

USES OF INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS FOR ELEMENTAL INVESTIGATION OF SOME DOMESTIC FERTILIZERS

A. M. Hassan¹, H. A. Abdel Ghany², W. M. Abdel-Aziz¹, T. El-Zakla³

¹*Reactors Physics Dept. N.R.C, AEA, Cairo, Egypt*

²*Physics Dept. Faculty of Girls for Art, Science and Education, Ain-Shams University, Cairo, Egypt*

³*Hot Laboratory Center, AEA, Cairo, Egypt*

The neutron irradiation facilities offered by the ET-RR-2 and the HPGe-detection system are used for elemental investigation of three samples of domestic fertilizers. A total of 31 elements have been identified as major and minor concentration values. The Pneumatic Irradiation Transfer Systems (PITS) and the core of the reactor are used for short and long time irradiation. The data obtained for some of the identified elements are compared with the corresponding values obtained by the XRF and the ICP-MS techniques for the same samples. The range of the concentration values of these elements was in between several parts per million (ppm) up to 45 %. A discussion on the elemental concentration values and their biological effect are given.

1. Introduction

Neutron activation analysis (NAA) is one of the most important analytical techniques, which yield very accurate and precise results for trace and ultra-trace elemental concentrations in complex samples. Along the past several decades, this has been applied for determination of a great variety of elements in many disciplines including environmental [1], biological [2], geological as well as material science [3]. It is considered as a method for qualitative and quantitative determination of elements based on the measurement of characteristic radiation from radionuclides formed directly or indirectly by neutron irradiation of samples [4]. The most suitable source of the neutrons is usually the nuclear research reactor. The high resolution gamma-ray detection systems are used as well for analysis of the complex gamma-ray spectra obtained by neutron capture.

A neutron flux in the order of $1.3 \cdot 10^{11} \text{ cm}^{-2}\text{s}^{-1}$ for long and $2.7 \cdot 10^{11} \text{ cm}^{-2}\text{s}^{-1}$ for short irradiation times are still quite acceptable for many NAA purposes, as has been demonstrated by many laboratories [5].

The research reactor facilities offer more advantages for INAA, such as, relatively low gamma-ray dose and allowing for relatively long irradiation with samples packed in Al-foils and plastic capsules.

Because phosphate fertilizers are manufactured from rock phosphates, they may contain various trace and minor elements [6]. These elements, when applied to the soil, may persist due to their long life-time in soils, and could be readily available for plants, especially in acidic soils [7] with a potential risk of transfer to the plants and human food chain [8]. Biologically, these heavy metals are toxic to living systems particularly when present in high concentrations. Thus the objective of this work is to evaluate the concentrations of elements in some domestic fertilizer samples [9].

Pollution of agricultural soils with fertilizers can be solved by limiting the total load of each heavy metal, taking into consideration pH, organic matter and clay contents, and other properties that reflect binding capacity of soil components, so that soil could be maintained as a multi-functional system, without affecting biodiversity, another important quality that could be adversely affected by fertilizers [6].

Knowledge of metal concentrations in fertilizers must be assessed in the case of fertility trials or in continuous cropping systems where phosphate fertilizers are added to soils. These concerns are very important in agricultural systems

2. Experimental

2.1. Samples preparation

Three samples of domestic fertilizers from Delta Gypsum Company and the International Company of Fertilizers were grinded and prepared for irradiation. The weight of each sample in case of short irradiation was 0.1 gm while in case of long irradiation was 0.3 gm. The samples were encapsulated in the polyethylene containers and irradiated for different times.

2.2. Irradiation

2.2.1. Short time irradiation facility. In ET-RR-2 there are two computerized PITS to give precise timing for irradiation for analysis of short lived radionuclides. The samples requiring short irradiation times (9000 s) and moderate flux densities ($10^{11} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$), then packed in virgin polyethylene vials and placed into larger transport device known as rabbit. The rabbit travels from the laboratory sending station to the irradiation position in the reactor core. After irradiation the rabbit was extracted from the core and transported back to the laboratory, where the vials are removed, and transferred to non-irradiated vials for completion of the analytical process and data collection.

2.2.2. Long time irradiation facility. The samples requiring longer irradiation times (several hours or days), with high flux densities, (10^{13} n/cm² · s) were packaged in high purity quartz vials. After irradiation, the sample holder is removed just to allow short-lived radioactivity to dissipate for safe handling levels.

