

Diammonium Phosphate Fertilizer from Wet-Process Phosphoric Acid

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A process for making diammonium phosphate fertilizer from ammonia and wet-process phosphoric acid was developed on a pilot plant scale. Acid containing about 24% phosphorus pentoxide was ammoniated to a pH of about 5. The resultant precipitate, which consisted principally of iron and aluminum phosphates, was separated from the solution by filtration. The filtrate and additional ammonia were fed to a continuous vacuum crystallizer to produce diammonium phosphate. The crystals were centrifuged, mixed with filter cake, and dried to produce a granular fertilizer containing 18% nitrogen and 47% phosphorus pentoxide. Alternatively, the crystals and filter cake may be dried separately to produce fertilizer materials of about 20-48-0 and 9-40-0 grades, respectively. Potassium chloride and ammonium nitrate or concentrated superphosphate may be mixed with the crystals and filter cake to produce a variety of grades of granular fertilizers such as 18-18-18 or 9-27-27. A major portion of the work was concerned with the development of satisfactory conditions for the precipitation step and for crystallizing diammonium phosphate in the system ammonia-phosphoric acid-sulfuric acid-water.

MONOAMMONIUM AND DIAMMONIUM PHOSPHATES are attractive for fertilizer use because of their favorable physical properties and high concentration of plant food, and because they provide an economical method for fixing ammonia in solid form. In the latter connection, diammonium phosphate is the more attractive because it fixes twice as much ammonia per unit of phosphorus pentoxide. Greenhouse tests by MacIntire and coworkers (4) showed diammonium phosphate to be an effective fertilizer and comparable to monoammonium phosphate as a source of nitrogen and phosphorus pentoxide. A recent review of agronomic data (6) shows that monoammonium phosphate and diammonium phosphate are particularly suited for use on alkaline soils and on acid soils when their residual acidity is neutralized with limestone.

Monoammonium phosphate fertilizer has been made commercially in Canada since 1931 and in the United States since 1946 from ammonia and wet-process phosphoric acid by a process described by Atwell (7). That process is unattractive for production of diammonium phosphate because of the high vapor pressure of ammonia over diammonium phosphate solutions, which can result in excessive loss of ammonia at the temperatures encountered.

Thompson and coworkers (8) have described a process for making diam-

monium phosphate from electric furnace phosphoric acid. By crystallizing diammonium phosphate from acidic mother liquor (mole ratio ammonium-phosphoric acid about 1.6; pH about 6.0), they were able to eliminate any need for recovering ammonia from the crystallizer vapors. The process was carried out satisfactorily in an atmospheric saturator and in a vacuum crystallizer. Attempts to substitute impure wet-process acid for electric furnace acid were unsuccessful in both types of equipment (8). The impurities in the wet-process acid precipitated in an unfilterable form and caused eventual gelling of the solution.

In the present work, innovations were developed for separating and handling the impurities in wet-process acid to overcome their interference with the process. The work resulted in the development of a process that consists in ammoniating wet-process acid, filtering off the resultant precipitate, and feeding the filtrate and additional ammonia to a continuous vacuum crystallizer. Diammonium phosphate is crystallized from acidic mother liquor, as in the process described by Thompson, and separated by centrifuging. The damp crystals from the centrifuge are mixed with the filter cake, which contains a significant proportion of the phosphorus and some nitrogen, and then are dried in a rotary dryer. The mixture of crystals and filter cake granulates on drying, so that the particle size of the product can be controlled without regard for the size of the crystals.

The capacity of the pilot plant was about 1 ton per 24 hours. A flow diagram of the process is shown in Figure 1.

