

Epiboron Neutron Activation Analysis with Nigeria Research Reactor -

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Abstract

Epiboron neutron activation analysis is optimized using Nigeria Research Reactor-1. Data are given for 6 elements using boron as shielding. Boron shield are of particular practical value for rapid instrumental analysis. Advantage factors for the following elements: I, Br, Cl, K, Mn and Na under boron shield are given. Concentrations of the mentioned elements is reexamined and compared with certificate values. The results obtained, showed good precision.

Keywords: Epiboron Neutron Activation, Nigeria Research Reactor-1, Boron shield, Boron Ratio and Quality control assurance.

1.0 Introduction

The importance of irradiating samples surrounded by neutron shield has been demonstrated by many researchers. The activation of many elements can be greatly reduced in this way, whereas the activation of a number of other elements is only reduced by a small amount, since the activation of these is largely due to higher energy neutrons which are not absorbed appreciably by the shield [1]. The advantage of having Epithermal Neutron Activation Analysis (ENAA) technique in a given situation depends upon whether or not any significant advantage over interfering elements can be obtained for the elements that are to be determined. ENAA is a useful extension of Instrumental Neutron Activation Analysis (INAA) in that it enhances the activation of a number of trace elements relative to the major matrix elements. In general, the activation cross sections of the matrix elements of environmental samples are inversely proportional to the neutron energy ($1/v$ low).

This paper presents results of an examination of ENAA utilizing NIRR-1. Data obtained under consistent experimental conditions are given for 6 elements using boron as a shield, so that results may be directly compared in a practical way.

2.0 Principal Of Neutron Activation Analysis

Neutron Activation Analysis is an analytical method which is based upon the properties of the nucleus. The process of neutron capture (activation) and decay are shown in figure 2.1 below.

During irradiation, an incident neutron is captured by a target nucleus in the sample forming a compound nucleus. The compound nucleus is in a highly excited state due to the high binding energy of the neutron and will rapidly return to the ground state (in about 10^{-15} second) by emission of prompt gamma rays.

For the target isotope, the reaction involved is:



The resultant nucleus usually radioactive has a unique half-life, mode of decay and energy for emitted radiation(s) during decay. In most cases, the radioactive nucleus undergoes beta decay which is accompanied by decay gamma

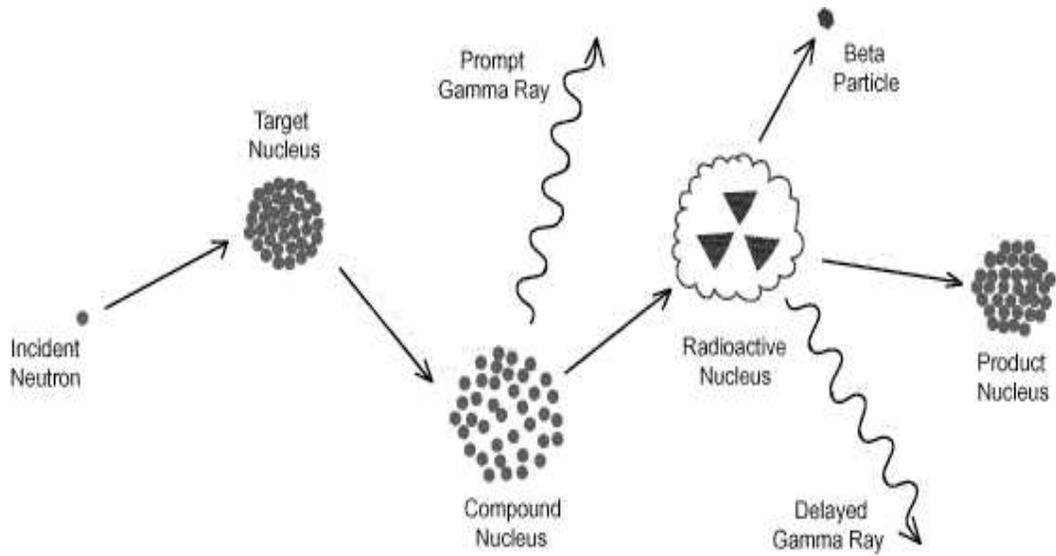


Figure 2.1 Sketch showing activation of a nucleus by an incident neutron, prompt and decay gamma rays which are emitted following neutron capture.

rays. Because the energies of the prompt and decay gamma rays are discrete and distinctive for each radioisotope, multi-elements NAA is feasible by use of high-resolution semiconductors such as germanium detectors to identify and measure the emitted gamma rays.

