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Neutron activation analysis of certified samples by the absolute method

F. Kadem[a], N. Belouadah and Z. Idiri

Faculté de physique, USTHB BP 32 El-Alia BEZ Alger Algeria

Abstract. The nuclear reactions analysis technique is mainly based on the relative method or the use of activation cross sections. In order to validate nuclear data for the calculated cross section evaluated from systematic studies, we used the neutron activation analysis technique (NAA) to determine the various constituent concentrations of certified samples for animal blood, milk and hay. In this analysis, the absolute method is used. The neutron activation technique involves irradiating the sample and subsequently performing a measurement of the activity of the sample. The fundamental equation of the activation connects several physical parameters including the cross section that is essential for the quantitative determination of the different elements composing the sample without resorting to the use of standard sample. Called the absolute method, it allows a measurement as accurate as the relative method. The results obtained by the absolute method showed that the values are as precise as the relative method requiring the use of standard sample for each element to be quantified.

1 Introduction

In order to validate our data calculated cross sections, we have made an application to the data for the quantitative analysis of certified standard samples using the Neutron Activation Analysis (NAA) and the absolute method. Once validated this method does not require the use of standard samples and just need to know the cross section for the nuclear reaction induced by fast neutrons. This approach is complemented by the use of data of the cross section quickly calculated by the semi- empirical formulas systematic studies. The neutron activation analysis of samples certified by the IAEA was carried out using the absolute method based on using our data calculated cross sections.

2 Treatment data

The photopeak area of an intensity I_γ and efficiency ε_γ, corresponding to the activity of a Y radioisotope with decay constant λ formed in a X (n, b) Y reaction (b = n, 2n, p np, d, t, a, …) is given by:

$$Net = n\sigma\phi I_\gamma \varepsilon_\gamma \frac{1}{\lambda}\left(1-\exp(-\lambda t_i)\right)\exp(-\lambda t_d)\left(1-\exp(-\lambda t_c)\right) \qquad (1)$$

where σ is integrated cross section of the reaction, Φ is the incident neutron flux, n is the number of target nuclei X, λ is the radioactive decay constant and t_i, t_d and t_c are respectively the time of irradiation , the cooling time and the time counting [1] . The number n of the target nucleus is given is related to the isotopic abundance θ and the mass m of the element constituting the sample by:

$$n = \frac{m\theta N_a}{M} \qquad (2)$$

where N_a is Avogadro's number and M the atomic mass. To determine the efficiency with gamma ray energy dependence the following expression is used:

$$\varepsilon(\%) = \varepsilon_0 + A_1 \exp(E_\gamma / B_1) + A_2 \exp(E_\gamma / B_2) \qquad (3)$$

The fitting parameters ε_o, A_i and B_i were determined by using the least squares method. The difference between the experimental points and the calculated points is generally less than 1%.

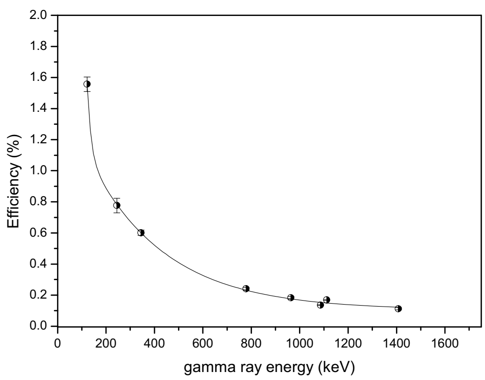

Figure 1. Efficiency curve via gamma ray energy.

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[a] Corresponding author: kademassia@yahoo.fr

3 Experimental results

The neutron activation analysis allows to determine the mass of samples constituents using the absolute method [4]:

$$m = \frac{N.M}{N_a \theta \sigma \phi I_\gamma \varepsilon \frac{1}{\lambda}\left(1-\exp(-\lambda t_i)\right)\exp(-\lambda t_d)\left(1-\exp(-\lambda t_c)\right)} \quad (4)$$

where N is the net area of the photopeak.

3.1 Measurement of neutron flux

To measure the flux of incident neutrons we used pure aluminum sheets. In order to measure the flux to which the sample is exposed we placed the sample between two aluminum sheets so that together form a sandwich. The reference reaction ^{27}Al (n, α)^{24}Na was used.

The detailed characteristics of this reaction are given as follows: σ=114 mb, E_γ = 1369 keV, I_γ= 100 %, $T_{1/2}$ = 53900 s.

