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# **k. -MEASUREMENTS AND RELATED NUCLEAR DATA COMPILATION** FOR  $(n, \gamma)$  REACTOR NEUTRON ACTIVATION ANALYSIS

#### IIIa: EXPERIMENTAL

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The determination of  $k_0$ -factors has been continued covering now the relevant gamma-lines of 112 analytically interesting radionuclides. Experimental details are given, and an extended tabulation is presented of complex activation decay types and the associated expressions for the parameters involved in the  $k_0$ -method.

#### **Introduction**

The former publications in this series, dealing with the  $k_0$ -factors for a total of 35 and 72 radionuclides of interest in  $(n, \gamma)$  reactor neutron activation analysis, date from 1980 (Part I)<sup>1</sup> and 1984 (Part II),<sup>2</sup> respectively. Science 1984, the cooperative work at the analytical laboratories of the Institute for Nuclear Sciences (INW, Gent/Belgium) and the Central Research Institute for Physics (KFKI, Budapest Hungary) has led to a significant extension and updating of the material available, now covering 112 radionuclides.

In the present paper, the new or revised output of the  $k_0$ -measurements is given together with the relevant experimental details, the knowledge of which is a prerequisite in order to ascertain the traceability of  $k_0$ -based NAA-results.<sup>3</sup> For that reason, essential input nuclear data (half-lives,  $Q_0$ - and  $\overline{E}_n$ -values) are to be found also in Part IIIb.<sup>4</sup> Additionally, an extended version is presented of the activation decay types and associated expressions for the parameters involved in the k<sub>o</sub>-method.

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#### **Theoretical**

Let it be recalled that the fundamental equations of the  $k_0$ -standardization method are:

*for ko-determination (INW/KFKI):* 

$$
k_{0,c}(s) = \frac{A_{sp,s}}{A_{sp,c}} \cdot \frac{G_{th,c}f + G_{e,c}Q_{0,c}(\alpha)}{G_{th,s}f + G_{e,s}Q_{0,s}(\alpha)} \cdot \frac{\epsilon_{p,c}}{\epsilon_{p,s}}
$$
(1)

or, according to the Cd-subtraction method:

$$
k_{0,c}(s) = \frac{[A_{sp,s} - [(A_{sp})_{Cd}]_s / F_{Cd,s}]/G_{th,s}}{[A_{sp,c} - [(A_{sp})_{Cd}]_c / F_{Cd,c}]/G_{th,c}} \cdot \frac{\epsilon_{p,c}}{\epsilon_{p,s}}
$$
(2)

with  $s$  - standard,

 $c - coirradiated comparator.$ 

*in actual analysis (concentration p):* 

$$
\rho_{\mathbf{a}}, \text{ ppm} = \frac{\left[\frac{N_{\mathbf{p}}/t_{\mathbf{m}}}{\text{SDCW}}\right]_{\mathbf{a}}}{A_{\text{sp},\mathbf{m}}} \cdot \frac{k_{0,c} \text{ (m)}}{k_{0,c} \text{ (a)}} \cdot \frac{G_{\text{th},\mathbf{m}}f + G_{\text{e},\mathbf{m}}Q_{0,\mathbf{m}}(\alpha)}{G_{\text{th},\mathbf{a}}f + G_{\text{e},\mathbf{a}} Q_{0,\mathbf{a}} \text{ (a)}} \cdot \frac{\epsilon_{\mathbf{p},\mathbf{m}}}{\epsilon_{\mathbf{p},\mathbf{a}}} \times 10^6 \quad (3)
$$

or, when applying ENAAi

$$
\rho_{a}, \text{ ppm} = \frac{\left[\left(\frac{N_{p}/t_{m}}{SDCW}\right)_{Cd}\right]_{a}}{\left[(A_{sp})_{Cd}\right]_{m}} \cdot \frac{k_{0,c} \text{ (m)}}{k_{0,c} \text{ (a)}} \cdot \frac{F_{Cd,m} G_{e,m} Q_{0,m} (\alpha)}{F_{Cd,a} G_{e,a} Q_{0,a} \text{ (a)}} \cdot \frac{\epsilon_{p,m}}{\epsilon_{p,a}} \times 10^{6} \tag{4}
$$

where a – analyte in sample, with  $k_{0,c}$  (a) =  $k_{0,c}$  (s),  $m$  - coirradiated monitor, with experimentally determined  $k_{0,c}$  (m) [if m = c, then  $k_{0,c}$  (m)  $\equiv$  1].

