STUDIES OF ELEMENTAL ANALYSIS AND NATURAL RADIOACTIVITY OF SOME COMMONLY-USED FERTILIZERS IN SAUDI ARABIA

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الخلاصــة :

تم دراسة التحليل العنصري لبعض عينات من السماد المنتج محلياً بطريقة التحليل باستخدام التنشيط النيوتروني البطيء وطريقة القياس الطيفي لأشعة جاما الطبيعية. وقد تم تبطئة النيوترونات (طاقتها ٢,٥ مليون الكترون فولت) المنتجة عن طريق تفاعل عنصر الدتريوم مع عنصر دتريوم آخر في المسارع الأيوني لجامعة الملك فهد للبترول والمعادن، وقد تَمَّ الحصول على فيض نيوتروني بطيء قدره ٢,٥٠٠,٠٠٠ نيوترون / سم٢ / الثانية. وقيس النشاط الإشعاعي المنتج بالحَثِّ النيوتروني في عينات السماد بواسطة كاشف عالي التميز والمصنوع من الجرمنيون العالي النقاوة ومحلل معلومات مرتبط بحاسوب شخصي. وقد أُنشئتْ منحنيات المعايرة لجميع العناصر الموجودة في عينات السماد وذلك بتشعيع عينات عيارية مماثلة لتركيبة السماد التي تُصنّعها هيئات عالمية مثل المنظمة العالمية للطاقة الذرية والمعهد العالمي للمعايرة والتقنية. وباستخدام منحنيات المعايرة تم تقدير تراكيز عناصر بوتاسيوم ومنجنيز وفانديوم والمنيوم وصوديوم وكلور. وتراوح تركيز عنصر البوتاسيوم مابين صفر إلى ١٦,٥٪ وزن مئوي. وتركيز عنصر المنجنيز مابين ٢٧ إلى ٥٧ جزيء من المليون. وتركيز عنصر الفانديوم مابين ٩٣ إلى ٣٢٠ جزيء من المليون. وتركيز عنصر الألنيوم مابين ١٤٨١ إلى ٢٥٤٧ جزيء من المليون. وتركيز عنصر الصوديوم مابين ٧٩٧ إلى ٣٥٠٧ جزيء من المليون. وتركيز عنصر الكلور مابين صفر إلى ١٧٠٠ جزيء من المليون. وقد تَمَّ قياس العناصر المشعة طبيعياً وهي البوتاسيوم –٤٠ واليورانيوم-٢٣٨ والثوريوم-٢٣٢ باستخدام كاشف عالي الكفاءَة من نوع صوديوم أيوداين. وقد أظهرت نتائج الطريقتين توافقاً في تركيز عنصر البوتاسيوم. وقد تم حساب كمية الرديوم المكافئة في عينات السماد وتراوحت مابين ٢٦ إلى ٤٢٠ بيكريل / كجم.

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ABSTRACT

A selected number of commonly used local fertilizer samples were studied for elemental analysis using slow neutron activation analysis and natural gamma ray spectroscopy. A slow neutron flux of about 2.5×10^6 n/cm²-s was available, which was obtained by slowing down 2.5 MeV neutrons from the $D(d,n)^3He$ reaction at the KFUPM 350 keV Ion Accelerator. The induced activities were measured by a HP-GMX detector and a PC-based data acquisition and analysis system. Calibration curves were established for the elements present in the fertilizer samples by activating certified standards of similar matrix from NIST and IAEA. From the calibration curves, the absolute concentrations of the elements K, Mn, V, Al, Na, and Cl were determined. The K concentration varied from 0 to 16.5 wt.%. The Mn concentration was between 27–57 ppm; V concentration between 93–320 ppm; Al concentration between 1481–2547 ppm, Na concentration between 797–3507 ppm, and the Cl concentration between 0–1700 ppm. The amounts of natural gamma rays from ^{40}K, ^{238}U, and ^{232}Th were determined by measuring their natural radioactivities using a 5″×5″ NaI(Tl) detector. The results of K from both methods were found to be similar. Radium equivalent natural radioactivities of the fertilizer samples were calculated to be in the range from 26 to 420 Bq/kg.

STUDIES OF ELEMENTAL ANALYSIS AND NATURAL RADIOACTIVITY OF SOME COMMONLY-USED FERTILIZERS IN SAUDI ARABIA

1. INTRODUCTION

Fertilizers are widely used in agriculture. Studies of elemental concentrations in fertilizers are important for a better understanding of the effect of each element on the end products of the fertilizer [1, 2]. To satisfy the strong need for fertilizers, a large fertilizer industry has been established in the Kingdom of Saudi Arabia, including companies such as Ibn Al-Baytar Fertilizer Company [3]. In order to support the local industry, it is interesting as well as useful to study the elemental concentrations of the local fertilizer samples. This will not only bring more information to the field, but will also help in the quality and possibly process control of the fertilizers [1–4].

