ESTIMATION OF PHOSPHORIC ACID IN SOILS BY DOUBLE PRECIPITATION WITH MOLYBDIC SOLUTION AND TITRATION OF THE AMMONIUM PHOSPHOMOLYBDATE WITH STANDARD ALKALI.

BY C. B. WILLIAMS.

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THE accurate estimation of the small quantities of phosphoric acid usually present in the acid extract of soils is a question that has given no small amount of trouble in soil analysis. There seems to be two main difficulties. First, in the precipitation with molybdic solution in the presence of large quantities of iron and aluminum salts, compounds of these latter elements are also often precipitated with the ammonium phosphomolybdate, and are either dissolved by the ammonia wash or remain in the cone of the filter as phosphates, thus giving rise to high or low results, as the case may be, unless special precautions are taken. In the second place very small quantities of phosphoric acid are not precipitated readily by magnesium chloride mixture and usually require long standing to be complete, in which case the precipitate is very liable to contain an excess of magnesia, thus giving rise to higher results.

Some work in the laboratory of the North Carolina Experiment Station upon the samples sent out by Prof. A. M. Peter, reporter of the A. O. A. C. on soils, indicate that these difficulties are readily overcome by the following procedure: The hydrochloric acid extract of the soil is obtained[1] by digesting the soil in acid of 1.115 sp. gr. at the temperature of boiling water and under atmospheric pressure for ten hours. The organic acid (one per cent. citric acid and 0.63 per cent. oxalic acid solutions) extracts[2] are obtained by digestion at laboratory temperature for five hours. Care must be taken to destroy all organic matter in the hydrochloric acid extract as well as in the organic acid extracts. This is done in the former case by adding about one cc. concentrated nitric acid for every three cc. of the portion taken for analysis and evaporating to two or three cc. concentration, and in the latter cases by evaporating the extracts to dryness and igniting with the addition of a small quantity of

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[1] U. S. Dept. Agr. Div. Chem., Bul. 43, page 387.

[2] See report of A. O. A. C. Reporter on Soils for 1895.

nitric acid until organic matter is completely destroyed. These residues are now dissolved up to convenient volumes, and portions corresponding to eighteen, twenty or more grams of soil, and portions of the hydrochloric acid extract corresponding to one or more grams of soil (according to the richness in phosphoric acid) are precipitated, after adding about fifteen grams ammonium nitrate, at 40° C., with a large excess of molybdic solution, (thirty cc. is usually enough), let stand four hours, filter and wash with water twice. Now dissolve the precipitate into the beaker used for precipitation with dilute ammonia, wash the filter with dilute nitric acid and add concentrated nitric acid until precipitate begins to re-form; add ten grams ammonium nitrate, digest in water-bath at 65° C., add two cc. strong nitric acid with vigorous stirring, let stand five minutes, add two cc. molybdic solution, let stand eight minutes more and filter, wash and titrate according to volumetric method in this laboratory.[1]

The results presented in the table were obtained in the course of this investigation and show very close agreement with each other on the same solutions.

VOLUMETRIC DETERMINATION OF PHOSPHORIC ACID COMPARED WITH OFFICIAL GRAVIMETRIC METHOD.

		Hydrochloric acid solution.		Citric acid solution.		Oxalic acid solution.	
No. of sample.	Official gravimetric method.	First solution.	Second solution.	First solutiou.	Second solution.	First solution.	Second solution.
I	0.4505	0.4170	0.4069	0.0282	0.0290	....	0.0505
	....	0.4170	0.4069	0.0282	0.0287	....	0.0505
	....	0.4170	0.4069	0.0282	0.0287	....	0.0510
	....	0.4170	0.4069	0.0282	0.0287	....	0.0510
2	0.1720	0.1826	0.1826	0.0119	0.0146	....	0.0060
	....	0.1775	0.1826	0.0119	0.0146	....	0.0060
	....	0.1826	0.1928	0.0119	0.0146	....	0.0065
	....	0.1826	0.1928	0.0122	0.0152	....	0.0065
3	0.3956	0.3767	0.3867	0.0233	0.0255	....	0.0475
	....	0.3869	0.3867	0.0233	0.0255	....	0.0475
	....	0.3869	0.3867	0.0233	0.0255	....	0.0483
	....	0.3767	0.3818	0.0233	0.0261	....	0.0483
	....	0.3767	....	....	....	....	....
	....	0.3818	....	....	....	....	....
4	0.1932	0.1828	0.1929	0.0157	....	....	0.0087
	....	0.1878	0.1929	0.0157	....	....	0.0087
	....	0.1928	0.1929	0.0157	....	....	0.0087
	....	0.1878	0.1878	0.0162	....	....	0.0084

N. C. EXPERIMENT STATION, Raleigh, N. C

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[1] See description of volumetric methods in methods of the A. O. A. C. for 1895.