

Figure 1. General view of pilot plant

## QUICK CURING OF SUPERPHOSPHATE

### Pilot Plant Studies

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A process for quick curing of normal superphosphate previously investigated on a laboratory scale was further developed on a pilot plant scale. It was demonstrated that the process can be carried out continuously to produce product suitable for either direct application or mixed fertilizer production.

LABORATORY investigations (3, 4) have been described of a process for quick curing of normal superphosphate, which consists of mixing phosphate rock and 55% sulfuric acid, denning, disintegrating, and drying in a Roto-Louvre dryer.

The present investigation was carried out to demonstrate that the process could be carried out continuously on a pilot plant scale, to confirm the effects of certain process variables on the properties of the product, and to obtain engineering information that would be useful in designing a larger plant (6). A brief summary of the pilot plant studies has been published previously (5).

#### Description of Pilot Plant

A photograph of the pilot plant is shown in Figure 1 and a flow sheet is shown in Figure 2.

Mixing was carried out batchwise in an 8-gallon Day pony mixer, size 0. The

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mixing blades operated at 64 r.p.m. and the pan rotated at 40 r.p.m.

The den and disintegrator simulated a Sturtevant den, which is widely used in the industry. The den was an open wooden box with removable wooden sides on a platform which was mounted on a track and could be moved slowly into a disintegrator consisting of a series of blades on a vertical shaft rotating at 58 r.p.m.

The disintegrated superphosphate was conveyed to a Model 106-6 Link-Belt Roto-Louvre dryer by means of a belt conveyor. The drying section was 6 feet in length and the inside diameter of the louvres varied from 12 inches at the feed end to 14 inches at the discharge end. Figure 3 shows the discharge end of the dryer with the cover plate removed to indicate construction of the louvres. The exit end of the dryer normally had a retaining ring with an inside diameter of 10 inches, which caused the bed of superphosphate to be 2 inches deep at this point. The outside diameter of the dryer shell was 18 inches. The dryer was heated by combustion of natural gas in a separate combustion chamber.

Inlet air, exit air, and product temperatures were measured by thermocouples placed in the inlet air duct near the dryer entrance, the discharge hood, and the bed of product immediately before it discharged from the louvres, respectively.

In all but the first few runs, the dryer was rotated at 12 r.p.m.

#### Materials and Procedure

The phosphate rock used was a Florida pebble containing 33.5% phosphorus pentoxide dry basis. One batch of rock was ground to 86% -100 mesh and 61% -200 mesh, and another batch was ground to 80% -100 mesh and 52% -200 mesh. Sulfuric acid of the desired concentration, usually 55% was made by diluting commercial 66° Bé. acid.

The acidulation used was 1.81 pounds of sulfuric acid (100% basis) per pound of phosphorus pentoxide in the rock. A total of about 45 pounds of rock and acid was mixed in each batch and 15 batches were required to fill the den. The mixing time was found to influence the ease

