Effect of Particle Size of Raw Materials on Granulation of Fertilizers

A. B. PHILLIPS, G. C. HICKS, J. E. JORDAN, and T. P. HIGNETT
Tennessee Valley Authority, Wilson Dam, Ala.

The efficiency of granulation of certain grades of fertilizers, such as 4–16–16, 8–16–16, and 5–20–20, was improved when a portion of the solid raw materials was of a particle size nearly equal to the desired granule size. The large particles serve as nuclei for the formation of granules and decrease the requirement of other aids to granulation such as steam, water, and acid. Coarse ordinary or triple superphosphate was as effective as coarse potassium chloride in promoting granulation. No advantage resulted when coarse materials were used in formulations that granulated well without nucleation; in some cases the granules were more homogeneous with fine raw materials.

IN PREVIOUS PILOT-PLANT STUDIES (*1*) of the production of granular high-analysis fertilizers, the particle size of the raw materials had a pronounced effect on the efficiency of granulation of some grades. Coarse or granular potassium chloride is commonly used to improve granulation of some grades that are difficult to granulate—5–20–20 and 4–16–16 are typical (*2*). The particle sizes of "granular" and "coarse" potassium chloride that are offered by the potash industry vary widely. Both the potash industry and the fertilizer industry are interested in finding the best particle size of potassium chloride for use in granulation processes.

To obtain more information on the effect of particle size of raw materials on the granulation characteristics of fertilizers, tests were made in the TVA pilot plant in which the particle size of potassium chloride or superphosphate was varied and the granulation efficiency was studied for several grades.

Test Methods

The tests were carried out in the TVA ammoniation-granulation pilot plant (*1*). The production rate in most tests was 2 tons per hour.

For the purpose of describing the particle size of the raw materials, a number was devised that was related to the mean particle size. This "particle-size index" was calculated from the screen analysis. For instance, a 6-mesh Tyler standard screen has 0.131-inch openings, and a 10-mesh screen has 0.065-inch openings. The mean between these two numbers is 0.098 inch, which is called the particle-size index of the fraction that passes a 6-mesh screen and is retained on a 10-mesh screen. Particle-size indexes were calculated similarly for each screen fraction and a weighted average particle-size index was calculated for each material based on its screen analysis (Table I).

Effect of Particle Size of Potassium Chloride on Granulation of 4–16–16. The 4–16–16 grade was selected for a detailed study of the effect of particle size of potassium chloride because it is one of the important grades that has proved difficult to granulate. Also, the granulation efficiency of this grade was strongly affected by the particle size of the potassium chloride. The basic formulation used in these tests follows.

Formulation for 4–16–16

Material	Lb./ton
Nitrogen solution[a]	53
Anhydrous ammonia	77
Ordinary superphosphate	736
Triple superphosphate	379
Sulfuric acid (95% H_2SO_4)	...[b]
Potassium chloride	533

[a] 21.7% ammonia, 65.0% ammonium nitrate, and 13.3% water.
[b] As required for granulation control.

The standard degree of ammoniation used in previous work (*1*) was 5.8 and 3.8 pounds of free ammonia per unit of available phosphorus pentoxide in ordinary and triple superphosphate, respectively. In the above formulation, the standard degree of ammoniation would be 4.7 and about 40 pounds of sulfuric acid would be required to react with the excess ammonia. In the current tests, the amount of sulfuric acid was varied from 100 to 180 pounds as required to control granulation. Water also was added to obtain granulation; however, it was not feasible to control granulation with water alone. In most tests, the amount of water added was about 200 pounds. The water, ammonia, and nitrogen solution were metered separately and premixed in the pipe line leading to the ammoniator.

In the first series of tests, four closely sized fractions of potassium chloride were used. The superphosphates were reduced to −10 mesh to accentuate the effect of nucleation by potassium chloride granules. No recycle was used.