One of the best nondestructive bulk analysis methods is Neutron Activation Analysis (NAA). The wide use of this technique in various areas in the last 30 years proves that the technique is very well suited for elemental analysis [5]. In this paper, a study of some commonly-used local fertilizer samples using the NAA technique is described, and the results of the absolute concentrations of the elements K, Mn, V, Al, Na, and Cl are presented. The results for K are compared with those from natural gamma ray measurements [6, 7]. Furthermore, the natural radioactivities of the fertilizers are also calculated.

2. EXPERIMENTAL METHOD

Six samples of commonly used compound fertilizers produced in Saudi Arabia by Ibn Al-Baytar National Chemical Fertilizer Company were collected. The samples were in the form of granular substances of 1–4 mm size with 1–2 kg crush strength. They were ammonia-based compound fertilizers with various grades of NPK (Nitrogen, Phosphorus, and Potassium). Table 1 shows the list of the samples used in the present experiment with the values of total nitrogen (NT), P_2O_5, and K_2O in weight percentages (wt.%). The samples were uniform admixtures of Di-Ammonium Phosphate, Mono-Ammonium Phosphate, Potassium Sulfate, and Urea [3].

Slow Neutron Activation Analysis

A slow neutron flux of about 2.5×10^6 n/cm²-s was obtained by slowing down the 2.5 MeV neutrons from the $D(d,n)^3He$ reaction at the KFUPM 350 keV Ion Accelerator. Deuterons of 200 keV and 1 mA bombarded a solid deuterium-on-scandium target with a 0.50 mm copper backing target. The fast neutrons were moderated by a cylindrical piece of polyethylene 25 cm in diameter and 30 cm in length. In order to keep the same geometry, each sample was placed in a plastic container of 3.5″ diameter and 0.5″ thickness. Each sample was then placed in the middle of the cylinder at 5 cm from the target, where the thermal neutron flux was maximum [8]. For determination of short-lived radioisotopes such as V and Al (with half-lives less than 5 min, see Table 2), the irradiation, delay, and counting sequence for each sample was 20 min/1.5 min/ 20 min respectively. For K, Mn, Na, Cl, and Gd determinations, that sequence was 2 h/1.5 min/2 h. Each sample was hand-carried and placed at 1 cm distance in front of the detector. The induced activities were measured by the counting station consisting of a HP-GMX detector, a 672 spectroscopy amplifier, a 919 multichannel buffer (all from EG&G Ortec), and a PC-based data acquisition and analysis system. The resolution of the detector was 1.95 keV at 1333 keV of ^{60}Co and its relative efficiency was 23%. The MCA, with 4096 channels, was calibrated using ^{22}Na and ^{60}Co γ-sources. The counting dead time of the system was less than 1%. The experimental setup is described in detail elsewhere [7–10].

Table 1. The List of the Fertilizer Samples Used in the Present Experiments [3].

Sample No.	Description (in wt.%)			Mass (g)
	TN	P_2O_5	K_2O	
1	14	38	10	78.815
2	18	46	0	71.841
3	12	27	18	76.939
4	12	35	8	42.970
5	18	18	5	42.126
6	28	28	0	36.530

Calibration curves were established for the elements present in the fertilizer samples by activating certified standards of similar matrix from NIST (USA) and IAEA (Vienna). The standards used in the experiments and the corresponding concentrations for the elements studied are summarized in Table 3. The neutron flux during irradiations was monitored using a 5"×2" NE213 liquid scintillation detector and a multichannel scaling unit (MCS) with a dwell time of 2 s for short and 5 s for long irradiations. The neutron flux monitor detector was placed at 227 cm from the target at about 90° with respect to the beam direction.

We estimated the thermal neutron flux attenuation due to neutron absorption by Gd which has the highest neutron absorption cross section. Our estimate was made for the highest Gd concentration present in our standard which was 12 ppm, as shown in Table 3. It is also important to notice that Gd is present in only two standards with 5 and 12 ppm concentration. We used the following standard formula to estimate the flux attenuation:

$$\phi(d) = \phi_o\, e^{-C\, d\, \sigma}, \tag{1}$$

where $\phi(d)$ is the neutron flux at the distance d in the standard, ϕ_o is the flux at the front end of the standard, C is the concentration of Gd nuclei/cm^3 in the standard, and σ is the neutron capture cross section for Gd. For 12 ppm concentration of Gd in one standard with a volume of 79 cm^3, we find $C = 1.4 \times 10^{16}$ Gd-nuclei/cm^3. The Gd neutron absorption cross section is $\sigma = 39\,100$ barns [16]. From these values, we calculated the flux attenuation at the back end of the standard (0.5 inch thick) to be:

$$\phi\ (d{=}1.25\ \text{cm})\ /\ \phi_o = 99.9\%.$$

Table 2. Nuclear Data Used in the Present Experiments for the Fertilizer Samples [12].

