

fertilizer. This product was dried to 0.2% moisture prior to storage. Conditioning with limestone or calcined dolomite, although not tested, probably would have eliminated bag rot.

Microscopic and petrographic studies of several of the products formulated with calcium metaphosphate as the sole source of phosphate showed that, during storage, the granules develop dense surface coverings of monoammonium phosphate crystals in the form of radiating needles. Where caking occurred, it was due to intergrowth of these crystals between granules. The curing period prior to bagging apparently provided time for development of these surface crystals and thereby decreased the amount of intergrowth that occurred in subsequent storage.

### Conclusions

The process described in this paper is a feasible method of utilizing calcium

metaphosphate in the production of a wide variety of high-analysis granular fertilizers. The water solubility of the calcium metaphosphate was increased to a range of 20 to 40% and unusually tough granules were produced. Efficient granulation was obtained at relatively low moisture content. The low moisture input and the water utilized in the hydrolysis of the calcium metaphosphate resulted in unusually dry products, which had superior storage properties. The process can be carried out in facilities now installed in many fertilizer plants without major changes in equipment.

The disadvantage of the process is the relatively large amount of acid required to fix free ammonia. As presently developed, the process does not permit ammoniation of the hydrolyzate to a degree which exceeds the ammonia-retaining capacity of the acid used in the hydrolysis. Further studies designed to increase the ammonia-retention ca-

capacity of the hydrolyzate are in progress.

### Acknowledgment

M. M. Norton assisted in carrying out the pilot-plant tests and George Hoffmeister, Jr., supervised testing of the physical properties of the products. The authors are indebted also to T. P. Hignett, Chief of the Development Branch, whose advice helped direct the course of the work. Most of the analytical work was done by T. C. Woodis and D. R. Miller.

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Table IV. Results of Bag-Storage Tests

Grade	Run No. <sup>b</sup>	Days Cured <sup>c</sup>	Moisture Content, % H <sub>2</sub> O	Condition after 3 Months			
				Unconditioned		Conditioned <sup>a</sup>	
				Bag set	Lumps <sup>d</sup>	Bag set	Lumps <sup>d</sup>
Products with Calcium Metaphosphate as Sole Source of Phosphate							
13-13-13	AM-4/25	0	0.2	Hard	15	Hard	0
		7	...	Hard	0	Light	0
9-18-18	AM-2/9 <sup>e</sup>	0	0.9	Hard	22	Hard	7
		1	...	Hard	0	Light	0
		7	...	Medium	0	None	0
11-22-11	AM-5/18	0	0.3	...	...	...	...
		7	...	Medium	0	Light	0
Products with Calcium Metaphosphate and Ordinary Superphosphate as Source of Phosphate							
12-12-12	AM-4/27	0	1.1	Hard	4	Medium	0
		7	...	Medium	0	Light	0
5-20-20	AM-3/9-5	0	1.1	Hard	0	Light	0
		7	...	Hard	1	None	0
6-24-12	AM-4/4	0	2.2	Hard	12	Hard	2
		7	...	Medium	0	Light	0

<sup>a</sup> 9-18-18 product was conditioned with 5% kaolin; all others were conditioned with 2.5% kaolin.

<sup>b</sup> For formulations and production data, see Tables II and III.

<sup>c</sup> Cured in steam-heated bin at 110° F.

<sup>d</sup> Per cent plus 2-mesh lumps after dropping four times from 3-foot level, once on each face and side of bag.

<sup>e</sup> Similar to run AM-3/2 (Table II).

## PHOSPHORUS AVAILABILITY

### Effect of Preparation Method and Water Solubility of Nitric Phosphates on Uptake by Millet in Greenhouse Culture

NITRIC PHOSPHATES are a group of fertilizers produced by the reaction of rock phosphate and nitric acid, either alone or in mixtures with sulfuric or phosphoric acid, followed by ammonia-

tion and other process steps (7). The production processes are technologically attractive (8) because they permit the formation of polynutrient fertilizers in one integrated continuous operation.

