A NOTE ON THE FORMATION OF TRI-CALCIUM PHOSPHATE ON MIXING GROUND LIMESTONE WITH ACID PHOSPHATE

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About two years ago one of us, while visiting a fertilizer plant, was attracted by a large pile of ground limestone, which, upon inquiry, was found to be used as a filler in preparing lower grade acid phosphate from higher grade. It appeared somewhat strange that manufacturers should deliberately revert the water-soluble phosphoric acid, which they had been at so much pains and expense to produce. Besides, it seemed not improbable that there would be some insoluble phosphoric acid formed with a consequent danger of the manufacturer falling below his guarantee of available, if he took no account of this possibility. The chemist of the plant stated, however, that there was no such danger, as he had tried experiments and did not find any insoluble phosphoric acid formed.

In a recent discussion of the advisability of using ground limestone as a filler in fertilizers carried on in Atlanta, Ga., and published in "The American Fertilizer" during the past two years, the statement has been made that no insoluble phosphoric acid is produced on mixing ground limestone with acid phosphate.

In view of these statements, and because we had not been able to find any published experiments giving figures, it seemed worth while to make a carefully conducted set of experiments to determine whether any insoluble phosphoric acid is produced and, if so, whether the amount would be sufficient to endanger an available phosphoric acid guarantee. In the spring of 1913, therefore, we procured 100 lbs. each of acid phosphate and ground limestone, and after making a careful analysis of each, prepared mixtures as follows:

No.	1	2	3	4
Lbs. acid phosphate.....	14.0	15.5	17.0	10.0
Lbs. ground limestone.....	6.0	4.5	3.0	10.0

The materials were, after grinding separately, weighed as accurately as possible on a grocer's scale,

mixed thoroughly on paper and finally in wooden boxes; the contents of the boxes were then run through a small grinder, and sacked. Samples were drawn from the sacks with an Indiana fertilizer sampler, such as is used by our Fertilizer Inspectors in taking samples. The 4- or 5-pound sample thus drawn was quartered down to about 8 oz. and bottled for analysis.

On making the mixtures a considerable rise of temperature was noted, though it was not measured. Evolution of carbon dioxide took place immediately and continued for many days, as was shown by connecting a bottle of one of the mixtures with a bottle containing lime-water. This experiment was not carried to completion, owing to an unavoidable accident. The mixtures were analyzed at set intervals as shown below.

ACID PHOSPHATE	Per cent	GROUND LIMESTONE	Per cent
Total phosphoric acid.....	16.84	Lime.....	43.62
Water-soluble phosphoric acid.....	13.55	Phosphoric acid	Trace
Insoluble phosphoric acid.....	1.33		

PERCENTAGE ANALYSES OF MIXTURES

Mixture	No. 1 W-S Ins.	No. 2 W-S Ins.	No. 3 W-S Ins.	No. 4 W-S Ins.
April 21st, immediately after mixing.....	6.15 1.07	8.30 1.15	10.50 1.24	2.40 0.83
May 10th.....	3.05 1.30	5.84 1.26	8.66 1.37	0.99 0.98
June 12th.....	3.31 1.39	6.39 1.66	8.15 1.59	1.10 1.11
Sept. 13th.....	3.38 1.46	3.31(a) 1.33	8.05 1.46	0.87 1.08

(a) This is probably an error as an analysis in November, 1913, gave about five per cent water-soluble.

All figures are the mean of closely agreeing duplicates. These results seem to warrant the following conclusions:

I—Tri-calcium phosphate is produced on mixing acid phosphate and ground limestone.

II—The formation of the tri-calcium phosphate begins immediately on mixing, but increases slightly on standing, showing that the amount formed is a function of both time and temperature.

III—Considering the closeness with which manufacturers guarantee today, it appears necessary that the formation of insoluble in such mixtures be taken account of in making guarantees.

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LABORATORY AND PLANT

APPARATUS FOR THE DETERMINATION OF SULFUR IN GAS¹

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The sulfur apparatus which is shown in the accompanying illustration is the same in principle as the well-known Drehschmidt apparatus and its many modifications, the gas being burned and the oxides of sulfur collected by passing the products of combustion through an alkaline absorbing solution. The novel features of the apparatus illustrated are the burner and the combustion chamber.

The burner is similar to one described in a technological paper of this Bureau.² The method of igniting the gas and the arrangement of the secondary air in-

lets, however, are different. The platinum spiral which was heated to incandescence in order to ignite the gas has been replaced in the new type of burner with a pair of platinum terminals forming a spark gap. The inconvenience caused by the occasional burning out of the platinum wire is thus avoided. The platinum terminals are sealed to the porcelain burner tube by means of "sealing in" glass. Only a short length of platinum wire is necessary, nickel leads being soldered to the platinum terminals, just below the point where the latter are sealed to the porcelain tube. The stopper which closes the lower end of the combustion chamber also serves as a connector, the porcelain burner tube and the glass T piece being firmly fastened into it by means of Khotinsky or sealing wax. The small tip through which the gas enters just above the primary air inlet is also

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² "Determination of Sulfur in Illuminating Gas," McBride and Weaver, *Tech. Paper No. 20*, Bureau of Standards.