Raw Materials

The wet-process acid used in the experimental work was made in a pilot plant from uncalcined Florida phosphate rock. Typical compositions of the rock and the acid made from it are as follows:

	Composition, Weight %			
	P ₂ O ₅	SO ₃	F	Fe ₂ O ₃
Rock	32.6	...	3.8	1.4
Acid	24.3	2.58	2.06	1.23

	Acid Insoluble			
	Al ₂ O ₃	CaO	H ₂ O	
Rock	1.8	47.3	6.0	0.5
Acid	1.29	0.26	...	59.4

Precipitation

The reaction of wet-process acid with ammonia causes the precipitation of most of the iron and aluminum and part of the fluorine. The precipitate contains complex orthophosphates of iron, aluminum, and ammonia. Exploratory work showed that precipitation of the impurities under the conditions that exist in the crystallizer always resulted in materials that could not be separated readily from the mother liquor by settling, filtration, or centrifuging. Therefore, it appeared desirable to precipitate those materials in a separate operation before crystal-

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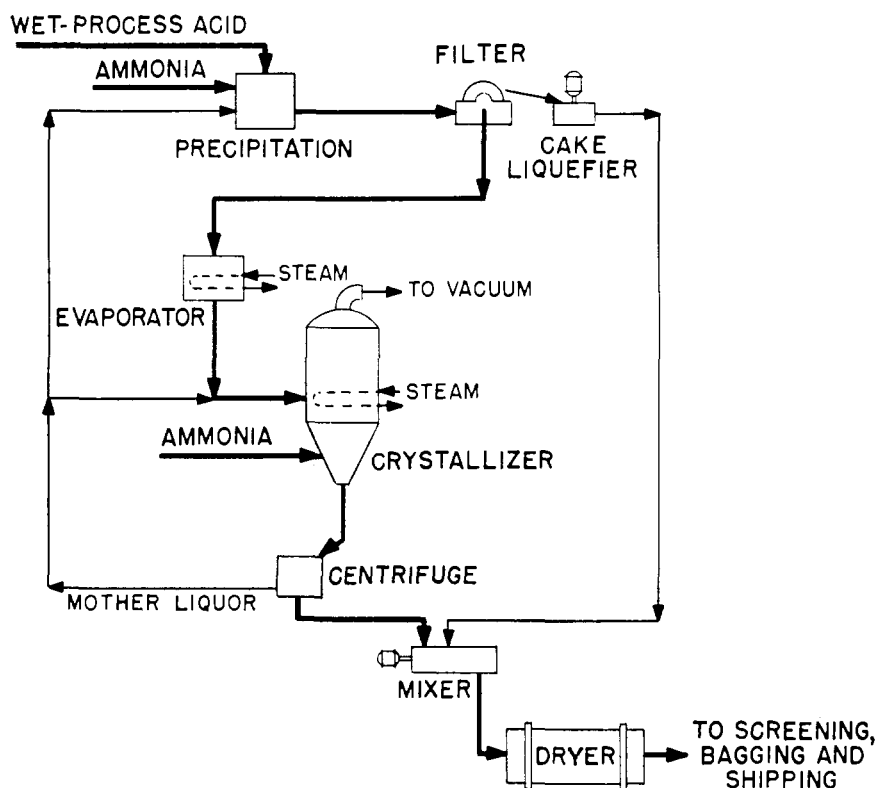


Figure 1. Flow diagram of process for making diammonium phosphate fertilizer from wet-process phosphoric acid

lization of the diammonium phosphate. Both batch and continuous precipitation were used in the laboratory tests of the effects of variables. Batch precipitation was used in the pilot plant tests. In the pilot plant, gaseous ammonia was added through an open-end pipe to a batch of wet-process acid contained in a cylindrical, cone-bottom tank without agitator until the pH reached 5.0. The conditions were selected on the basis of laboratory tests of the effect of variables discussed later. The batch was then filtered in a rotary vacuum filter. Typical data are shown in Table I.

Effect of Degree of Ammoniation. Laboratory tests were made in which wet-process phosphoric acid was ammoniated to various pH values, using batch or continuous ammoniation, to determine the effect of degree of ammoniation on the degree of precipitation and on the filtering rate. Batchwise ammoniation tests were made in a beaker in which 3 liters of acid were ammoniated by introducing gaseous ammonia through a glass tube until the desired pH was reached. The continuous ammoniation tests were made in a 3-liter wide-mouthed flask provided with a side-outlet tube. Ammonia and wet-process phosphoric acid were introduced separately through two glass tubes located near the bottom of the flask. The slurry left the flask continuously through the side outlet, and the first 9 liters were discarded before making filtering tests to ensure that steady-state conditions had been reached. No mechanical agitation

was used in either type of ammoniator. The only agitation was that resulting from natural turbulence. Filtering tests were made in replicate with a test-leaf vacuum filter. The data are shown in Table II.