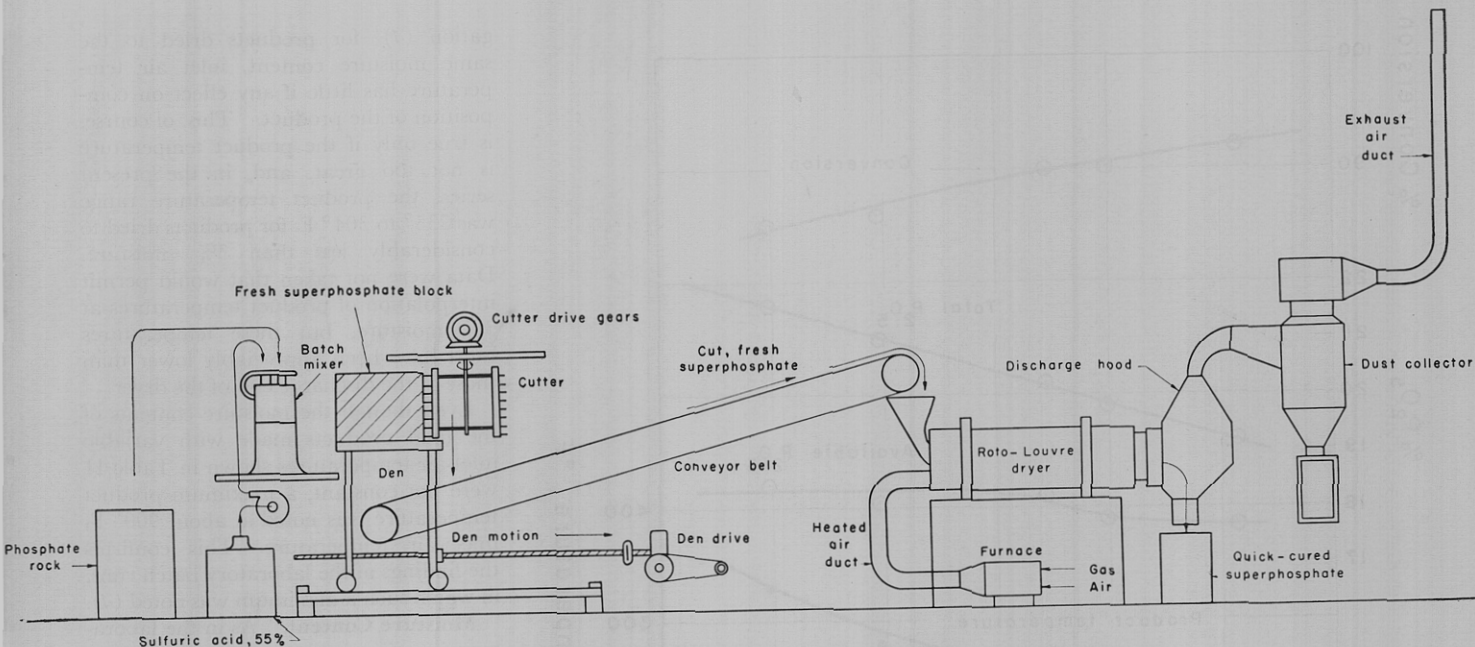


Figure 2. Flow diagram of pilot plant

with which the denned product could be disintegrated. The best mixing procedure was to add the acid to the mixer first and then add the rock gradually over a 1-minute period with the mixer operating. After an additional 2 minutes of mixing, the batch, which was still in the form of a thin slurry, was poured into the den. This total mixing cycle of 3 minutes was found better than cycles of 2, 4, or 5 minutes. About 2 hours was required to fill the den.

With acid and rock at room temperature, the final temperature of the mixture was 155° to 165° F. The maximum temperature developed in the den, which was reached when the den was completely filled, was 200° to 210° F.

Within 15 minutes after the last batch had been poured into the den, the sides were removed and disintegration could be started. This was a marked improvement over the laboratory studies, in which 12 to 20 hours of denning was required before disintegration could be accomplished. The difference is doubtless due to both the higher temperature developed in the pilot plant den and the differences in mechanical handling and disintegration between laboratory and pilot plant.

The disintegrated material was fed to the dryer; the conditions of operation varied widely and are described in subsequent tables.

The products were analyzed by methods of the Association of Official Agricultural Chemists (7), except that phosphate determinations were made by the colorimetric method of Bridger, Boylan, and Markey (2). The oven method for moisture was used (5 hours at 100° C.). Analyses of samples designated as "0 day" were begun within 2 hours from

sampling time, and the "2-day" analyses were made 2 days after sampling.

#### Effect of Experimental Conditions

**Acid Concentration.** Pilot plant runs were made with sulfuric acid concentrations of 50, 55, 60, and 72%. In the run made with 50% acid, considerable stratification took place immediately after the mixture was poured into the den. After 3 hours of standing, the block of fresh superphosphate was very wet and difficult to disintegrate. With the other concentrations, however, the material set up quickly and could be disintegrated satisfactorily 1 hour after the den was filled.

**Inlet Air Temperature.** Laboratory studies (4) had indicated that over the inlet air temperature range of 500° to 1000° F., little variation in composition of the product occurred within a product temperature range of 174° to 311° F., but a marked difference in particle size of the product was observed. Two series of pilot plant runs were made in which inlet air temperature was varied; in one feed rate was constant and in the other a constant product moisture content was attempted.

The results of the series at a constant feed rate of 2.2 pounds of wet superphosphate per minute are shown in Table I and Figure 4. Conversion decreased with increasing inlet air temperature but this effect is due primarily to the lower moisture content of the products dried at the higher temperatures. Total phosphorus pentoxide increased with increasing inlet air temperature, which also reflects the loss of moisture, and available phosphorus pentoxide content changed very little with inlet air temperature.