Determinations are based on the detection of the highly penetrating γ -photons of discrete energies. Gamma energies of different nuclides are spread over the interval from some keV to some MeV. For qualitative analysis the measurable parameters are the energy of the emitted gamma quanta (E_γ) and the half life of the nuclide $T_{1/2}$. For quantitative analysis the intensity γ is used, which is the number of gamma-photons of energy E_γ , measured per unit time.

2.1 Kinetics Of The Reaction

The irradiation of a nuclide in a neutron flux, where two flux components are present namely the conventional sub-cadmium flux f_{th} and the conventional epithermal flux f_{ep} , is characterized by a reaction rate given by:

$$R = s_{th}f_{th} + I_0f_{ep} \tag{2.2}$$

where, s_{th} is thermal absorption (n, g) cross-section for neutrons with $v = 2200ms^{-1}$

I_0 is resonance integral defined by [2]:

$$I_0 = \int_{E_{cd}}^{\infty} s(E) \frac{dE}{E} \text{ with } E_{cd} = 0.55eV$$

If f_{th} and I_0 are known equations 2.2 can now be written as

$$m = \frac{N_p M}{t_m (s_{th}f_{th} + I_0f_{ep}) S.D.C.N_A a e_p g} \tag{2.3}$$

Values of s_{th} and I_0 are given by Decorte *et al* [3].

If epithermal neutron activation analysis is applied only the conventional epithermal flux f_{ep} is considered. The thermal neutrons will be shielded by the cadmium bars or boron shields. Equation 2.3 becomes.

$$m = \frac{N_p}{t_m} \cdot \frac{M}{N_A \cdot a \cdot g \cdot e_p \cdot I_0 f_{ep} \cdot S.D.C.} \quad (2.4)$$

The deviation of the epithermal flux from ideality influences the resonance response [4]. This means that

$$f_{ep} \propto \frac{1}{E^{1+\alpha}} \quad (2.5)$$

As the epithermal flux distribution deviates from the ideal $1/E$ function, the resonance integral is modified with an

α – dependent term as follows:

$$I_0(a) = \int_{E_a}^{\infty} s(E) \frac{E_a^\alpha}{E^{1+\alpha}} dE \quad (2.6)$$

E_a = arbitrary energy (usually 1 eV) and α = an experimentally determined characteristics of the reactor site [5]. In order to convert I_0 to $I_0(a)$ so as to use I_0 values in real $1/E^{1+\alpha}$ (non –ideal) circumstances, the effective resonance energy E_r has to be introduced. E_r is defined as the energy of a single virtual resonance which gives the same resonance activation rate as all actual resonance for isotope⁶ and is an α dependent parameter.

$$E_r(a) = E_a \frac{I_0(a)}{I_0} \quad (2.7)$$

Where $I_0(a)$ and I_0 = reduced resonance

$$\text{Integral, } E_a = 1eV \text{ and } \frac{E_r(a)I_0}{E_a} = I_0(a)$$

The conversion of I_0 to an α dependent term taken the form as given by[6]

$$I_0(a) = \frac{I_0 - 0.429\sigma_{th}}{\bar{E}_r^a} + \frac{0.429\sigma_{th}}{(2a + 1)\bar{E}_{cd}^a} \quad (2.8)$$

with \bar{E}_r^a , \bar{E}_{cd}^a and \bar{E}_{cd}^a in eV

σ_{th} = (n, γ) cross section (2200ms^{-1} neutron velocity).

From equation 2.7 it can be observed that E_r is not a constant but α - dependent.