With batch ammoniation the precipitation of iron and aluminum leveled off at about 97% by the time pH 4.5 was reached. Data on that point were not obtained for continuous ammoniation. In the case of batch ammoniation there was a progressive decrease in filtering rate with increase in pH from 4.5 to 6.0, whereas continuous ammoniation did not follow this trend, but a maximum filtering rate was obtained at pH 5.5.

Acid Concentration. The effect of

acid concentration on the filtering rate of slurries prepared by batch and continuous ammoniation of the acid at a rate of 10 pounds of ammonia per hour per cubic foot of liquor in vessel is shown in Figure 2. The highest filtering rate was obtained with slurry prepared from acid containing about 23% phosphorus pentoxide. Acid concentrations in the range 26 to 30% phosphorus pentoxide had a decided detrimental effect on filtering rate, for both batch and continuously ammoniated slurries. It therefore would be desirable to avoid operating in that range. The usual concentration of wet-process acid produced commercially is around 27% phosphorus pentoxide.

Temperature of Precipitation. In most of the laboratory and pilot plant work the thermal conditions were such that the heat of neutralization brought the solution to the boiling point in the course of ammoniation. Little precipitation occurred during the early stages of batch ammoniation, but eventually a point was reached where heavy precipitation occurred. Significant variations in the filtering rates obtained with slurries in which heavy precipitation was controlled to take place at different temperatures are shown in Table III. Decreasing the temperature only slightly below boiling caused a significant decrease in the filtering rate.

Rate of Ammoniation. The effect of rate of batch ammoniation, expressed as pounds of ammonia per hour per cubic foot of acid, is shown in Figure 3. This variable was not fully investigated in continuous ammoniation, although a few points obtained for the latter do not deviate much from the curve in Figure 3. Although increase in ammoniation rate caused a decrease in filtering rate, favorable filtering rates were obtained at all ammoniation rates below 15 pounds of ammonia per hour per cubic foot. A rate of 5 to 10 pounds of ammonia was used in most of the pilot plant work. Choice of a specific rate would be deter-

Table I. Pilot Plant Data on Precipitation and Filtration

Initial temperature of wet-process acid, ° F.	150
Size of batch, gal.	75
Ammonia rate, lb. NH ₃ /(hr.)(cu. ft.)	5
Temperature at time of heavy precipitation, ° F.	220 ^a
Terminal pH	5.0
Filtering rate, gal. filtrate/(hr.)(sq. ft. submerged)	100 ^b
Wash water rate, lb./lb. slurry	0.18
Ratio of wet filter cake to filtrate, by weight	0.234

Composition of Materials, Weight %

	P ₂ O ₅	NH ₃	SO ₃	F	Fe ₂ O ₃	Al ₂ O ₃	CaO	H ₂ O
Phosphoric acid	24.3	...	2.58	2.06	1.23	1.29	0.26	59.4
Slurry to filter ^c	25.1	9.2	2.67	2.13	1.27	1.33	0.27	48.5
Filtrate plus wash	21.3	8.3	2.55	1.03	Trace	Trace	Trace	57.8
Filter cake	20.0 ^c	5.3	0.88	5.00	5.58	5.58	1.17	50.0

^a Boiling.

^b Eimco Corp. rotary vacuum filter, 18-inch diameter by 12 inches; monofilament polyethylene cloth; vacuum, 10 to 14 inches Hg; filtering temperature, 180° F.

^c About 50% of P₂O₅ was water-soluble.

Table II. Effect of Degree of Ammoniation^a

Terminal pH	Degree of Precipitation, % ^b			Filtering Rate ^c , Gal. Filtrate/(Hr.)(Sq. Ft.)	
	Fe ₂ O ₃	Al ₂ O ₃	F	Batch	Continuous
				ammoniation	ammoniation
3.9	91.0	75.1	15.0
4.5	97.3	96.0	54.0	150	115
5.0 ^d	97.8	96.9	54.4	135	115
5.5	98.1	91.6	65.5	120	185
6.0	97.5	95.8	71.4	90	170

^a Acid concentration, 24% P₂O₅; ammoniation rate, about 10 lb. NH₃/(hr.)(cu. ft.); unagitated; boiling.

