

ELEMENTAL ANALYSIS OF SOME PHARMACOLOGICAL MATERIALS USED IN SAUDI ARABIA BY NEUTRON CAPTURE GAMMA-RAY TECHNIQUES

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In this paper the elemental analysis of four pharmacological materials used in the Kingdom of Saudi Arabia are performed. This has been done by means of the prompt and delayed gamma-ray spectroscopy techniques. ²²⁶Ra/Be and ²⁵²Cf isotopic neutron sources are used for irradiation. The hyper-pure germanium detection systems installed at Physics Department of Jeddah College are used for gamma-ray detection. The concentration percentage values by weight for some major, minor and trace elements for each sample have been estimated. A comparative study between the obtained data and that measured by other techniques for the same samples is given.

Keywords: *PGNAA and DGNAA techniques, Pharmacological samples, ²²⁶Ra/Be and ²⁵²Cf neutron sources.*

INTRODUCTION

In the last few decades, more applications on the use of neutron capture gamma-ray spectroscopy have been developed dealing with the elemental analysis of many of strategic materials [1-30]. A great attention has been made for the analysis of pharmacological samples [2-7,11-15] due to its importance for treatment.

The neutron capture gamma-ray systems installed at the Faculty of Girls Education of Jeddah [20,21], are used for investigation of such materials. The delayed gamma-rays neutron activation analysis (DGNAA) and the prompt gamma-rays due to neutron capture activation analysis (PGNAA) techniques are used.

Four pharmacological samples have been elementally investigated qualitatively and quantitatively. Several advanced computer programs were applied for data analysis [20,21]. The samples under investigation are irradiated by means of isotopic neutron sources ($^{226}\text{Ra}/\text{Be}$ and ^{252}Cf).

For the sake of comparison, the concentration percentage values obtained in this work for many elements have been compared with the data obtained for the same samples by Induced Coupling Plasma-Mass Spectrometer (ICP-MS) and by the Energy Dispersive X-ray [EDX-ray] systems [24-25].

EXPERIMENTAL

Samples

Four pharmacological samples used in the Kingdom of Saudi Arabia named Cix, Sie, Dit and Saf have been submitted in tablets form Saudi Pharmacological and Manufacturing Drug Company at Al-Kasseem industrial area.

The first sample Cix (Cibrofloxacin hydrochloride) is used as antibiotic material of chemical formula of $\text{C}_{19}\text{H}_{16}\text{Cl}_2\text{N}_3\text{NaO}_5\text{S}\cdot\text{H}_2\text{O}$. The second sample Sie (Paracetamol and Pseudoephedrine) is used as analgesic antipyretic material with chemical formula of $\text{C}_{10}\text{H}_{15}\text{NO}\cdot\text{HCl}$ and $\text{C}_8\text{H}_9\text{NO}_2$. The third sample Dit (Glucosamine) which is used as antidiabetic of chemical formula of $\text{C}_{23}\text{H}_{28}\text{ClN}_3\text{O}_5\text{S}$. The fourth sample is Saf (Epi-Brofen) is used as potent analgesic, it has a chemical formula of $\text{C}_{13}\text{H}_{18}\text{O}_2$.

The weight of each sample in grams used in this work for (DGNA) and (PGNA) was as follows:- (36.7, 163.6) for Cix samples, (38.24, 179.43) for Sie samples, (34.30, 150.05) for Dit sample and (34.15, 165.30) for Saf samples, respectively.

Irradiation

In case of (DGNA), each sample is encapsulated in a polyethylene container and positioned in the irradiation cell using $^{226}\text{Ra}/\text{Be}$ and ^{252}Cf isotopic neutron sources [22]. The thermal neutron flux at the irradiation position was about $\sim 8.84 \times 10^6 \text{ n/cm}^2\cdot\text{s}$ as estimated by means of gold foil technique [20,21]. Each sample was irradiated for 4 to 42 days. In case of using the (PGNA) technique, the prompt gamma-rays estimated due to neutron capture were detected directly during the irradiation. The ^{252}Cf isotopic neutron source is used for irradiating the samples (placed at the optimum position) in the prompt gamma-ray system. The thermal neutron flux at the target position was $\sim 8 \times 10^6 \text{ n/cm}^2\cdot\text{s}$. A special polyethylene container for each sample is used.

