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(54) **Method for the simultaneous determination of nitrogen, phosphorus and potassium**

(57) A method for the simultaneous determination of the nitrogen, phosphorus and potassium contents of materials containing them, such as vegetable materials and fertilisers, comprises exposing samples to be analysed and standard samples to neutron irradiation, recording the spectra of gamma radiation induced in the samples, laying the samples aside for a period of time determined by the half-life of the gamma ray-emitting isotopes, again recording the spectra of the samples being analysed and those of the standard samples, superimposing the first and second spectra of the samples being analysed and standard samples and shifting them relative to each other along the energy axis, and determining the content of the elements being analysed by comparing the spectra of the samples being analysed with those of the standard samples.

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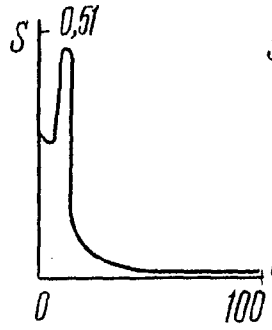

FIG. 1

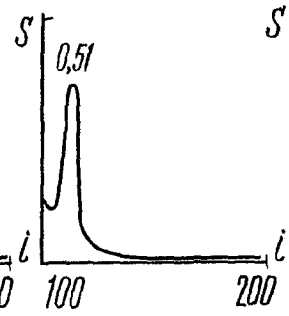

FIG. 2

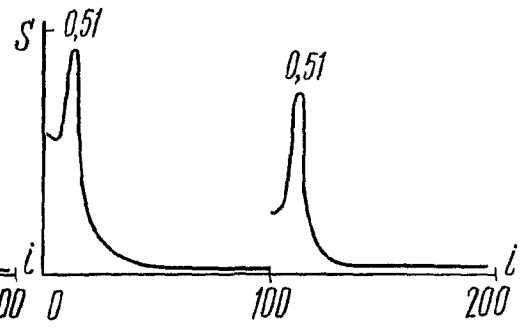

FIG. 3

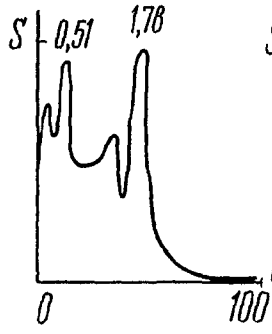

FIG. 4

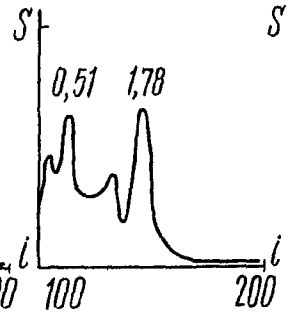

FIG. 5

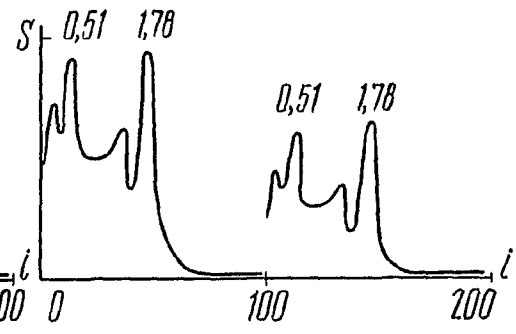

FIG. 6

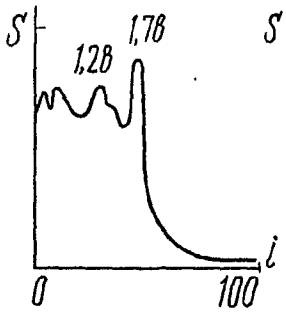

FIG. 7

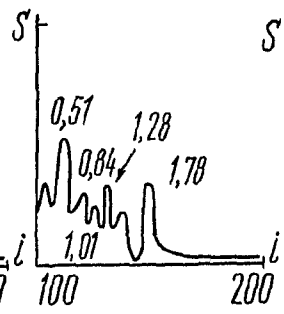

FIG. 8

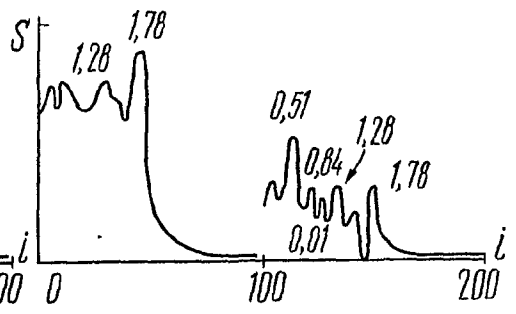

FIG. 9

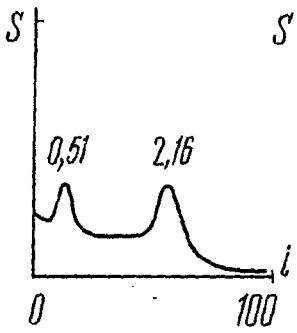

FIG. 10

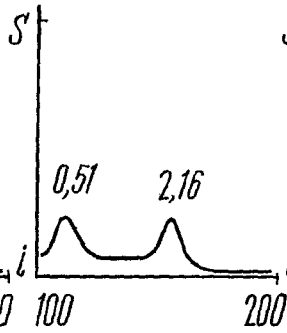

FIG. 11

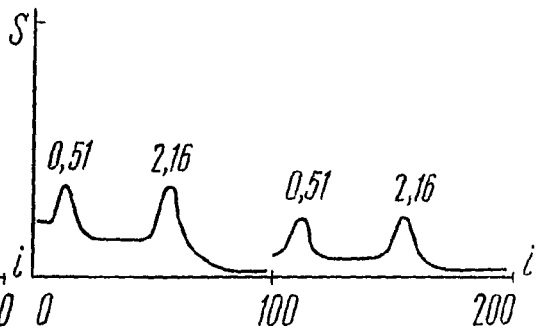

FIG. 12

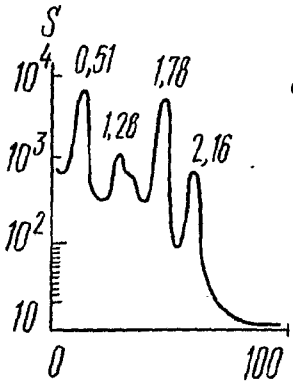

FIG. 13

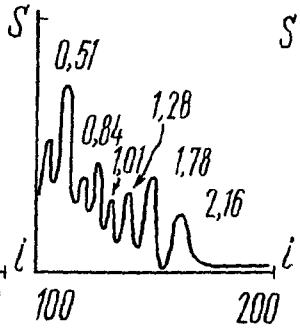

FIG. 14

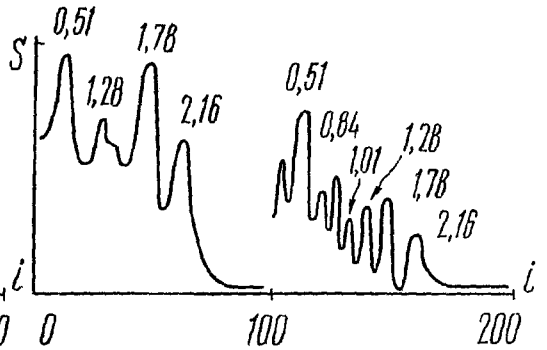

FIG. 15

SPECIFICATION

Method for the simultaneous determination of nitrogen, phosphorus and potassium

The present invention is concerned with the determination of the content of chemical elements in natural and artificial materials containing them and, more particularly, with a method for the simultaneous determination of the nitrogen, potassium and phosphorus content in vegetable materials and fertilizers.

According to the present invention, there is provided a method for the simultaneous determination of the nitrogen, phosphorus and potassium contents of materials containing them, which comprises exposing samples to be analysed and standard samples to neutron irradiation, recording the spectra of gamma radiation induced in the samples, laying the samples aside for a period of time determined by the half-life of the gamma ray-emitting isotopes, again recording the spectra of the samples being analysed and those of the standard samples, superimposing the first and second spectra of the samples being analysed and standard samples and shifting them relative to each other along the energy axis, and determining the content of the elements being analysed by comparing the spectra of the samples being analysed with those of the standard samples.