A conversion formula given by [3]:

$$Q_o(a) = \frac{Q_o - 0.429}{\bar{E}_r^a} + \frac{0.429}{(2a + 1)\bar{E}_{cd}^a} \quad (2.9)$$

Since $E_a = 1eV$, \bar{E}_r^a can be expressed as

$$\bar{E}_r^a = \frac{(Q_o - 0.429)(2a + 1)\bar{E}_{cd}^a}{(2a + 1)\bar{E}_{cd}^a Q_o(a) - 0.429} \quad (2.10)$$

If the flux ratio parameter is defined as:

$$f = \frac{f_{th}}{f_{ep}} = Q_o(a)[R_{cd} - 1] \quad (2.11)$$

where f_{th} = thermal flux

f_{ep} = epithermal flux and

$$R_{cd} = \frac{A_b}{F_{cd}A_{cd}} \quad (2.12)$$

where R_{cd} = cadmium ratio
 A_b = activity of bare cadmium
 A_{cd} = activity of cadmium covered foil, and
 F_{cd} = cadmium transmission factor

From equation 2.11 one can write:

$$Q_o(a) = \frac{f_{th}}{(R_{cd} - 1)f_{ep}} \quad (2.13)$$

Then equation 2.10 becomes:

$$\overline{E}_r^a = \frac{(Q_o - 0.429)(R_{cd} - 1)f_{ep}(2a + 1)\overline{E}_{cd}^a}{(2a + 1)\overline{E}_{cd}^a f_{th} - (R_{cd} - 1)0.429f_{ep}} \overline{E}_a^{-a} \quad (2.14)$$

where $Q_o = \frac{I_o}{S_{th}}$

Values of \overline{E}_r^{-a} can be calculated from equations 2.7, 2.10 and 2.14

Equation 2.4 can be written as:

$$m = \frac{N_p}{t_m} \cdot \frac{M}{N_A a g e_p f_{ep} I_0(a) S.D.C} \quad (2.15)$$

2.3 Standardization

The two features of neutron induced reaction –high penetrability for neutrons and gamma radiation –ensure that its standardization is potentially easy and accurate. As the signal to concentration ratio is nearly matrix independent, the preparation is rather easy; therefore, the risk of systematic or random errors is reduced. The method of standardization used in work is the classic relative method. Samples and standards are irradiated simultaneously in identical positions. Standard and samples must have similar composition. The induced intensities are measured for both samples and standards. Individual mono-element standards can be used for relative standardization as well as synthetic or natural multi-element standards. The value of the unknown quantity (m) is obtained with the help of the following equations.

$$m_x = \frac{I}{I_{sp}} \quad (2.16)$$

where $I_{sp} = \frac{N_{p.sp}}{[S.D.C.M]_p}$ and $I = \frac{N_p}{S.D.C}$

(st) refers to standard all other terms have their usual meaning. Thus,

$$m_x = m_{st} \frac{\frac{N_p}{S.D.C}}{\frac{N_p}{S.D.C}} \cdot \frac{S.D.C}{N_p} \quad (2.17)$$

where (x) refers to sample or unknown

The standard preparation procedure determines the accuracy of the relative method. The advantage of the relative classic method lies in the multi-element application. The procedure of the preparation of standard and counting is laborious. In addition to the occasional loss of information appearance of an element for which no standard has been irradiated is a hindrance. The commercial standard reference materials (SRMs) aim at improving the accuracy of the measurement and allowing a proper assurance in the laboratories.

3.0 Experimental

The samples were standard reference material obtained from National Institute of Standard Technology (NIST). The samples were homogenized. Homogenized samples were prepared for irradiation according to the samples preparation procedure developed for NIRR-1[7]. Samples were irradiated using NIRR-1 at the Centre of Energy Research and Training(CERT) ABU, Zaria.

The quantitative determination of trace elements in the standard is achieved by the use of ENAA with NIRR-1 facilities. The method consists of obtaining the concentrations of the elements in standard sample and comparing it with the literature values which are the predetermined concentration of the appropriate trace element(s) of interest.