Table I. Screen Analyses of Raw Materials Used in Studies of Effect of Particle Size on Granulation of 4–16–16 Fertilizer

Material Designation	Particle-Size Index, In.	Screen Analysis, % by Weight: −6 +10	−10 +16	−16 +28	−28 +48	−48
Potassium chloride						
Coarse	0.083	67	33	0	0	0
Medium coarse	0.048	0	80	19	1	0
Medium fine	0.022	0	0	43	49	8
Fine A	0.009	0	0	0	13	87
Granular A	0.049	21	33	28	12	6
−10-mesh granular	0.034	1	32	46	14	10
Regular	0.016	0	2	19	38	41
Granular B	0.068	42	44	10	3	1
Fine B	0.008	0	0	0	19	81
Extra fine	0.007	0	0	0	7	93
Granular superphosphate						
Ordinary	0.073	50	41	7	1	1
Triple	0.080	67	23	7	2	1

Table II. Effect of Particle Size of Potassium Chloride on Granulation and Acid Requirement in Production of 4–16–16

Potassium Chloride			Granulator Product, %			
Particle-size index, in.	Size designation[a]	Sulfuric Acid Rate, Lb./Ton	Oversize, +6 mesh	On-size, −6 +16 mesh	Undersize, −16 mesh	Yield of On-size Granules after Crushing Oversize,[b] %
Closely Screened Size Fractions—Acid Rate Constant						
0.083	Coarse	107	27	63	10	79
0.048	Medium coarse	101	12	63	25	70
0.022	Medium fine	98	6	31	63	35
0.009	Fine A	100	6	24	70	28
Closely Screened Size Fractions—Acid Rate Varied						
0.083	Coarse	107	27	63	10	79
0.048	Medium coarse	128	22	70	8	83
0.022	Medium fine	156	21	49	30	62
0.009	Fine A	179	66	28	6	68
Commercial Granular and Mixed Particle Sizes—Acid Rate Constant						
0.049	Granular A	133	27	55	18	71
0.034	−10-mesh granular A	133	8	46	46	51
0.032	Mixed, 1:1[c,d]	130	23	44	33	58
Commercial Granular and Mixed Particle Sizes—Acid Rate Varied						
0.049	Granular A	133	27	55	18	71
0.034	−10-mesh granular A	150	26	55	19	71
0.038	Mixed, 2:1[d]	155	31	59	10	78
0.032	Mixed, 1:1[d]	155	40	54	6	78
0.027	Mixed, 1:2[d]	160	27	63	10	79

[a] See Table I for screen analyses.
[b] Based on 60% recovery of on-size from crushed oversize.
[c] Water rate was 257 pounds per ton of product in this test only; in all other tests the rate was 205 pounds per ton.
[d] Mixtures of granular A and regular potassium chloride in the proportions shown.

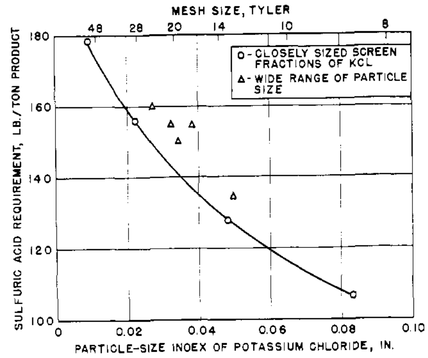

Figure 1. Effect of particle-size index of potassium chloride on amount of acid required for granulation of 4–16–16 fertilizer

The series was started with coarse potassium chloride, and the proportions of sulfuric acid and water were adjusted to give what appeared to be the best granulation. Each of the other size fractions of potassium chloride was then tested with about the same acid and water rate (Table II). The recovery of on-size (−6 +16 mesh) product after crushing the oversize decreased from 79 to only 28% as the particle size of the potassium chloride was decreased from coarse to fine.