2.3. Gamma ray spectra-detection

Gamma-ray spectrometers were used to record the gamma-ray spectra. A coaxial hyper-pure germanium (HPGe) detector (1.71 keV FWHM for the 1332.5 keV gamma-ray line of ⁶⁰Co and 25 % detection efficiency) attached to preamplifier, spectroscopy amplifier and a computerized 4096-multichannel analyzer provided with a printer and plotter to record the measured gamma-ray spectra. The sample-to-detector distance was about 10 cm. Energy and efficiency calibration curves up

to about 3 MeV at the experimental conditions were carried out using the multi-gamma-ray standard sources MGS-4 [10]. All measurements were analyzed both qualitatively and quantitatively.

3. Results and discussion

Qualitatively, the results revealed a total of 31 elements. Six elements of isotopes: ²⁴Na, ²⁸Al, ⁵²V, ⁵⁶Mn, ⁸⁶Rb, and ^{137m}Ba were determined. All these isotopes are short half-life isotopes which were measured by rabbit technique as shown in Table 1. Other 25 elements have the isotopes (¹⁵²Eu, ¹⁰⁹Pd, ⁵¹Cr, ¹⁸¹Hf, ¹³¹Ba, ¹²⁴Sb, ⁹⁵Zr, ⁴⁶Sc, ⁵⁹Fe, ¹⁸²Ta, ⁶⁰Co, ⁷⁷Ge, ¹⁴²Ce, ¹⁶⁰Tb, ¹⁶⁹Yb, ¹⁴⁷Nd, ¹⁹⁴Ir, ¹⁰³Ru, ⁴⁷Ca, ^{124m}Sb, ⁷⁶As, ^{71m}Zn, ⁸⁵⁹Sr, ¹³⁴Cs, and ⁸⁶Rb) were determined. These isotopes have long half-life isotopes which are measured by delayed technique as shown in Tables 2, 3 and 4.

Table 1. The concentration values for the short lived radionuclide in the samples under investigation

Sample	Element	Nuclear reaction	γ-ray, keV	I _γ , %	T _{1/2}	INAA (present work)	XRF, %	ICP, ppm
S1	⁵⁶ Mn	⁵⁵ Mn(n, γ) ⁵⁶ Mn	847.8	98.9	2.5 h	0.109 % ± 0.007	0.321 ± 0.11	–
	²⁴ Na	²³ Na(n, γ) ²⁴ Na	1368.5	100	14.96 h	20.6 ppm ± 1.90	–	–
	⁵² V	⁵¹ V(n, γ) ⁵² V	1434.1	100	3.75 m	20.7 ppm ± 1.91	–	8.63
	²⁸ Al	²⁷ Al(n, γ) ²⁸ Al	1779	100	2.25 m	0.614 % ± 0.04	0.714 ± 0.34	–
	⁸⁶ Rb	⁸⁵ Rb(n, γ) ⁸⁶ Rb	2111.2	0.12	17.8 m	58.8 ppm ± 5.44	–	80.5
S2	⁵⁶ Mn	⁵⁵ Mn(n, γ) ⁵⁶ Mn	847.8	98.9	2.5 h	0.158 % ± 0.01	0.504 ± 0.17	–
	²⁴ Na	²³ Na(n, γ) ²⁴ Na	1368.5	100	14.96 h	0.051 % ± 0.003	–	–
	⁵² V	⁵¹ V(n, γ) ⁵² V	1434.1	100	3.75 m	31.5 ppm ± 2.91	–	8.87
	²⁸ Al	²⁷ Al(n, γ) ²⁸ Al	1779	100	2.25 m	3.53 % ± 0.23	3.366 ± 0.66	–
	⁸⁶ Rb	⁸⁵ Rb(n, γ) ⁸⁶ Rb	2111.2	0.12	17.8 m	80.6 ppm ± 7.46	–	–
S3	⁵⁶ Mn	⁵⁵ Mn(n, γ) ⁵⁶ Mn	847.8	98.9	2.5 h	3.99 ppm ± 0.36	–	–
	²⁸ Al	²⁷ Al(n, γ) ²⁸ Al	1779	100	2.25 m	60.67 ppm ± 5.61	–	–
	^{137m} Ba	¹³⁶ Ba(n, γ) ^{137m} Ba	661.6	28.4	3.94 h	16.78 ppm ± 1.55	–	16.25