Gamma-Ray Measurements and Identification of Elements, (Qualitative Analysis)

The delayed and prompt gamma-ray spectra were measured for the samples under investigation by using the hyper-pure germanium detection systems which are described in details in references [20] and [21] published by the same authors. In case of the delayed gamma-ray technique, the delayed gamma-ray spectra were measured two times for each sample. The first run was collected in 2000 s (measuring time) directly when the sample was taken from the irradiation cell, while the second run has been measured under the same conditions after a cooling time of 52 hours, just to identify the short lived isotopes in the region of detection.

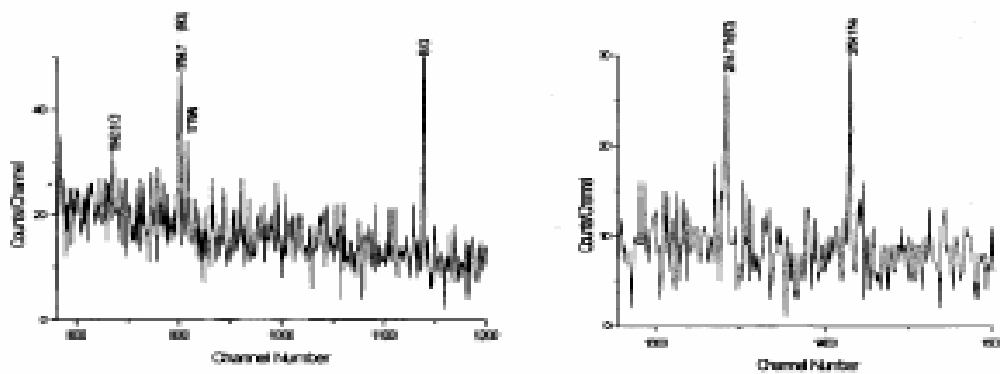


Figure 1-a. A portion of the single γ -ray spectrum of (Sie) sample measured directly after removing from the irradiation cell.

As an example Figure 1-a. shows a portion of the single gamma-ray spectrum of one of the Sie samples measured directly after removing from the neutron sources irradiation cell, while Figure 1-b. shows the same portion of the gamma ray spectrum for the same sample measured after 52 hours cooling time.

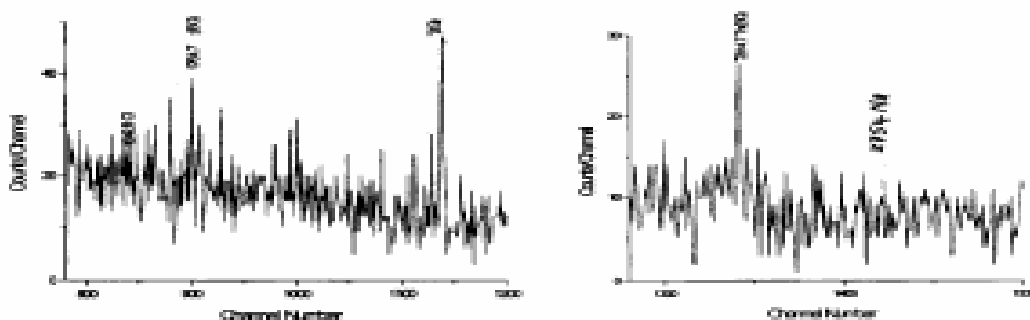


Figure 1-b. The same portion of the single γ -ray spectrum of (Sie) sample after 52 hours cooling time.

These measurements have been done for each sample and repeated under the same conditions for several times up to energy of about 3 MeV. The gamma-ray lines of some different standard radioactive sources were used for the construction of an energy and efficiency calibration curves covering this energy region [20,21].

A total of 17 gamma-ray lines appeared in the spectra of the four samples under investigations which are belonging to the following 15 elements: Na, Al, Cl, K, Mn, Fe, Zn, Rb, In, Cd, Sb, Te, Ho, Lu and Ir. A simple computer program is used for identifying these elements after ignoring the energies appeared in the background spectra. Table 1. summarizes the qualitative elemental analysis of the four samples depending on the gamma-ray energies E_γ appeared in the spectra obtained due to the decay of the radioactive isotopes of these elements. Also, it gives the values of the (n,γ) cross-section (σ) and intensities of the gamma-ray lines I_γ .