In the next series of tests, the acid rate was adjusted to give—by visual inspection—the best granulation obtainable with each size fraction of potassium chloride (Table II). The amount of acid required to obtain best granulation increased as the particle size of the potassium chloride was decreased. The granulation efficiency was highest when the medium-coarse fraction was used as judged by either the percentage of on-size granules formed or the yield of on-size particles after crushing the oversize. Granulation was good also when using coarse potassium chloride, but poorer results were obtained with the finer fractions. Operation with the finest material was very difficult to control, and granulation was erratic.

In another series of tests the effect of using potassium chloride containing a relatively wide range of particle sizes with varying proportions of fines was studied. The potassium chlorides used were a commercial granular material, the −10-mesh portion of the granular material, and mixtures of the commercial granular and commercial regular materials in proportions of 2 to 1, 1 to 1, and 1 to 2 (Table II).

Commercial granular A potassium chloride was used in the first test of this series, and the acid rate was adjusted to that which gave the best granulation. The granulation efficiency was satisfactory but not as good as that obtained with the medium-coarse potassium chloride, which had about the same particle-size index but a narrower range of sizes. When using the −10-mesh fraction of granular A and the same acid rate as was best for granular A, much poorer granulation resulted.

Poorer results also were obtained when a 1 to 1 mixture of granular A and regular potassium chlorides was used with the acid rate that was best for granular A alone. In this test, the water rate was increased to 257 pounds per ton of product as compared with 205 pounds for the other tests. Increasing the water rate did not give good granulation.

When acid rate was adjusted to that which appeared to give the best granulation, granulation efficiency was approximately equal for granular A, −10-mesh granular A, and all mixtures of granular A and regular potassium chloride. There was some indication of better granulation with the mixtures than with granular A alone, but the difference may not be significant.

In Figure 1, the acid requirements for best granulation with the various sizes of potassium chloride are plotted against the particle-size index. The amount of acid required generally decreased as the particle-size index was increased. The amount required with the potassium chloride of wide particle-size range was in all cases higher, but was fairly close to the amount required with the closer sized fractions of the same particle-size index.

Effect of Particle Size of Superphosphate on Granulation of 4–16–16. When granular particles are needed to assist in the granulation of fertilizers, it may be convenient to supply them by using granular ordinary or triple superphosphate. Tests were made in which

Table III. Comparative Effects of Using Granular Superphosphates or Granular Potassium Chloride on Granulation of 4–16–16

Granular Raw Material Used				Granular Product, %			
Kind	Quantity, lb./ton	Particle-size index, in.	Sulfuric Acid Rate, Lb./Ton	Oversize, +6 mesh	On-size −6 +16 mesh	Undersize, −16 mesh	Yield of On-size Granules after Crushing Oversize, %
Triple superphosphate	421	0.080	173	36	54	10	76
Potassium chloride	548	0.083	107	27	63	10	79
Ordinary superphosphate	788	0.073	90	34	64	2	84