Standards are usually those of identical matrices or materials as close as possible to the composition of samples. Standard are usually referred to as “certified” or simply Standard Reference Materials (SRMs). SRMs for National Institute of Standard and Technology (NIST) that were used in this work are:

- a. NIST SRM 1548a, Typical Diet
- b. NIST SRM 1573a, Tomato Leave
- c. NIST SRM IAEA, Soil-7

Care was taken during the sample preparation in order to minimise the contamination of the standards samples by avoiding excessive direct contact. As these could give rise to errors in the measured concentrations of trace elements in the samples and standards. All standards samples were prepared in identical manners. This is necessary in order to reduce matrix effect, as well as ensuring uniform neutron attenuation and absorption.

An assembly of polyethylene sample containers, a boron shield and associated polyethylene cushions were transferred in the reactor “NIRR-1” in a rabbit capsule transport done by pneumatic transfer systems A and B. The irradiation time is 5 min at a neutron flux setting of $5 \times 10^{11} \text{ n cm}^{-2} \text{ s}^{-1}$. Every sample was irradiated separately.

The irradiated samples were removed from the boron shield after irradiation. Induced activities in detector foil was measured on a *GEM – 30195 HPGe* waxial, vertical dipstick detector (*ORTEC*), which has a relative efficiency of 30% and resolution of 1.95 keV, at 1.33 MeV, ^{60}Co . The gamma –ray acquisition system consist of *MAESTRO* multi-channel Analyzer (*MCA*) emulation software card, coupled to the detector via electronic modules, all manufactured by *ORTEC*. The multi-purpose Gamma ray analysis software *Win SPAN* [8] was used for peak identification and evaluation.

4.0 Results And Discussion

The nuclear reactions, the Q_0 values, half- life of activation products and the principal gamma ray energies are listed in table 1.

Table 1: Relevant Nuclear Data

Nuclides	Nuclear Reactions	Halfe-life(min)	$E_\gamma(\text{KeV})$	$\sigma_{\text{th}}(\text{barns})$	$I_0(\text{barns})$	Q_0
^{128}I	$^{127}\text{I}(\text{n}, \gamma)^{128}\text{I}$	24.99	442.9	100	4.04	24.75
^{82}Br	$^{81}\text{Br}(\text{n}, \gamma)^{82}\text{Br}$	17.68	619.11	49.8	2.58	19.3
^{38}Cl	$^{37}\text{Cl}(\text{n}, \gamma)^{38}\text{Cl}$	37.24	1642.69	0.29	0.423	0.68
^{42}K	$^{41}\text{K}(\text{n}, \gamma)^{42}\text{K}$	722160	1524.58	1.41	1.45	0.97
^{56}Mn	$^{55}\text{Mn}(\text{n}, \gamma)^{56}\text{Mn}$	154.74	846.8	13.9	13.2	1.05

^{24}Na	$^{23}\text{Na} (n, \gamma) ^{24}\text{Na}$	897.84	1368.6	0.303	0.513	0.59
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Table 2, 3 and 4 gives the experimentally determined boron ratios for elements of interest in three standard reference materials, where the boron ratio is the peak area obtained for the unshielded sample divided by the peak area obtained when the sample was irradiated under the boron shield.

Table 2: Boron ratio for IAEA soil 7

Element	Isotope	ACTIVITY INAA(Bq/g)	ACTIVITY ENAA(Bq/g)	R _{BN} Experimental
Sodium	^{24}Na	7268.57	95.07	76.45
Manganese	^{56}Mn	31045.82	575.45	53.95
Iodine	^{128}I	36.07	5.86	6.15

Table 3: Boron ratio for NIST 1548a Typical Diet

Element	Isotope	ACTIVITY INAA(Bq/g)	ACTIVITY ENAA(Bq/g)	R _{BN} Experimental
Sodium	^{24}Na	17938.99	430.05	41.71
Chlorine	^{38}Cl	834.56	35.16	23.73
Potassium	^{42}K	422683.4	NA	NA
Manganese	^{56}Mn	379.62	9.24	41.08
Bromine	^{82}Br	993.34	67.05	14.82
Iodine	^{128}I	NA	0.53	NA