Another series of runs was made in which the feed rate was increased as inlet air temperature was increased, in an attempt to produce products of constant moisture content. This was not accomplished, but samples were taken in each run at various points throughout the length of the dryer so that the composition at the desired moisture content of 3% could be interpolated. Figure 5 shows the results of a typical run and indicates the method of interpolation to a constant moisture content. The appearance of this figure is very similar to those for batch runs made in the laboratory investigations (4), in which composition of sample was plotted against time of drying. A point of maximum conversion was noted at a moisture content of about 11% after 2 days of sample storage.

The results of all the runs in this series, shown in Table II and Figure 6, confirm those of the laboratory investi-

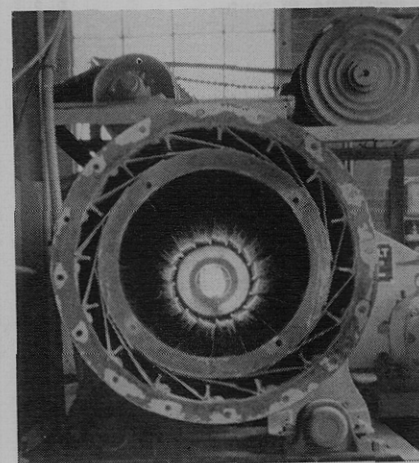


Figure 3. Discharge end of dryer

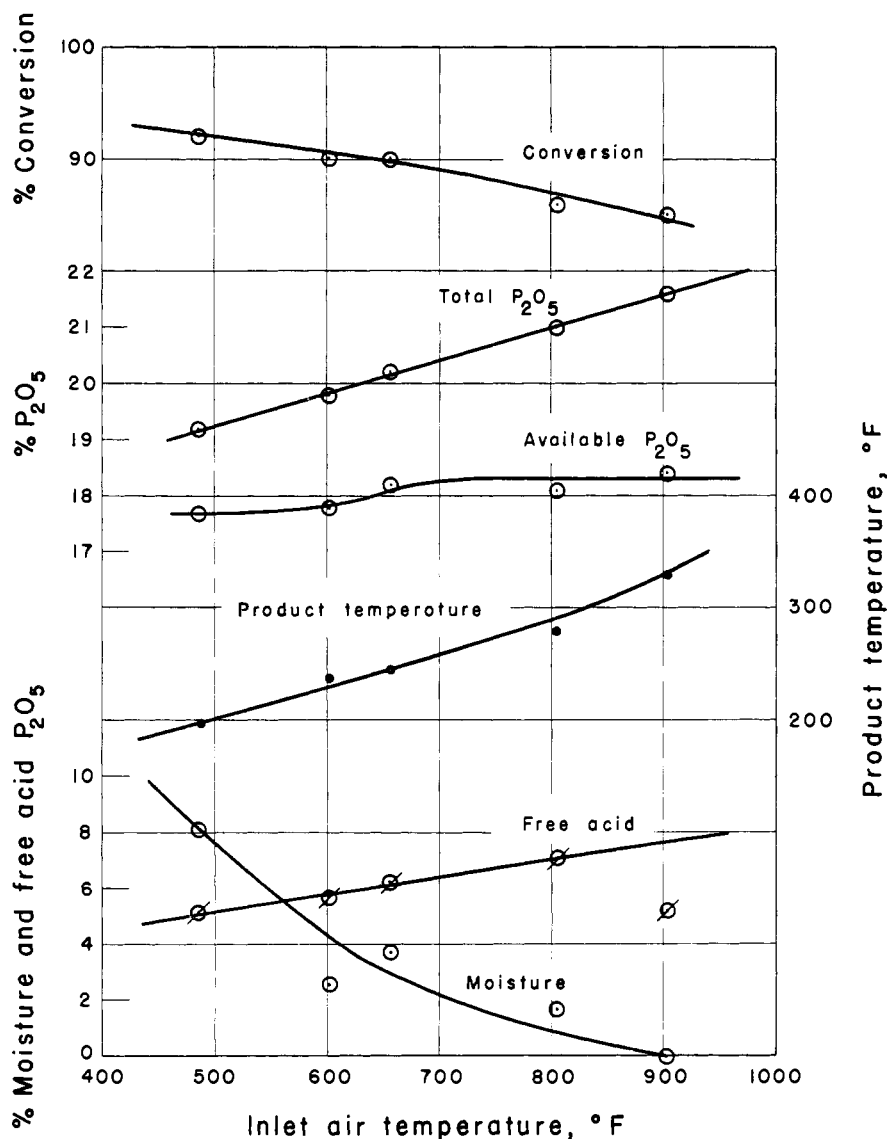


Figure 4. Effect of inlet air temperature at constant feed rate

Table I. Pilot Plant Runs at Various Inlet Air Temperatures and Constant Feed Rate

Operating conditions					
Inlet air temp., °F.	486	602	657	805	903
Feed rate, lb./min.	2.2	2.2	2.2	2.2	2.2
Product temp. at outlet of dryer, °F.	198	238	245	280	330
Exit air temp., °F.	177	205	220	256	287
Inlet air rate, lb./min.	7.9	6.9	7.2	6.8	6.3
Inlet air velocity through dryer bed, ft./sec.	0.57	0.52	0.56	0.56	0.54
Inlet air rate through dryer bed, c.f.m.	205	189	200	200	195
Fuel gas consumption, c.f.m. <sup>a</sup>	1.51	1.90	2.26	2.61	2.93
Residence time in dryer, min.	16.8	19.8	19.5	21.4	18.4
Dust collected in cyclone, % of dry solids processed	3.7	4.0	5.9	0.0	7.1
Product composition at 2 days					
Total P <sub>2</sub> O <sub>5</sub> , %	19.2	19.8	20.2	21.0	21.6
Citrate-insoluble P <sub>2</sub> O <sub>5</sub> , %	1.5	2.0	2.0	1.9	3.2
Available P <sub>2</sub> O <sub>5</sub> , %	17.7	17.8	18.2	18.1	18.4
Conversion, %	92	90	90	86	85
Free acid (as P <sub>2</sub> O <sub>5</sub> ), %	5.1	5.7	6.2	7.1	5.2
Moisture, %	8.1	2.6	3.7	1.7	0.0

<sup>a</sup> Cubic feet per minute at pressure equivalent to 11 inches of water.

