Available online at www.derpharmachemica.com

ISSN 0975-413X
CODEN (USA): PCHHAX

Der Pharma Chemica, 2016, 8(5):250-255
(http://derpharmachemica.com/archive.html)

Calculating concentrations of elements in sample and compare with standard certified results of the International Atomic Energy Agency (IAEA) Soil-7

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ABSTRACT

After the discovery of the neutron (1932) and of artificial radioactivity (1934), the neutron activation analysis (1936) will become the method of choice, (INAA: Instrumental Neutron Activation Analysis) is a exclusively method of elemental analysis. Its implementation involves irradiating the sample for analysis in a flux of suitable particles, neutrons, charged particles ... and identify, then after irradiation, radioactive isotopes created from the elements to be determined. The purpose of this study is calculated the concentrations of the elements of each element in the sample irradiated, using the absolute and comparative methods, this results are Compared with results certified standard of the International Atomic Energy Agency (IAEA) Soil-7, in the aim of validity the research, and repeat the need of some requirement in the different sectors such as the pollution of the air and water by heavy metals, food, medicine archaeology, geochemistry, biology, geology, agronomy and the hydrology.

Keywords: INAA; IAEA; detector of germanium; neutron activation; standard material; gamma spectrometry

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INTRODUCTION

INNA is a sensitive method capable of quantifying the many elements at the same time [1-10], so the laboratory of neutron analysis activation of Maamoura, uses this method in research and nuclear instrumentation.

The neutron activation analysis (NAA) is to make the radioactive elements of a material by subjecting them to a stream of neutrons. Therefore the atomic nuclei are activated by the neutrons. Their changes, during this process, allow us to identify and quantify each element and determination of the concentration of an element in is called dosage [1-15], of its disadvantages such as the dependence of the irradiation facility (a nuclear reactor) and the threshold imposed by the rules of legal security for the handling of radioactive materials. The additional obstacle to its use lies in the slowness of the procedure of analysis. Indeed, the duration of irradiation of the analysis traces of radionuclides of long period, may take several hours also for the extent and this for a single sample. The only way to remedy these disadvantages consists in the use of large samples to higher fluxes and metering devices more effective. Not only the presence of the sample and standard, but also the time used for their preparation, counting and calculation.

Bitter the analysis and the calculation of the concentrations of the components of a sample [16], are compared with the results given by a confidence interval certified of the International Atomic Energy Agency (IAEA), [17-19].

MATERIALS AND METHODS

2.1 Experimental study

This publication for the purpose of analyzing the spectra after their counting and the calculation of the on centrations of the components and compared with a standard material of reference appointed (Soil-7) (Reference Sheet Reference Material) issued by the IAEA. The chain of detection provides a spectrum of energy representing the number of photons which has interacted with the detector in the function of a channel number [20-25]. When all of the samples of the shuttle are counted, the corresponding spectra are available for the treatment qualitative and quantitative [16].

The calibration phase of energy is a very important approach to have a good result in placing the sample in the detector as follows [26]: It opens the enclosure of lead which surrounds the detector of germanium (Ge-HP), [27-28]. After the registration of the spectrum in the spectrometer and transferring it on a computer to perform the analysis, If it's known that an element emits gamma radiation with an energy known, it is then necessary to sweep the whole spectrum, there goes directly to the peak want to analyze,

Once all peaks of a selected spectrum, the results are saved in a file text as a table or form curves, [27-28]**.** The files text containing the following information: name and geometry of detector used for the counting, dead time [27], real time, energy, radionuclide's are gamma emitters with the same energy, width has mid-heights, the area gross, net area, and the error in %. On the other hand, we have seen the representations made for the irradiation of a sample up to the detection of gamma radiation emitted by a source. AAN is an analytical method very sensitive [11-15], particularly well adapted to characterize the purity of a sample. It involves irradiating a sample by a stream of neutrons. The analysis is done by a theoretical and practical study. All previous data will be operated to calculate the concentrations of the incoming elements in the chemical composition of sample irradiated.

After all measurement taken is prepared sheets calculated using Microsoft Excel, to use the results of qualitative analysis, the spectra find in our goal is to determine the concentration of the elements. The spreadsheet comes in the form of a workbook containing four tabs: (Lib): a sheet contains all data associated with all nuclear radioisotopes such as the atomic number Z, the atomic weight A, time half-life and gamma ray energy in Kev. These data will be used in an indirect manner in calculating the concentrations. Tab; sheet containing the data concern the irradiated sample are: the code and the type of liquid and solid sample, weight, the irradiation time, the cooling time and the count, table 1.

Table 1: Containing information on the irradiated samples

	Code Lab	Type of simple	weight (mg)	weight (g)	Tire (sec)	Tre (sec)	Tce (sec)
Simple	B183D21K	Powder	103,763	0,10376	30	1159	1272
	B185D21K	Powder	91,01	0,09101	30	1321	1477
	B191D21K	Powder	103 ,16	0,10316	30	1044	2489

These data were recorded in a register or in an Excel spreadsheet when preparing and irradiating the sample to counting.

