

the volumetric results. In this work, 2-gram samples were used, and the amount of organic matter being insufficient of itself to complete the combustion, 0.5 gram of flowers of sulphur was used with each charge.

TABLE II.—TOTAL CARBON IN SOILS.

Sample No.	Volumetric. Per cent.	Average. Per cent.	Gravimetric. Per cent.	Variation from gravimetric. Per cent.
59	{ 3.50 3.32 3.71 3.44 }	3.42	3.49	-0.05
119	{ 3.23 3.11 3.08 }	3.14	3.24	-0.10
121	{ 0.76 0.73 }	0.75	0.68	+0.07
473	{ 1.06 1.02 }	1.04	0.91	+0.13
474	{ 0.28 0.31 0.31 }	0.30	0.37	-0.06
475	{ 0.14 0.08 0.11 }	0.11	0.13	-0.02
630	{ 2.00 2.00 }	2.00	1.94	+0.06
661	{ 4.25 4.07 }	4.16	4.15	+0.01
662	{ 2.69 2.49 }	2.59	2.57	+0.02
690	{ 1.32 1.29 }	1.31	1.31	-0.00

A sample of oxalic acid gave 19.08 per cent. carbon against 19.04 per cent., the theoretical amount.

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[CONTRIBUTIONS FROM THE UNIVERSITY OF ILLINOIS AGRICULTURAL EXPERIMENT STATION, No. II.]

**THE DIRECT DETERMINATION OF POTASSIUM IN THE ASH OF PLANTS.**

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IN CONNECTION with a large amount of analytical work relating to soil investigations, it has been found necessary to have many determinations made of the elements nitrogen, phosphorus,

and potassium, in foodstuffs, in manure, etc. In making these determinations it was desirable to use short, direct methods, by which the elements required might be estimated without any long, intervening operations, in which elements which seemed to be unimportant in our work are determined.

In the preparation of the ash, on the reliability of which must depend the succeeding phosphorus and potassium determinations, all published methods were carefully considered. The quickest and most satisfactory method used, and a method giving very reliable results, was with the addition of ammonium nitrate to facilitate combustion.<sup>1</sup> The sample was moistened with a 20 per cent. solution of ammonium nitrate and dried on a hot plate. It was then incinerated in a muffle for about two hours at a very low heat. (The muffle had hardly a tinge of red.) The ammonium nitrate furnishes a sufficient amount of oxygen for the complete combustion of the carbon, and at a temperature not sufficiently high to endanger a loss of phosphorus or alkalies, but in some cases leaves nitrates instead of carbonates and chlorides, as was shown by Verdeil.<sup>1</sup> In this method it was found to be saving time and trouble to run three determinations for each sample, using a counterpoise dish and weighing out from 2 to 3 grams, depending upon the kind of sample. One portion is transferred to a weighing bottle, dried at 104°, and the nitrogen determined by the regular Kjeldahl method. The two other portions are transferred to unweighed crucibles incinerated and used for determinations of phosphorus and potassium, respectively.

The methods in use for the direct determinations of phosphorus and nitrogen are all that could be wished in speed and reliability. One man may easily weigh out and complete 40 nitrogen determinations in one day of eight hours, and weigh out, ignite, and complete twenty phosphorus determinations in a like length of time. In the direct determination of potassium, however, difficulties were encountered: First, too much time was consumed in making double alkali determinations which were not needed; second, errors occurred in the determination. In the official method for fertilizers (Lindo-Gladding), there is some danger of mechanical loss of potassium in the evaporation of sulphuric acid. In the official method for alkalies in ashes, the addition of ammonia and ammonium carbonate does not take out

<sup>1</sup> *Ann. Chem. u. Pharm.*, 69, 89.

all of the barium<sup>1</sup> even with a number of filtrations, and the method is long and tedious.

