DETERMINATION OF THE NITROGEN CONTENTS OF WHEAT GRAIN SAMPLES BY MEANS OF FAST NEUTRON ACTIVATION ANALYSIS

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ABSTRACT

The nitrogen contents of wheat grain samples were determined by the use of fast neutron activation analysis employing the reaction ^{14}N (n,2n) ^{13}N. Barley grain samples with known nitrogen contents were used as standards for the comparative activation analysis. As a result, the nitrogen amounts in fifteen varieties of wheat samples were found between, (2.69∓0.10) % and (1.53∓0.08) %.

ÖZET

Bu çalışmada, Türkiye topraklarında yetiştirilen bazı buğday türlerine ait çeşitli nümunelerdeki ozat miktarları, 14.7 MeV enerjili nötronların azotla etkileşmesiyle oluşan ^{14}N (n,2n) ^{13}N reaksiyonu yardımı ile tayin edilmiştir. Uygulanan karşılaştırmalı nötron aktivasyon metodunda içerisindeki azot miktarları bilinen arpa taneleri standart olarak kullanılmıştır. Onbeş tür buğdaya ait nümunelerde, çalışma sonucu elde edilen değerler % (2.69∓0.10) ve % (1.53∓0.08) arasında bulunmuştur.

INTRODUCTION

The nitrogen contents of grains contribute mainly to the amount of contained protein which is important from the standpoint of nutrition. Therefore the nitrogen contents of several varieties of wheat samples grown in Turkey were found of great importance for determination, since it has widely been used for breeding, feeding, baking, etc.

Aside from classical Kjeldahl technique, various nuclear methods have been applied for nitrogen determination in grain samples [1, 2, 3, 4]. The Kjeldahl method is precise but requires rather a long time. Neutron activation analysis was replaced with this method to diminish the risk of environmental pollution which occurs due to catalysts used, and to reduce the time for the complete analysis [4]. Furthermore, since the

alternative method is non-destructive the seed can be retained in viable condition [3].

Using the fast neutron activation analysis (FNAA) the nitrogen contents can be determined utilizing the reaction $^{14}N(n,2n)\,^{13}N$. Because of the low cross section value of this reaction for 14.7 MeV neutrons, large samples or high neutron flux is required for the optimization of the induced activities. The emitted positrons from ^{13}N radionuclide with a half-life of 10 minutes produce two annihilation photons. By using a NaI (Tl) detector and related electronics these radiations are detected and measured.

EXPERIMENT

The induced activities of the irradiated samples and standards were evaluated by determination of peak areas of 0.511 MeV gamma-rays and utilizing the gamma-gamma coincidence counting technique. Peak areas were calculated according to Covell method [7], and coincidence counting between two annihilation photons was adopted [8] in the case of gamma-gamma coincidence method.

The samples were contained in two kinds of containers. Three vials each containing 5-6 grams of sample and a fourth for the standard were used for the simultaneous irradiation using a geometrical arrangement similar to that of ref. [3]. The neutron flux was normalized by copper threads mounted axially in each vial. In the case of coincidence method, polyethylene bottles, each containing 30-35 grams of sample were used in front of the target for irradiation (Fig. 1). A BF_3 detector located in a paraffin block was used for flux monitoring.

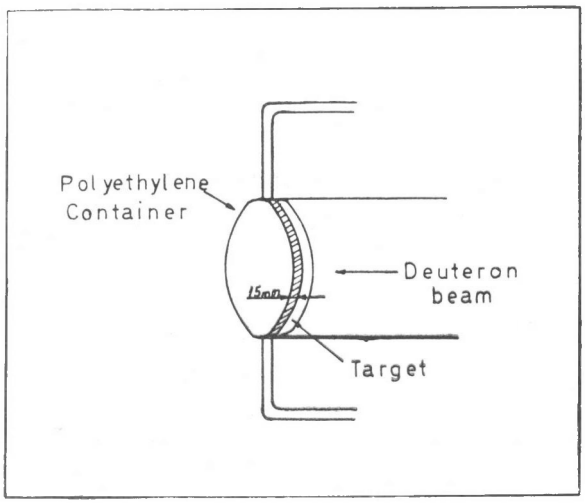


Fig. 1. Geometrical arrangement for irradiation of samples at coincidence counting technique.

The samples were irradiated for periods of 10 minutes with 14.7 MeV energy neutrons which produced in ^{3}H (d,n) ^{4}He reaction by 150 kV neutron generator. The average neutron flux was 1.28×10^{8} n.cm^{-2}. sec^{-1} close to the target. The neutron flux attenuation was found negligible. Moreover, since the sample and standard have the same bulk density, the effect of neutron self-shielding during irradiation and the absorption and scattering of 0.511 MeV annihilation photons during counting were also neglected.