Table 1. The gamma-ray lines – appeared in the decay spectra of the four pharmacological samples and parameters of (H.L), (σ), (f), (I_γ) for each (E_γ).

Serial No.	Product Isotope	H.L Of the product isotope	σ barn	f % isot. abund.	I_γ %	<i>Ey</i> lines presented in DGNAAspectra (keV)			
						Cix	Sie	Dit	Saf
1	^{24}Na	15 hr	0.53	100	100	1368.4	1368.4	1368.4	1368.4
					98.6	2753.98	2753.98	2753.98	2753.98
2	^{28}Al	2.24	0.235	100	100	-----	1779*	-----	-----
3	^{38}Cl	37.3min	0.565	24.4	76	1642.8	1642.8	1642.8	1642.8
4	^{42}K	12.56 hr	1.1	6.8	1.68	-----	-----	-----	312.4
5	^{56}Mn	2.6 hr	13.3	100	-----	-----	846.8	-----	846.8
6	^{56}Fe	44.6 d	0.9	0.33	100	1099.3	1099.3	-----	-----
7	^{71}Zn	2.4 min	0.10	0.63	21	-----	910.5	-----	910.5
8	^{86}Rb	18.7 d	0.85	72.2	100	-----	1076.7	-----	-----
9	$^{114\text{m}}\text{In}$	49.5 d	63	4.2	28.5	558.4	558.4	558.4	558.4
10	^{115}Cd	44.8 d	1.24	28.8	43	1290.6	-----	-----	-----
11	^{124}Sb	60 d	3.46	43	100	602.1	602.1	602.1	602.1
12	^{131}Te	25 min	0.21	34.4	15	-----	-----	-----	452.4
13	^{166}Ho	27 hr	64.00	100	20	-----	-----	1581.9	-----
14	$^{177\text{m}}\text{Lu}$	160.9 d	3200	2.6	31	-----	-----	-----	327.7
15	^{192}Ir	74.2 d	1000	38.5	35	-----	-----	295.95	-----
					7	612.5	612.5	612.5	612.5

For the measurements using the (PGNAAs) technique, each sample is positioned in its special container of polyethylene material and exposed to thermal neutrons emitted from the ^{252}Cf isotopic neutron source for 2000 s. The prompt gamma-rays emitted due to neutron capture in the nuclei of each element of the sample have been detected by the hyper-pure germanium detection system directly during irradiation. The measurements have been repeated with the same way for each sample, under the same conditions.

Some gamma-ray lines appeared in the spectra, due to background emitted from the shielding materials. These have been subtracted from each spectrum by using an empty container and measured at the conditions of samples measurement.

A sample of (NaCl) in granules form have been measured as well at the same conditions just to calibrate the system for energy and efficiency up to 10 MeV [20,21] and identifying the background gamma-ray lines.

The gamma-ray lines belonging to H, C, N, Fl, Na, Mg, Al, Si, P, S, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Cu, Zn, Ga, As, Se, Rb, Sr, Ag, Cd, Sb, Fe, Au, Hg and Pb including the background elements have been appeared in the measured spectra. The prompt gamma-ray spectra of the samples under investigation were very complicated in the high energy region from 2-10 MeV where the single and double escape peaks for each photo-line were appeared. So a huge number of gamma-rays lines could be identified in each spectrum, for each sample.

As an example Figure 2. shows a portion of the prompt gamma-ray spectrum of one of the Saf sample taken for 2000 s. using ^{252}Cf neutron source. The sensitivity of each gamma-ray line of each element is considered as the most important factor for identifying the elements which is equal $(I\sigma/M)$ where I is the intensity of the gamma-ray line, σ is the neutron capture cross-section and M is the atomic weight of the element [20,21]. A special computer program is used for identifying the elemental constituents of each sample under investigation.