gation (4)—for products dried to the same moisture content, inlet air temperature has little if any effect on composition of the product. This, of course, is true only if the product temperature is not too great, and, in the present series, the product temperature range was 233° to 304° F. for products dried to considerably less than 3% moisture. Data were not taken that would permit interpolation of product temperatures at 3% moisture, but these temperatures must have been appreciably lower than those at the discharge end of the dryer.

Even though the moisture contents of the final products made with variable inlet air temperatures shown in Table II were not constant, a maximum product temperature was noted at about 900° F. inlet air temperature. This confirms the findings of the laboratory batch runs, in which such a maximum was noted (4).

**Moisture Content.** As in the laboratory investigations, the most important single variable affecting conversion was moisture content of the dried sample. Figure 7 shows the conversion and available phosphorus pentoxide contents of all the 2-day samples from the pilot plant runs plotted against their moisture content. Although there is considerable scattering, due to the influence of other variables which were not constant, there is an unmistakable correlation showing increasing conversion as moisture increases. In spite of this, the available phosphorus pentoxide contents of the samples decrease slightly with increasing moisture content because of the dilution effect of the moisture. The point of maximum conversion is not so clearly defined as in the laboratory studies (4), and appears to be at a slightly higher moisture content—namely, 12 to 15%.

The conversion and available phosphorus pentoxide content of storage-cured samples made in the laboratory and the estimated conversion and available phosphorus pentoxide content of similar product made by storage curing in a large plant where conversion would not be quite complete are also shown.

As indicated in the previous study (4), the degree to which superphosphate should be dried depends on the use for which it is intended. For direct application, it would probably be better to dry to a low moisture content, so that available phosphorus pentoxide content would be high and handling properties better. For ammoniation or mixing, however, it would be better to dry to an intermediate moisture content and take advantage of the higher conversion; this assumes that facilities are available for drying the ammoniated or mixed fertilizer.