The flux, which is exposed, the sample is calculated using the expression:

$$\phi = 4\frac{x^2}{\left(2x+d\right)^2}\phi_1 e^{-\mu d/2} \quad (5)$$

where d is the thickness of the sample, ϕ_1, ϕ_2 are respectively the flux measured by the aluminum sheets 1 and 2, x is the distance from the neutron source and the aluminum foil measuring the flux ϕ_1 and μ is the absorption coefficient of the neutrons in the sample. μ is deduced from the following equation:

$$\phi_2 = \phi_1 \frac{x^2}{\left(x+d\right)^2} e^{-\mu d} \quad (6)$$

3.2 Analysis of certified samples

The irradiation time was 20 min for each samples of animal blood, milk and hay. The components samples have been identified, through the gamma rays characteristic of a radionuclide produced via a nuclear reaction induced by fast neutron on the sample target.

Table 1. Component characteristics of animal blood sample (A-13).

IAEA-A-13 element	reaction	$T_{1/2}$ (s)	Eγ (keV)	Iγ (%)
Fe	^{56}Fe (n,p)^{56}Mn	9290	847	98.9
Mg	^{24}Mg (n,p)^{24}Na	53900	1369	100
P	^{31}P (n,p)^{28}Al	117	1779	100
K	^{39}K (n,2n)^{38}K	458	2168	99.9
Rb	^{85}Rb(n,2n)^{84m}Rb	1220	248	59

3.3 Experimental results

The neutron activation analysis of samples of animal blood, milk and hay were identified qualitatively and quantitatively and the components of these samples using an absolute method were carried out. Indeed, this method is based to the fundamental equation of neutron activation and using the known cross section for the nuclear reaction. The experimental results were compared with data certified by the IAEA. The following tables present the results of the counting of gamma spectra of samples of blood, milk and hay.

Table 2. Component characteristics of hay sample (V-10).

IAEA-V-10 element	reaction	$T_{1/2}$ (s)	Eγ (keV)	Iγ (%)
Fe	^{56}Fe(n,p)^{56}Mn	9290	847	98.9
Mg	^{24}Mg(n,p)^{24}Na	53900	1369	100
P	^{31}P(n,p)^{28}Al	117	1779	100
K	^{39}K(n,2n)^{38}K	458	2168	99.9
Rb	^{85}Rb(n,2n)^{84m}Rb	1220	248	59
Sr	^{88}Sr(n,2n)^{87m}Sr	10100	388	81.8
Ca	^{44}Ca(n,p)^{44}K	1330	1157	58.2

Table 3. Component characteristics of milk powder sample (A-11).

IAEA-A-11 element	reaction	$T_{1/2}$ (s)	Eγ (keV)	Iγ (%)
Cl	^{37}Cl (n,p)^{37}S	303	3103	94.1
Mg	^{24}Mg (n,p)^{24}Na	53900	1369	100
P	^{31}P (n,p)^{28}Al	117	1779	100
K	^{39}K (n,2n)^{38}K	458	2168	99.9

Table 4. AAN results for Animal blood and powdered milk samples (A-13 & A-11).

AEA-A-13 element	reaction	σ_{cal} (mb)	Experimental massic concentration (mg/Kg)	Certified massic concentration (mg/Kg)
Fe	^{56}Fe(n,p	107	2410 ± 250	2400
Mg	^{24}Mg(n,p)	258	55.4 ± 16.1	99
P	^{31}P(n,p)	160	1310 ± 230	940
K	^{39}K(n,2n)	4	1820 ± 650	2500
Rb	^{85}Rb(n,2n)	470	40.5 ± 17.3	23
IAEA-A-11				
Cl	^{37}Cl(n,p)	36	11.2 ± 1.4	9.08
Mg	^{24}Mg(n,p)	258	0.8 ± 0.2	1.01
P	^{31}P(n,p)	160	11.4 ± 1.6	9.10
K	^{39}K(n,2n)	4	10.6 ± 5.3	17.2

3.4 Discussion

Within the error bars the measured concentrations in this study are in agreement with the certified values by the IAEA. A slight disagreement recorded for some items is mainly due to the low sensitivity of the activation method probably because of the small cross section and the inadequacy of the half live radioisotope irradiation with time so chosen to consider short and long half live.

Table 5. AAN results for hay sample (V-10)

AEA-V-10 element	reaction	σ_{cal} (mb)	Experimental massic concentration (mg/Kg)	Certified massic concentration (mg/Kg)
Fe	^{56}Fe(n,p)	107	128 ± 36	185
Mg	^{24}Mg(n,p)	258	1530 ± 224	1360
P	^{31}P(n,p)	160	2790 ± 427	2300
Ca	^{44}Ca(n,p)	33.7	20500 ± 2130	21600
K	^{39}K(n,2n)	4	25700 ± 2710	21000
Rb	^{85}Rb(n,2n)	474	23.5 ± 11.6	7.6
Sr	^{88}Sr(n,2n)	262	55.7 ± 13.8	40

4 Conclusion

The absolute neutron activation method is as reliable as the relative method using standards. This approach is complemented by the use of data of the cross section quickly calculated by the semi-empirical formulas systematic studies.

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