CONCLUSIONS

I—The freezing-point figure of milk is the most constant one yet obtained and the safest basis upon which to draw conclusions as to the presence or absence of added water.

II—The freezing-point figures on the milks of sixteen individual cows examined show that the presence of 5 per cent added water can be detected in the majority of cases by this method and in any case place the sample in the suspicious class.

III—The presence of water added to fresh milk in excess of 5 per cent can be detected with certainty by the freezing-point measurement. The use of sufficient formaldehyde for preservation was found to lower the freezing point.

IV—It is essential that the test be applied only to reasonably fresh milk as the presence of acidity to the extent of 0.1 per cent beyond the normal for fresh milk (0.15 per cent) counteracts the amount of decrease in the freezing point depression produced by the presence of approximately 5 per cent of added water.

V—The method is practical in milk control work in that the test need be applied only to samples of doubtful character.

DAIRY LABORATORY AND LABORATORY OF FOOD CONTROL
BUREAU OF CHEMISTRY, WASHINGTON, D. C.

INSOLUBLE PHOSPHORIC ACID IN ORGANIC BASE GOODS

By E. O. THOMAS

Received June 2, 1917

The Official Method[1] for the determination of the citrate-insoluble phosphoric acid in fertilizers while giving all the available phosphoric acid in acid phosphate does not give all of the available in certain classes of organic materials. However, when this same material is batched into a complete fertilizer more available phosphoric acid (or what is the same thing, less insoluble phosphoric acid) is obtained, due to a relatively larger amount of citrate solution acting on the material. Particular reference is here made to base goods made by acidulating garbage tankage without any addition of phosphate rock. By the Official Method this material ranges from 0.95 per cent to 1.80 per cent insoluble phosphoric acid. That the Official Method does not give the true available phosphoric acid in this material is shown by the following experiments. A sample of this base goods containing 17.82 per cent moisture showed by the Official Method 1.56 per cent insoluble phosphoric acid; by increasing the amount of citrate solution used per 2.0 g. of base the results in Table I were obtained.

That the Official Method gives all of the available phosphoric acid in acid phosphate is shown by the tests (Table I) on two samples of acid phosphate.

A complete fertilizer made on the following formula using no phosphatic material but base goods (Table I) and acid phosphate No. 2 (Table I) gave the results given in Table I on insoluble phosphoric acid when deviating from the Official Method.

500 lbs. Base Goods (1.56% Insol. P_2O_5)
900 lbs. Acid Phosphate No. 2 (0.12% Insol. P_2O_5)
520 lbs. Kanona Nitrogene
80 lbs. Muriate of Potash

The calculated amount of insoluble phosphoric acid in the complete fertilizer mixture is 0.44 per cent, or 0.09 per cent more than that found. As the Official Method is shown to take care of all the available phosphoric acid in acid phosphate it is fair to assume that this gain of 0.09 per cent available is derived from the 500 lbs. of base goods, or a gain to the buyer of 0.36 unit of available phosphoric acid per ton. Leaving out of consideration the other materials in this mixture, of the 2.0 g. taken for analysis, 0.5 g. is base goods which is acted on by 100 cc. of citrate solution; *i. e.*, the ratio of 2.0 g. base goods to 400 cc. citrate solution. The insoluble phosphoric acid found in the base goods using 400 cc. citrate solution (Table I) was 1.05 per cent, a gain in available of 0.51 per cent over that found by the Official Method.

TABLE I—RESULTS ON 2-GRAM SAMPLES WITH VARYING AMOUNTS OF NEUTRAL AMMONIUM CITRATE SOLUTION, GRAVITY, 1.09
(100 Cc. Neutral Ammonium Citrate Solution Used in Official Method)

Cc. Neutral Ammonium Citrate Solution	Per cent Insoluble Phosphoric Acid (P_2O_5)			
	Base Goods	Acid Phosphates No. 1	Acid Phosphates No. 2	Complete Fertilizer
100	1.56	1.41	0.12	0.35
133	1.44	1.44	0.09	0.28
200	1.38	1.35	0.10	0.26
400	1.05	1.42	0.08	0.28
800	0.88	1.37	0.08	0.20
1200	0.80	....	....	....
1600	0.70	....	....	....

By following all details of the Official Method, except the using of 100 cc. of $N/10$ normal citric acid in place of the 100 cc. ammonium citrate as suggested by Rudnick,[1] this sample of base showed 1.78 per cent insoluble phosphoric acid. Using 100 cc. 2.0 per cent citric acid to replace the ammonium citrate this same base showed 1.15 per cent insoluble. This latter figure is comparable with that obtained when using 400 cc. citrate solution in the digestion, namely, 1.05 per cent, and is also comparable with the figures obtained when this garbage base is batched (*viz.*, in complete fertilizer, Table I) not to exceed 500 lbs. to the ton. As shown in Table I, there is a gain of 0.36 per cent available phosphoric acid per ton of base goods when this material is batched at the rate of 500 lbs. per ton of complete fertilizer and the Official Method is used. This gain of 0.36 per cent plus the 1.15 per cent found when using the 2.0 per cent. citric acid solution gives 1.51 per cent insoluble phosphoric acid, very close to the amount found by the Official Method, *viz.*, 1.56 per cent.

CONCLUSION

The Official Method for the determination of citrate-insoluble phosphoric acid gives the true value of acid phosphate, but some modification should be used for the analysis of materials of the character of acidulated garbage tankage.

814 PAUL-GALE-GREENWOOD BUILDING
NORFOLK, VIRGINIA

[1] *Jour. A. O. A. C.*, **1**, No. 4, Part 2, p. 4.

[1] THIS JOURNAL, **6** (1914), 486.