Tab (Stand), sheet envelope all the information on the standard as the weight, the net surfaces (Net Area) of each peak, irradiation time, cooling and metering standard used for the assay and their concentrations in ppm.

Tab (Calc_Const), this sheet has been designed to calculate the elemental concentrations. The next step after this one data filtering job to select the one we need in our calculation. Also in the same tab, the third step involves a "Design-Const" macro, the program written in Microsoft Visual Basic responsible for calculating the concentrations of each element in the sample using a mathematical formula whose main variable is the peak areas (Area Net) characterizing standards and radioactive elements that constitute the sample.

When processing and give the results to find a mathematical process involved, starting with the calculated activity to the concentration of elements in a sample.

2.2 Theoretically study

The fundamental equation of neutron activation

Suppose a target nucleus **A** is subjected to neutron flux **n**, and that the nuclear reaction creates the radioisotope **B***
of period **Tb**, and a particle **b**

$$A + {}^{1}n \longrightarrow B^{*} + b$$

The number of nuclei **A**, affected by the reaction, is infinitesimal. Then a nucleus **A** is assumed to remain constant during the irradiation. The number (***Nb***) of nuclei **B** created during a time of radiation ***dt*** is equal to:

$$N_B = N_A\, \sigma\, \Phi\, dt \quad (1)$$

Where:

σ = cross section of reaction by Barn

Φ = neutron flux $n.s^{-1}.cm^{-2}$

N_A = nombre de noyaux cibles

N_A Can be written with:

$$N_A = p\, m \frac{N}{A} \quad (2)$$

Where:

m Weight of simple

p Fraction of the useful isotope

N Number of Avogadro

A Number de weight

However, the amount of cores **B*** created, decreases in the radioactive decay constant λ_B. The number of nuclei **B*** which disappears during the time ***dt*** is equal to:

$$N_B(t)\, \lambda_B\, dt \quad (3)$$

Increasing dN_B number of nuclei **B***during ***dt*** equals:

Where:

The resolution of this equation gives us the number of nuclei N_B expressed as a function of the irradiation time t_i:

$$N_B(t) = \frac{N_A\, \sigma\, \Phi}{\lambda_B} \left(1 - e^{-\lambda_B t_i}\right) \quad (4)$$

Can thus be calculated, depending on the irradiation time t_i the activity generated by the resulting radionuclide.

$$A(t_i) = N_B(t_i)\, \lambda_B = N_A\, \sigma\, \Phi \left(1 - e^{-\lambda_B t_i}\right) \quad (5)$$

The time t_r elapsed between the end of the irradiation and the start of counting (the sample cooling time), activity time $t_i + t_r$ is equal to:

$$A(t_i) = N_A\, \sigma\, \Phi \left(1 - e^{-\lambda_B t_i}\right) e^{-\lambda_B t_r} \quad (6)$$

For a counting time t_c, the theoretical number of shots is given by:

$$\Delta M(t = t_c) = \frac{A}{\lambda_B}(1 - e^{-\lambda_B t_c}) \quad (7)$$

For a detection efficiency ε and ray intensity I_γ, the number of counts is equal to

$$M = \frac{N_A .\sigma\, .\Phi\, .\varepsilon\, .I_\gamma}{\lambda_B} \left(1 - e^{-\lambda_B t_i}\right)\left(1 - e^{-\lambda_B t_c}\right).e^{-\lambda_B t_r} \quad (8)$$

From the relations (2) and (8) the number of counts (peak area) is expressed as follows:

$$M = p.m\frac{N}{A}\frac{\sigma.\Phi.\varepsilon.I_\gamma}{\lambda_B}\left(1-e^{-\lambda_B t_i}\right)\left(1-e^{-\lambda_B t_c}\right).e^{-\lambda_B t_r} \quad (9)$$

In terms of weight concentration peak area can be expressed as follows:

$$M = C.\ p.m\frac{N}{A}\frac{\sigma.\Phi.\varepsilon.I_\gamma}{\lambda_B}\left(1-e^{-\lambda_B t_i}\right)\left(1-e^{-\lambda_B t_c}\right).e^{-\lambda_B t_r} \quad (10)$$

With,

C: The concentration of an element in the sample

The concentration of an element, [29-33] in a sample is proportional to the area of photoelectric (number of counts M) γ-rays peaks. This method (absolute method) used to directly determine the concentration of the desired element in sample from the following equations:

$$C = \frac{\lambda_B.A.M.e^{\lambda_B t_r}}{m.N.p.\sigma.\Phi.\varepsilon.I_\gamma\left(1-e^{-\lambda_B t_i}\right)\left(1-e^{-\lambda_B t_c}\right)} \quad (11)$$