#### Particle Size of Product

The laboratory investigations had indicated that particle size of product could

be controlled by inlet air temperature (4). In the pilot plant runs, there was not so marked a difference in product particle size for the different inlet air temperatures. The average particle size of the product from all the runs was about 30% +4, 40% -4 +40, and 30% -40 mesh. At the higher inlet air temperatures, the -40-mesh fraction increased somewhat at the expense of the -4 +40-mesh fraction. Probably one reason why particle size was not influenced more by inlet air temperature in the pilot plant runs was that the feed to the dryer was not disintegrated so finely, and was not so uniform in size as in the laboratory studies. The +4-mesh material could be milled and screened to recover an additional -4 +40-mesh fraction, so that at least half of the final product would be in a particle size range that would permit it to be considered a granular product.

The bulk densities of the various size fractions were as follows: +4 mesh, 57 pounds per cubic foot; -4 +40 mesh, 63 pounds per cubic foot; and -40 mesh, 68 pounds per cubic foot.

#### Storage of Product in Bags

Superphosphate from the pilot plant runs, screened to -4 +40 mesh and containing about 5% moisture, was stored for 1 year in 80- or 100-pound bags of several types under pressure equivalent to a stack of 12 bags. Identical tests were made with superphosphate mixed with 1% of slaked lime and superphosphate containing no lime. Storage-cured superphosphate was used as a control for one type of bag.

The best type of bag tested was a polyethylene-lined four-ply paper bag; these bags were in good condition after the tests for both limed and unlimed products. Double asphalt-laminated four-ply paper bags were in fair condition for both limed and unlimed products. Single asphalt-laminated three-ply paper bags failed with unlimed product, but were in fair condition with the limed product. Bags made of paper as an inner layer and burlap as an outer layer, cemented together with asphalt, failed with both limed and unlimed products.

The storage-cured superphosphate did not seriously deteriorate the single asphalt-laminated three-ply paper bags.

It is concluded that the quick-cured superphosphate is more deleterious to bags than storage-cured superphosphate, and that polyethylene-lined or double asphalt-laminated bags should be used for its storage.

#### Conclusions

It was demonstrated on a pilot plant scale that normal superphosphate made with 55% sulfuric acid can be quick-

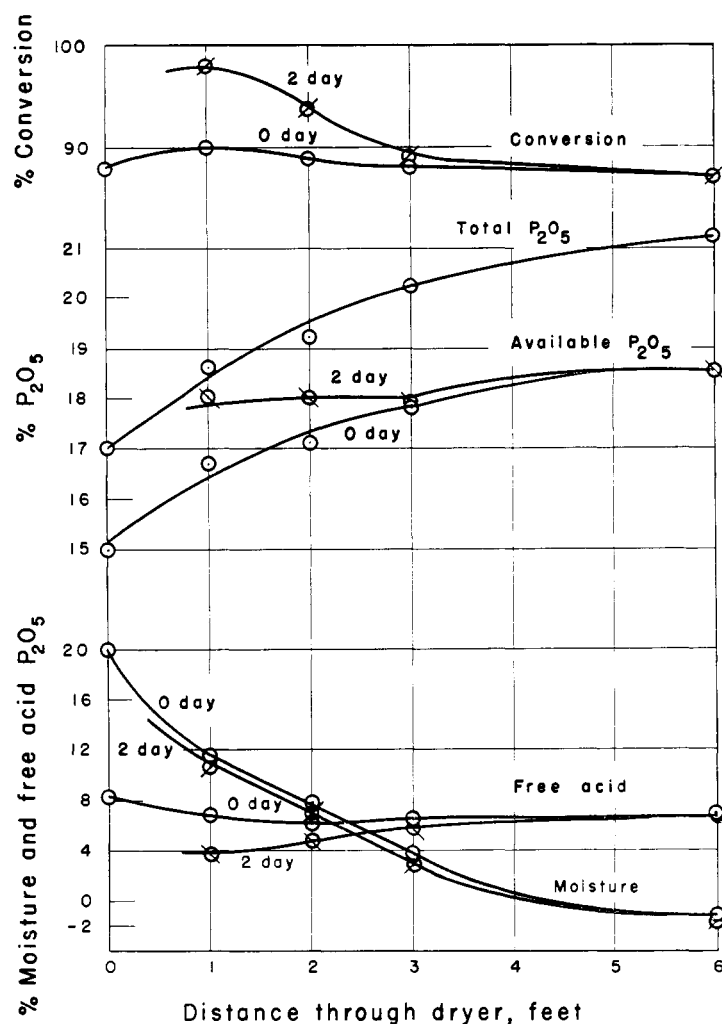


Figure 5. Change in composition of superphosphate during passage through dryer

Table II. Pilot Plant Runs at Various Inlet Air Temperatures and Constant Product Moisture

