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Preliminary report on estimating potassium content of soils and minerals

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EXPERIMENTAL METHOD AND RESULTS

Methods and Materials

The gravimetric determination of potassium by the perchlorate, platinochloride and cobaltinitrite methods has several disadvantages. However, the tetraphenylboron method, developed in the last few years, represents an important advance for this determination. The low solubility of potassium tetraphenylboron, its stability, favourable conversión factor and selectivity are the principal advantages of this method.

This method was introduced by Wittig and his collaborators in 1949. Potassium was determined by precipitation from an aqueous solution at 70°C, with lithium tetraphenylboron as precipitant.

To eliminate the adsorption of alkaline earths by the potassium presipitate, Raff and Brota in 1951 precipitated potassium from cold dilute acetic acid solution. Flaschka applied the method to micro amounts of potassium. Schoter and Fricker, and Spier introduced some modification to determine potassium in biological materials. Rudorf and Zannier recommended carrying out the precipitation at 70°C from an acetic acid solution of pH 5 to 6. Kohler introduced a major innovation by using a 0,1 N mineral acid solution and limiting the time of standing of the precipitate to make the method more selective. Geilmann and Gebauhr precipitated potassiun tetraphenylboron at pH 4 to 5 at a temperature between 30 to 40°C.

The basis of the method proposed by Belcher, Nutten and Thomas is the precipitation with sodium tetraphenylboron from the water extract obtained from a J. Lawrence Smith decomposition.

G. S. Cooper studied the conditions necessary for obtaining the best stability of sodium tetraphenylboron solutions.

Several volumetric methods have been used in connection with the precipitation of potassium tetraphenylboron: the conductimetric titration of G. Jander and Anke, the turbidimetric methods of A. Windaus, the alka-

limetric estimation in presence of mercuric chloride of R. Montequi, etc. E. D. Schall precipitated with an excess of sodium tetraphenylboron and determined the excess with a standard solution of quaternary ammonium salt in the presence of bromo-phenol blue as an indicator.

Belcher's method

This author recommended extracting potassium, after a Lawrence Smith decomposition with 100 ml. water and adding one or two drops of concentrated HCl. A 25 ml. aliquot of this solution was pipetted into a beaker and 0.2 ml. of conc. HCl and 10 ml. of 0.6 sodium tetraphenylboron solution were added; after stirring and allowing to stand for 10 or 15 mins. the precipitate was filtered and washed with 5 × 3 ml. portions of a saturated aqueous solution of potassium tetraphenylboron and dried at 120°C for 30 minutes.

This method was used to estimate potassium standard solution of KCl. The results are presented in Table 1.

TABLE 1

K taken (mg.)	K found (mg.)		% recovery (mean)
5	4.85	4.69	95.6
10	10.09	9.60	98.5
15	15.05	14.45	98.4

H. J. Cluley in 1955 criticised the above mentioned methods and established conditions under which a tractible precipitate can be obtained without risk of errors due to the decomposition of the precipitant. He recommended two methods at pH 2 and pH 6.5. The pH 2 method is as follows: To the potassium chloride solution of volume 80 ml. were added 10 ml. of 0.1 N hydrochloric acid. Then 8 ml. of the 3.4 % W/V sodium tetraphenylboron solution were added dropwise from a burette to the solution which was stirred for about 3 minutes during the addition of the reagent. After 30 minutes the precipitate was collected on a No. 4 sintered glass crucible, washed with 2 to 3 ml. portions of saturated potassium tetraphenylboron solution and then once with 1 to 2 ml. of water. The precipitate was dried for 1 hour at 120°C and weighed.

The results we have obtained by this method with a standard solution of KCl are presented in Table II.

TABLE II

K taken (mg.)	K found (mg.)		% recovery (mean)
5	4.94	4.95	98.6
10	9.82	9.98	98.6
15	14.79	14.80	99.5

When Cluley's method was used to estimate potassium in soils and minerals, incomplete extractions were obtained with 100 ml. of water; we recommend using 250 ml. If the material has a high potassium content it is absolutely necessary to increase the volume to obtain quantitative extractions. If the amount of potassium is very small it may be convenient to add 5 to 10 ml. of a standard solution of potassium chloride (see Table III).

TABLE III

Moscovite: % K_2O

Extract with 100 ml.		Extract with 250 ml.	
6.5	7.2	9.11	9.20

The following are the results obtained in Saxmundham soils, using Cluley's method with the modification indicated above:

TABLE IV

K content (*g.*/100 *g.*)

Saxmundham Soil No	HF + Flame photom			Lawr. Smith + Na TPB		
A 10117	1.27	1.27	1.25	1.17	1.12	1.20
A 10122	1.07	1.07	1.12	1.01	1.00	
A 10124	1.15	1.15		1.09	1.03	
A 10126	1.15	1.15		1.07	1.00	
A 10131	1.40	1.30		1.29	1.34	
A 10141	1.20	1.27		1.28	1.33	
A 10148	1.22	1.21		1.31	1.37	

The analysis of potassium in different clay's minerals: muscovite, Glauconite, Wyoming Bentonite and K-Bentonite (prepared by treatment of Wyoming Bentonite with KCl), are presented in Table V as follows:

TABLE V

Mineral	Mesh size	% g. of K		Mean
Muscovite	100	9.11	9.20	9.15
Glauconite	100	4.63	4.65	4.64
Bentonite (Wyoming)	100	0.15	0.17	0.16
K-Bentonite	100	2.49	2.58	2.53

Determination of Radioactivity

To measure the activity of these cray minerals and different mixtures of them we used a Geiger counter for liquid samples filled to a fixed volume with the samples of different densities and pecking. Sometimes quartz, which was nearly inert, was used to change the density; if the percentage of quartz in the mixture is high it is necessary to correct the total activity for that present in this mineral. There results are shown in Tables VI, VII, VIII.