Element	Reaction	Half-life	Cross section (barns)	$E\gamma$ (keV)	γ-Intensity (%)
K	^{41}K$(n,\gamma)^{42}$K	12.36 h	1.45	1524.6	18.8
Mn	^{55}Mn$(n,\gamma)^{56}$Mn	2.58 h	13.2	846.8	98.9
V	^{51}V$(n,\gamma)^{52}$V	3.75 m	4.79	1434.1	100.0
Al	^{27}Al$(n,\gamma)^{28}$Al	2.24 m	0.23	1779.0	100.0
Na	^{23}Na$(n,\gamma)^{24}$Na	14.96 h	0.51	1368.6	100.0
Cl	^{37}Cl$(n,\gamma)^{38}$Cl	37.24 m	0.42	1642.7	31.0
Gd	^{160}Gd$(n,\gamma)^{161}$Gd	3.66 m	1.51	315	22.7
Dy	^{164}Dy$(n,\gamma)^{165}$Dy	2.33 h	2725.0	94.7	3.58

Table 3. The Standards Used in the Experiments With the Certified Concentrations of the Elements Concerned.

Standard code & name	Mass (g)	K wt.%	Mn ppm	V ppm	Al wt.%	Na wt.%	Cl wt.%	Gd ppm
NIST SRM-278 Obsidian rock	69.787	3.454	403		7.489	3.591		(5.3)*
NIST SRM-694 Western Phosphate Rock	90.041	0.423	90	1737	0.953	0.638		
NIST SRM-1633a Coal Fly Ash	73.464	1.88	179	297	14.3	0.17		
IAEA SL-1 Sediment lake	24.491	(1.5)	3400	170	(8.9)	0.172		(12)
IAEA A-11 Milk powder	6.665	1.72				0.442	0.908	

* The values in the brackets are for information only.

We estimate that the flux attenuation due to Mn, V, and Dy are all less than 0.1% because of their low concentration and/or lower neutron absorption cross section. We may therefore conclude that the errors due to flux attenuation are within our statistical uncertainties.

The total integrated and background subtracted counts were normalized to the integrated neutron flux measured.

Natural Gamma Ray Spectroscopy

The amount of natural gamma rays from ^{40}K, ^{238}U, and ^{232}Th was determined by measuring their natural radioactivities using a 5"×5" NaI(Tl) detector and a PC-based data acquisition and analysis system. The setup was calibrated for the determination of K, U, and Th by measurements of 12 certified standards from NIST and IAEA. The calibration procedure is explained elsewhere [7]. The samples and standards were placed in a cylindrical plastic container of 3.5" diameter and 0.5" thickness and were sealed to prevent any escape of the radon gas (^{222}Rn) which has 3.8 days half-life. The samples were then kept for 3 weeks for ^{222}Rn to reach equilibrium in the ^{238}U decay series. Each fertilizer sample was then counted for one day to reach counting statistics around 1%. The absolute concentrations of K, U, and Th were then determined from a comparison of the samples with the standards. The radioactivity due to K, U, and Th as well as the total radioactivity was calculated from the results. Then, the radium equivalent activity of the samples was calculated using the following formula [11]:

$$Ra_{eq} (Bq/kg) = A_{226} + 1.43 \times A_{232} + 0.077 \times A_{40} \tag{2}$$

where A_{226} = activity of ^{226}Ra (or that of ^{214}Bi since the latter was in equilibrium with the former), A_{232} = activity of ^{232}Th, and A_{40} = activity of ^{40}K.

3. RESULTS AND DISCUSSION

A typical spectrum for a fertilizer sample is shown in Figures 1a and 1b. Figure 1a shows the induced delayed gamma lines for short irradiations of 20 min. The gamma lines of the short-lived radioisotopes ^{52}V and ^{28}Al as well as that of the medium-lived radioisotope ^{38}Cl are clearly visible. Figure 1b shows gamma lines for long irradiations of 4 hours where the ^{56}Mn, ^{24}Na, and ^{42}K lines are clearly visible. The nuclear data of the γ-ray considered for each element are shown in Table 2 [12], where the most prominent peak was selected for each element. It is interesting to mention that a low concentration of dysprosium and gadolinium was observed in the fertilizer samples from the presence of γ-lines at 94.7 keV of ^{165}Dy and 315 keV of ^{161}Gd, respectively.

The minimum detection limit (MDL) for each element was calculated using the following Equation [13, 14]:

$$MIDL = \frac{m}{NC} \times 3.3\sqrt{B} \tag{3}$$

where NC and B are the background-subtracted net counts for a sample of mass m and the background counts under the peak of interest, respectively.