Table 2. Elemental content of sample No. 1 as obtained by INAA, XRF and ICP, for long lived isotopes

Element	Nuclear reaction	γ-ray, keV	I _γ , %	T _{1/2}	INAA (present work)	XRF, %	ICP, ppm
¹⁵² Eu	¹⁵¹ Eu(n, γ) ¹⁵² Eu	121.8	28.4	13.3 y	0.3185 ppm ± 0.02	–	0.62
¹⁰⁹ Pd	¹⁰⁸ Pd(n, γ) ¹⁰⁹ Pd	311.4	0.03	13.46 h	0.383 ppm ± 0.03	–	0.37
⁵¹ Cr	⁵⁰ Cr(n, γ) ⁵¹ Cr	320.1	9.83	27.7 d	21.1 ppm ± 1.95	–	16.13
¹⁸¹ Hf	¹⁸⁰ Hf(n, γ) ¹⁸¹ Hf	482	85.5	42.4 d	0.17 ppm ± 0.015	–	0.11
¹³¹ Ba	¹³⁰ Ba(n, γ) ¹³¹ Ba	496.3	47.1	11.8 d	130.5 ppm ± 12.08	–	115.3
¹²⁴ Sb	¹²³ Sb(n, γ) ¹²⁴ Sb	602.7	98.4	60.2 d	0.2 ppm ± 0.018	–	0.5
⁹⁵ Zr	⁹⁴ Zr(n, γ) ⁹⁵ Zr	724.2	43.7	64.03 d	2.9 % ± 0.26	–	–
⁴⁶ Sc	⁴⁵ Sc(n, γ) ⁴⁶ Sc	889.2	100	83.82 d	0.49 ppm ± 0.04	–	–
⁵⁹ Fe	⁵⁸ Fe(n, γ) ⁵⁹ Fe	1099.3	56.3	44.5 d	9.14 % ± 0.63	6.261 ± 0.09	–
¹⁸² Ta	¹⁸¹ Ta(n, γ) ¹⁸² Ta	1121.3	35.0	115.0 d	1.04 % ± 0.07	–	–
⁶⁰ Co	⁵⁹ Co(n, γ) ⁶⁰ Co	1173.2	99.9	5.27 y	11.8 ppm ± 1.08	–	10.5
⁷⁷ Ge	⁷⁶ Ga(n, γ) ⁷⁷ Ge	2089.6	0.33	11.3 h	0.662 ppm ± 0.05	–	0.87
⁴⁷ Ca	⁴⁶ Ca(n, γ) ⁴⁷ Ca	530.4	0.1	4.54 d	42.4 % ± 2.76	42.329 ± 0.45	–

Table 3. Elemental content of sample No. 2 as obtained by INAA, XRF and ICP, for long lived isotopes