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The resultant fertilizer is an intimate mixture of fine-grained compounds that shows little tendency toward segregation of nutrients. However, the water solubility of the phosphorus is notably lower

Nitrogen-phosphate fertilizers were prepared by three methods in which the citrate- and water-soluble phosphorus were similar in content. Millet was grown in greenhouse culture as a test crop on three soils: Ida silt loam, pH 7.6; Fort Collins silt loam, pH 7.8; and Miami silt loam, pH 5.1. Three criteria of measurement were used: plant response, phosphorus uptake, and A values. The plant uptake of phosphorus correlated closely with water solubility of the fertilizer when the nitrogen-phosphate was prepared by nitric acid dissolution of phosphate rock. However, the method of nitrogen-phosphate preparation was found to be as important as the water solubility in determining the plant availability of the applied fertilizer.

than that in most polynutrient fertilizers manufactured with superphosphates.

Agronomic evaluation of these materials has been conducted at various locations. Thorne, Johnson, and Seatz (7) and Rogers (5) recently summarized many of the experiments with nitric phosphates in the United States, and Cooke (7) and Mulder (4) compiled summaries of experiments in Europe. Their summaries were averages of widely differing responses under individual conditions, indicating that, for acid soils, nitric phosphates with a water solubility of over 10% were nearly as effective as superphosphate. Nitric phosphates with water solubilities below 10% were less effective than superphosphate. For neutral or alkaline soils, higher water solubilities were necessary to obtain results comparable with superphosphate.

Fertilizer laws in the United States require the manufacturer to state the total available phosphorus pentoxide—defined as that which is soluble in neutral ammonium citrate. The manufacturer may thus produce a product of high available guarantee, but this product may not be the most efficient agronomically. In Great Britain, available phosphorus is defined in the fertilizer trade as that phosphorus which is soluble in water (6). However, neither the citrate solubility nor the water solubility nor both may adequately define a nitric phosphate fertilizer with respect to plant-available phosphorus.

The purpose of this report is to assess the importance of water solubility and to determine how different nitrogen-phosphate preparations with similar water and citrate solubilities may compare agronomically.

### Methods

Nitrogen-phosphates with similar water and citrate solubilities were prepared by two methods, rock phosphate dissolution and slurry mix.

The rock phosphate-dissolution method consisted of dissolving rock phosphate with nitric acid. Phosphoric acid, monoammonium phosphate, and ammonium nitrate were then added to the slurry in such proportions as to arrive at predetermined water solubilities and nitrogen-to-phosphorus ratios. These

materials, similar to commercial nitric phosphates, were then dried and the size fraction 28-80 mesh was sieved out for use.

The slurry-mix materials were made by adding monoammonium phosphate, ammonium nitrate, and dicalcium phosphate in a slurry to arrive at the same water solubilities and nitrogen-to-phosphorus ratios as for the rock phosphate-dissolution materials. After thorough mixing, the preparation was dried and the size fraction 28-80 mesh was sieved out for use.

A third set of materials prepared by a dry-mix method was also used. This consisted of a physical dry mixture of dicalcium phosphate, ammonium nitrate, and monoammonium phosphate in proportions to arrive theoretically at the same water and citrate solubilities and nitrogen-to-phosphorus ratios as the slurry-mix materials.

Three soils were used: Fort Collins silt loam, with a pH of 7.8 and containing free calcium carbonate; Ida silt loam, with a pH of 7.6 but no free calcium carbonate; and Miami silt loam, with a pH of 5.1. Millet was grown in 7 pounds of soil contained in No. 10 cans. Individual treatment comparisons were made for each material at the different levels of water solubility for each method of preparation. Each material was applied at the rate of 100 pounds of phosphorus pentoxide per acre (2,000,000 pounds of soil). This furnished about 100 pounds of nitrogen per acre from the material. Where there was less, supplemental nitrogen, as ammonium nitrate, was added to furnish 100 pounds of nitrogen per acre to all pots.