After irradiation the samples were cooled in accordance with decay of the ^{30}P, ^{28}Al and ^{16}N radionuclides formed from the interfering reactions ^{31}P (n,2n) ^{30}P, ^{28}Si (n,p) ^{28}Al and ^{16}O(n,p) ^{16}N respectively, and suitable counting times were chosen taking the half-life of the produced ^{13}N radioisotope and the counting statistics as well, into consideration.

RESULTS AND DISCUSSION

Since the annihilation radiations have been measured and used for the quantitative determination, the accuracy of the resulting nitrogen amounts would certainly be influenced due to presence of impurities in the samples which also cause the positron annihilation, or emit gamma radiation with an half-life and/or energy around to that of annihilation photons of ^{13}N. The main interfering sources in wheat were observed and removed in a following way :

The nitrogen contamination due to the air of irradiated capsule was avoided by transferring the pressurized nitrogen gas through the drilled sample containers. Thus the effect of atmosphere nitrogen [2] was neglected considerably. On the other hand, since the location of the grains in the containers has remained same during the irradiation and counting position, the error which would be produced because of the sample being transferred to a new container has been eliminated.

The interference of potassium contents in samples arising from ^{38}K ($T_{1/2}=7.7$ min.) which is formed by the reaction ^{39}K(n,2n) ^{38}K was corrected by the use of a correction factor. This factor was determined as the ratio of 0.511 MeV peak area to 2.16 MeV peak area calculated from the gamma-ray spectrum of an irradiated K_2CO_3 sample (Fig. 2).

The recoil protons formed by the scattering of fast neutrons and by the reaction ^{12}C(n,p) ^{12}B on hydrogen and carbon nuclei of the sample, respectively, cause an apparent nitrogen content produced from the reactions ^{16}O(p,α) ^{13}N, ^{13}C(p,n) ^{13}N and ^{12}C(p,γ) ^{13}N [5, 6]. In order to eliminate this effect the following procedure was carried out : The container was irradiated as empty and filled with pure saccarose, $C_{12}H_{22}O_{11}$. The measured gamma-ray spectra are given in Figs. 3, 4. The 0.511 MeV peak

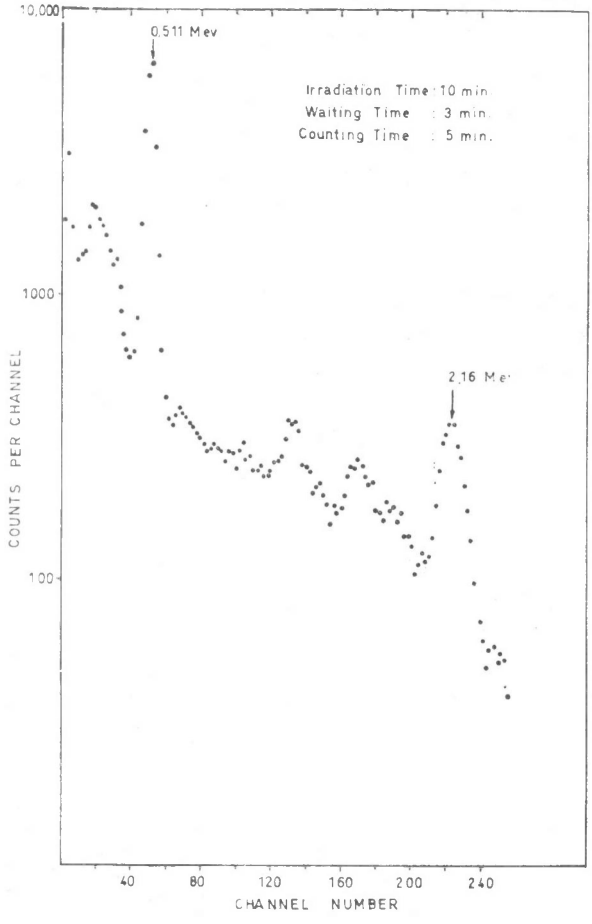


Fig. 2. Gamma-ray spectrum of K_2CO_3.

area for the empty container was assumed as background and subtracted from those of saccarose and sample to find the net peak areas. This last value for saccarose was multiplied with a coefficient calculated from the ratio of carbon, hydrogen and oxygen contents of the sample to those of saccarose, and then subtracted from the net peak area of each sample. Thus the contribution of unwanted ^{13}N activity was eliminated.

The interference of copper contents in samples was neglected due to very low ($\sim$25 ppm) copper concentration [2].

