{176}\text{Lu}(n,\gamma)^{177}\text{Lu}$ should also cross the other curves at the same point. Thus, α and T are adjusting parameters of the crossing point between all curves, fact that provides the basis for their calculation. In practice, all the curves do not cross the others at exactly the same point, due to experimental uncertainties and some degree of inconsistency in the input data.

The values of α and neutron temperature are adjusted until a minimum value is obtained for the variance of the mean thermal neutron flux or, alternatively, when an arbitrary selected crossing point is reached. Then the value of f is calculated as the ratio of the average values for the thermal and epithermal neutron fluxes.

An example of the application of the new method was performed, using simultaneously ^{45}Sc , ^{50}Cr , ^{55}Mn , ^{58}Fe , ^{59}Co , ^{64}Zn , ^{94}Zr , ^{96}Zr , ^{98}Mo , ^{176}Lu and ^{197}Au , as monitors. The input data was taken from [14, 23]. It was observed that the sets ^{94}Zr - ^{96}Zr - ^{197}Au and ^{50}Cr - ^{98}Mo - ^{197}Au behaved different, and that the thermal neutron fluxes determined by the set of $1/v$ monitors spread over an interval of about 6%. Moreover, the fitting of the set ^{50}Cr - ^{98}Mo - ^{197}Au required a neutron temperature of 24 °C, which was not in consonance with the expected value, slightly higher than 37 °C (nominal moderator temperature). These disappointing results were difficult to explain, because the uncertainty budget [24] was low enough to assure an adequate application.

A second test was accomplished, applying the thermal cross sections and resonance integrals published by Mughabghab [25] to the same experimental data. Other literature data were extracted from the following references: atomic weights, [23]; isotopic abundances, effective resonance energies, half lives and gamma intensities, [2, 14-15]. The results showed that the agreement between the $1/v$ monitors considerably improved, with the only astonishing departure of ^{64}Zn . The ^{176}Lu curve agreed with the set of $1/v$ monitors, for a neutron temperature of 40 °C, and the agreement between the Zr, Mo and Au monitors was

also pretty good; nevertheless, the difference between this set and the $1/v$ monitors persisted.

On the basis of the aforementioned tests, it was decided to keep the data from [2, 14-15] for ^{64}Zn , ^{94}Zr and also for the thermal cross section of ^{96}Zr , whereas the data published in ref. [25] were selected for the remaining monitors and for the resonance integral of ^{96}Zr . The results (Figure 2) show that that this eclectic choice of nuclear data leads to a good agreement for all the used monitors.

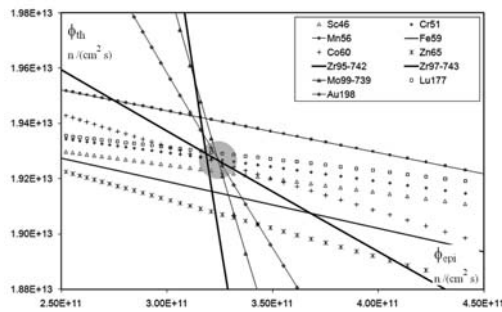


Figure 2. Consistent determination of f , α and neutron temperature. Selected crossing point: ^{60}Co – ^{99}Mo – ^{198}Au . Results: $f = 60.9 \pm 1.2$, $\alpha = 0.033 \pm 0.004$, neutron temperature = $(40 \pm 2)^\circ\text{C}$ (1σ).

A relevant feature is that the new crossing points for the ^{60}Co - ^{99}Mo - ^{198}Au and the ^{51}Cr - ^{99}Mo - ^{198}Au curves are nearly coincident; both are close to the crossing point for the set: ^{95}Zr - ^{97}Zr - ^{198}Au . In addition, the ^{177}Lu curve agrees with the whole set monitors for a neutron temperature of 40°C .

