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## DETERMINATION OF POTASH AND PHOSPHORIC ACID IN FODDERS.

CONTRIBUTED BY H. W. WILEY.

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IN the comparative analyses of soils during the past three years we have grown a great number of pot cultures and determined the mineral plant foods in the resulting crops. The following modified potash method, devised by Mr. K. P. McElroy, while not sacrificing accuracy, has made it possible for one analyst to determine the potash, often in duplicate, in more than ten samples a day. Since the quantity of the crop harvested from a poor soil is often small, it is desirable that the phosphoric acid and potash be determined in the same sample.

The method in use for the determination of potash in feeding stuffs, in the laboratory of the United States Department of Agriculture is a simple modification of the ordinary Lindo-Gladding method, as prescribed by the Association of Official Agricultural Chemists. It is as follows:

Burn eight grams of the substance over a low flame to approximate whiteness. Burning after addition of sulphuric acid does not give more potash than burning alone, and it is more troublesome. Transfer the ash to a 200 cc. flask, using about fifty cc. of water, add five cc. of strong hydrochloric acid and place on the steam-bath for an hour, or boil from five to ten minutes. Add a little iron chloride to precipitate all phosphoric acid as fer-

ric phosphate, then ten cc. of strong ammonia and then from five to ten cc. of ammonium carbonate solution (200 grams per liter of the commercial salt). Replace on the steam-bath and heat for an hour, and allow to stand over night. Complete the volume to the 200 cc. mark with water and shake three times at intervals of five or ten minutes. Grease the inside of the neck of the flask and pour its contents on a dry folded filter. When all is transferred to the filter and run through, wash down the neck of the flask with a little water, put the funnel into the flask, and stand aside till the filter dries. Then roll up the filter and push down into the flask. Add dilute nitric acid, digest, make volume up to the mark, and use an aliquot part for the determination of phosphoric acid.

Transfer fifty cc. of the filtrate containing the potash, equivalent to two grams of material, to a platinum dish, cover and heat on the steam-bath till evolution of gas ceases. Remove the cover and rinse it and the sides of the dish with a stream from the wash bottle. Evaporate to dryness and heat in an air-bath till all water is removed in order to avoid loss by decrepitation in the subsequent ignition. Heat over a low gas flame till the bulk of the ammonium chloride is removed, cool and add one cc. of sulphuric acid (1 : 1), then heat on a hot plate till fuming begins, then over a flame till all the sulphuric acid is driven off and the residue in the dish is white. Every portion of the dish should reach a low but distinct red heat, the bottom first and then the sides. The reason for the preliminary driving off of the bulk of the ammonia as sal-ammoniac is that ammonium sulphate melts and sputters, involving danger of loss. Cool the dish and add one or two drops of strong hydrochloric acid, then from fifty to seventy-five cc. of water, washing down the sides of the dish with a jet from the wash bottle. Add platinum chloride solution in amount equivalent to 150 mm. of metallic platinum, for materials not containing over four per cent. of potash. Very few reach this limit. Evaporate on the water-bath as usual and take up with alcohol of eighty (volume) per cent. Filter through a Gooch crucible, keeping the insoluble material in the dish as far as possible. Wash with four more portions of alcohol, decanting through the crucible each time. Finally rinse down the sides of the crucible with a stream of alcohol from a

wash bottle. Cover the residue in the dish with the half-saturated solution of ammonium chloride prescribed in the official method for the determination of potash, and stir thoroughly. Decant through the Gooch crucible and treat with five or more portions of sal-ammoniac solution, decanting through the crucible each time. Finally wash into the crucible with eighty per cent. alcohol. When the transfer is complete, rinse the sides of the crucible thoroughly and finally fill it twice with alcohol, of course, constantly filtering with a vacuum. Dry for an hour at  $100^{\circ}$  and weigh.

Pour about 150 cc. of boiling water through the weighed Gooch crucible. If the platinum potassium chloride is not wholly dissolved, again bring the filtrate to a boil and pour through once more. Store this filtrate finally in a large flask, containing aluminum clippings, to reduce the platinum. Bring a fresh portion of water (150 cc.) to a boil and pour through the Gooch crucible. Remove the crucible from the vacuum apparatus, wipe, and dry in an air-bath, with good ventilation, for two hours, at  $110^{\circ}$ . Weigh once more. The loss in weight is the double chloride. The second portion of hot water is used to dissolve the double salt in the next crucible operated upon, after being once more brought to a boil.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF MICHIGAN.]

### PERIODIDES OF PYRIDINE.<sup>1</sup>

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*Pyridine Methyl Pentiodide*,  $C_5H_5N.CH_3.I_5$ .—This preparation has been reported,<sup>2</sup> but the present method of preparation is of interest. It was obtained in two experiments, when pyridine was added to an alcoholic solution of iodine containing methyl iodide. The precipitate of the pyridine tetriodide<sup>2</sup> was filtered off and the liquor set aside for partial evaporation. The crystals obtained were purified by recrystallizations from alcohol. Melting-point  $44.5^{\circ}$  C.

	Calculated for $C_5H_5N.CH_3.I_5$ .	Found.	
		1.	2.
Total iodine <sup>3</sup> .....	87.06	86.24	87.19
Titrated iodine.....	69.65	69.64	69.21

<sup>1</sup> Continuation of paper by Prescott and Trowbridge. This Journal, 17, 859.

<sup>2</sup> This Journal, 17, 859.

<sup>3</sup> This Journal, 17, 867, for methods of analysis.