The above-mentioned corrections were made separately and the results about fifteen varieties of wheat grain samples with their standart deviations calculated from the repetitive measurements were given in Table—1 and Table—2. As it can be seen from the tables. the nitrogen contents change between the values of (2.69 ± 0.10) % and (1.53 ± 0.08)

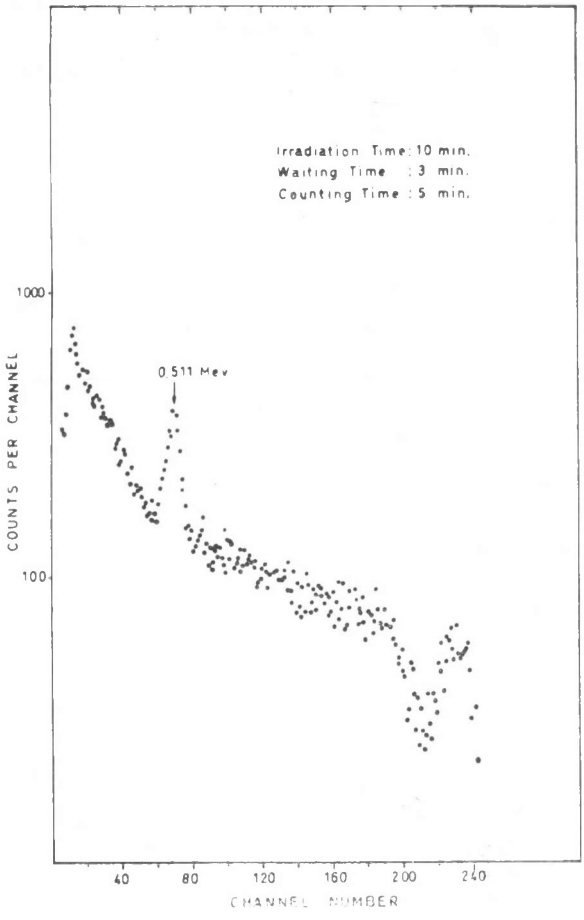


Fig. 3. Gamma-ray spectrum of irradiated empty container

TABLE—1. The percentage of nitrogen in turkish and foreign varieties of wheat grain samples determined by FNAA

Sample	Nitrogen (%)
Anadolu Beyazı	1.82 ± 0.07
Kızılca	1.89 ± 0.06
Topbaş	1.76 ± 0.05
Sert	1.93 ± 0.06
Makarnalık Sert	1.75 ± 0.05
Tır	1.60 ± 0.05
Sünter	2.05 ± 0.08
Penjoma	1.89 ± 0.06
Yektay	1.96 ± 0.06
Floransa	1.93 ± 0.06
Wanser	1.53 ± 0.08
Bezostaja	1.94 ± 0.08

% per sample weight. Certainly, these rather different values depend not only on the variety of the wheat but also on soil, fertilizer and climate conditions and the arrangement of the wheat grains on ear. Therefore,

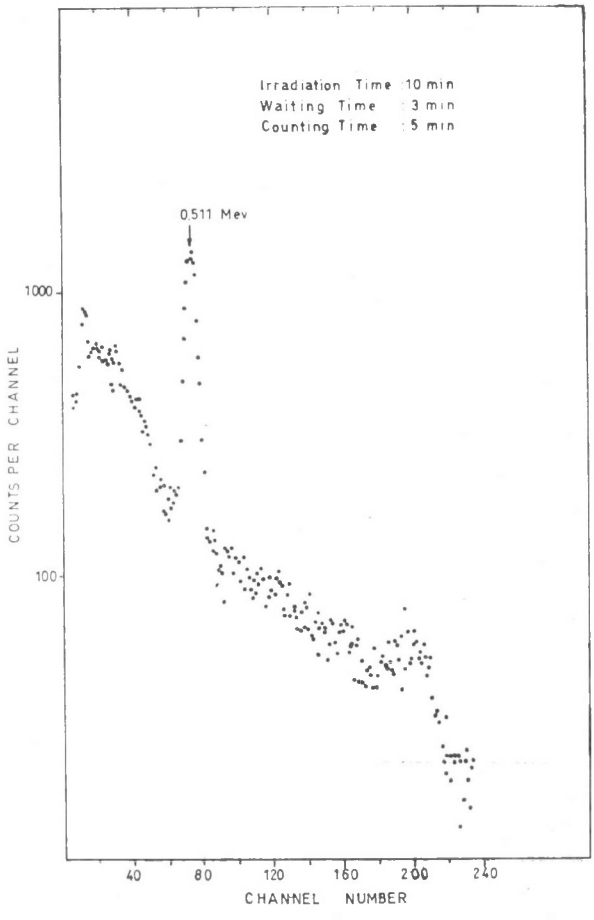


Fig. 4. Gamma-ray spectrum of saccarose.

the samples being analyzed quantitatively have to be selected with regard to these criteria. On the other hand a good agreement can be observed between the results obtained by FNAA and those obtained by the classical Kjeldahl method (Table—2).

TABLE—2. Comparison of nitrogen analysis in wheat grains by activation and by the Kjeldahl method

Sample	% N (activation analysis)	% N (Kjeldahl)	Deviation %
1	2.69∓0.10	2.688	+0.4
2	2.13∓0.08	2.037	+4.0
3	2.15∓0.06	2.107	+2.0

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