A preferred procedure for carrying out the method according to the invention will now be described with reference to the accompanying drawings, in which the Figures are portions of the gamma radiation spectra obtained from different samples, the curves being plots of the number of pulses recorded in a given channel, S, against the channel number proportional to the energy of gamma radiation, i.

Standard samples of materials containing nitrogen, phosphorus, potassium and silicon and samples to be analysed are exposed to a neutrom flux emitted by a neutron generator. After 1 to 5 minutes, i.e. after a period of time sufficiently long for decay to take place, the gamma radiation spectrum of the irradiated samples is recorded with spectrometric equipment.

The standard samples and those being analysed are then laid aside for a second period of 5 to 25 minutes and their spectra are then recorded again. The first and second spectra are superposed, the discontinuities of the first spectra are filled with portions of the second spectra of the respective samples, and the first and second spectra are shifted relative to each other along the energy axis. The information thus recorded is illustrated in the Figures of the accompanying drawings.

The nitrogen, phosphorus and potassium content is determined by comparing the spectra of the standard samples with those of the samples being analysed using any suitable conventional mathematical method, such as the least squares method.

The method according to the invention is fundamentally different from conventional methods serving the same purpose in that instead of measuring the actual gamma radiation spectrum, the object of the measurements is a composite spectrum produced by superposing two spectra taken after different periods of time. Apart from information as to the gamma radiation energy distribution, the spectra thus superposed also carry information as to the change of this distribution with time, which leads to a greater accuracy of the analysis.

The following table compares the results of analysing plant samples using a conventional method and the method according to the invention. The analysis covered 4 types of control plant samples, namely, oats, barley, maize and grass mixture. The N, P and K content of the samples was determined by chemical analysis.