Element	Nuclear reaction	γ -ray, keV	I_γ , %	$T_{1/2}$	INAA (present work)	XRF, %	ICP, ppm
¹⁵² Eu	¹⁵¹ Eu(n, γ) ¹⁵² Eu	121.8	28.4	13.3 y	0.184 ppm \pm 0.06	–	0.15
¹⁴² Ce	¹⁴¹ Ce(n, γ) ¹⁴² Ce	145.4	48.4	32.5 d	12.7 ppm \pm 1.26	–	5.12
¹⁶⁰ Tb	¹⁵⁹ Tb(n, γ) ¹⁶⁰ Tb	197	6.79	2.73 y	0.15 % \pm 0.01	–	0.0013
¹⁶⁹ Yb	¹⁶⁸ Yb(n, γ) ¹⁶⁹ Yb	307.7	11.1	32.02 d	1.5 % \pm 0.1	–	–
¹⁴⁷ Nd	¹⁴⁶ Nd(n, γ) ¹⁴⁷ Nd	319.4	1.95	10.98 d	0.52 ppm \pm 0.02	–	0.75
¹⁹⁴ Ir	¹⁹³ Ir(n, γ) ¹⁹⁴ Ir	328.4	92.8	171 d	48.7 ppm \pm 4.8	–	–
¹⁷⁵ Hf	¹⁷⁴ Hf(n, γ) ¹⁷⁵ Hf	344.04	86.6	70 d	0.17 ppm \pm 0.01	–	–
¹⁰³ Ru	¹⁰² Ru(n, γ) ¹⁰³ Ru	443.8	0.32	39.25 d	0.14 ppm \pm 0.01	–	0.5
¹³¹ Ba	¹³⁰ Ba(n, γ) ¹³¹ Ba	486.5	2.09	11.8 d	31.04 ppm \pm 3.08	–	24.38
⁴⁷ Ca	⁴⁶ Ca(n, γ) ⁴⁷ Ca	530.4	0.1	4.54 d	41.4 % \pm 2.76	41.329 \pm 0.45	–
^{124m} Sb	¹²³ Sb(n, γ) ^{124m} Sb	602.7	20.0	60.2 d	0.25 % \pm 0.01	–	–
⁵⁹ Zr	⁹⁴ Zr(n, γ) ⁹⁵ Zr	724.2	43.7	64.03 d	0.072 % \pm 0.005	0.073 \pm 0.06	0.073
⁷⁶ As	⁷⁵ As(n, γ) ⁷⁶ As	867.6	0.13	26.32 h	1.34 ppm \pm 0.10	–	1.25
⁴⁶ Sc	⁴⁵ Sc(n, γ) ⁴⁶ Sc	889.2	100	83.82 d	0.149 % \pm 0.01	–	–
⁷⁷ Ga	⁷⁶ Ga(n, γ) ⁷⁷ Ga	925.5	0.74	11.3 h	0.78 ppm \pm 0.06	–	0.73
^{71m} Zn	⁷⁰ Zn(n, γ) ^{71m} Zn	964.7	4.7	3.94 h	4.6 % \pm 0.32	1.012 \pm 0.11	–
⁵⁹ Fe	⁵⁸ Fe(n, γ) ⁵⁹ Fe	1099.3	56.3	44.5 d	3.42 % \pm 0.23	8.881 \pm 0.15	–
⁶⁰ Co	⁵⁹ Co(n, γ) ⁶⁰ Co	1173.2	99.9	5.27 y	10.2 ppm \pm 0.83	–	8.25

Table 4. Elemental content of sample No. 3 as obtained by INAA, XRF and ICP, for long lived isotopes

Element	Nuclear reaction	γ -ray, keV	I_γ , %	$T_{1/2}$	INAA (present work)	XRF, %	ICP, ppm
⁴⁷ Ca	⁴⁶ Ca(n, γ) ⁴⁷ Ca	489.2	6.74	4.54d	45.3 % \pm 3.02	59.098 \pm 0.17	–
⁸⁵⁹ Sr	⁸⁴ Sr(n, γ) ⁸⁵⁹ Sr	514	99.3	64.84d	0.193 % \pm 0.007	0.119 \pm 0.02	–
¹³⁴ Cs	¹³³ Cs(n, γ) ¹³⁴ Cs	604.7	97.6	2.06y	0.58 ppm \pm 0.11	–	0.5
¹⁵² Eu	¹⁵¹ Eu(n, γ) ¹⁵² Eu	778.9	13.0	13.33y	7.7 ppm \pm 0.76	–	–
¹⁶⁰ Tb	¹⁵⁹ Tb(n, γ) ¹⁶⁰ Tb	879.4	30.0	72.3d	7.25 ppm \pm 0.71	–	–
⁴⁶ Sc	⁴⁵ Sc(n, γ) ⁴⁶ Sc	889.2	100	83.82d	28.59 ppm \pm 2.83	–	–
⁸⁶ Rb	⁸⁵ Rb(n, γ) ⁸⁶ Rb	1076.6	8.78	18.66d	8.8 ppm \pm 0.87	–	2.25
⁵⁹ Fe	⁵⁸ Fe(n, γ) ⁵⁹ Fe	1099.3	56.3	44.5d	18.02 % \pm 1.20	–	–
¹⁸² Ta	¹⁸¹ Ta(n, γ) ¹⁸² Ta	1121.3	35.0	115.0d	0.128 ppm \pm 0.02	–	0.37
⁶⁰ Co	⁵⁹ Co(n, γ) ⁶⁰ Co	1173.2	99.9	5.27y	28.5 ppm \pm 2.82	–	9.25
⁷² Ga	⁷⁶ Ga(n, γ) ⁷⁷ Ga	1596.7	4.24	14.1h	5.342 ppm \pm 0.52	–	6.125
¹²⁴ Sb	¹²³ Sb(n, γ) ¹²⁴ Sb	1691	49.0	60.2d	10.3 ppm \pm 1.02	–	–