 $\ddot{4}$ 

In Eqs  $(1)$ - $(4)$ :

$$
A_{sp} = \frac{N_p/t_m}{SDCw}
$$

- with  $N_p$  peak area corrected for pulse losses (true and random coincidence; dead time),
	- $t_m$  measuring time,
	- S, D, C  $-$  saturation, decay and counting factor, to be modified in case of complex activation and decay (see Table 1),
	- $w$  mass of irradiated element  $(g)$ ,
	- $W =$ sample mass  $(g)$ ,

 $f = \Phi_{s}/\Phi_{e}$ , thermal (subcadmium) to epithermal neutron flux-ratio,

 $G_{\text{th}}$  - thermal neutron self-shielding correction factor.

$$
Q_0(\alpha) = (Q_0 - 0.429)\bar{E}_r^{-\alpha} + 0.429/[(0.55)^{\alpha}(2\alpha + 1)],
$$

- with  $\alpha$  representing the non-ideal  $1/E^{1+\alpha}$  epithermal neutron flux distribution.<sup>5</sup>
	- $\overline{E}_r$  effective resonance energy,<sup>5</sup>
	- $Q_0 = I_0/\sigma_0$ ,
		- $I_0$   $(n, \gamma)$  resonance integral,
		- $\sigma_0$  2200 m · s<sup>-1</sup> (n,  $\gamma$ ) cross-section, to be modified in case of complex activation-decay (see Table 1),
		- $G_e$  epithermal neutron self-shielding correction factor,
		- $F_{Cd}$  correction factor for Cd transmission of epithermal neutrons,<sup>6</sup>
		- $\epsilon_p$  -full energy peak detection efficiency, including gamma-attenuation.

A more detailed discussion of the experimental parameters  $(f, \alpha, \epsilon_{p}, \ldots)$  can be found in a recent review paper<sup>7</sup> and references therein.

In the above, the  $k_0$ -factor is defined as a compound nuclear constant (for modifications, see Table 1)

$$
k_{0,c} (s) = \frac{M_c \Theta_s \sigma_{0,s} \gamma_s}{M_s \Theta_c \sigma_{0,c} \gamma_c}
$$
 (5)

with  $M -$  molar mass;

- $\Theta$  isotopic abundance;
- $\gamma$  absolute gamma-intensity.

## *Modifications in case of complex activation decay*

 $N_p/L_m$ In the above, the terms  $\frac{1}{SDC}$  and Q<sub>0</sub>, as well as the k<sub>0</sub>-definition, should be

modified in case of branching activation and mother-daughter decay. An extended version of the formerly treated<sup>1</sup> practical activation-decay cases encountered in  $(n, \gamma)$ activation analysis is presented in Table 1, together with relevant expressions for the parameters involved.

 $N_p/t_m$ It should be remarked that the expressions for  $\frac{1}{1}$  are not at all specific to SDC

to the ko-method, but are to be used inevitably in all types of standardization, the relative method included. This implies that, also there, such data as  $\gamma_2/F_2 \gamma_3$  (type II/d),  $F_{24}/F_2F_3$  (type III/a),  $F_2\sigma_0^m/\sigma_0^g$  and  $[f+Q_0^m(\alpha)]/[f+Q_0^g(\alpha)]$  (type IV/a), etc., have to be known occasionally.

#### Results

#### *Experimental k<sub>0</sub>-determination*

The experimental results of the present work are shown in Table 2. Some of the radionuclides were already included in References 1 and 2, but improved remeasurements are given now. All ko-factors are expressed versus Au as comparator  $[c = 197 \text{Au}(n, \gamma)^{198} \text{Au}; E_{\gamma} = 411.8 \text{ keV}$ . Note that each entry under the heading "Measured  $k_{0, Au}$ " is the average of 3-5 repetitions.

It should be emphasized that, as a first essential principle with respect to quality assurance, the  $k_0$ -factors were determined in a parallel but independent way at the INW, Gent and at KFKI, Budapest. This means that in both laboratories, use was made of different experimental setups, viz.:

Chemical and physical characteristics of standards and comparators (see Table 2).

Reactors (THETIS, Gent and WWR-M, Budapest), and irradiation positions with different f and  $\alpha$  values (see Refs 1 and 2). For a number of isotopes, the average of 4 determinations in channel R4V4 of the DR-3 reactor (Risø, Denmark) is included: the high thermal-to-epithermal neutron flux ratio in this channel (f  $\simeq$  320) was especially interesting when studying  $(n, \gamma)$  reactions with a high  $Q_0$ -value  $[{}^{96}Zr(n, \gamma) {}^{97}Zr$ , <sup>98</sup> Mo(n,  $\gamma$ <sup>99</sup>Mo, <sup>124</sup> Sn(n,  $\gamma$ )<sup>125</sup> Sn, etc.], the k<sub>0</sub>-factors of which were determined by the Cd-subtraction method.<sup>8</sup>

Ge-detectors and counting geometries. In general, the gamma-measurements in each laboratory were performed with 2 different Ge detectors (1 GAMMA-X detector at Risø), with an accurately determined "reference" full-energy peak detection efficiency curve for point-geometry at 15-20 cm distance to the detector  $(e_p^{\text{ref}})$ ; see below).