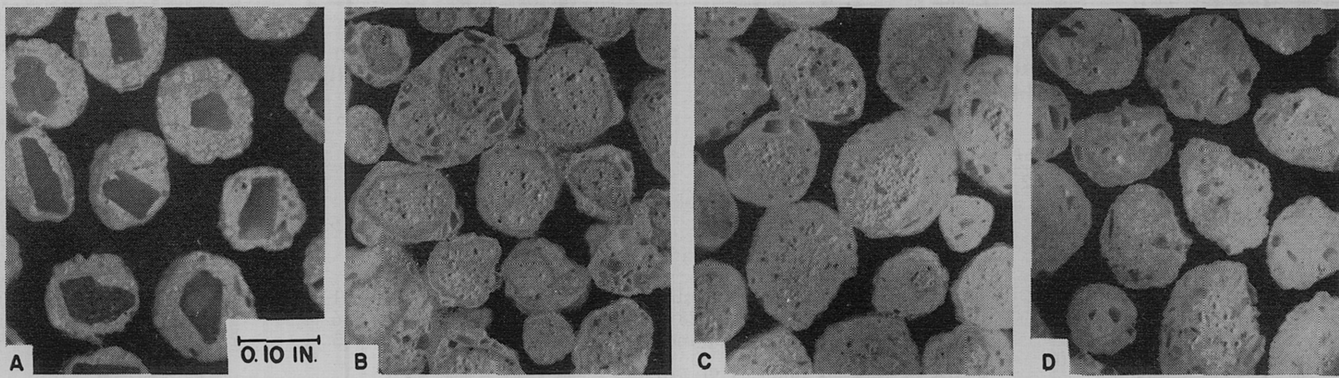

Figure 2. Cross sections of granules of 4–16–16 fertilizer

A. Made with granular potassium chloride
B. Made with granular ordinary superphosphate
C. Made with granular triple superphosphate
D. Made with nongranular materials

either granular ordinary or granular triple superphosphate was used in making 4–16–16. The method for producing granular superphosphates in the pilot plant has been described (*4*).

Table III compares the results when using granular ordinary superphosphate, granular triple superphosphate, and granular potassium chloride. In each case, only one of the three materials was granular. The test with coarse potassium chloride was used for comparison because it was the closest in particle size to the granular superphosphates. The data show that the yield of on-size product increased as the quantity of granular raw materials was increased. The kind of granular raw material did not have any important effect on granulation. The amount of sulfuric acid required for best granulation decreased as the amount of granular raw material was increased.

In the tests with granular potassium chloride and granular triple superphosphate, the water rate was 200 pounds per ton. With granular ordinary superphosphate, this much water caused a muddy condition in the ammoniator, and better operation was obtained when the water rate was decreased to 100 pounds per ton. With this reduced moisture input, the moisture content of the product leaving the granulator was 5.4% as compared with about 9% for the other runs.

Inspections of granules of 4–16–16 that were made with granular raw materials showed that the granular raw material formed a core or nucleus, and that the other ingredients formed a coating or hull around this core. Photomicrographs of cross sections of typical 4–16–16 granules, shown in Figure 2, illustrate this effect. Very few uncoated original granules were found in the product.

Uniformity of Composition of Screen Fractions of 4–16–16. Some difficulties, due to lack of uniformity in analyses of screen fractions of granular fertilizers, have been reported by fertilizer manufacturers. Conical storage piles form when the fertilizer is fed to a single point. The finer particles tend to remain near the center of the pile, and the larger particles tend to roll toward the edge of the pile. If there is a difference in the chemical analyses of the size fractions, various portions taken from the piles may be off-grade.

In the current work, most of the runs were made without recycle. For this reason, the analyses of screen fractions would not be typical of the final product of a plant in which the oversize is crushed and returned to the screens and the fines recycled. However, the potash contents of screen fractions of the product from the granulator were determined. The oversize (+6 mesh) usually contained less potash than the on-size (−6 +16 mesh) when either granular or nongranular potassium chloride was used. The difference increased as the particle size of the potassium chloride was increased. The fines (−16 mesh) contained less potash than the on-size fraction when granular or coarse potassium chloride was used, and more potash than the on-size when potassium chloride of smaller particle size was used. The disparity in potash contents of screen fractions was accentuated by the use of closely screened potassium chloride; it was minimized by use of blends of granular and nongranular potassium chloride.

When granular superphosphates were used in producing 4–16–16, the phosphorus content of the on-size screen fraction was high and the phosphorus content of the fines was low.

These results indicate that some care should be taken to recycle the fines at the rate they are produced. If a semigranular product is made, attention should be given to prevention of segregation in the storage pile.

Two runs were made in which the fines—produced after crushing the oversize—were recycled. In one run, granular potassium chloride was used; in the other run a 1 to 2 blend of granular and nongranular potassium chloride was used. The distribution of potash in the screen fractions of the product from the granulator, shown in Table IV, was about the same in both runs. There was a difference of 1 to 1.5 units of potash between the −6 +10- and the −10 × 16-mesh portions. Analyses of the final −6 +16-mesh product, which contained crushed oversize, showed a variation of about one unit of potash between the +10- and the −10-mesh fractions in both runs.

