

XXV.—*A Quick Method for the Estimation of Phosphoric Acid in Fertilisers.*

By J. S. WELLS.

As I have had to make a great many analyses of fertilisers, and the ammonium molybdate method is so long and tedious, I was led to seek a process which would be more expeditious and at the same time give equally good results. The method to be generally applicable must be one in which the presence of iron and alumina does not interfere, as these substances are very often present in fertilisers. While examining the different volumetric methods, the idea occurred to me that Joule's citric acid process might be so modified as to be used gravimetrically; in this way I have obtained results corresponding very closely with those obtained by means of ammonium molybdate.

The modified process is as follows:—Two solutions are made up, one, which we will call "A," consisting of citric acid 900 grams, ammonia (sp. gr. 0.92) 1400 c.c., and water 500 c.c. The second, or solution "B," is simply a strong solution of magnesium citrate, prepared by dissolving carbonate of magnesia in citric acid.

*Total Phosphoric Acid.*—1 gram of the fertiliser is taken and fused with a mixture of 3—4 grams of sodic carbonate and 3—4 grams of sodic nitrate; the product is dissolved in nitric acid, and any insoluble residue removed by filtration. If the fusion has not been continued too long, any silica contained in the fertiliser will remain insoluble, and will be left in the residue.

To the filtrate, enough of the solution "A" is added, usually about 10 c.c., so that on making the mixture alkaline with ammonia no precipitate is formed, except possibly a slight one of silica.

It is now allowed to remain for about half an hour, and if any silica has separated it is filtered, and to the filtered solution suffi-

cient of solution "B" is added to precipitate the phosphoric acid; it is stirred well and allowed to stand for some time. The precipitate obtained is washed and weighed in the usual way. The result gives the total phosphoric acid present.

*Soluble Phosphoric Acid.*—In order to obtain the soluble phosphoric acid, another gram of the phosphate is taken and treated with water in the usual way.

To the solution thus obtained sufficient of "A" is added to prevent precipitation by the ammonia, which is now added in excess, and then enough of "B" to precipitate the phosphoric acid. The precipitate is washed and weighed as usual, and gives the amount of soluble phosphoric acid.

*Reverted Phosphoric Acid.*—The residue left after the extraction of the acid soluble in water is then treated with a solution of ammonium citrate in the usual manner to extract the reverted acid.

The solution thus obtained cannot be used to determine the amount of reverted phosphoric acid, as I found that the results obtained by precipitating the phosphoric acid contained in it are untrustworthy; sometimes the acid is completely precipitated, and at other times only partly. I think the reason of this is the very large quantity of ammonium citrate present in the solution.

Finding that the acid could not be readily determined in this solution, I have obtained it by difference, *i.e.*, the sum of the percentages of the soluble and insoluble acid is subtracted from the percentage of the total phosphoric acid, and the difference is of course the reverted acid.

*Insoluble Phosphoric Acid.*—The residue left after extracting the reverted acid now contains only the insoluble acid. It is fused with sodium carbonate and nitrate, and the phosphoric acid determined in exactly the same way as the total phosphoric acid was.

My first experiments were made on a solution containing a known amount of phosphoric acid. This solution was made up of sodium phosphate, calcium chloride, and ammonium iron alum, so as to contain  $P_2O_5$  19.83 per cent.,  $CaO$  25 per cent.,  $Fe_2O_3$  3 per cent. Equal portions of this solution were taken for each determination, and the results obtained were as follows:—

No.	Time.	Per cent. found.	Theory.
1.	2 hours	19.62 per cent.	19.83 per cent.
2.	"	19.77	" "
3.	18 "	19.84	" "
4.	"	19.84	" "
5.	"	19.74	" "
6.	"	20.06	" "

Numbers 1 and 2 show that the precipitation was nearly complete at the end of two hours. In No. 5 a solution of sodium silicate was added, then solution "A," and then ammonia to alkaline reaction. After standing one hour the separated silica was filtered off, and the phosphoric acid precipitated by solution "B." To No. 6 sodium silicate was added, and the phosphoric acid precipitated without previous removal of silica. After weighing, the precipitate was boiled with strong nitric acid, the insoluble residue of silica filtered and weighed, and the weight deducted from that of the first precipitate.

The results obtained being so satisfactory, I then tried the method on several fertilisers, with the following results:—

	Ammonium citrate method.		Ammonium molybdate method.
No. 1. ....	4.23 per cent.		4.23 per cent.
„ 2. ....	18.36 „		18.18 „
„ 3. ....	3.42 „		3.64 „
„ 4. ....	14.01 „		13.92 „

In making an analysis, care must be taken not to mistake a possible precipitate of magnesian phosphate for one of silica, when the alkaline solution is allowed to stand so as to separate silica. The difference in the appearance of the two will, however, enable one to easily distinguish between them. If silica and magnesia should happen to occur together in the same phosphate, it would be best to remove the former in the usual way by evaporating the acid solution to dryness.

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