Peak area evaluation methods:  $SEOAL<sup>9</sup>$  (INW, Risø), HYPERMET<sup>10,11</sup> (KFKI), MARKER/CAOS<sup>12</sup> (INW), SAMPO80<sup>13</sup> (INW, Risø), ND-604A<sup>14</sup> (Risø) and TRAP, based on the trapezoidal method (INW, KFKI, Ris $\phi$ );

Correction procedures for dead-time and/or pulse pile-up, including the use of a dead-time stabilizer<sup>15,16</sup> (INW), a CANBERRA 2020 AMPLI/PUR (INW, KFKI) and a pulser (KFKI, Ris $\phi$ ).

As a rule, a Gent-Budapest meeting was organized each year, where it was decided for which elements, isotopes and gamma-lines  $k_0$ 's had to be determined in the course of the following year, and where the fully documented  $k_0$ -results of the past year were critically compared and examined. Possible discrepancies were tracked down, sources of error discovered and repetitions imposed. The sources of error could be attributed to problems associated with the stoichiometry of the chemical compounds, micropipetting, contamination or losses, neutron self-shielding, reaction or spectral interferences, peak area evaluation of multiplets, accuracy of  $Q_0$ -values, etc.

Apart from the  $k_0$ -determinations performed in different experimental conditions  $(INW, KFKI, Risk)$ , in general the following precautions were taken with respect to accuracy and traceability:

The volumes of compaxator and standard sources were kept as small as possible, and they were counted at the reference distance of 15-20 cm from the Ge-detector. This procedure rendered true-coincidence effects negligible,<sup>17</sup> and it allowed the introduction in Eqs (1) and (2) of  $\epsilon_0^{k}$  corrected for slight gamma-attenuation effects-, or of  $\epsilon_n^{\text{geo}}$  (geo = actual geometry) calculated from  $\epsilon_n^{\text{ee}}$  according to the program  $SOLANG<sup>18</sup>$  with minor conversions only.

Whenever possible, use was made of sufficiently thin and/or dilute sources so as to make thermal and epithermal neutron self-shielding effects negligible  $(G<sub>th</sub>$  and  $G_e = 1$ ): thin metallic foils or wires, fine powders spread out over a large area or mixed with inert (i.e. low cross-section) materials, dried spots of solutions on Al-foil or W-41 paper, dilute Al-aUoys, etc. [e.g. A1-Au alloyed wires of different composition and production  $(0.097-0.503\%$  Au content,  $0.1-1$  mm diameter, manufactured by ATEC or CBNM/Belgium); for the wires with  $\simeq 0.5\%$  Au,  $G_{th} = 1$  but  $G_e$  is  $0.985$ .]<sup>9</sup> Occasionally, isotopically enriched compounds (supplied by ORNL) were used to eliminate neutron shielding caused by other isotopes of the element studied. Owing to the depletion of the Shielding isotope(s), a limited dilution can be sufficient, so that adequate activities of the studied isotope could be obtained. This was the case for  $115 \text{Cd} / 115 \text{m}$  In [98.55%  $114 \text{Cd-enrichment}$ ; depleted to 0.60%  $113 \text{Cd}$  $(\sigma_0 = 20600 \text{ barn})$ , for <sup>159</sup> Gd and <sup>161</sup> Gd [81.0% <sup>158</sup> Gd, 98.71% <sup>160</sup> Gd enrichment; depleted to, respectively, 1.72% and 0.14% <sup>155</sup>Gd ( $\sigma_0$  = 60900 barn), and 9.72% and 0.24% <sup>157</sup>Gd ( $\sigma_0$  = 254000 barn)], and for <sup>171</sup>Er [96.89% <sup>170</sup>Er enrichment; depleted to 0.72% <sup>167</sup>Er ( $\sigma_{\theta}$  = 659 barn)]. The choice of the final sample composi-

tion (dilution, thickness, etc.) was based on calculation or estimation of the neutron self-shielding effects.