Operating conditions						
Inlet air temp., ° F.	400	517	604	801	1009	1141
Feed rate, lb./min.	0.8	0.8	1.6	2.1	3.1	4.1
Product temp. at outlet of dryer, ° F.	233	256	266	292	304	280
Exit air temp., ° F.	203	233	222	267	274	263
Inlet air rate, lb./min.	7.9	7.6	7.3	6.7	6.3	5.7
Inlet air velocity through dryer bed, ft./sec.	0.60	0.58	0.57	0.54	0.52	0.46
Inlet air rate through dryer bed, c.f.m.	216	210	205	194	189	167
Fuel gas consumption, c.f.m. <sup>a</sup>	1.35	1.72	2.06	2.84	3.25	4.00
Residence time in dryer, min.	—	—	25.5	—	19.6	—
Product composition at 2 days						
Total P <sub>2</sub> O <sub>5</sub> , %	20.0	20.5	20.9	21.7	21.2	21.2
Citrate-insoluble P <sub>2</sub> O <sub>5</sub> , %	1.8	2.0	2.2	3.3	2.8	2.8
Available P <sub>2</sub> O <sub>5</sub> , %	18.2	18.5	18.7	18.4	18.4	18.4
Conversion, %	91	90	89	85	87	87
Free acid (as P <sub>2</sub> O <sub>5</sub> ), %	5.8	6.3	6.3	6.4	6.6	6.4
Moisture, %	3.5	1.3	1.5	-0.5	-1.1	1.4
Interpolated composition of product at 2 days having 3.0% moisture content						
Total P <sub>2</sub> O <sub>5</sub> , %	20.5	19.4	20.7	20.0	20.2	20.4
Citrate-insoluble P <sub>2</sub> O <sub>5</sub> , %	2.1	1.6	2.2	2.2	2.2	2.3
Available P <sub>2</sub> O <sub>5</sub> , %	18.4	17.8	18.5	17.8	18.0	18.1
Conversion, %	92	92	89	89	89	88
Free acid (as P <sub>2</sub> O <sub>5</sub> ), %	6.2	6.3	6.0	4.8	5.8	6.0
Moisture, %	3.0	3.0	3.0	3.0	3.0	3.0

<sup>a</sup> At pressure equivalent to 11 inches of water.

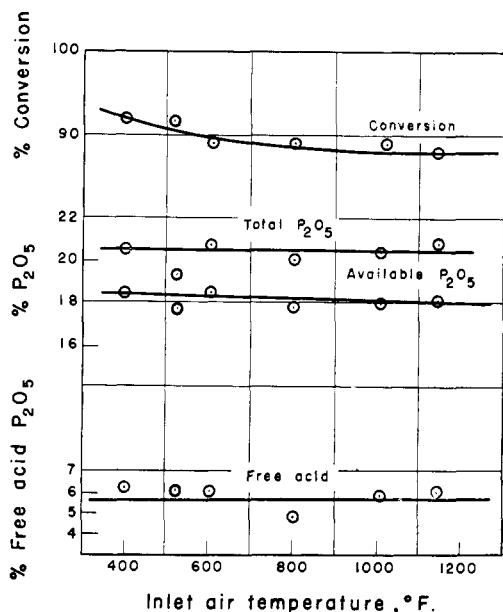


Figure 6. Effect of inlet air temperature at product moisture of 3%

cured continuously in a Roto-Louvre dryer to give an available phosphorus pentoxide content equivalent to that made by the storage-curing process, although phosphorus pentoxide conversion was not so high. There was no evidence of build-up of material in the dryer. The quick-cured superphosphate

could be screened and milled so that about half of it would be in the  $-4 + 40$ -mesh size range.

A product that should be especially suitable for ammoniation and mixing can be made by drying the superphosphate to an intermediate moisture content and storing for 2 or 3 days.