Estimation of the Concentration Percentage Values of Some Elements (Quantitative Analysis)

The concentration percentage values of eight elements; Na, Al, Cl, Fe, Rb, In, Sb and Ir have been estimated using the data of the delayed gamma-ray spectra. The well known formula of the concentration percentage values estimation for the elemental constituents of the complex samples [21] is used. The formula contained the following important parameters: the half life of isotope selected for this calculations (H.L), the thermal neutron capture cross-section (σ), the fractional abundance of the isotope (f), the intensity of the selected gamma-ray line (I_γ), the thermal neutron flux (ϕ_{th}), the absolute efficiency of the detection system (ϵ), the irradiation time (t_i), cooling time (t_c) and the detection time (t_d). A list of the concentration values of the 8 elements by weight is shown in Table 2. The error in each value includes the error in the net peak area of the selected gamma-ray line, the error in the neutron flux, the error in the efficiency of the system and the error in the intensity of I_γ .

In case of the (PGNAA), the concentration percentage values by weight for the following 12 elements, Na, Mg, Al, Si, S, Cl, Ti, Co, Zn, Rb, Sr and Pb have been estimated.

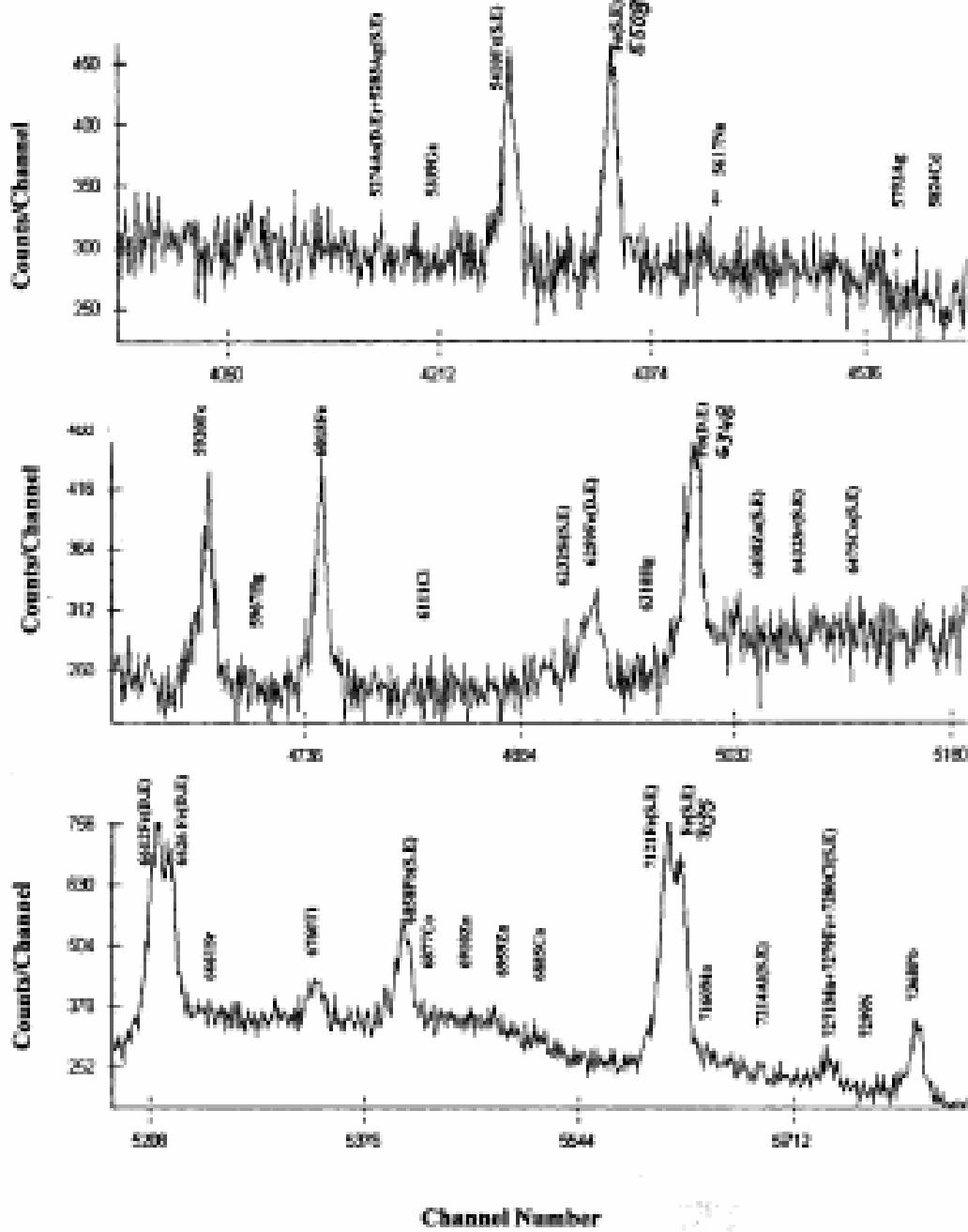


Figure 2. A portion of the prompt gamma-ray spectrum of Saf sample for 2000 s. using ^{252}Cf source.

The net peak area of the selected gamma-ray line $A_{(i)}$, the efficiency of the system $\epsilon_{(i)}$, the thermal neutron capture gamma-ray cross-section (σ) and the intensity of the gamma-ray line $I_{(i)}$ [calculated for 100 neutron capture of the gamma-ray line (i)], are used for $C_{(n)}$ calculations [20,21] as the concentration value by weight for each element in the sample under investigation. The details of the derivation of the applied formula used in this work are found in many of