A yield response of millet to applied phosphorus was determined on each soil. Twenty-five-pound-per-acre increments of phosphorus pentoxide from 0-

**Table I. Total Nitrogen and Total Phosphorus Pentoxide in Water- and Citrate-Soluble Form in Phosphorus-Fertilizer Materials Used**

Rock Phosphate-Dissolution Materials				Slurry-Mix and Dry-Mix Materials			
P <sub>2</sub> O <sub>5</sub>				P <sub>2</sub> O <sub>5</sub>			
Water soluble, % <sup>a</sup>	Citrate soluble, % <sup>a</sup>	Total, %	Total N, %	Water soluble, % <sup>a</sup>	Citrate soluble, % <sup>a</sup>	Total, %	Total N, %
				3	100	21	20
15	86	21	18	16	100	21	19
35	90	22	20	34	100	23	18
42	91	22	20	47	100	25	18
59	93	23	21	59	100	25	21
74	96	24	22	77	100	26	21
				100	100	26	24

<sup>a</sup> Percentage of total P<sub>2</sub>O<sub>5</sub>. The citrate-soluble percentage includes the water-soluble.

The water and citrate solubilities, phosphorus pentoxide content, and nitrogen content of these materials are listed in Table I. The fertilizers prepared by the rock phosphate-dissolution procedure were similar in water and citrate solubilities to those prepared by the slurry-mix procedure. Almost all of the rock phosphate-dissolution materials were citrate-soluble (86 to 96%) while all of the slurry-mix materials were citrate-soluble (100%). Sufficient potassium dihydrogen phosphate carrying radioactive phosphorus-32 was added to each material in its preparation to give a specific activity of 0.15 mc. per gram of phosphorus pentoxide.

to 100<sup>1</sup>-pound rates were applied as monoammonium phosphate and mixed with the soil. Ammonium nitrate fertilizer was added in solution at the time of seeding to furnish 100 pounds of nitrogen per acre to all pots.

Materials prepared by rock phosphate dissolution and slurry mix were applied two ways—banded and mixed. Materials prepared by the dry-mix method were applied by the mixed procedure only. Banding consisted of placing the fertilizer in two bands about 3/4 inch below the soil surface and about 1 1/2 inches apart. The seeds were then placed between the bands. The treatments were replicated three times on