For quantitative analysis, the well-resolved and pronounced γ -ray lines have been selected to measure the concentrations of 31 elements of the fertilizer samples. In order to estimate the concentration value of each element, the well-known analytical equation was used [11].

$$m = \frac{\lambda CM}{\left[(\epsilon I_\gamma \sigma_{th} f N_0 \phi_m) e^{-\lambda t_w} (1 - e^{-\lambda t_{irr}}) (1 - e^{-\lambda t_c}) \right]}$$

where m is the mass of the element; ϕ_m is the thermal neutron flux (measured by Au foils); λ is the decay constant; C is the activity (net peak area of the interested gamma-rayline); M is the atomic mass; ϵ is the efficiency of the system at the selected full energy peak, I_γ is the absolute intensity of the

gamma-ray line; σ_{th} is the thermal (n, γ) cross-section; f is the isotopic abundance fraction; t_w is the cooling time; t_{irr} is the irradiation time, t_c is the counting time; and N_0 is the Avogadro's number. The main factors used in the calculation were the isotopic abundance of the selected isotopes for each element, half-life, cross-section of the (n, γ) reaction and the intensity of the selected γ -ray line [12].

Fig. 1 shows major elements in the investigated samples. It shows that Ca and Fe have the highest concentrations in sample No. 3 (45.3 %) and (18.02) respectively. While Fig. 2 shows trace elements in the investigated samples. It shows that Ba has the highest concentration in sample No. 1 (130.5 ppm). While Rb has the highest concentration in sample No. 2 (80.6 ppm).

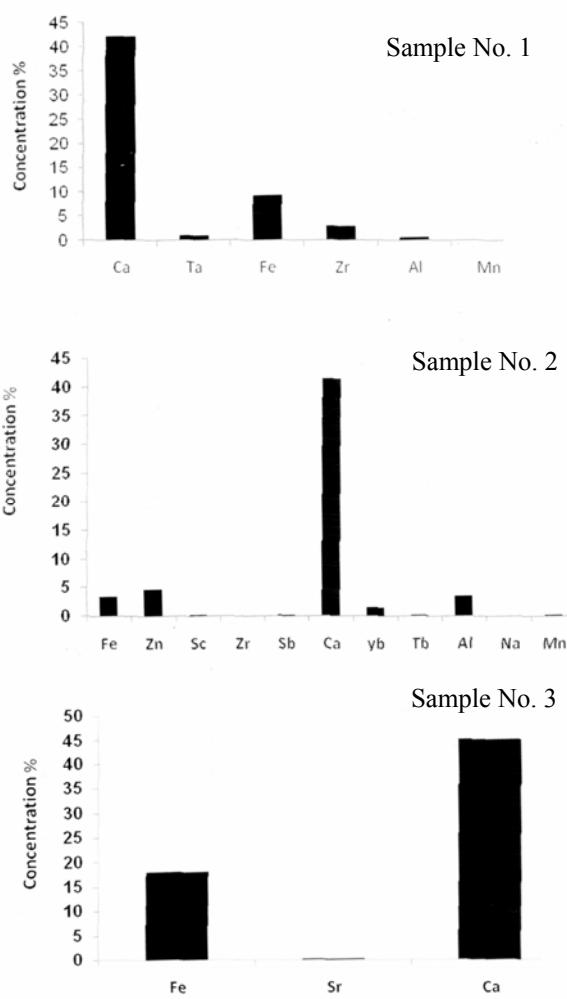


Fig. 1. Major elements in the investigated samples.