references dealing with the prompt gamma-ray neutron activation analysis technique [14-23]. Table 3. shows the concentration percentage values for the 12 elements according to their presence in the four samples under investigation. Also, the error in each value includes the errors in the net peak area $A_{(i)}$, efficiency $\epsilon_{(i)}$ intensity of the gamma-ray line $I_{(i)}$.

Table 2. The concentration percentage values of some selected elements identified in the four samples under investigation by (DGNAA) technique.

Serial number	Element	Nuclear reaction	Selected E_{γ} peak keV	value of element concentration present work %			
				Cix	Sie	Dit	Saf
1	Na	$^{23}\text{Na}(n,\gamma)^{24}\text{Na}$	1368.4	0.25 ± 0.01	0.27 ± 0.01	0.087 ± 0.004	0.21 ± 0.01
2	Al	$^{27}\text{Al}(n,\gamma)^{28}\text{Al}$	1779	-----	0.14 ± 0.01	-----	-----
3	Cl	$^{37}\text{Cl}(n,\gamma)^{38}\text{Cl}$	1642.8	14.3 ± 0.71	2.50 ± 0.13	0.72 ± 0.03	0.11 ± 0.01
4	Fe	$^{58}\text{Fe}(n,\gamma)^{59}\text{Fe}$	1099.3	0.31 ± 0.01	0.43 ± 0.02	-----	-----
5	Rb	$^{85}\text{Rb}(n,\gamma)^{86}\text{Rb}$	1076.7	-----	0.00042 ± 0.00003	-----	-----
6	In	$^{113}\text{In}(n,\gamma)^{114\text{m}}\text{In}$	558.4	0.010 ± 0.001	0.0088 ± 0.0005	0.011 ± 0.001	0.010 ± 0.001
7	Sb	$^{123}\text{Sb}(n,\gamma)^{124}\text{Sb}$	602.7	0.0050 ± 0.001	0.0045 ± 0.0002	0.0055 ± 0.0003	0.0056 ± 0.0003
8	Ir	$^{191}\text{Ir}(n,\gamma)^{192}\text{Ir}$	612.5	0.00026 ± 0.0001	0.00044 ± 0.00003	0.00074 ± 0.00004	0.00039 \pm 0.00002

RESULTS AND DISCUSSION

Table 4. summarizes the data obtained in this work using both techniques (DGNAA and PGNA) for the four pharmacological samples. Also, it gives a

comparison between these concentration percentage values and the data obtained by ICP and EDX-Ray techniques for the 12 elements in the same samples.

Table 3. The concentration percentage values by weight for some elements identified in four samples under investigation by PGNAA.