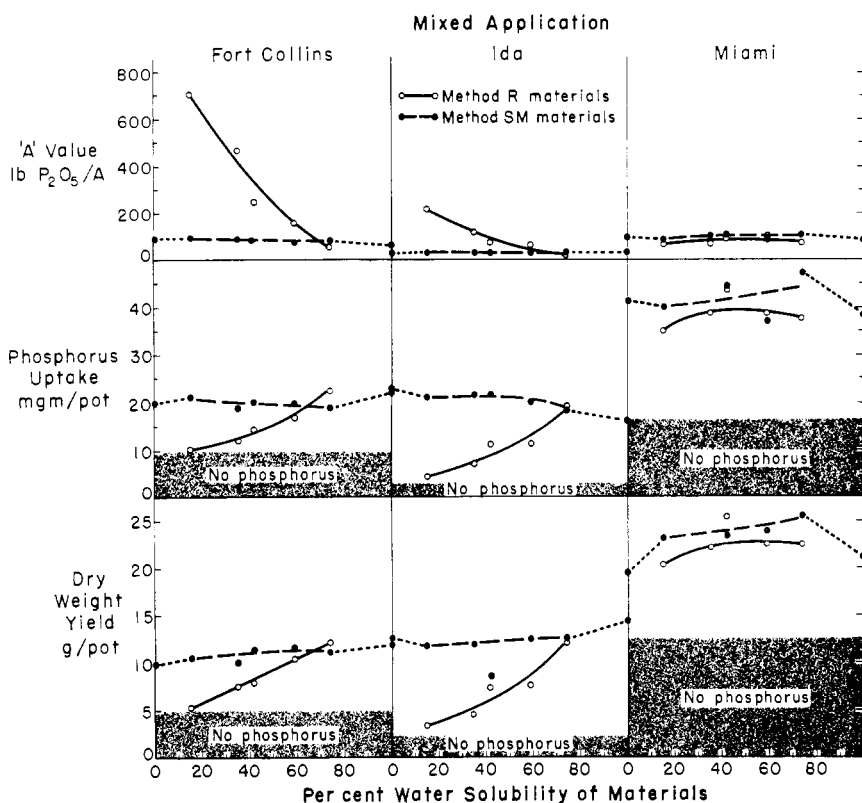


Figure 1. Relationship of water solubility of nitrogen-phosphate fertilizers, mixed with various soils, on yield, phosphorus uptake of millet, and A value

Method R refers to rock phosphate-dissolution materials  
Method SM refers to slurry-mix materials

Table II. Response of Millet to 100% Water-Soluble Fertilizer (Monoammonium Phosphate) Thoroughly Mixed with Ida, Fort Collins, and Miami Soils

Rate P <sub>2</sub> O <sub>5</sub> , Lb./Acre <sup>a</sup>	Yield, G./Pot			Total P Uptake in Tops, Mg./Pot			A Value		
	Ida	Ft. Collins	Miami	Ida	Ft. Collins	Miami	Ida	Ft. Collins	Miami
0	2.3	6.3	12.4	2.9	11.2	16.5	..	..	..
25	4.6	8.5	16.0	6.2	13.8	21.7	22	59	71
50	7.1	9.8	19.2	10.9	16.9	29.6	22	50	91
75	7.8	10.6	21.4	12.3	18.2	36.1	24	58	88
100	10.3	12.5	21.1	15.8	21.4	41.0	23	61	75
Rate LSD (0.05)		1.19			2.06		Not significant		
Av.	6.4	9.5	18.0	9.6	16.3	29.0	23	57	81
Soil LSD (0.05)		0.92			1.60		1.22		

<sup>a</sup> Nitrogen as NH<sub>4</sub>NO<sub>3</sub> was added to supply 100 pounds of N per acre to each pot.

each soil and, in the mixed procedure, the soil and fertilizer were mixed in a split-shell Patterson-Kelley mixer.

Thirteen days after germination, 120 pounds of potassium, as potassium sulfate, and 100 pounds of nitrogen, as ammonium nitrate, were added in solution and watered into the soil. After 12 days more, 80 pounds of potassium, as potassium sulfate, and 200 pounds of nitrogen, as ammonium nitrate, were added in the same way. The above-ground portion of the plants were harvested after 5 weeks, dried in an oven at 65° C., and ground in a Wiley mill. An aliquot sample was ashed at 500° C.

and made up to volume with 1N nitric acid. Counting of the radioactivity was done with a solution counter, and an assay of phosphorus was made by the vanadomolybdate procedure. A values were calculated by the method of Fried and Dean (3).

### Results

The level of soil phosphorus and the response of millet to added monoammonium phosphate is presented in Table II. The order of response was Ida > Fort Collins > Miami. In general, significant increases in yield of dry matter

and phosphorus were obtained for each 25-pound increment of phosphorus pentoxide. The yield of dry matter on the Miami soil appeared to reach its maximum close to the 100-pound rate.

The effects of water solubility of the nitrogen-phosphate fertilizer mixed in the soil on the yield, phosphorus uptake, and A value obtained on all three soils are shown in Figure 1. An increase in water solubility of the phosphorus fertilizer prepared by rock phosphate dissolution increases yield and phosphorus uptake and decreases the A value. The increase in yield and phosphorus uptake was small for the Miami soil and was statistically significant only at the first level of water solubility. However, it was marked in both the Ida and Fort Collins soils. There was no decrease in A value in the Miami soil, but a very large and highly significant increase occurred in the Ida and Fort Collins soils. This A-value decrease has been shown by Fried (2) to reflect an increase in fertilizer efficiency.