A particular advantage of the quick-curing process is its ability to use sulfuric acid as low as 55% in concentration. This suggests the possibility of using spent acids from various processes without reconcentration.

A patent on the process has been applied for and assigned to the Iowa State College Research Foundation.

#### Acknowledgment

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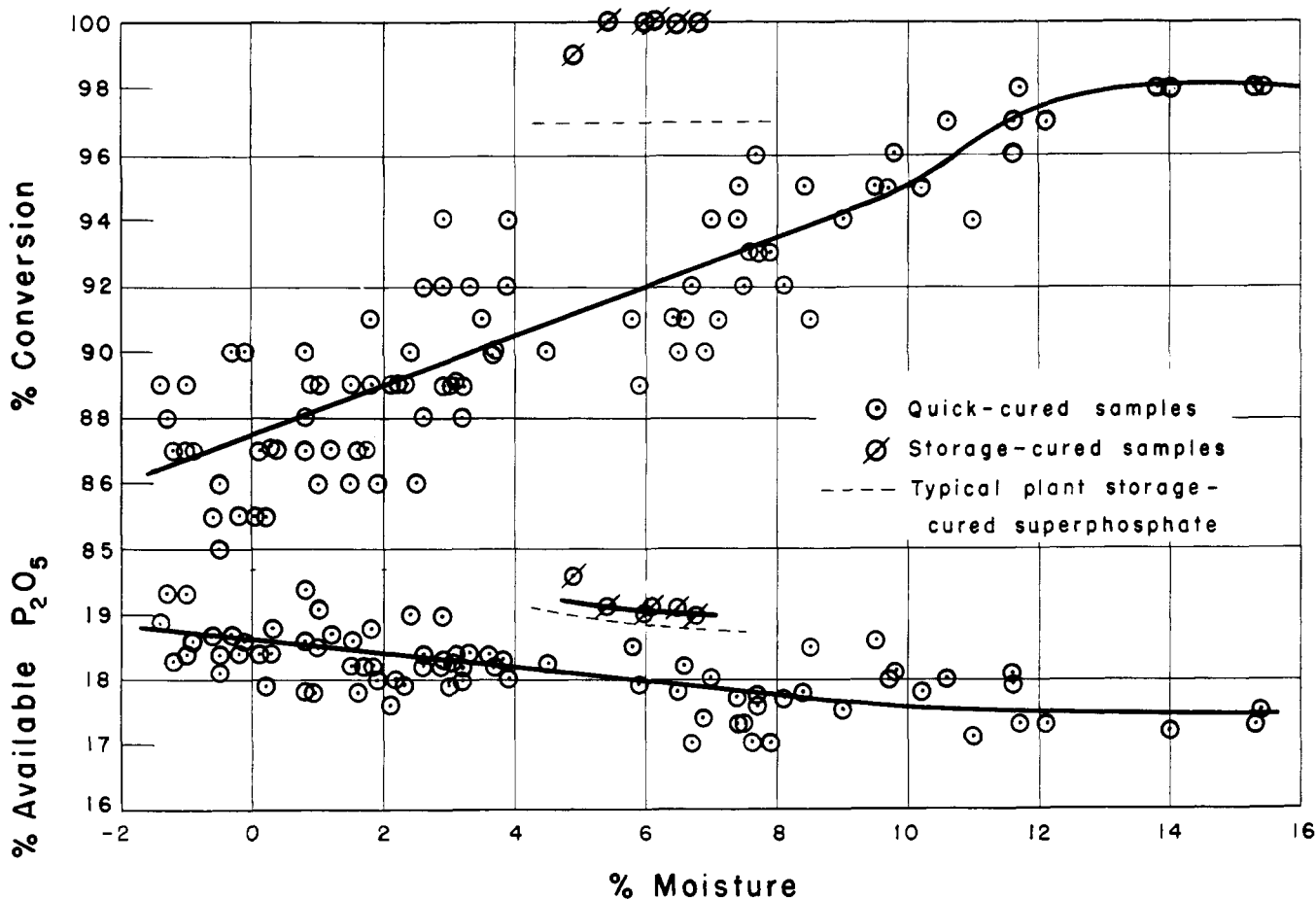


Figure 7. Effect of moisture content on composition of 2-day samples