Indeed the use of the equation (11) requires knowledge of all parameters involved as the flux and cross section σ, which usually are not precisely known. In addition, the number of detected photons γdepends on the detection efficiencyε. To reduce the risk of error, we use the comparative method of simultaneously irradiating the sample and standard and effective succession during the same time. The equation of relation (11) between the element concentration in the sample and in the standard gives:

$$\frac{C_{éch}}{C_{ét}} = \left(\frac{M_{éch}}{M_{ét}}\right)\left(\frac{m_{éch}}{m_{ét}}\right)e^{-\lambda(t_{r.éch}-t_{r.ét})} \quad (12)$$

This equation gives the content $C_{éch}$ of an element in a sample in mg/kg by the equation:

$$C_{éch} = C_{ét}\left(\frac{M_{éch}}{M_{ét}}\right)\left(\frac{m_{éch}}{m_{ét}}\right)e^{-\lambda(t_{r.éch}-t_{r.ét})} \quad (13)$$

$M_{ét}$ and $M_{éch}$ are the areas of the photoelectric peaks of the gamma line representing the element in the standard spectrum and that of the sample.

The mathematical formula used in our calculation is written as follows:

$$C_{éch} = C_{ét}\ \frac{m_{ét}}{m_{éch}}\ \frac{e^{-\lambda t_{r.ét}}}{e^{-\lambda t_{r.éch}}}\ \frac{\left(1-e^{-\lambda_B t_{i.ét}}\right)\left(1-e^{-\lambda_B t_{c.ét}}\right)}{\left(1-e^{-\lambda_B t_{i.éch}}\right)\left(1-e^{-\lambda_B t_{c.éch}}\right)}\frac{M_{éch}}{M_{ét}} \quad (14)$$

$C_{éch}$The concentration of element in the sample

$C_{ét}$ Standard element concentration.

$M_{ét}$ and $M_{éch}$: are the peak areas (Net Area) in the same photoelectric gamma ray representing the element in the standard spectrum and that of the sample. It should be noted that the areas of the standard and sample peaks are issued automatically by the Genie 2000 software.

$t_{r.ét}$ and $t_{r.éch}$: are the cooling time of the standard and of the sample

$t_{i.ét}$ and $t_{i.éch}$: are the irradiation time of the standard and of the sample

$t_{c.ét}$ and $t_{c.éch}$: Are of the standard counting time and that of the sample

$\frac{m_{ét}}{m_{éch}}$: is the ratio of the weight of the standard to the sample weight

$\frac{M_{éch}}{M_{ét}}$: is the ratio of the net area of the standard to the net surface of the sample

The formula (14) is deduced from the equation of relation (11) is between the element concentration in the sample and in the standard.

The portion of equation (11) to the formula (14) is justified when the intensity of the neutron flux is stable and that the neutron spectrum energy does not vary with time, and the effectiveness detection is made constant.

Uncertainty about the concentration of the sample takes into account the uncertainties related to the standard concentration, the net surface of the standard peak and the net surface of the sample peak are calculated as follows:

$$< C_{éch} > = \left(\sqrt{\left(\frac{<C_{ét}>}{C_{ét}}\right)^2 + \left(\frac{<M_{ét}>}{M_{ét}}\right)^2 + \left(\frac{<M_{éch}>}{M_{éch}}\right)^2} \right) . C_{éch} \qquad (15)$$

RESULTS AND DISCUSSION

Table 2 presents some elemental concentrations of simple (Vanadium, Potassium, Aluminium, Magnesium, Sodium and Barium) calculated with their uncertainties and compared with the standards environmental Soil-7 IAEA

	Calculated value		values certified by IAEA	
Element	**Concentration (mg/Kg)**	**Error (+/-)**	**Confidence Interval**	**certified Value**
$^{52}_{23}V$	64.08	3.43	59-73	66
$^{42}_{19}K$	11 351.96	873.54	11300-12700	12100
$^{28}_{13}Al$	48 530.01	1 150.84	44000 - 51000	47000
$^{56}_{25}Mn$	623.65	8.27	604-650	631
$^{24}_{11}Na$	2 421.24	41.37	2300-2500	2400
$^{139}_{56}Ba$	161.13	30.14	131-196	159

Elemental concentrations calculated and certified standard Soil7 (mg/Kg),

We made a comparison between the calculated values and the values certified by the IAEA also found good agreement between the concentrations found by our calculations and the data delivered by the International Atomic Energy Agency.

CONCLUSION

The comparison between the values of the certified concentrations and those calculated by our method shows great consistency and hence the neutron activation analysis (NAA) is a competitive technology to other analytical technique. The AAN allows us to analyze accurately and simultaneously a large number of elements.

The neutron activation analysis at laboratory of Nuclear Studies Centre Maâmora (CENM) uses the comparative method and the absolute method which require the use of reference standards. This obligation is seen as a disadvantage, since these standards are expensive and are not always available. In addition, the determination of the elements is determined by the existing elements in the standard, while other elements (peaks) present in the gamma spectrum from the irradiated sample cannot be measured because of the absence of these peaks in the standard. For all these reasons, we must think of opportunity another method such as K_0 method. Indeed, this method does not require the use of standard and allows dosing all existing peaks in the spectrum.

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