In contrast, millet fertilized by materials prepared by the slurry-mix method (data for dry-mix materials are not shown as they behaved the same as slurry-mix materials in all cases) did not show any statistically significant response differences or A-value differences to change in water solubility when the fertilizer was mixed in the soil (Figure 1). Water solubility of the fertilizer as a criterion of availability to millet was important only with the rock phosphate-dissolution materials.

As 100 pounds of phosphorus pentoxide per acre of each material was added, the non-water-soluble fraction of the slurry-mix materials furnished phosphorus to the millet plants equally as well as the water-soluble fraction. However, for rock phosphate-dissolution materials on the alkaline soils, a higher water solubility was associated with increase in both dry-weight yield and phosphorus uptake and a decrease in A value. Only at the 74% water solubility was the rock phosphate-dissolution material equivalent in fertilizer value to the slurry-mix material. Materials prepared by both methods behaved similarly in the acid Miami soil.

Figure 2 shows the yield and uptake of phosphorus obtained when the rock phosphate-dissolution and slurry-mix-fertilizer materials were banded. Both the yield and phosphorus uptake in the Ida and Fort Collins soils were not equal to that produced by mixing the fertilizers and soils. In contrast to the two basic soils, both the yield and phosphorus uptake in the very acid Miami soil was nearly as good for the band placement as for the mixed placement. With the Ida and Fort Collins soils, there was a parallel behavior of the rock phosphate-dissolution and slurry-mix materials with band application which was absent when

the fertilizers and soils were mixed (compare with Figure 1). On the other hand, with the Miami soil there was a parallel behavior of the two material preparations when mixed, which was absent when banded. Band placement allowed for less interaction of soil and fertilizer than mixed placement so the yield results more closely reflected the fertilizer qualities.

### Discussion and Conclusions

The uptake of phosphorus by a plant is not necessarily related to the citrate solubility and phosphate content of a nitric phosphate. These measurements may be enough if the nitric phosphate is mixed with a very acid soil such as the Miami silt loam of pH 5.1. When applied to less acid soils or in band placement, other factors are necessary for agronomic characterization of the fertilizer. One of these factors is water solubility. However, even this additional measurement may not be adequate.

In the preparation of nitric phosphate the process is controlled to minimize the citrate-insoluble phosphorus because the market is based on legally defined available phosphorus. Nevertheless, some citrate-insoluble or legally defined unavailable phosphorus is usually present and Table I shows that the rock phosphate-dissolution materials had some of this component. However, it was too small to account for the differences in results between rock phosphate-dissolution and slurry mix materials. Some other factor such as the relative availability of the non-water-soluble fraction could account for these differences.

The relative availability of the non-water-soluble portion of the fertilizer was calculated from the data in Table II (where 25-pound increments of 100% water-soluble fertilizer were added to each soil and the resultant dry-weight yield, uptake of phosphorus was measured and the *A* value calculated) and Figure 1 (where each fertilizer material was added at a 100-pound-phosphorus pentoxide rate). If the water-soluble phosphorus of two fertilizers is equally available to plants, 100 pounds of 50% water-soluble fertilizer should be equivalent to 50 pounds of 100% water-soluble fertilizer. If the uptake from 100 pounds of a 50% water-soluble fertilizer exceeded that of 50 pounds of a 100% water-soluble fertilizer, the increase could be attributable to the water-insoluble fraction or some other factor present in the 50% water-soluble fertilizer. The relative availabilities of the materials were calculated by this technique. The results are presented in Table III.