Table 4: Boron ratio for NIST 1573a Tomato Leaves

Element	Isotope	ACTIVITY INAA(Bq/g)	ACTIVITY ENAA(Bq/g)	R _{BN} Experimental
Sodium	^{24}Na	NA	NA	NA
Chlorine	^{38}Cl	459.32	16.68	27.53
Potassium	^{42}K	829490.2	11664.16	71.11
Manganese	^{56}Mn	10338.13	403.08	25.65
Bromine	^{82}Br	68536.83	9133.99	7.50
Iodine	^{128}I	NA	NA	NA

Iodine and Bromine are shielded more effectively by the boron shield. In general, a given sample type will exhibit several isotopes which dominate the gamma-ray spectrum (e.g. K, Cl, Mn, Na). These will often cause serious interference in determining other isotopes of interest, particularly ones which give minor peaks in the spectrum. It is noteworthy that the elements that typically cause interference can be highly shielded. ^{24}Na is often taken as an example to estimate the advantage factor obtainable using the ENAA technique. The advantage factor is calculated as the boron shield ratio

for sodium divided by the boron shield ratio for the isotope of interest. The advantage factor represents the degree to which the particular interference will be reduced by using the boron shield. The advantage factors over Sodium for elements of interest under the boron shield are 1.76, 1.02, 2.82 and 12.43 for Cl, Mn, Br, and I respectively. Iodine and Bromine show better advantage.

Thus it is indeed possible to optimize the shielding conditions to discriminate among the elements present in a sample, in order to give the largest advantage to elements of particular interest.

In this work, Iodine and Bromine give better advantage factors showing that their determination will not have any hindrance as the activities of the interfering elements are highly reduced by the boron shield. This can clearly be shown from the calculated values of the advantage factors of Iodine and Bromine with respect to the entire possible interfering element, namely Na, K, Cl and Mn (Table 5). They were calculated by dividing the boron ratios of the interfering elements by those of the elements of interest (Iodine and Bromine). All the values obtained are greater than one. This indicates that Iodine and Bromine can be determined with better detection limits in the presence of the interfering element such as Na, Cl, K and Mn by ENAA.

Table 5: Advantage Factor of Iodine and Bromine with Respect to the Interfering Element (NIST 1548a)

Element	Isotope	Advantage factor	
		Iodine	Bromine
Sodium	²⁴ Na	7.28	2.82
Chlorine	³⁸ Cl	4.14	1.60
Potassium	⁴² K	NA	NA
Manganese	⁵⁶ Mn	7.17	2.77

Due to relatively low half-life (25 min) of the activation product of Iodine (¹²⁸I), the irradiation time has to be limited, else interferences from high Compton background from the activation products of interfering elements like Na, K, Cl, Mn and Br will become significant. Thus, due to high Compton background, determination of low levels of Iodine in biological samples like food and food products cannot reliably be measured by INAA using whole reactor neutrons. In order to overcome this problem, ENAA is used. Thermal neutrons are filtered off by covering the sample with thermal neutron absorber like boron. The ENAA is effective if the isotope of interest has higher Q₀ value than those of the interfering elements. The Q₀ value for a nuclide is defined as the ratio of resonance integral (I₀) to its thermal neutron absorption cross-section (σ_{th}) of the isotope of interest [9]. Any nuclide having Q₀ value >10 is a good candidate for its determination by ENAA [10]. The Q₀ value for ¹²⁷I is 24.75 which is much larger than that for some of the interfering nuclides such as ²³Na, ³⁷Cl, ⁴¹K and ⁵⁵Mn whose Q₀ values are 0.59, 0.69, 0.97 and 1.053, respectively.