A method was worked out, however, by which a determination may be finished in one-half the time of the old method, with less danger of loss, giving satisfactory and more closely agreeing results. The procedure is as follows:

Prepare ash from a 2 or 3 gram sample by the above method. Transfer to a beaker with only a few drops of hydrochloric acid and heat to boiling. Add to the hot solution, barium hydroxide solution saturated at 32°, sufficient to precipitate interfering salts. From 3 to 5 cc. are usually enough, providing the hydrochloric acid has been used sparingly. Digest one hour, filter *hot*, and wash with hot water. Precipitate the barium as sulphate by a sodium sulphate solution made up of strength equivalent to the barium hydroxide solution used. Digest five hours, filter, and wash. Evaporate down to about 25 cc. in a platinum dish. Add a drop or two of hydrochloric acid and the calculated amount of chlorplatnic acid needed to convert all the alkalis into chlorplatnates. Proceed as in the Lindo-Gladding method for fertilizers. Transfer all the double salt to the filter, after the first addition of 10 cc. of the ammonium chloride solution. The interfering salts of magnesium and calcium will all be dissolved on the first application. After transferring, five washings of 5 cc. each are sufficient to clear the double salt from impurities; then wash with alcohol, dry, and wash through the filter with hot water, as usual.

This method gave entire satisfaction in speed and reliability. It should be noted that the success of the method depends, in a measure, upon keeping the quantity of reagents used as small as possible. The use of sodium sulphate does away with the use of ammonium salts,<sup>2</sup> and the error which is sometimes caused by driving them off. It also removes all traces of barium as the insoluble sulphate. Sometimes a slight amount of barium goes through the filter; this should not occur, if the liquid has been sufficiently digested; it will, however, be filtered out from the double salt when finally washed through into the platinum dish with hot water. Potassium chlorplatinate is not dissolved in ap-

<sup>1</sup> This Journal, 25, 490 (1903).

<sup>2</sup> When both sodium and potassium are to be determined, ammonium sulphate is used to complete the precipitation of barium (see this Journal, 25, 496 (1903)).

preciable amount by the ammonium chloride wash, as was determined by the A. O. A. C.,<sup>1</sup> and the double salt remaining after its use in all cases appeared perfectly pure when examined under a microscope. It may easily be seen in comparing this with the official method that at least two filtrations, and the time and annoyance of driving off ammonium salts are saved. It has also been ascertained that if ordinary care be taken in the laboratory, no ammonium salts will be absorbed.<sup>2</sup> The following table will show how duplicates agree by this method:

Air-dried sample.	Amount taken. Grains.	Per cent. K.	
		1.	2.
Oats .....	2	0.506	0.506
Corn .....	2	0.315	0.311
Gluten meal .....	2	0.245	0.232
Oil meal .....	2	1.233	1.220
Clover hay .....	3	1.463	1.452
Corn silage .....	3	0.809	0.798
Dung No. 1 .....	3	0.485	0.488
Dung No. 2 .....	3	0.510	0.515
Dung No. 3 .....	3	0.585	0.588
Dung No. 4 .....	3	0.477	0.473

## NOTE ON A METHOD OF DETERMINING SMALL QUANTITIES OF MERCURY.

BY THEODORE WILLIAM RICHARDS AND SIDNEY KENT SINGER.

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IN A recent investigation, Richards and Archibald<sup>3</sup> had occasion to detect and estimate small quantities of mercury in the presence of cadmium. The different solution-tensions of these metals at once suggested an electrolytic procedure, but it was found more convenient to conduct the separation with the help of a third metal than with the application of outside electromotive force. Among the many other metals which might have been chosen, copper was selected as the most suitable to precipitate the mercury; because, although its solution-potential is over 0.4 volt greater than that of mercury, it is nevertheless so hard to

<sup>1</sup> 4th An. Cou. Bull., 16, Div. of Chem., p. 44. Also 5th An. Cou. Bull., 19, Div. of Chem., p. 39.

<sup>2</sup> It should be noted that the ammonium double salt is soluble in the Lindo-Gladding wash.

<sup>3</sup> *Proc. Am. Acad.*, 38, 347 (1902); *Ztschr. phys. Chem.*, 40, 385 (1902) (?).