When 100 pounds of phosphorus pentoxide per acre of the 74% water-soluble material of the rock phosphate-

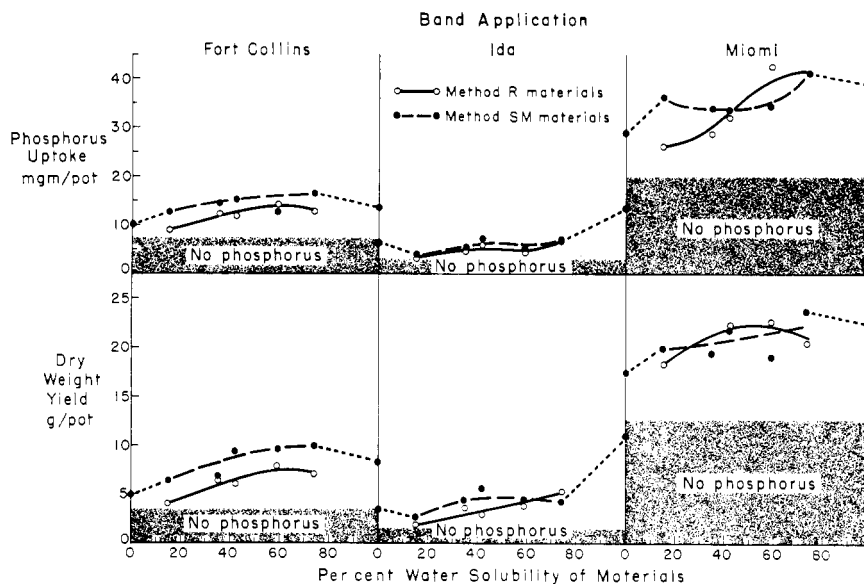


Figure 2. Relationship of water solubility of nitrogen-phosphate fertilizers, band applied with various soils, on yield and phosphorus uptake of millet

Method R refers to rock phosphate-dissolution materials  
Method SM refers to slurry-mix materials

Table III. Relative Availability of Citrate-Soluble, but Water-Insoluble Phosphorus in Rock Phosphate-Dissolution and Slurry-Mix Materials

Water solubility, %	Rock Phosphate-Dissolution Materials			Water solubility, %	Slurry-Mix Materials		
	Relative Availability, % <sup>a</sup>				Relative Availability, % <sup>a</sup>		
	Ft. Collins	Ida	Miami		Ft. Collins	Ida	Miami
15	0	0	90	3	80	100	100
35	0	0	100	16	80	100	100
42	0	30	100	34	80	100	100
59	30	30	100	47	90	100	100
74	100	100	100	59	90	100	100
				77	90	100	100

<sup>a</sup> Monoammonium phosphate = 100.

dissolution materials were mixed throughout the Fort Collins and Ida soils, the yield of phosphorus was equal to that obtained when 100 pounds of phosphorus pentoxide per acre of 100% water-soluble material was added to the same soil. At all levels of water solubility, the rock phosphate-dissolution materials when applied to the Miami soil gave similar results. The citrate-soluble but non-water-soluble fraction appeared to be as readily utilized by the millet as was the water-soluble fraction, and its relative availability was 100. However, when the rock phosphate-dissolution materials of lower water solubility were applied to the Fort Collins and Ida soils, the uptake of phosphorus was lower than that obtained with 100% water-soluble material.

With the 42% water-soluble fertilizer of the rock phosphate-dissolution materials on the Fort Collins soil, the total uptake of phosphorus by the millet

plants can be totally accounted for by the water-soluble fraction, and the relative availability of the citrate-soluble, but non-water-soluble, fraction is then 0%. Likewise, the citrate-soluble phosphorus of the 35 and 15% water-soluble materials did not contribute to phosphorus uptake in the Fort Collins soil. At the 59% water solubility, the relative availability of the citrate-soluble, but non-water-soluble, fraction of the fertilizer was only 30%.

The Ida soil gave slightly different results. The water-soluble, but citrate-soluble, fraction appeared to contribute phosphorus down to the 42% water-solubility level although this contribution was only 30% at the 42 and 59% water solubilities. At the 35 and 15% water-soluble levels the phosphorus uptake by the millet could be totally accounted for by the water-soluble fraction alone.