Calibration Curves for Absolute Concentrations

A background run with an inactivated sample was acquired for the same counting period as each activated sample. These were especially important for subtraction of natural γ-ray background from the neutron induced activities. Then, the background-subtracted and flux normalized net counts were obtained for all the elements studied. The calibration curves were then established for the elements present in the fertilizer samples by activating certified standards of similar matrix from NIST and IAEA. For each element, a minimum of two standards was used. The integrated net counts from the induced activities were then normalized to 100 g mass for each sample. The content of each element *versus* number of counts was plotted and the data were fitted with the best least-square linear function. Figure 2 shows the calibration curves for vanadium and aluminum, which are short-lived radioisotopes, while calibration curves for manganese, sodium, and potassium are shown in Figure 3. The uncertainties were from counting statistics corresponding to one standard deviation, and varied as follows:

K: 3–7%; Mn: less than 1%; Na: 0.2–3%; V: 0.7–8%; Al: 2–6%; and Cl: 3%.

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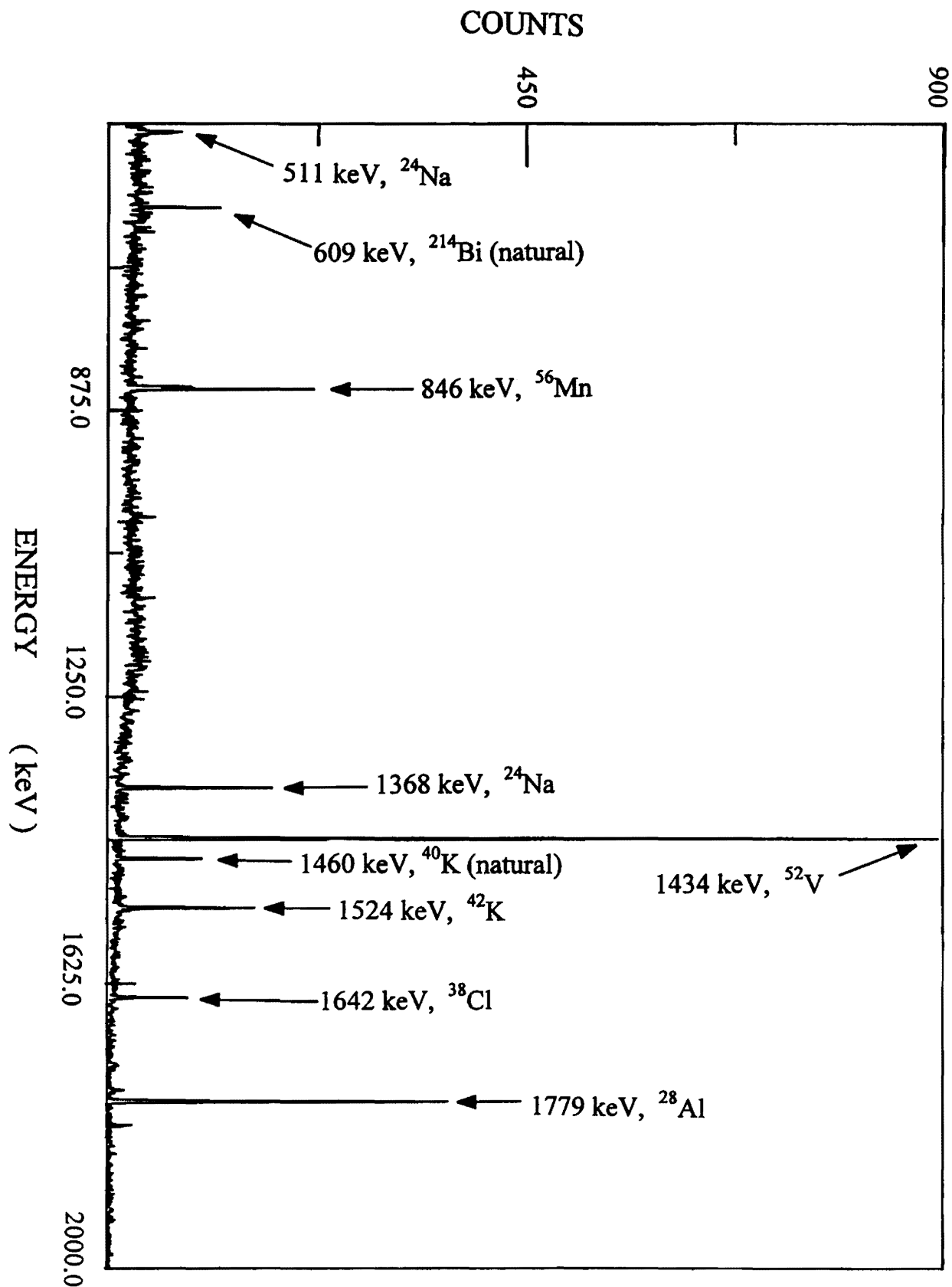
COUNTS