Table

Type of Sample	Chemical Element	Result of Chemical Analysis, wt.%	Average Value, wt.%		Variation Coefficient of Single Measurement, % Relat.		Absolute difference from Results of Chemical Analysis %	
			Method According to Invention	Prior Art Method	Method According to Invention	Prior Art Method	Method According to Invention	Prior Art Method
1	2	3	4	5	6	7	8	9
Oats	N	2.02	2.02	2.18	1.5	3.0	0.0	+8.0
	P	0.43	0.46	0.30	8.1	13.0	+7.0	-3.02
	K	0.51	0.56	0.57	8.8	15.1	+9.8	+11.8
Grass Mixture	N	3.35-3.41	3.50	3.39	0.9	2.8	+2.6	0.0
	P	0.30-0.34	0.35	0.38	6.5	9.6	+2.9	+11.8
	K	2.10-2.30	2.26	2.60	4.3	6.9	0.0	+13.0
Barley	N	1.94	1.95	1.94	3.0	2.7	+0.5	0.0
	P	0.39	0.40	0.41	7.2	16.6	+2.5	+5.1
	K	0.43	0.45	0.48	6.0	12.6	+4.6	+11.6
Maize	N	1.62	1.64	1.64	3.2	2.7	+1.2	+1.2
	P	0.37	0.38	0.39	7.8	10.7	+2.7	+5.4
	K	0.38	0.35	0.24	6.9	11.3	-10.2	-36.8

It is clear from the foregoing table that the discrepancy between the results of chemical analysis and those obtained using the method according to the invention is not greater than 10%; the average discrepancy is 3% for N and 5 to 7% for P and K. The convergence of the results is also satisfactory, being 3% for K and 6 to 9% for P and K. On the contrary, the prior art method shows large deviations in determining P and K, which in a number of cases are as high as 11 to 30%; the variation coefficients are often as high as 10 to 15%, which is unacceptable.

In order that the invention may be more fully understood, the following examples are given by way of illustration only.

Example 1

Standard samples of materials containing nitrogen, phosphorus, silicon and potassium and samples of oats were placed in standard volume polyethylene capsules, exposed to a neutron flux of 14.5 Mev, and put aside for 1 to 2 minutes for decay to take place. A gamma radiation spectrum of each sample was recorded with a scintillation detector and a multi-channel pulse height analyser; the spectrum was taken in 100 channels of the analyser. The standard samples and those being analysed were then laid aside for a second period of 16 minutes and were then transferred to a second detector.

A scintillation gamma radiation spectrum was again recorded in 100 channels of the analyser and added to the first spectra of the respective standard samples and samples being analysed as channels 101 to 200.

Figs. 1 to 15 show the information thus obtained.

Fig. 1 shows the spectrum of the standard sample of nitrogen, recorded 2 minutes after the end of the irradiation, Fig. 2 shows the spectrum of the standard sample of nitrogen recorded 16 minutes after the end of the irradiation, and Fig. 3 shows a composite spectrum of the standard sample of nitrogen, produced by adding the first and second spectra together.

Figs. 4 to 15 show first and second spectra, and the composite spectra of the standard samples of the phosphorus (Figs. 4–6), silicon (Figs. 7–9) and potassium (Figs. 10–12) and those of the sample being analysed (Figs. 13–15).

In order to find the concentrations of nitrogen, phosphorus and potassium, the least squares method was used to divide the composite spectrum of the sample subjected to analysis into those of the standard samples.

The period of time between the end of irradiation and the moment the first spectrum was taken was 2 minutes. This period was sufficiently long for short-lived isotopes to decay (this applies, for example, to N^{16} of which $T^1/2 = 7.4$ sec) and was short enough for the activity of Al^{28} produced from phosphorus and silicon to be maintained at a reasonably high level ($T^1/2$ of Al^{28} is 2.3 min). The second period of time during which the samples were put aside was 16 minutes and was selected with due regard for the decay of the activity of Al^{28}.

20 weighed portions of oats were used in the determination of the nitrogen, phosphorus and potassium content. The content of these elements (%age by weight) were as follows: N, 2.02; P, 0.46; K, 0.56.

Example 2

Samples of grass mixture were analysed by the procedure described in Example 1.

The content of the three elements (% by weight) were as follows: N, 3.50; P, 0.35; K, 2.26.

Example 3

Samples of barley were analysed by the procedure described in Example 1.

The content of the three elements (%ages by weight) were as follows: N, 1.95; P, 0.40; K, 0.45.

Example 4

Samples of maize were analysed by the procedure described in Example 1.

The content of the three elements (%ages by weight) were as follows: N, 1.64; P, 0.38; K, 0.35.

CLAIMS

1. A method for the simultaneous determination of the nitrogen, phosphorus and potassium contents of materials containing them, which comprises exposing samples to be analysed and standard samples to neutron irradiation, recording the spectra of gamma radiation induced in the samples, laying the samples aside for a period of time determined by the half-life of the gamma ray-emitting isotopes, again recording the spectra of the samples being analysed and those of the standard samples, superimposing the first and second spectra of the samples being analysed and standard samples and shifting them relative to each other along the energy axis,

and determining the content of the elements being analysed by comparing the spectra of the samples being analysed with those of the standard samples.

2. A method for the simultaneous determination of the nitrogen, phosphorus and potassium contents of materials containing them, substantially as described in any of Examples 1 to 4.

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