Effect of Raw Material Particle Size on Granulation of Various Grades. A recent paper (*3*) reported a study of the use of ammoniating solutions containing urea in the production of several grades of granular fertilizer. In the current work, three of the tests were repeated using granular potassium chloride. The formulations selected (Table V) were those which were considered likely to show improvement in granulation due to use of the granular material. Table VI compares the results obtained with granular and nongranular potassium chloride. In these tests, the "on-size" fraction was taken to be 6 to 28 mesh rather than the 6 to 16 mesh used in the previously described tests with 4–16–16. Also, the superphosphates were −4 mesh in the present test and −10 mesh in the previous tests. Both of these factors would tend to decrease any improvement in granulation owing to the use of

Table IV. Distribution of Potash in Granulator Product When Producing 4–16–16 Fertilizer

Screen Fraction of Granulator Product, Tyler Mesh	Product Made with Granular KCl: Proportion of product in indicated fraction, %	Product Made with Granular KCl: K_2O content, %	Product Made with Blend of 1 Part Granular KCl to 2 Parts Regular: Proportion of product in indicated fraction, %	Product Made with Blend of 1 Part Granular KCl to 2 Parts Regular: K_2O content, %
+ 6	26	12.7	20	13.3
− 6 +10	50	16.3	41	16.2
−10 +16	17	17.8	23	17.4
−16	7	19.5	16	19.5

Table V. Formulations for Granular Fertilizers in Which Use of Granular Potassium Chloride Was Tested

	Pounds Per Ton of Product		
Grade	5-20-20	6-12-12	8-16-16
Nitrogen solution	244[a]	300[a]	342[b]
Superphosphate			
Ordinary	262	1224	1009
Triple	747	—	—
Phosphoric acid	—	—	190
Potassium chloride	655	406	528
Sulfuric acid	134	—	—
Filler	—	94	—
Total	2042	2024	2069

[a] Containing 24.5% ammonia, 56.0% ammonium nitrate, 10% urea, and 9.5% water.
[b] Containing 30.6% ammonia, 43.1% urea, and 26.3% water.

Table VI. Effect of Particle Size of Potassium Chloride on Granulation of Three Grades of Fertilizer

	5-20-20		6-12-12		8-16-16	
	Regular[a]	Granular B[a]	Regular[a]	Granular B[a]	Fine B[a]	Granular B[a]
Particle-size index of potassium chloride, in.	0.015	0.068	0.015	0.067	0.008	0.068
Screen analysis of granulator product, %						
Oversize (+6 mesh)	32	24	31	23	39	12
On-size (−6 + 28 mesh)	66	75	66	75	47	82
Undersize (−28 mesh)	2	1	3	2	14	6
Screen analysis of product from cooler or dryer, %						
Oversize (+6 mesh)	37	31	23	23	22	6
On-size (−6 +28 mesh)	60	69	67	75	37	73
Undersize (−28 mesh)	2	0	10	2	41	21
Yield of on-size product after crushing oversize, %	88	92	84	92	53	78

[a] Potassium chloride designation, see Table I for screen analyses.

granular potassium chloride. However, in each case there was some improvement. The improvement was least in 5–20–20, greatest in 8–16–16, and intermediate in 6–12–12.

In the 5–20–20 tests, excess sulfuric acid was used for controlling granulation. The use of granular potassium chloride decreased the requirement of acid from 137 to 131 pounds per ton of product. The smallness of this decrease may be attributed to the presence of appreciable quantities of +28-mesh nuclei in both the superphosphates and the "regular" potassium chloride.