Serial number	Element	Nuclear reaction	Value of element concentration %			
			Cix	Sie	Dit	Saf
1	Na	$^{23}\text{Na}(n,\gamma)^{24}\text{Na}$	0.29 ± 0.01	-----	-----	0.23 ± 0.01
2	Mg	$^{24}\text{Mg}(n,\gamma)^{25}\text{Mg}$	-----	-----	1.52 ± 0.05	0.51 ± 0.03
3	Al	$^{27}\text{Al}(n,\gamma)^{28}\text{Al}$	1.20 ± 0.03	-----	0.60 ± 0.03	1.90 ± 0.05
4	Si	$^{28}\text{Si}(n,\gamma)^{29}\text{Si}$	3.05 ± 0.09	0.12 ± 0.01	8.10 ± 0.24	2.30 ± 0.06
5	S	$^{32}\text{S}(n,\gamma)^{33}\text{S}$	0.20 ± 0.01	-----	0.51 ± 0.02	0.70 ± 0.03
6	Cl	$^{35}\text{Cl}(n,\gamma)^{36}\text{Cl}$	15.10 ± 0.45	2.00 ± 0.06	0.70 ± 0.03	0.09 ± 0.01
7	Ti	$^{48}\text{Ti}(n,\gamma)^{49}\text{Ti}$	15.06 ± 0.451	-----	-----	12.30 ± 0.37
8	Co	$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$	0.000301 ± 0.00018	0.000201 ± 0.00012	0.000261 ± 0.00015	0.00018 ± 0.00001
9	Zn	$^{64}\text{Zn}(n,\gamma)^{65}\text{Zn}$	0.00346 ± 0.0002	0.000801 ± 0.000048	0.000801 ± 0.000048	0.005752 ± 0.000345
10	Rb	$^{85}\text{Rb}(n,\gamma)^{86}\text{Rb}$	0.000335 ± 0.00002	0.000406 ± 0.000024	0.000400 ± 0.00002	0.000400 ± 0.00002
11	Sr	$^{88}\text{Sr}(n,\gamma)^{89}\text{Sr}$	0.000320 ± 0.000019	0.000301 ± 0.000018	0.000298 ± 0.00001	0.000209 ± 0.000012
12	Pb	$^{207}\text{Pb}(n,\gamma)^{208}\text{Pb}$	0.000203 ± 0.000012	0.00018 ± 0.00001	0.000250 ± 0.00001	0.000179 ± 0.000011

One can notice the good agreement in many of elemental concentration values, and in the mean-time, the high values of Cl in Cix sample and Al in Saf sample while it is low in other samples comparing with the other techniques. Also, in case of Si which has a relatively high concentration in Dit sample in comparison with the other samples. The Ti element is presented in Cix and Saf samples and not appeared in Sie and Dit samples. The Co, Zn, Sr and Rb elements were presented in all samples in a trace level comparing with other elements. The agreements and the other some discrepancies, may

be due to the high accuracy of the applied techniques taking into consideration the percentage error in each.

By all means, some of these elements are very important from the point of industrial view, and other may have some toxic influences depending on the dose rate recommend by the specialists.

Table 4. A comparison between the concentration values obtained in this work and the other techniques for the same samples.

S. No.	Element	Cix Sample			Sie Sample		
		Present Work		EDX-Ray & ICP-MS	Present Work		EDX-Ray & ICP-MS
		DGNAA	PGNAA		DGNAA	PGNAA	
1	Na	0.25 ± 0.01	0.29 ± 0.01	0.33 EDX	0.27 ± 0.01	-----	-----
2	Mg	-----	-----	-----	-----	-----	-----
3	Al	-----	1.20 ± 0.03	1.05 EDX	0.14 ± 0.01	-----	-----
4	Si	-----	3.05 ± 0.09	0.30 EDX	-----	0.12 ± 0.01	0.06 EDX
5	S	-----	0.20 ± 0.01	0.16 EDX	-----	-----	-----
6	Cl	14.3 ± 0.7	15.1 ± 0.45	14.16 EDX	2.50 ± 0.12	2.0 ± 0.1	1.2 EDX
7	Ti	-----	15.06 ± 0.45	14.6 EDX	-----	-----	-----
8	Co	-----	0.00030 ± 0.0001	0.0002 ICP	-----	0.00020 ± 0.00001	0.00017 ICP
9	Zn	-----	0.00346 ± 0.0002	0.0034 ICP	-----	0.00080 ± 0.00004	0.00075 EDX
10	Rb	-----	0.00034 ± 0.0001	0.00037 ICP	0.00042 ± 0.0001	0.00041 ± 0.00002	0.00038 ICP
11	Sr	-----	0.00032 ± 0.0001	0.00029 ICP	-----	0.00301 ± 0.00002	0.00040 ICP
12	Pb	-----	0.00020 ± 0.0001	0.000151 ICP	-----	0.00018 ± 0.00002	0.00014 ICP