TABLE VI

Muscovite + Quartz

Sample	c. p. m./gr. of mixt.	c. p. m./gr. of K	density
100 M	21.1	268.2	.28
80 + 20	17.3	288.0	.31
70 + 30	13.8	262.3	.36
60 + 40	11.7	255.9	.39
50 + 50	10.0	254.2	.50
40 + 60	7.7	248.6	.56
30 + 70	5.7	239.6	.61
20 + 80	3.8	230.5	.70
10 + 90	1.9	208.3	.87
100 Q	0.4	—	.98

TABLE VII

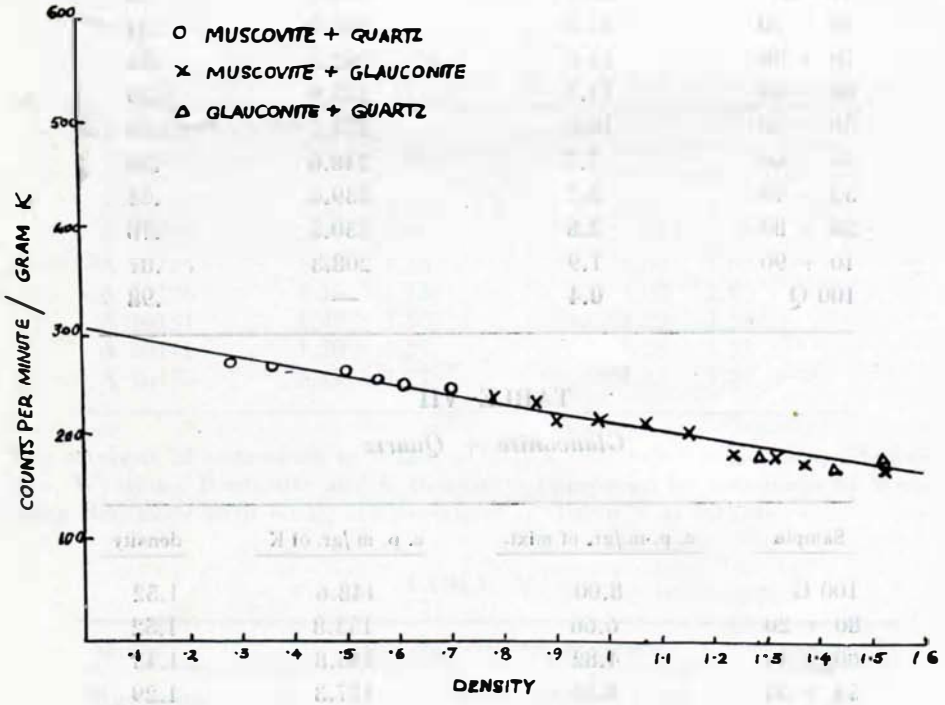
Glauconite + Quartz

Sample	c. p. m./gr. of mixt.	c. p. m./gr. of K	density
100 G	8.00	148.6	1.52
80 + 20	6.66	153.8	1.52
60 + 40	4.82	145.8	1.43
50 + 50	4.38	157.3	1.29
40 + 60	3.46	152.8	1.29
20 + 80	2.00	170.0	1.29
100 Q	0.23	—	1.29

TABLE VIII

Glauconite + Muscovite

Sample	c. p. m./gr. of mixt.	c. p. m./gr. of K	density
100 G	8.00	148.6	1.52
95 + 5	8.41	153.0	1.37
90 + 10	9.19	164.2	1.24
90 + 10	10.36	185.0	1.15
90 + 10	10.89	194.8	1.07
80 + 20	11.84	204.6	.98
80 + 20	12.10	200.3	.90
80 + 20	12.62	217.6	.86
80 + 20	12.82	225.2	.78



RESUMEN

En el presente trabajo se determina el contenido en potasio en varios minerales de la arcilla (moscovita, glauconita y bentonita — K) siguiendo el método de H. J. Cluley. A partir de esos valores se establece una relación entre el contenido en potasio y la radioactividad correspondiente al K-40 en función de la densidad. Esa relación nos ha permitido determinar radiométricamente el contenido en K en diversos suelos de Inglaterra (Trabajo en prensa J. RODRIGUEZ. G. E. G. Mattingly y O. Talibudeen. Rothamsted Experimental Statión, Harprenden. Inglaterra).

SUMARY

The potassium contents in clay minerals has been estimated by the Cluley's method. On stablish the relation between the % of K and the K-40 radio-activity for different densitys. This reation has been also used to determine the total amount of K in soils of England (JULIO RODRIGUEZ, G. E. G. Mattingly y O. Talibudeen; paper sended to Journal of Science of Food and Agriculture).

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