Screen analyses of the 5–20–20 granulator product showed that 68% of the granules were in the 6- to 14-mesh fraction, when using granular potassium chloride, as compared with 38%, when using nongranular potassium chloride. If 6 to 14 mesh were the desired size of the final product, the use of granular potassium chloride would have a much greater effect on the yield than when 6 to 28 mesh was the desired product size.

In the runs of 6–12–12, steam was used to control granulation. About 17% less steam was required with granular potassium chloride than with nongranular potassium chloride. The on-size recovery was increased from 84 to 92% by the use of granular potassium chloride.

In producing 8–16–16, the granules were dried from about 6 to 2% moisture. There was a considerable increase in the amount of undersize after the product passed through the dryer. However, the increase in undersize was less in the test with granular potassium chloride, which indicated that the granules formed around nuclei of granular potassium chloride may be stronger than granules containing no nuclei.

To obtain further information on the stability of granules as affected by particle size of potassium chloride, samples of the six products listed in Table VI were subjected to an abrasion test in which they were tumbled under controlled conditions. In each case, products made with granular potassium chloride formed 33 to 37% fewer fines than products of the same grade made with nongranular potassium chloride.

In other tests, good results were obtained when granular triple superphosphate was used in production of 5–20–20. Granular triple superphosphate also was tested in production of 12–12–12. This grade usually granulates satisfactorily without using any granular raw material, and it is often difficult to prevent excessive agglomeration. In the test with granular triple superphosphate, about half of the product was on-size and half was oversize. This was somewhat more oversize than usually obtained with all nongranular raw materials, but use of the granular triple superphosphate was not seriously detrimental to control of granulation of 12–12–12.

The use of regular and extra-fine potassium chloride was compared in the the production of 8–16–32 and 6–18–36. These grades were made by the ammonium phosphate–nitrate process (*5*). Raw materials, other than potassium chloride, were ammonia, nitrogen solution, and wet-process phosphoric acid. Granulation was controlled by the quantity of recycle. The particle-size indexes of the regular and extra-fine potassium chlorides were 0.016 and 0.007 inch, respectively. There was no appreciable difference in the granulation efficiency; in each case, about 70% of the granulator product was on-size (6 to 16 mesh) and about 24% was oversize. Also, there was no appreciable difference in the quantity of recycle required for granulation control.

The granules of 6–18–36 made with extra-fine potassium chloride were more nearly round, more uniform in shape, and more nearly homogeneous than those made with regular potassium chloride.

Conclusions

Fertilizer formulations may be divided into two classes: formulations that require some aid to induce sufficient granule formation and formulations that granulate readily without aid and may require some control to prevent excessive agglomeration. Formulations of the first class are more readily granulated when a portion of the solid raw materials is of a size nearly equal to the desired granule size. These large particles serve as nuclei around which the fertilizer granules form. The presence of these nuclei increases the percentage of on-size granules, increases the strength of the granules, and decreases the amount of other aids such as steam, water, or acid required for granulation.

In the present work, the quantity of granular raw material used in the production of 4–16–16 fertilizer was varied from 0 to 40%. Within this range, granulation efficiency increased and the amount of sulfuric acid required for granulation decreased as the quantity of granular raw material was increased. Increasing the particle-size index of potassium chloride or the superphosphate in the range of 0.009 to 0.08 inch improved granulation and decreased the acid requirement. Granular superphosphate and granular potassium chloride were equally effective as an aid to granulation.

In all tests with 4–16–16, there was considerable disparity in chemical composition of screen fractions of the granulator product. No combination of potassium chloride particle sizes would entirely avoid this disparity. However, when the oversize was crushed and returned to the screens and the fines were recycled, the difference in the analysis of the upper and lower screen fractions of a

6- to 16-mesh product was not large. Thus, close sizing of the final product appeared to be the most promising solution to the problem of variation in chemical composition of screen fractions.

The presence of coarse raw materials was not advantageous in formulations that granulated readily without nucleation. It was not seriously detrimental in these formulations, but in some cases the granules appeared more nearly homogeneous and more regular in shape when fine raw materials were used.