Figure 1a. A Sample Spectrum of the Induced Activities of the Short-Lived Elements in a Fertilizer Sample for Irradiation and Counting Periods of 20 m Each. All the emitted γ-lines of V, Cl, and Al have been well separated.
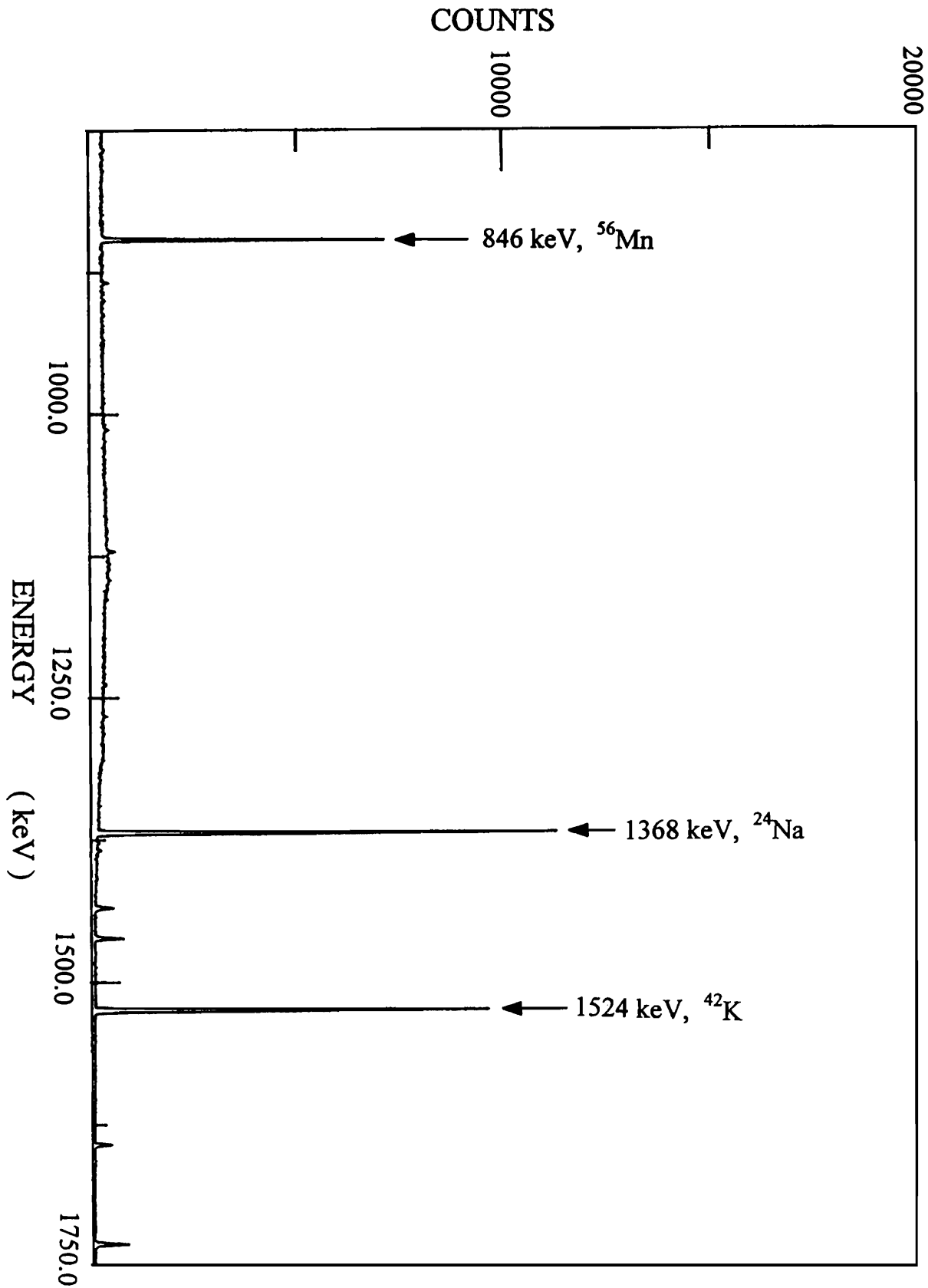
Figure 1b. A Sample Spectrum of the Induced Activities of the Long-Lived Elements in a Fertilizer Sample for Irradiation and Counting Periods of 4h Each. The γ-lines of Mn, Na, and K are clearly seen.

The MDL for K, Mn, V, Al, Na, and Cl were determined and are shown in Table 4. The absolute concentrations for K, Mn, V, Al, Na, and Cl were determined from the calibration curves. Although in Table 3 we show data for three certified standards for aluminum, only two of them were actually used for calibration, as shown in Figure 2. The reason that we did not use the data point of 14.3% was due to the high concentration of aluminum which resulted in a high Compton continuum in the spectra, and consequently increased the sample-related background. Moreover since the concentrations of Al in our samples are already low, using standards with low aluminum concentration is more adequate and results in a cleaner (less Compton continuum) spectrum.