Since the beginnings of 2008, the ^{59}Co - ^{98}Mo - ^{197}Au set is being using at the authors' laboratory, instead of the ^{50}Cr - ^{98}Mo - ^{197}Au set proposed by Koster-Ammerlaan *et al.* [20]

for routine determinations of the f and α parameters. Although the ^{59}Co is not a pure $1/v$ precursor, it is a neutron flux standard monitor; both its thermal cross section and resonance integral values for the capture reaction are very well determined. In contrast, our data indicates that the right value of the thermal cross section for ^{50}Cr seems to be more likely 15.9 barns [25] rather than 15.1 barns, as reported in [14]. The relative difference of about 5% between those values is high enough as to produce discrepant values for f , α and neutron temperature, when different sets of neutron flux monitors are used.

The determination of k_0 values from a set of data is a useful tool to check the overall quality of an experiment [26]. Consequently, a good agreement between experimental and literature k_0 values is an indicator of the suitability of the experimental conditions and the input data. The experimental k_0 values for the studied monitors, determined from the values: $f = 60.9$, $\alpha = 0.033$, and neutron temperature = 40°C (Figure 2) are shown in Table 2. All the values obtained from the experimental data of the present work are in good agreement with the theoretical values calculated from the nuclear data taken from [25], while the agreement with the values reported in [15] exhibit some cases with deviation $\geq 3\%$, (data marked with an asterisk). The discrepancies observed in the present work for the k_0 values of Cr and Sc are in agreement with those reported by Molnár [27].

Table 2. Comparison between the k_0 values obtained in the present work, with those reported in the literature, and the values calculated from atomic and nuclear data.

Nuclide	E (keV)	k_0 observed	k_0		k_0 observed/ k_0 literature	k_0 observed/ k_0 calculated
			literature [20]	calculated [24]		
Co-60	1173.2	1.312E+00	1.32E+00	1.317E+00	0.9941	0.9967
Co-60	1332.5	1.316E+00	1.32E+00	1.318E+00	0.9971	0.9987
Cr-51	320.1	2.727E-03	2.62E-03	2.732E-03	1.0407 *	0.9980
Fe-59	1099.2	7.698E-05	7.77E-05	7.695E-05	0.9907	1.0004
Fe-59	1291.6	5.839E-05	5.93E-05	5.938E-05	0.9846	0.9833
Lu-177	208.4	6.930E-02	7.14E-02	7.111E-02	0.9706	0.9746
Mn-56	846.8	5.094E-01	4.96E-01	5.001E-01	1.0271	1.0186
Mn-56	1810.7	1.364E-01	1.35E-01	1.375E-01	1.0107	0.9921
Mo-99	181.1	4.363E-05	4.15E-05	4.377E-05	1.0513 *	0.9968
Mo-99	739.5	8.799E-05	8.46E-05	8.740E-05	1.0401 *	1.0067
Sc-46	889.3	1.256E+00	1.22E+00	1.264E+00	1.0294	0.9937
Sc-46	1120.5	1.261E+00	1.22E+00	1.264E+00	1.0338 *	0.9979
Zn-65	1115.5	5.662E-03	5.72E-03	5.730E-03	0.9898	0.9881
Zr-95	742.2	2.006E-04	2.00E-04	2.010E-04	1.0032	0.9984
Zr-97	743.4 (Nb-97m)	1.309E-05	1.24E-05	1.319E-05	1.0554 *	0.9922

5. Present and future of k_0 -based INAA on the applied research in Peru

Some examples of the current participation of the authors' laboratory in applied research projects, mostly connected with the fields of archaeology and environmental monitoring, have already been referred [4 - 8]. A common feature of both fields is the need of processing large batches of samples, and to produce reliable results for the concentrations of a number of elements per sample. While the participation and the impact of the NAA technique in environmental studies will be always somewhat hindered because some limitations (e.g. its unsuitability for determining lead) the increasing participation in archaeological projects, as the main analytical tool, is one of the best strategic opportunities, particularly because of the importance of this kind of studies in Peru.

It is possible to affirm that the main proportion of the INAA activities at the authors' laboratory in the future will be performed by the k_0 method. These activities will not restrict to the accomplishment of the analytical task: a permanent research related to the own method is and will be always necessary to ensure its continuous improving and updating. In that sense, the laboratory will intend to contribute to this progress by an active participation in forthcoming developments.

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