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PHOSPHORUS AVAILABILITY

Effect of Flooding on Plant Availability of Phosphorus from Various Phosphate Rocks

RAYMOND E. SHAPIRO and W. H. ARMIGER

Soil and Water Conservation Research Division, Agricultural Research Service, U. S. Department of Agriculture, Beltsville, Md.

The phosphate rocks tested under greenhouse conditions on a Cecil sandy loam and a Crosby silt loam fall into three plant-availability groups (60, 30, or 5% as plant available as resin phosphate), whether flooded or unflooded, when applied at the rate of 1 ton per acre. The effect of flooding on the plant availability of phosphate rock varied with the soil and the source of material. The relative plant availability of 10 phosphate sources was measured by four methods: yield of plants, uptake of phosphorus, *A* values, and resin phosphate equivalents. The methods lead to essentially the same groupings of the sources on the basis of phosphorus availability.

UNDER MOST AGRICULTURAL SITUATIONS, the phosphorus of phosphate rock is less readily available to plants than that of superphosphate. Phosphate rocks also vary markedly as to plant availability of the phosphorus when obtained from different sources. Armiger and Fried (*1*), using various sources of phosphate rock on several United States soils, showed marked differences among the sources in their ability to supply phosphorus to buckwheat and alfalfa. The relative magnitudes of the differences among the various sources depended upon the soil to which they were applied. For example, although both a Cecil sandy loam and a Crosby silt loam responded to phosphate application, the differences in the effectiveness of the various rocks as phosphate sources were much greater on the Crosby than on the Cecil soil.

Scattered evidence dealing with submerged soils indicates that phosphate rock may serve as a source of phosphorus for rice. In rice culture, an estimate of the relative effectiveness of various sources of phosphate rock requires agronomic data on flooded rather than on aerated unflooded soils.

This experiment compares various phosphate rocks, under flooded and unflooded conditions, in their ability to supply phosphorus to rice. Two soils from the same series used by Armiger and Fried were used to determine whether the interaction found between soil and phosphate rock applied also when the soil was flooded. The soils were a Cecil sandy loam, a high-phosphate fixing soil, and a Crosby silt loam, a medium-phosphate fixing soil. Seven of the phosphate rocks are the same as those used by Armiger and Fried (*1*).

Experimental Procedure

Colusa, a lowland variety of japonica rice, was grown during the summer in No. 10 cans under greenhouse conditions. When the soil was flooded, about 1 inch of water was maintained on the soil surface; when it was not flooded (unflooded culture), the moisture content was adjusted approximately to field capacity daily. The soils used were a Cecil sandy loam, pH 5.4, from South Carolina, and a Crosby silt loam, pH 5.1, from Indiana. Each pot contained 7 pounds of Cecil soil or 6 pounds of Crosby soil. The treatments, in triplicate, were: 1 ton per acre of each of 10 phosphate rocks (100 to 150 mesh, except for Hyperphosphate, a commercial grade of Tunisian phosphate rock, which was less than 300 mesh) mixed with the entire soil before seeding. A base application of 20 pounds of phosphorus pentoxide per acre in the form of a saturated phosphate resin (IRA 400) tagged with phosphorus-32 was mixed with the soil of all pots (including the check pots). The sources and phosphorus pentoxide contents of the 10 rocks are listed in Table I. The characteristics and surface areas for seven of these materials are given by Caro and Hill (*2*) and Hill, Caro, and Wieczorek (*5*).

Standard phosphorus response curves, flooded and unflooded, were obtained in another series of pots at the same time. The conditions of handling and fertilization were exactly the same except for phosphate application. In this series, saturated phosphate resin (IRA 400) was the only source of phosphorus. The treatments, in triplicate, were: 0, 40, 80, 160, 320, and 640 pounds of phosphorus pentoxide per acre.