Table 4. Continued

S. No.	Element	Dit Sample			Saf Sample		
		Present Work		EDX-Ray & ICP-MS	Present Work		EDX-Ray & ICP-MS
		DGNAA	PGNAA		DGNAA	PGNAA	
1	Na	0.087 ± 0.004	-----	-----	0.21 ± 0.0105	0.23 ± 0.0115	0.29 EDX
2	Mg	-----	1.52 ± 0.04	1.46 EDX	-----	0.51 ± 0.03	0.41 EDX
3	Al	-----	0.60 ± 0.03	0.38 EDX	-----	1.90 ± 0.06	1.52 EDX
4	Si	-----	8.10 ± 0.24	4.6 EDX	-----	2.30 ± 0.07	2.43 EDX
5	S	-----	0.51 ± 0.02	0.37 EDX	-----	0.70 ± 0.04	0.74 EDX
6	Cl	0.72 ± 0.04	0.70 ± 0.04	0.47 EDX	0.11 ± 0.0055	0.09 ± 0.01	0.10 EDX
7	Ti	-----	15.06 ± 0.45	14.6 EDX	-----	-----	-----
8	Co	-----	0.00030 ± 0.0002	0.0002 ICP	-----	0.00020 ± 0.00001	0.00017 ICP
9	Zn	-----	0.00346 ± 0.0002	0.0034 ICP	-----	0.00080 ± 0.00004	0.00075 EDX
10	Rb	-----	0.00034 ± 0.0002	0.00037 ICP	0.00042 ± 0.00003	0.00041 ± 0.00002	0.00038 ICP
11	Sr	-----	0.00032 ± 0.0002	0.00029 ICP	-----	0.00301 ± 0.00002	0.00040 ICP
12	Pb	-----	0.00020 ± 0.00001	0.000151 ICP	-----	0.00018 ± 0.00001	0.00014 ICP

CONCLUSIONS

On view of the obtained results in this work, one can point out the following: -

- 1- The success of the [DGNAA and PGNAA] techniques using the isotopic neutron sources for elemental analysis of such complex samples as pharmacological samples is very clear.
- 2- Using the HPGe detection systems with some special software programs can facilitate the complete analysis in short time with high accuracy.

- 3- The comparative study between the obtained data and other one using different sophisticated techniques gives a real evidence for our results.
- 4- More efficient and reliable isotopic neutron sources and more advanced software programs can give more accurate results with the scope of DGNAA and PGNAA for other elements.

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التحليل العنصري لبعض المواد الدوائية المستخدمة في المملكة العربية السعودية باستخدام تقانة جاما أسر النيوترونات

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^٢تقسم طبيعة المفاعلات، شعبة المفاعلات الذرية، مركز البحوث النووية، هيئة الطاقة الذرية، مصر

تم في هذا البحث إجراء الفحص العنصري لأربعة من المواد الدوائية المستخدمة في المملكة العربية السعودية، وقد تم ذلك باستخدام الطيف الجامي الفوري والطيف الجامي الأجل الناتج عن أسر النيوترونات الحرارية في نوي هذه المواد.

إستخدم لذلك فيض النيوترونات المنطلق من مصادر النيوترونات، (^{252}Cf)، ($^{226}\text{Ra/Be}$). كما أستخدم نظام الكشف الجامي والذي يحوي كواشف من الجرمانيوم بالغ النقاوة والإلكترونيات المكملة له وإستخدمت النظم المعدة لهذا الغرض بمعامل جده. وقد تم تعيين نسب تركيز غالبية العناصر المكونة لهذه المواد ذات الوفرة العالية ومتوسطة الوفرة وكذلك العناصر شحيحة التواجد، وقورنت النتائج التي حصلنا عليها بتقانات أخرى لنفس العينات.