^b Data apply to batch ammoniation only.

^c Filter medium, polyfilament saran; filter vacuum, 15 inches Hg; test leaf; single cycle; replicate tests.

^d 10% of P₂O₅ precipitated at pH 5.0.

Table III. Effect of Temperature of Precipitation^a

Temp. of Precipitation, ° F.	Ammoniation Rate, Lb. NH ₃ /(Hr.)(Cu. Ft.)	Filtering Rate ^b , Gal. Filtrate/(Hr.)(Sq. Ft.)
220 ^c	10	175
215	10	116
170	10	19
220 ^c	5	226
170	5	31

^a Acid concentration, 23% P₂O₅; batch ammoniation; unagitated; terminal pH, 5.

^b Filter medium, polyfilament saran; filter vacuum, 15 inches Hg; test leaf; single cycle.

^c Boiling.

mined by costs of ammoniation tanks as compared with costs of filters.

Agitation. In the course of the experimental work on precipitation it became evident that vigorous agitation was detrimental to the filterability of the precipitate. To obtain a quantitative indication of the effect of agitation, a test was run in which 3 liters of wet-process phosphoric acid containing 24% phosphorus pentoxide was ammoniated batchwise to pH 5.0 at a rate of 5 pounds of ammonia per hour per cubic foot. The ammoniation vessel was a glass battery jar 7 inches in diameter. The filtering rate of the resulting slurry, determined on a test-leaf filter, was 160 gallons of filtrate per hour per square foot. A motor-driven propeller was then placed in the slurry and four 1-inch baffles were installed in the jar. After agitation for 30 minutes, the filtering rate had dropped to 28 gallons per hour per square foot. Aging of the slurry for 30 minutes without agitation had no effect on filterability.

As the natural turbulence of ammonia sparging and boiling was adequate for the reaction, mechanical agitation was avoided in the pilot plant ammoniator. The action of a centrifugal pump also was detrimental to the filterability of the slurry, so gravity flow was used rather than pumping to transfer the slurry from the ammoniator to the pilot plant filter.

Filtration

The variables discussed were evaluated on the basis of test-leaf filtering tests using polyfilament saran and single-cycle operation, without washing. That procedure was convenient and gave good reproducibility and good correlation. However, it was found that the polyfilament saran was subject to blinding when used on the pilot plant rotary vacuum filter. Monofilament polyethylene was not subject to blinding and, therefore, was the preferred medium in the pilot plant and also for design of a large plant. Other monofilament materials may be equally satisfactory. Data obtained with monofilament polyethylene, as presented in Table I, are therefore more reliable for purposes of design extrapolation than those presented elsewhere in this report.

In the pilot plant the filter cake was washed on the filter by means of water sprays. Although the filter cake is not discarded in the process, but is included in the final product, it is desirable to remove as much of the water-soluble phosphorus pentoxide as can be readily removed by washing, since it is economically desirable to ammoniate it further in the crystallizer.

The filter cake had a tendency to crack very soon after emerging from the slurry, and the cracking hampered washing. The use of a drag cloth improved washing by 15 to 20%, as did the use of a smoothing roller to seal the cracks. The residual moisture in the cake was around 50% by weight, and little improvement in dewatering could be obtained by either drag cloth or roller.

When mother liquor was recycled to the precipitation step (see crystallization) satisfactory precipitation and filtration were maintained when the variables discussed in the foregoing were held within the recommended ranges.

Laboratory tests of continuous ammoniation gave results that were comparable to those obtained in batch, but continuous ammoniation was not tested in the pilot plant. It is believed, however, that continuous ammoniation would be the preferred method in most plants and that no serious problems would be

encountered in its use. It is planned to test it in the pilot plant.

Crystallization

In the original process, in which electric furnace acid was used, the mole ratio of ammonia to phosphoric acid in the mother liquor was controlled by controlling the pH. Thompson *et al.* (8) present data on the relation of pH to mole ratio for saturated solutions in the system ammonia-phosphoric acid-water. When wet-process acid is used, the system is altered by other materials which are present in such acid. The most important of these is sulfate ion, practically none of which is removed in the precipitation step. The crystallizer system therefore would be essentially the system ammonia-phosphoric acid-sulfuric acid-water. The data available from the literature on that system in the diammonium phosphate range (3) were inadequate for purposes of crystallizer control and operation. Therefore, tests were run in the pilot plant crystallizer using pure materials to determine the relationship between pH and the composition of the mother liquor, the solid phases in equilibrium with the solution, and the ratio of ammonia to water in the vapors.