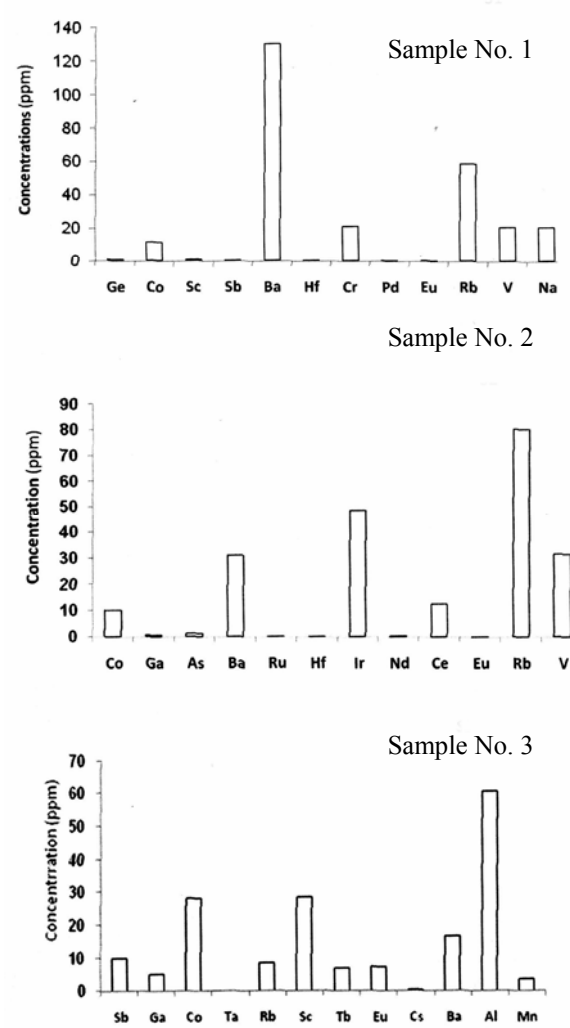
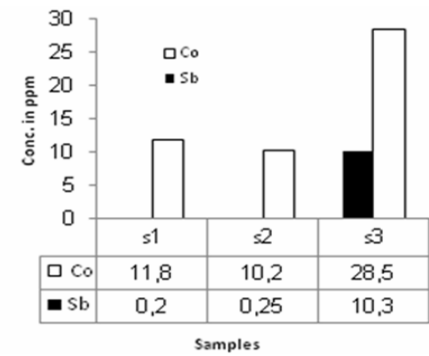
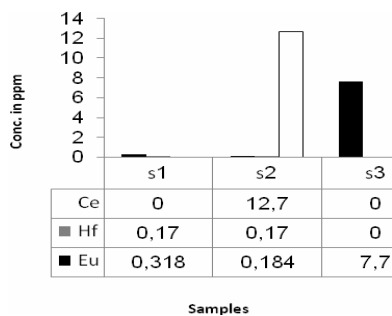


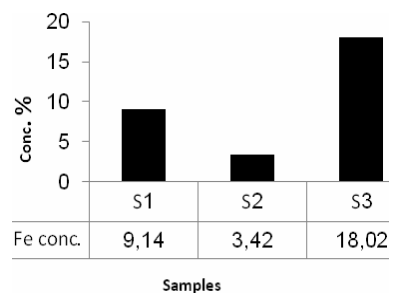
Fig. 2. Trace elements in the investigated samples.



a



b



c

Fig. 3. Heavy metals and rare earth elements in the investigated samples: a – Co and Sb concentrations in the investigated samples; b – contents of rare earth elements in the investigated samples; c – average iron concentration in three samples under investigation.

Toxic elements such as Co and Sb have the great importance in toxicological studies. The concentration of Cobalt in the samples under investigation ranges from 10.2 to 28.5 ppm. The highest concentration of Co was found in sample No. 3 (Fig. 3, a), also the level of Antimony in sample No. 3 was

much higher than other samples. Fig. 1, *b* shows the content of rare earth elements such as Ce, Hf, and Eu in the investigated samples, where Ce was appeared only in sample No. 2, while the concentration of Eu in sample No. 3 was much higher than others. But Hf was found slightly high in sample No. 1 and sample No. 2 and does not appeared in sample No. 3. Also, the contents of heavy metals Fe, Zn, Co, Cr, and Sc were determined, where Fe shows the highest concentration in sample No. 3 (Fig. 3, *c*). Biologically, Iron is known to be essential for different physiological bioprocesses in plants [13] and increase shoot dry weight in soil [14]. Zn element, on the other hand increase seeds yield [15]. While Ca is the major component of the phosphate rocks, because these rocks are mostly phosphorites of marine sedimentary origin, where the Ca was found in sample No. 2 and sample No. 3. Also the increase in Sr in sample No. 3 was expected, because its chemistry is similar to Ca. Where, Chromium is naturally found in the environment, occurring in soils, rocks and living organisms [16]. The biological effects associated with chromium uptake are diverse and depend on its oxidation state. The chromium state is non-carcinogenic because of its inability to bind with carriers encountered in cell membranes [17 - 25].