As shown in Table 3, there was only one certified standard available for chlorine determination and, thus, for calibration of chlorine that data point was utilized. The concentrations of these elements in the fertilizers are shown in Table 4. The errors correspond to one standard deviation. The K concentration varied from 0 to 16.5 wt.% with an average value of 6.31%. The Mn concentration was between 27–57 ppm, with an average value of 39 ppm; V concentration was between 93–320 ppm, with an average of 196 ppm; Al concentration varied between 1481–2547 ppm, with an average of 1993 ppm; Na concentration was between 797–3507 ppm, with an average of 1765 ppm; and the Cl concentration was between 0–1700 ppm, with an average of 781 ppm. The mean values which are the average of the concentrations measured, are also shown in Table 4. One can see from the results that the concentration of the elements from large to small can be ranged as K, Al, Na, and Cl then V and, with lesser content, Mn. It should be mentioned that there is also an indication of low concentrations of Dy and Gd in the samples, but their determination in the present experiments is only qualitative. Of course there might be other elements which could not have been activated by our neutron flux.
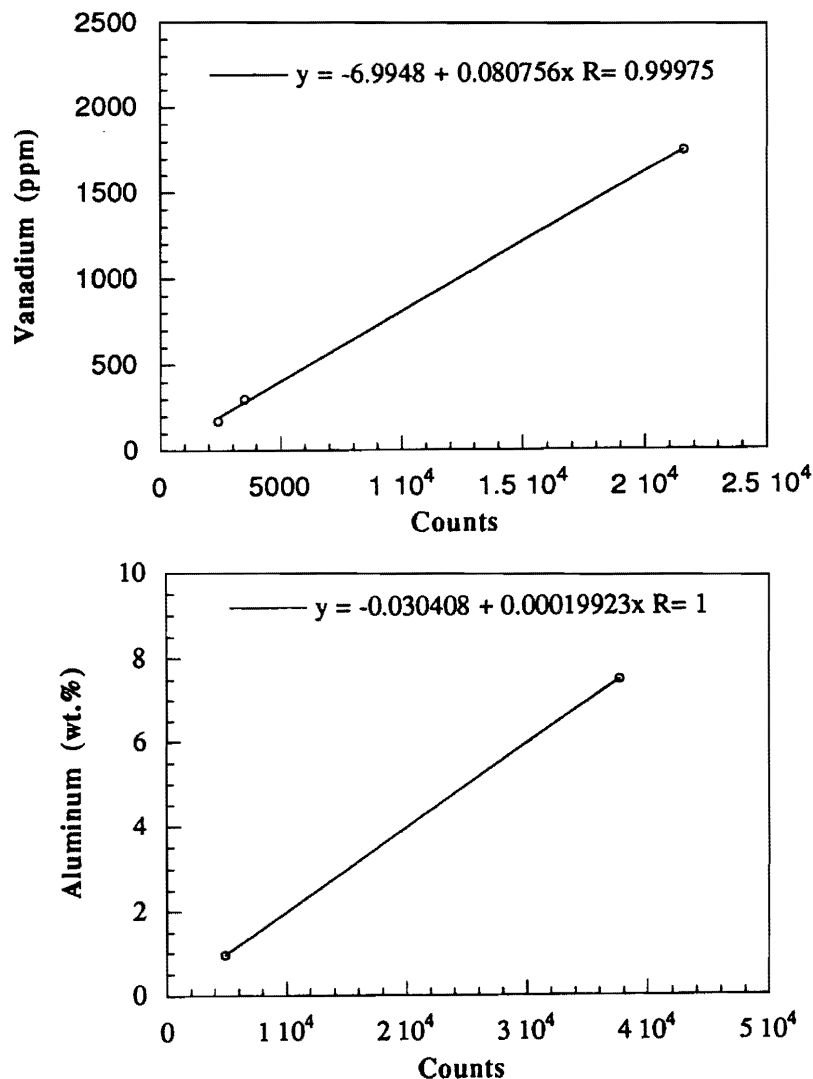