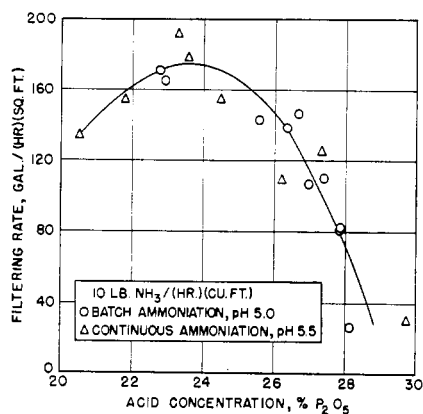


Figure 2. Effect of acid concentration on filtering rate of slurry

Those tests were run in the following manner. A mother liquor having a given pH was prepared in the crystallizer by feeding pure phosphoric acid and sulfuric acids and ammonia and evaporating water until crystals were present in appreciable amounts. The feed of acids and ammonia was then stopped, and water was fed to the crystallizer while being removed at the same rate by evaporation. The vapor from the crystallizer was sampled to determine ratio of ammonia to water in the vapor at the given pH while water was being fed. Samples of mother liquor and crystals were taken at the beginning and end of the period of vapor sampling. The pH was then changed by adding ammonia, and the procedure was repeated.

The results are shown in Table IV. Diammonium phosphate and ammo-

Table IV. System Ammonia-Phosphoric Acid-Sulfuric Acid-Water at 140° F.

Composition of Solution				Mole ratio NH ₃ H ₂ PO ₄ (cor. for H ₂ SO ₄)	pH	Composition of Vapor, Ratio NH ₃ H ₂ O by Weight	pH of 0.1M Solution of Solid Phase
Grams/100 Grams Saturated Solution							
NH ₃	H ₃ PO ₄	H ₂ SO ₄					
13.91	42.05	10.20	1.43	5.5	0.0029	4.5	
14.02	43.75	9.30	1.43	5.6	0.0026	5.4	
13.44	31.4	14.60	1.55	5.8	0.0076	7.7	
13.60	30.82	14.63	1.60	6.0	0.008	7.7	
13.10	28.01	14.51	1.66	6.15	0.012	7.7	
12.95	30.81	10.01	1.78	6.42	0.032	7.8	
12.63	28.86	9.76	1.85	6.62	0.03	7.8	

Table V. Pilot Plant Data on Crystallizer Operation

Temperature, ° F.	140
pH of slurry	6.0
Crystals in suspension, volume %	35-40
Crystallization rate, lb./hr.(cu. ft. mother liquor)	17-18
Loss of NH ₃ in vapors, % of input	2.6

	Composition of Materials, Weight %						
	P ₂ O ₅	NH ₃	SO ₃	F	Fe ₂ O ₃	Al ₂ O ₃	H ₂ O
Feed liquor ^a	27.1	10.5	3.2	1.3	Trace	Trace	46.5
Mother liquor	22.3	13.6	10.7	2.5	0.11	0.15	40.0
Crystals	46.1	24.0	5.6	2.3	Trace	Trace	3.3

^a Filtrate plus wash (Table I) preconcentrated by atmospheric evaporation.

mium sulfate were found to be the solid phases in equilibrium with the solution at pH 5.8 through 6.62 (Table IV). Monoammonium phosphate replaced diammonium phosphate as a solid phase at and below pH 5.6. The tests indicated that, from the standpoint of composition of the crystals, operation should be at or above pH 5.8. With regard to the ammonia-water ratio in the vapors, there was a fourfold increase in going from pH 6.0 to pH 6.4. It was convenient to operate the pilot plant crystallizer near pH 6.0, which provided an adequate margin to avoid monoammonium phosphate formation and prevent significant loss of ammonia in the vapors. Operation at pH 6.0 resulted in a loss of about 2.5% of the total ammonia. If desired, the feed liquor can be preconcentrated to around 26% phosphorus pentoxide by atmospheric evaporation without increasing the over-all loss of ammonia.