Irradiations were performed in reactor channels with sufficiently stable flux characteristics ( $\phi_s$ , f,  $\alpha$ ) so as to guarantee negligible errors not only from variations during one irradiation,<sup>7</sup> but also from possible differences between the irradiations of bare and Cd-covered samples (in the Cd-subtraction method). This stability was not only evident from the power recording in the reactor operation logbook, but also from repeated experimental determination - as a function of time - of  $\phi_s$ , f and a. In fact, this established stability allowed, in reactor THETIS/INW, to rely on the a priori determined flux characteristics, which were, however, checked regularly. Flux gradients within the irradiation container were accounted for by sandwiching each standard sample between two A1-Au wires (or by using an internal comparator; see below).

Significant errors caused by random coincidence (pulse pile-up) were avoided by keeping the count rates sufficiently small, even when a CANBERRA 2020 AMPLI/PUR was used. In case of short-lived radionuclides, errors due to a decrease of dead time during counting were minimized by using a dead time stabilizer<sup>15,16</sup> (INW), or by keeping the counting time below one tenth of the half-life of the measured radionuclide (KFKI, Riso).

Care was taken to have very accurate knowledge of the masses of eomparator and standard. Whenever possible, use was made of high purity substances as starting materials (especially feasible for metals, but also for sulfur, etc.). When using dilute Al-alloys, the homogeneity and the content of the alloyed element were carefully checked (e.g. for Au-AI; see Ref.<sup>3</sup>). As chemical compounds, preferably primary standards were selected with generally accepted well-known stoichiometry [e.g. NaC1,  $Na<sub>2</sub>CO<sub>3</sub>$ , KHCO<sub>3</sub>, KHC<sub>8</sub> O<sub>4</sub> H<sub>4</sub> (potassium biphthalate)]. Whenever necessary, these primary standards were pretreated according to the prescription, e.g.  $Na<sub>2</sub>CO<sub>3</sub>$  was heated for one hour at  $270-300$  °C. Other compounds were of specpure (J. M.) or ultrapure quality, and again a proper pretreatment was done; e.g. the rare earth oxides were ignited at 900  $^{\circ}$ C for 1 hour. If available, easily soluble specpure (J. M.) compounds were used with a certified concentration of the main element: e.g.  $(NH_4)$ <sub>2</sub> Ru(H<sub>2</sub>O)Cl<sub>5</sub> with 30.6% Ru content,  $(NH_4)$ <sub>2</sub>OsCl<sub>6</sub> with 43.6% Os content, etc. Occasionally, when significant discrepancies between the INW and the KFKI results were observed, the content of the main element in the compound used was redetermined experimentally: e.g. Ce in Ce(SO<sub>4</sub>)<sub>2</sub> · 4H<sub>2</sub>O (expected 34.7% Ce) by titration of Ce(IV) [after adding ammoniumpersulfate] with As(Ill), according to the procedure developed by  $GLEU<sup>20</sup>$ ; the Ce-content in the compound used was found to be 31.4%. In order to make suitable dilutions, the starting materials, weighed on a calibrated (micro) balance, were dissolved in ultrapure solvents, and the solu-

tion was transferred to a volumetric flask. At the KFKI, small aliquots were then spotted on high purity Al-foil by means of a calibrated micropipette. After drying, the Al-foil was folded, up and pressed to a small cylindrical pellet, usually 6.4 mm diameter  $\times$  2 mm height. At the INW, use was made of an internal comparator (index ic; with accurately known  $k_0$ -factor), a known amount of which was brought in solution together with the standard. From this solution,  $\simeq$  100 or 250  $\mu$ l was spotted on a circular W41 paper, which was then dried  $-$  usually under an I. R. lamp (but occasionally at room temperature, e.g. for mercury). The thus loaded W41 paper was folded up, enveloped in a second W41 paper and finally pressed to a pellet, usually 10 mm diameter  $\times$  4 mm height. By means of radioactive tracers it was shown that the spotted substance was homogeneously distributed over the W41 paper. With this technique of the internal comparator, the  $k_0$ -factor can be obtained as:

$$
k_{0,c}(s) = \frac{w_{ic}}{w_{s}} \cdot \frac{\left[\frac{N_{p}/t_{m}}{SDC}\right]_{s}}{\left[\frac{N_{p}/t_{m}}{SDC}\right]_{ic}} \cdot \frac{G_{t h,ic} \cdot f + G_{e,ic}Q_{0,ic}(\alpha)}{G_{t h,s} \cdot f + G_{e,s}Q_{0,s}(\alpha)} \cdot \frac{\epsilon_{p,ic}}{\epsilon_{p,s}} \cdot k_{0,c}(ic)
$$
(6)