Figure 2. The Linear Calibration Lines for Determination of Vanadium (Top) and Aluminum (Bottom).
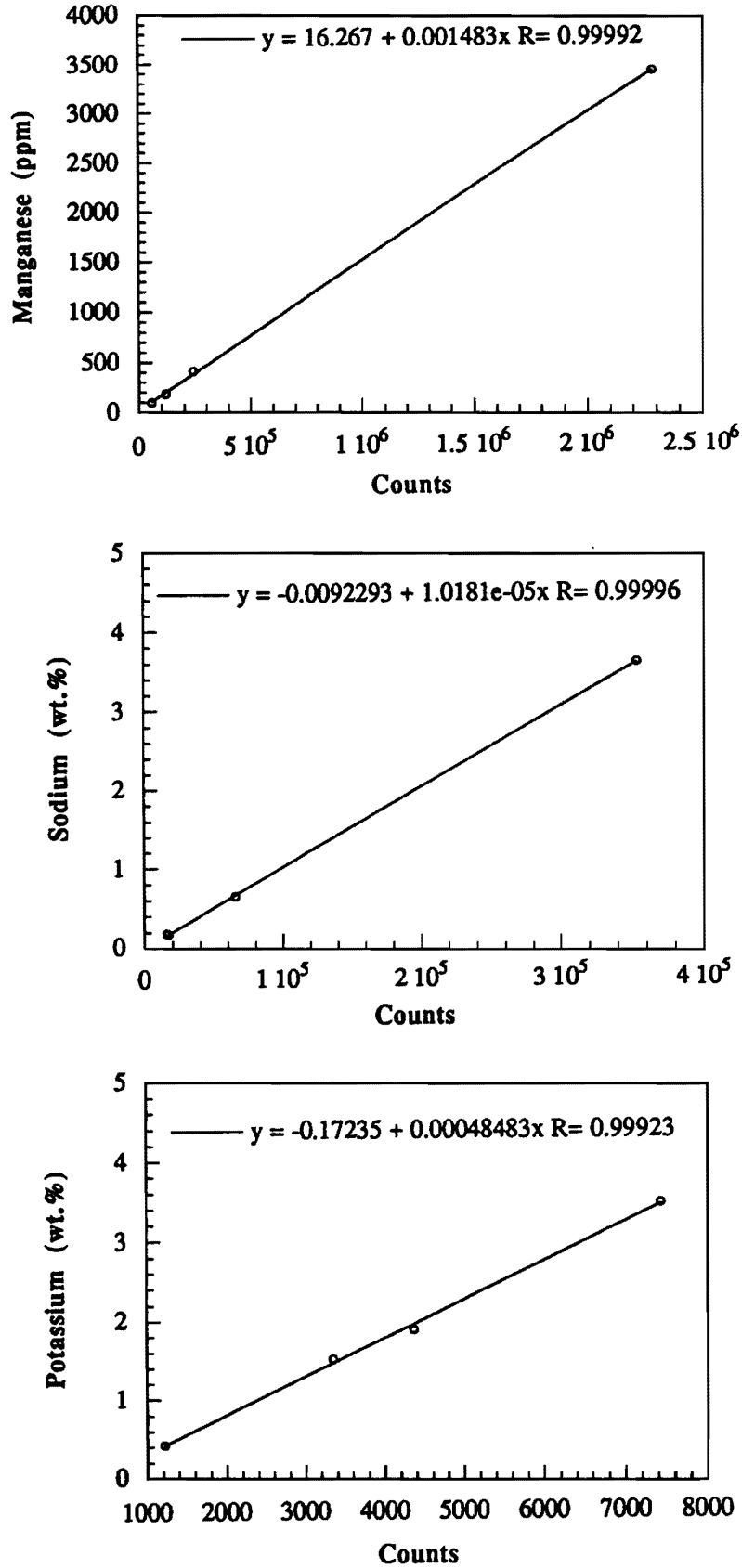
Figure 3. The Linear Calibration Lines for Determination of Manganese (Top), Sodium (Middle), and Potassium (Bottom).

Table 5. Results of the Concentrations of K, U, and Th as Well as Radioactivities in the Fertilizer Samples by Natural Gamma Ray Measurements.

Sample No.	K, Co.* (wt%)	K, exp** (wt%)	U (ppm)	Th (ppm)	K Activity (Bq/kg)	U Activity (Bq/kg)	Th Activity (Bq/kg)	Total Activity (Bq/kg)	Total Activity (nCi/kg)	Ra_{eq} (Bq/kg)
Fertilizer #1	8.30	8.33 ± 0.02	0.3 ± 0.04	2.4 ± 0.2	2912 ± 7	15 ± 2	5 ± 0.4	2932 ± 7	79	246.4
Fertilizer #2	0	0.14 ± 0.01	0.3 ± 0.04	1.3 ± 0.2	48 ± 3	18 ± 2	3 ± 0.4	69 ± 4	2	26.0
Fertilizer #3	14.94	14.58 ± 0.03	0.4 ± 0.04	2.0 ± 0.2	5099 ± 10	22 ± 2	4 ± 0.4	5125 ± 10	138	420.3
Fertilizer #4	6.64	6.77 ± 0.03	-	-	2368 ± 10	-	-	2368 ± 10	64	182.3
Fertilizer #5	4.15	5.26 ± 0.03	-	-	1840 ± 10	-	-	1840 ± 10	50	141.7
Fertilizer #6	0	-	-	-	-	-	-	-	-	-

* K Weight percentage values reported from Ibn Al-Baytar Company.
** K Experimental values from natural gamma ray measurements.