A point of difference between the system shown in Table IV and the system resulting from the use of wet-process acid is that the latter contains fluorine. The presence of fluorine did not, however, interfere with crystallizer operation. Calcium fluoride was identified as inclusions in the crystal product.

The pilot plant crystallizer, shown in Figure 4, was a continuous vacuum-type unit with internal agitation and without any positive classifying action. It was a cylindrical cone-bottomed vessel 18 inches in diameter and 6 feet in over-all height. Vapors were drawn from a top-entering duct through a cyclone-type mist trap to a barometric condenser. Vacuum was maintained with a two-stage steam jet ejector. Steam coils were provided on the inside of the

crystallizer to supply the necessary supplemental heat for evaporation. Feed liquor, sometimes preconcentrated by atmospheric evaporation to about 26% phosphorus pentoxide, was introduced at a constant rate, and gaseous ammonia was introduced at a manually controlled rate as required to maintain the pH at 6.0. The temperature was held at 140° F., which required an absolute pressure of about 4.5 inches of mercury. Crystal slurry was withdrawn at 20-minute intervals by means of a vacuum tank and was centrifuged. Production rates were around 70 pounds of crystals per hour (17 to 18 pounds per hour per cubic foot of mother liquor). Typical data from the pilot plant crystallizer are shown in Table V.

Because some precipitate passed the filter and a small proportion of the iron and aluminum was not precipitated in the prior ammoniation step, there always was some precipitate present in the crystallizer. Generally, the precipitate

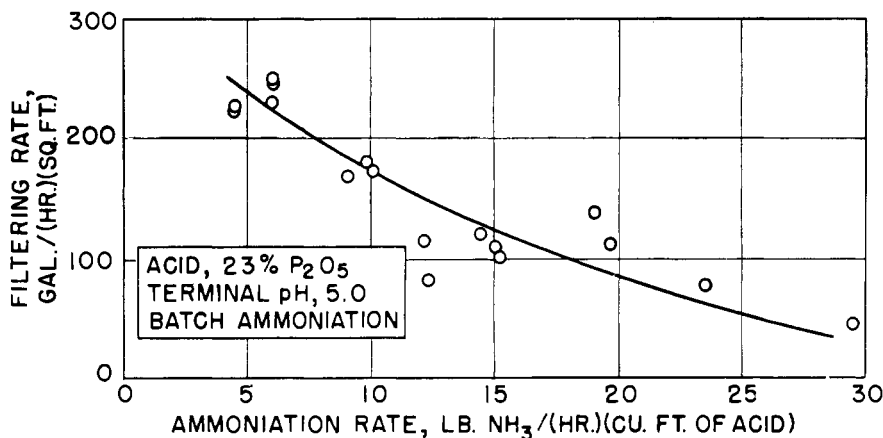
caused no trouble in the pilot plant and remained below 0.3% through incidental removal with the diammonium phosphate crystals. At times, however, difficulty was encountered in centrifuging. It was found that this difficulty could be avoided by continuously withdrawing a portion of the mother liquor and recycling it to the raw acid prior to ammoniation. A recycle ratio of 1 part of phosphorus pentoxide as mother liquor to 10 parts of phosphorus pentoxide as acid was found to be adequate. The data in Tables I and V include such recycling.

Centrifuging and Drying

The pilot plant centrifuge was a batch-type machine with a perforated basket 18 inches in diameter by 9 inches deep. Stainless steel 25-mesh screen wire was used to line the basket, which had a speed of 1800 r.p.m. The average batch was 50 pounds. It was not convenient to obtain design data with the pilot plant centrifuge because of lack of control of feeding and the time required for stopping the basket. Centrifuging tests were run in a smaller centrifuge 4.75 inches in diameter. With a cake thickness of $\frac{3}{8}$ inch and a centrifugal force of 850 × G, the moisture was brought to between 2.5 and 3% in 15 seconds.