While Aluminum was found in the three samples and have the concentrations of 0.614 %, 3.35 %, and 60.67 ppm in sample No. 1, sample No. 2, and sample No. 3 respectively [26].

The concentration values have been determined by XRF & ICP just for sake of comparison. Tables 1 - 4 show the discrepancies of the results which was not very high. The elements determined by inductively coupled plasma mass spectrometry (ICP-MS) were ^{52}V , ^{86}Rb , $^{137\text{m}}\text{Ba}$, ^{152}Eu , ^{109}Pd , ^{51}Cr , ^{181}Hf , ^{131}Ba , ^{124}Sb , ^{60}Co , ^{77}Ge , ^{142}Ce , ^{160}Tb , ^{147}Nd , ^{103}Ru , ^{131}Ba , ^{59}Zr , ^{76}As , ^{77}Ga , ^{134}Cs , ^{85}Rb , and ^{182}Ta , and also elements determined by x-ray fluorescence were ^{56}Mn , ^{28}Al , ^{59}Fe , ^{47}Ca , ^{59}Zr , $^{71\text{m}}\text{Zn}$, and ^{85}Sr .

Conclusion

The elemental concentration values of 31 elements in the fertilizer samples, have been determined by applying Sensitive nondestructive analytical techniques such as INAA. It has also shown enough sensitivity to determine the concentrations of several trace and rare earth elements, the concentration values of elements were compared with the corresponding elements obtained by the ICP-MS and XRF techniques, for the same samples.

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ВИКОРИСТАННЯ ІНСТРУМЕНТАЛЬНОГО НЕЙТРОННОГО АКТИВАЦІЙНОГО АНАЛІЗУ ДЛЯ ЕЛЕМЕНТНОГО ДОСЛІДЖЕННЯ ДЕЯКИХ ВІТЧИЗНЯНИХ ДОБРІВ

А. М. Хассан, Х. А. Абдель Гани, В. М. Абдель-Азиз, Т. Ель-Закла

Обладнання для нейтронного опромінення, що існує на Єгипетському дослідницькому реакторі ET-RR-2, та HPGe-детекторна система використовуються для елементного дослідження трьох зразків вітчизняних добрив. Усього було ідентифіковано 31 елемент як з великими, так і незначними значеннями концентрацій. Пневматична транспортна опромінювальна система (PITS) та активна зона реактора використовуються для короткотривалого та довготривалого опроміньєнь. Дані, отримані для деяких ідентифікованих елементів, порівнюються з відповідними значеннями, отриманими за допомогою рентгенофлуоресцентного аналізу (XRF) та методу мас-спектрометрії з індуктивно зв'язаною плазмою (ICP-MS), для тих самих зразків. Діапазон значень концентрації цих елементів був від декількох мільйонних часток (м. ч.) до 45 %. Обговорюються значення елементної концентрації та їх біологічний вплив.

ИСПОЛЬЗОВАНИЕ ИНСТРУМЕНТАЛЬНОГО НЕЙТРОННОГО АКТИВАЦИОННОГО АНАЛИЗА ДЛЯ ЭЛЕМЕНТНОГО ИССЛЕДОВАНИЯ НЕКОТОРЫХ ОТЕЧЕСТВЕННЫХ УДОБРЕНИЙ

А. М. Хассан, Х. А. Абдель Гани, В. М. Абдель-Азиз, Т. Ель-Закла

Установки для нейтронного облучения, которые существуют на Египетском исследовательском реакторе ET-RR-2, и HPGe-детекторная система используются для элементного исследования трех образцов отечественных удобрений. Был идентифицирован 31 элемент как с большими, так и с незначительными значениями концентраций. Пневматическая транспортная облучательная система (PITS) и активная зона реактора используются для кратковременного и длительного облучений. Данные, полученные для некоторых идентифицированных элементов, сравниваются с соответствующими значениями, полученными с помощью рентгенофлуоресцентного анализа (XRF) и метода масс-спектрометрии с индуктивно связанной плазмой (ICP-MS), для таких же образцов. Диапазон значений концентраций этих элементов был от нескольких миллионных долей (м. д.) до 45 %. Обсуждаются значения элементной концентрации и их биологическое влияние.

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