The citrate-soluble fraction of the

slurry-mix (and dry-mix) materials does not differ from the water-soluble fraction in availability to millet, and the relative availabilities approximate 100. This occurred on all three soils where the fertilizer was mixed with the soil. These materials thus behaved differently from the rock phosphate-dissolution materials.

The slurry-mix (and dry-mix) materials appear to be no different from the monoammonium phosphate, dicalcium phosphate, and ammonium nitrate constituents from which they were made. Petrographic analysis of these materials showed no evidence of solution erosion or chemical alteration.

Definition of rock phosphate-dissolution materials is more complex. As indicated before, the differences in response to these materials as compared to the dicalcium phosphates of the slurry-mix (and dry-mix) materials could be due to the relative unavailability of the citrate-soluble basic phosphates formed in the manufacturing process. This is also indicated by the high availability of the rock phosphate-dissolution materials in the very acid Miami soil where reaction with the soil should result in a greater similarity to the other materials.

The rock phosphate-dissolution materials were examined petrographically and the results are presented in Table IV. All samples contained a precipitated apatite with optical properties of cel'ophane as the major calcium phosphate constituent. Only the 74% sample contained appreciable quantities of dicalcium phosphate dihydrate.

In contrast to the results obtained with the rock phosphate-dissolution and

**Table IV. Chemical Compounds in Rock Phosphate-Dissolution Materials as Indicated by Petrographic Analysis<sup>a</sup>**

Water Solubility, %	Calcium Phosphate Compounds <sup>b</sup>
15	Ppt. apatite with optical properties of cel'ophane
35	Same as 15%
42	Same as 15% with a small amount of CaHPO <sub>4</sub> ·2H <sub>2</sub> O
59	Same as 15%
74	Same as 42% with a much greater content of CaHPO <sub>4</sub> ·2H <sub>2</sub> O

<sup>a</sup> NH<sub>4</sub>NO<sub>3</sub> and NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> found in all five materials.

<sup>b</sup> Small amounts of unreacted phosphate rock were present in all five materials.

slurry-mix materials when they were mixed with the soil, band application did not differentiate very well between methods of preparation though there were indications that the rock phosphate-dissolution materials were not as effective at low water solubilities as the slurry-mix materials on the acid Miami soil (Figure 2). Water solubility in the band treatment appeared to be the most important fertilizer criteria. Webb (9) has recently shown that corn-yield response from starter fertilizers in Iowa is closely related to water solubility of the phosphorus. The influence of mixing the soil and fertilizer was greater on the slurry-mix materials; thus, there was a dissimilarity of the non-water-soluble fraction of the rock phosphate-dissolution and the slurry-mix materials, and

the effect was greater than could be attributed to the citrate-insoluble fraction alone. This again illustrates the inability to predict the agronomic value of a material from chemical measurements of citrate solubility and even water solubility.

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## SURFACE AREA EFFECTS

### Greenhouse Crop Response to Water-Insoluble Phosphates with Different Surface Areas

Corn stover and alfalfa were grown in greenhouse cultures on slightly acid Chester soil to determine the effect of surface area of water-insoluble phosphorus in fertilizers upon yield and phosphorus uptake. Radioactive preparations of basic phosphate and of dicalcium phosphate with surface areas ranging from 0.7 to 22.8 square meters per gram were tested. Essentially the same yields of both corn and alfalfa were obtained with the dicalcium phosphates, although their surface areas differed by as much as 5 square meters per gram. Markedly lower yields resulted from using the basic phosphate despite the fact that its surface area was higher than that of any of the dicalcium phosphates. Thus, chemical composition appears to be more critical than surface area within the range studied.

THE REACTIVITY of a phosphate fertilizer, and perhaps its agronomic value also, is influenced by the extent of exposed surface, which in general depends on the fineness of the material as

determined by appropriate methods. Particle size, though relatively unimportant in the case of very soluble fertilizers, is prominent among those factors that influence the nutritive value of

water-insoluble phosphates. The fertilizer value of such materials is said to depend on particle size. Most of the water-insoluble phosphates present in mixed fertilizers possess finenesses that lie

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