It was desired to mix the crystals with the filter cake obtained earlier in the process because the filter cake contained around 18% of the phosphorus pentoxide introduced as phosphoric acid and also

Figure 3. Effect of ammoniation rate on filtering rate



some nitrogen. It was found that the filter cake could be liquefied readily by agitation, such as obtained with a high-speed propeller. That property simplified the handling of the filter cake. In the pilot plant, damp crystals from the centrifuge were fed by scoop to a small tank equipped with a propeller agitator. Liquefied filter cake was metered into the tank through an orifice. The mixture overflowed through a pipe which led into a rotary dryer, 1 foot in diameter by 8 feet long. Typical data from the dryer operation are shown in Table VI. The mixture of crystals and filter cake in the production ratio—about 2 pounds of crystals to 1 pound of wet cake—formed agglomerates in the feed end of the dryer, which resulted in a granular product in the 6- to 20-mesh range and permitted the use of relatively high gas velocities without loss of product. No loss of ammonia was detected by analysis of the material as long as the temperature of the material remained below 200° F. and the inlet gas temperatures did not exceed 550° F.

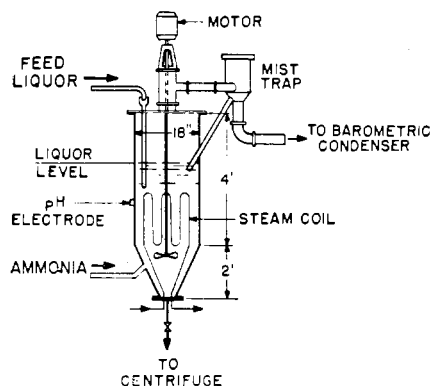


Figure 4. Pilot plant vacuum crystallizer

It may be desirable in certain instances to produce crystals without admixture of filter cake—for example, to make fertilizers that are completely water-soluble. In such cases the filter cake, produced as a by-product, should be useful as a phosphatic supplement in the preparation of mixed fertilizers. Drying tests made with filter cake gave results that were comparable to those obtained with mixtures of filter cake and crystals in terms of temperatures and gas rates per pound of water removed. As in the case of the mixture, the material granulated in the dryer, and there was negligible dust loading of the exit gases. The dried cake contained about 9% nitrogen and 40% phosphorus pentoxide, about 90% of which was citrate-soluble and 50% was water-soluble. It could be readily reduced to powder by crushing. Drying tests made with unmixed crystals showed that the maximum allowable inlet gas temperature was about 400° F. as compared with about 550° F. allowable for the mixture. The few per cent

Table VI. Pilot Plant Data on Drying^a

Lb. filter cake/lb. crystals (wet basis)	0.6
Moisture in feed, %	20
Temperature, ° F.	
Inlet gas	550
Exit gas	200
Exit material	180
Drying gas velocity, ft./sec.	7
Drying gas rate, lb./lb. material (dry basis)	4.3
Retention time, min.	20
Screen analysis ^b , %	
+4 mesh	0.5
-4 +35 mesh	81.4
-35 mesh	18.1

Composition of Product, Weight %

Total	P ₂ O ₅		N	SO ₃	F	Fe ₂ O ₃	Al ₂ O ₃	CaO	H ₂ O
	Available	W.S.							
47.1	47.0	42.6	18.0	4.9	3.9	2.3	2.4	0.5	0.7

^a Natural gas, cocurrent; 1-foot diameter by 8 feet long; 12 flights extending to within 2 feet of feed end; speed, 25 r.p.m.; slope, 2%.
^b Includes crushed oversize, amounting to 25%.

of associated impurities caused some agglomeration of the crystals during drying. The analysis was about 20% nitrogen and 48% phosphorus pentoxide.

Materials of Construction

All of the equipment in the pilot plant was made from stainless steel, with the exception of the dryer, which was made from aluminum. Corrosion tests run in the laboratory showed, however, that plain carbon steel should be suitable as a material for plant construction for the crystallizer, centrifuge, and dryer. The filter should be constructed of austenitic stainless steel (300 series) to permit periodic washing of the cloth with phosphoric acid. The results of corrosion tests using a slurry of mother liquor and crystals are shown in Table VII. That part of the plant required to handle phosphoric acid—namely, the acid storage tanks and the ammoniator—should be made from A.I.S.I. Type 316 or 317 stainless steel. Rubber-lined steel is an alternative for construction of storage tanks.