From Eq. (6) it is clear that only the mass ratio  $w_{ic}/w_s$  should be known, thus avoiding quantitative work after simultaneous dissolution of internal comparator and standard. Also, flux gradients are automatically accounted for, and the counting geometries of internal comparator and standard are identical, so that  $-$  in spite of the relatively large volume of the W41 pellets - it is allowed to introduce  $\epsilon_0^{ref}$ -values, which should only be corrected for gamma-attenuation in the paper. The gamma-attenuation factor was experimentally determined as a function of gamma-energy by measuring various point sources (at the reference distance from the detector) with and without screening by means of a 2 mm thick W41 pellet (half height of the usual pellet). To a good approximation, the gamma-attenuation function could be fitted as:

$$
F_{\text{att}} = 0.953 + 4.86 \cdot 10^{-5} E_{\gamma}
$$
 (keV)  $[r^2 = 0.9992]$ 

in the energy region 60 keV  $\lt E_{\gamma}$   $\le$  900 keV. Above 900 keV, gamma-attenuation was considered to be negligible  $(< 0.3\%$ ).

Experimental conditions were chosen so as to obtain, for the analytically interesting gamma-lines under consideration, a statistically acceptable number of counts  $(s < 1\%)$  in the full-energy peaks, with negligible spectral interference. This could be achieved using high purity-materials, and by optimizing the irradiation, decay and

counting times and the weight of the irradiated element (taking care to minimize neutron self-shielding effects). For instance, when determining the  $k_0$ -factor of the 279.2 keV-line of  $203$  Hg (46.612 d), it is mandatory to wait for the decay of  $197m$ Hg (23.8 h), the 279.0 keV line of which can cause of spectral interference. For  $k_0$ -determination of the  $239$ Np gamma-lines, use was made of a U-standard (a Al-0.443% U wire) depleted to a 0.0375% <sup>235</sup>U content (and enriched to 99.962% <sup>238</sup>U); this reduced strongly the dead-time, background and interfering lines due to the <sup>235</sup> U(n, f) fission products. For  $k_0$ -determination of the <sup>147</sup> Nd, <sup>149</sup> Nd/<sup>149</sup> Pm and  $151$  Nd/<sup>151</sup> Pm gamma-lines, use was made of isotopically enriched oxides (ORNL); this reduced drastically the complexity of the spectra, otherwise showing numerous interfering lines. Optimization of the irradiation, decay and counting times can also lead to considerable reduction of the uncertainty originating from T, due to (partial) compensation of terms in the function describing the propagation of the error on T towards  $k_0$ .

 $Q_0$ -values and  $F_{Cd}$ -factors were either critically selected from the literature or experimentally determined (see Part IIIb).<sup>4</sup> As to the  $Q_0$ -values, it follows from error propagation calculations that the residual uncertainties of  $k_0$ , determined according to Eq. (1), do not exceed 1%. When using the Cd-subtraction method [Eq.  $(2)$ ], no  $Q_0$ -values are involved.

In Table 2, a "recommended"  $k_0$ -factor is usually the average of 3-5 repetitions  $\times$  1-2 irradiation channels  $\times$  2 reactors, and the quoted uncertainty is the standard deviation on the mean (except for the  $k_0$ 's of the Zr-isotopes, where the weighted mean and the larger of the internal or external error was calculated; see Ref.<sup>8</sup>). It should be remarked that a  $k_0$ -factor, even when obtained according to the above outlined procedure, is only recommended when the standard deviation of the mean i8 less than 2%.

A  $k_0$ -factor is considered to be "tentative" in brackets in Table 2, and with no mention of the uncertainty) when the standard deviation is exceeding 2% or when for a particular isotope, or for a particular gamma-line of a given isotope, the determinations were only performed in one reactor. Even then, the average usually results from 3-5 measurements  $\times$  2 irradiation channels, and the accuracy is probably not worse than  $\simeq$  5%.

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Table 1



Table 1 (cont'd)



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Table 1 (cont'd)



Table 1 (cont'd)





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ierences 1 and 2 (for survey: Experimental determination of k0, Au-factors [extension and updating of results in F<br>see Part IIIb)<sup>\*</sup>; activation decay type: see Table I]



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 $Table 2\ (conf.d)$ 

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 $Table\ 2\ (conf\ d)$ 



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Table 2 (cont'd)





 $Table\ 2\ (cont'\ d)$ 



Table 2 (cont'd)



 $Table 2~(contd)$ 



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 $Table~2~(conl'd)$ 



Table 2 (cont'd)







 $Table\ 2\ (cont\ d)$ 



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 $Table~2~(cont'd)$ 



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