The peak area under the characteristics gamma ray peaks of the elements of interest were determined by peak fit software MAESTRO for Windows developed at ORTEC. The concentration calculation was done by the relative method of INAA. Using concentration of element in standard (C_{x,std}) and specific activity of standard (A_{sp,std}) and sample (A_{sp,sa}), the concentration of element present in the sample (C_{x,sa}) was calculated for the same counting period by the following equation:

$$C_{x,sa} = C_{x,std} \frac{A_{sp,sa}}{A_{sp,std}} \quad (4.1)$$

Here soil7 and tomato leaves standard reference materials were used as comparator standards. Concentrations of the following elements: I, Br, Cl, K, Mn and Na were reinvestigated and the values compared with literature values of Reference Materials as shown in Table 6.

Table 6: Determination of Concentration of elements in the Typical Diet Reference Material (NIST 1548a).

Element	Literature value	This work	Factor
I	0.759±0.103	0.745±0.32	0.98
Br	9.64±0.00	9.490±3.01	0.98
Cl	12078±356	13925±377	1.15
K%	0.6970±0.013	NA	NA
Mn	5.75±0.17	5.627±1.4	0.98
Na	8132±942	11181±3051	1.4

Factor= this work /literature value

For the kind of shield the concentration compared well with the literature quoted values. This shows that boron can be used as shielding material for ENAA of food samples.

The quality assurance result (Table 6) shows that the determinations for most of the elements were in good agreement with the Standard Reference Materials certified values. Other factors that can be used to assess quality assurance are coefficient of variation (COV%), or accuracy and precision as shown in Table 7 for NIST 1548a.

Table7: Summary of Internal Quality Control Programme

Element	COV%	Accuracy	Precision	LD(µg/g)
I	14.76	0.09	0.2	NA
Br	31.66	0.05	0.32	6.70
Cl	2.71	-3.56	0.04	NA
K	NA	NA	NA	NA
Mn	24.31	0.09	0.24	0.09
Na	27.29	-0.95	0.29	0.60

COV= Coefficient of Variation

The data generated during the study of six radiologically and nutritionally important elements indicate that most of the elements were analyzed with an average accuracy of 7% for boron shield. The average precision value obtained with boron as shielding material was found to be 24% .This value is acceptable since the maximum reported limit [11] is 25%.

Cl and Na showed a large deviation from the literature values, this is due to their being major interfering elements. The method is reported to be good for iodine and bromine. The interference effect of Cl and Na is highly reduced but not completely eliminated. Therefore interference effect need to taken into consideration by applying a method describe elsewhere [12]

The detection limit was calculated first using the Curie formula [13] given as

$$L_D = 2.71 + 3.29\sqrt{B}$$

(4.2)

where B is the back ground obtained by subtracting the net count given by the detector from the gross count. The values of the detection limit obtained for various elements were converted to $\mu\text{g/g}$ by dividing the obtained values calculated from the above equation by the sensitivity and sample mass. The results for typical diet reference material (NIST 1548a) are shown in Table 7.

The L_D values are in the range of $0.09 - 6.7 \mu\text{g g}^{-1}$ for the reference material (Table 7). The higher detection limits of Iodine in samples could be attributed to enhanced background due to sample matrix and longer decay time of counting. Salt contents are lower in these types of samples and hence the spectral interference is minimal [14]. Detection limits for Iodine can further be improved by suitably choosing the experimental conditions like irradiating for longer periods and using a higher efficiency detector for measurements so as to enhance sensitivity. Additionally, detection limit can be improved by the use of Compton suppressed gamma ray spectrometry in which there is a substantial reduction of Compton background.

5.0 Conclusion

An Epithermal Neutron Activation Analysis method using boron as thermal neutron shielding was optimized for the determination of trace levels of I, Br, Cl, K, Mn and Na. The concentrations obtained in NIST SRMs 1548a were found to be within 7% of their certified values, reflecting the accuracy of the ENAA method. The L_D values are in the range of $0.09 - 6.7 \mu\text{g g}^{-1}$ for the reference material. The method is simple, non-destructive and quick to arrive at Iodine and Bromine concentrations even in the presence of high salt matrix like NaCl.

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