Discussion

Estimates indicate that the production of diammonium phosphate from ammonia and wet-process phosphoric acid should be attractive economically. The principal requirements of the process are shown in Table VIII.

The composition of the phosphoric acid will affect the proportion of filter cake in the product as well as the filter requirements. The acid used in the experimental work was slightly less pure than most commercially produced acid made from Florida rock. Therefore, it is probable that phosphoric acid produced from the usual grades of Florida rock would be satisfactory in the process. The use of acid made from western rock should be preceded by some experi-

mental work, since less standardization has occurred in the western phosphate industry and less is known concerning rock composition. Laboratory tests with a commercially produced wet-process acid made from western rock indicated that no special problems would be involved when such acid is used in the process.

The phosphorus pentoxide in the product is practically all citrate-soluble and around 90% of it is water-soluble, as determined by official methods of analysis.

Bag-storage tests were made according

Table VII. Corrosion Rates in Diammonium Phosphate Mother Liquor^a

Material	Corrosion Rate, Mils/Year
A.I.S.I. Type 304 stainless steel	0.05
A.I.S.I. Type 430 stainless steel	0.02
A.I.S.I. Type 410 steel	0.08
A.I.S.I. Type 502 steel	0.80
Mild steel	0.20
Duriron	Nil
Everdur 1010	1.5

^a Laboratory tests, 1 month's duration, nonaerated, 140° F.

Table VIII. Requirements for Diammonium Phosphate Fertilizer from Ammonia and Wet-Process Acid

Item	Requirement per Ton of Product
Phosphoric acid (100% H ₃ PO ₄ basis)	0.64 ton
Ammonia	0.22 ton
Steam, for evaporation	1.00 ton
Cooling water, to barometric condenser	12.70 tons
Heat, to dryer	716,000 B.t.u.
Electric power, miscellaneous	30 kw.-hr.



Plant for making diammonium phosphate fertilizer from wet-process phosphoric acid

to a method described previously (5). The results showed that the granular product made from mixtures of crystals and filter cake remained free flowing after 6 months in five-ply plain paper bags. No loss of ammonia in long-term bag storage could be detected by chemical analysis, nor could the odor of ammonia be detected over the material. The product that was tested was 88% minus 6 plus 35 mesh and 12% minus 35 plus 60 mesh. The moisture was 0.7% on bagging.

The addition of other fertilizer materials such as ammonium nitrate, potassium chloride, or concentrated superphosphate during the mixing of the diammonium phosphate crystals and filter cake offers attractive possibilities of making granular high-analysis fertilizers such as 18-18-18, 15-30-15, and 9-27-27. The compatibility of pure diammonium

phosphate with common fertilizer materials has been discussed in an earlier paper from this laboratory (7). Although the early work indicated that the compatibility with ammonium nitrate was limited to nitrogen-phosphorus pentoxide ratios below 0.75, that point was found to be erroneous. Beeson (2) has discussed the reaction between diammonium phosphate and limestone in the presence of considerable moisture, which results in loss of ammonia. It is planned to make tests of the storage properties of several representative mixtures containing common fertilizer materials and diammonium phosphate made from wet-process acid.

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POTASH DETERMINATION IN FERTILIZERS

Flame Photometric Determination with Ion Exchange Separation of Interfering Anions

THE FLAME PHOTOMETER offers a rapid and inexpensive method for the determination of the potassium content of fertilizers and for this reason several studies have been made on the applicability of flame photometric methods. Schall and Hagelberg (7) adapted the official gravimetric procedure of the Association of Official Agricultural Chemists to flame determination, and reported on the interference of magnesium,

sodium, and phosphate. Several other investigators have studied the flame photometric determination of potassium and observed that various anions affect the intensity of the potassium line (1, 2, 6, 8).

This paper describes a procedure used successfully in the Missouri Agricultural Experiment Station laboratories, in which basic ammonium carbonate is used to remove the cations (calcium,

magnesium, and others) and an anion exchange resin is used to remove interfering sulfate and phosphate anions.

Reagents and Apparatus

Ammonium carbonate, analytical reagent, 1.5M solution.

Methyl red indicator, 0.2 gram of the indicator per 100 ml. of 95% alcohol.

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