Table 4. Minimum Detectable Limits (MDL), and Absolute Concentrations of the Elements Present in the Fertilizer Samples.

Minimum Detectable Limit (MDL) and Sample	K wt.%	Mn ppm	V ppm	Al ppm	Na ppm	Cl ppm	Natural γ-rays Results K wt.%
MDL	0.074	1	8	142	21	22	0.015
Fertilizer #1	9.16 ± 0.04	40 ± 0.3	247 ± 5	2547 ± 79	2025 ± 8	964 ± 31	8.33 ± 0.02
Fertilizer #2	0.08 ± 0.01	38 ± 0.3	320 ± 6	1809 ± 67	797 ± 5	52 ± 17	0.14 ± 0.01
Fertilizer #3	16.51 ± 0.05	33 ± 0.4	138 ± 4	2091 ± 74	3507 ± 12	1700 ± 44	14.58 ± 0.03
Fertilizer #4	7.45 ± 0.10	57 ± 0.9	263 ± 7	2354 ± 108	1328 ± 20	-	6.77 ± 0.03
Fertilizer #5	4.64 ± 0.08	27 ± 0.8	93 ± 4	1673 ± 85	1565 ± 21	406 ± 30	5.26 ± 0.03
Fertilizer #6	-	36 ± 0.6	112 ± 5	1481 ± 82	1369 ± 22	-	-
Average value	6.31	39	196	1993	1765	781	5.85

* Absolute concentrations were calculated for a sample mass of 100 g.

The results of the natural γ-ray measurements for K, U, and Th are shown in Table 5. The concentration of U was less then 1 ppm while that of Th was between 1 and 2.5 ppm, which implies that U and Th are at the background level, while the results for the concentration of K were varying between 0–14.6% with an average value of 5.9%. As can be seen, the results for K from activation (column 1) and natural γ-ray spectroscopy (last column in Table 4) agree with each other within 10%. It is also interesting to have the results of natural gamma ray activities in terms of Bq/kg and nCi/kg and these are shown in Table 5. The results for the six fertilizers for radioactivity due to K varied from 0 to 5099 Bq/kg with an average value of 2453 Bq/kg. These results can be compared with 461 Bq/kg reported from Heaton and Lambley [6] which shows that K radioactivity in our fertilizers is about 5 times higher than in theirs. It should also be mentioned that the P_2O_5 value of our fertilizers (see Table 1), on average, was 32%, while theirs was 3%. On the other hand, the radium equivalent activities of our fertilizer samples range from 26 to 420 Bq/kg. Only fertilizer sample #3 had an activity of 420 Bq/kg, which exceeds 370 Bq/kg, the maximum permissible limit for radiation dose. Furthermore, in a recent study by Noorddin Ibrahim for fertilizer samples in Malaysia [15] they found ^{40}K activity ranging from 13.4 Bq/kg to 6003 Bq/kg; uranium activity from 0.4 Bq/kg to 112 Bq/kg, and thorium activity from 0.8 Bq/kg to 48 Bq/kg. These show that the K activity range of their samples was similar to ours, while their uranium and thorium activities were more than ours. The radium equivalent activity of their samples was ranging from 3 Bq/kg to 643 Bq/kg, which show that their maximum activity was about 50% greater than that in this work.

4. CONCLUSION

Elemental analysis of six commonly used local fertilizer samples from Saudi Arabia was performed using the non-destructive bulk analysis method of neutron activation analysis. The samples were irradiated by thermal neutrons obtained from the slowing-down accelerator based 2.5 MeV neutrons from the D(d,n) reaction and the delayed induced activities were measured using a HP-GMX detector connected to a PC-based data acquisition and analysis system. The natural radioactivities from ^{40}K, ^{238}U, and ^{232}Th of these fertilizer samples were also measured using a 5″×5″ NaI(Tl) detector.

The results showed measurable concentrations of K, Mn, V, Al, Na, and Cl elements in the fertilizer samples studied. An appropriate calibration technique was established for the determination of the respective ratios of each element present in the fertilizer samples using five standards from NIST and IAEA. The concentration of K varied between 0–16.5%, Mn between 27–57 ppm, V between 93–320 ppm, Al between 1481–2547 ppm, Na between 797–3507 ppm, and Cl between 0–1700 ppm. The errors correspond to one standard deviation. A low concentration of Dy and Gd was also observed.

The results of K content determined by the natural radioactivity measurement was in agreement with those from activation analysis. U and Th contents were at the background level. Radium equivalent activities of the samples ranged from 26 to 420 Bq/kg. One fertilizer sample had a radium equivalent activity above 370 Bq/kg, the maximum permissible level. More studies are recommended for the measurement of elemental concentrations in other fertilizers used in the Kingdom as well as for the possible effect that these elements and the presence of natural radioactivity might have on the soil and plants using these fertilizers.

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