

NITRIC PHOSPHATES

Nitric Phosphates from Phosphate Rock, Nitric Acid, Ammonia, and Carbon Dioxide

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A process for making nitric phosphate fertilizers from phosphate rock, nitric acid, ammonia, potassium chloride, and carbon dioxide was demonstrated in a 4-ton-per-day pilot plant. The process differs from nitric acid processes described previously by TVA in that carbon dioxide is used to avoid the presence in the product of hygroscopic calcium nitrate. Gas from a scrubbing operation at the TVA ammonia plant was used as the source of carbon dioxide. Minus 35-mesh phosphate rock was extracted continuously in three stages with 42% nitric acid at a nitric acid-calcium oxide mole ratio of 1.8. Ammonia was added to the slurry in three successive stages in an amount calculated to precipitate the phosphorus pentoxide as dicalcium phosphate and the fluorine as calcium fluoride. The presence of a small amount of gypsum or soluble sulfate in the slurry reduced the tendency of phosphorus pentoxide to revert to a citrate-insoluble form during ammoniation. After ammoniation, carbon dioxide and ammonia were added simultaneously in each of two stages to convert the calcium nitrate in the slurry to calcium carbonate and ammonium nitrate. The slurry was dried after the addition of potassium chloride to give a N-P₂O₅-K₂O fertilizer of 14-11-11 grade. Only about 1% of the phosphorus pentoxide in the final product was in a water-soluble form; at least 97% was in a citrate-soluble form. A 12-12-12 grade fertilizer was made by replacing a small proportion of the nitric acid with sulfuric acid. Agronomic tests showed that these products are effective fertilizers. Estimates indicate that the process is attractive economically.

A PROCESS FOR PRODUCING NITRIC PHOSPHATE FERTILIZERS in which nitric acid is used to solubilize phosphate rock has been carried through the pilot plant stage by the Tennessee Valley Authority. In this process, carbon dioxide is used to avoid the presence of hygroscopic calcium nitrate in the product, whereas in the other processes developed by TVA, phosphoric acid, sulfuric acid, or potassium sulfate was used for this purpose (2, 3, 6, 9).

The use of ammonia and carbon dioxide to obtain nitrogen-phosphate fertilizers free of calcium nitrate was reported by the Netherlands State Mines

(4, 5) and others (1, 7), but little information was given regarding methods, equipment, and suitability of types of phosphate rock available in this country. The present work was done to obtain such information and because it was believed that process improvements could be made.

The work was carried out in the same pilot plant that was used by TVA in the development of other nitric phosphate fertilizer processes (3, 6, 9). The process, as finally developed, consists of five steps: (1) acidulating phosphate rock with nitric acid, (2) ammoniating the acidulate, (3) adding carbon dioxide and additional ammonia, (4) mixing potassium chloride with the slurry (optional), and (5) drying the slurry. A small amount of soluble sulfate or

calcium sulfate (gypsum) is added during the acidulation or early ammoniation stage to inhibit the reversion of phosphorus pentoxide to a citrate-insoluble form during ammoniation.

Most of the work was done with nitric acid, ammonia, carbon dioxide, and potassium chloride; the grade of the product was 14-11-11 (N-P₂O₅-K₂O). When no potassium chloride was added, the grade of the product was 17-13-0. A 12-12-12 nitric phosphate was made by replacing a small proportion of the nitric acid with sulfuric acid. All the products were granular and appeared to have satisfactory physical properties.

Acidulation

Florida phosphate rock (flotation concentrate) and nitric acid were fed simul-

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taneously to the first of the three acidulation tanks, which were arranged in series. The phosphate rock, which was fed to the system by a screw feeder at a rate of 150 pounds per hour, contained 34.6% phosphorus pentoxide and 49.5% calcium oxide. For most of the work the rock was ground to pass a 35-mesh screen. The nitric acid, which was of 42% concentration, was available from TVA operations. It was measured to the system with a rotameter. The ratio of acid to rock was such that the nitric acid-calcium oxide mole ratio in the acidulate was about 1.8.

The tanks, two of which were made of AISI Type 304 stainless steel and the other of AISI Type 316 stainless steel, were 3.5 feet high and 1 foot in diameter. Each tank was equipped with a motor-driven propeller-type agitator; a foam breaker was attached to the agitator shaft. The slurry level allowed 45% freeboard in the first tank for foaming and 20% freeboard in the remaining tanks.

The acidulate flowed by gravity through the three extraction tanks and into the first ammoniation tank. The total retention time was 50 minutes. Short tests, however, indicated that two tanks with a total retention time of 30 minutes would be adequate for essentially complete reaction of minus 20-mesh phosphate rock (Florida pebble or flotation concentrate). The acidulate flowed to the ammoniator at a temperature of 140° F. It contained about 6.5% nitrogen, 10% phosphorus pentoxide, 14% calcium oxide, and 42% water.

Ammoniation and Carbonation

The ammoniation and carbonation equipment consisted of a series of five stainless steel tanks 4 feet high and 2 feet in diameter. Each tank was provided with two turboimpellers, one 12 inches and one 10 inches in diameter, which were mounted on the same shaft and were rotated at 200 r.p.m. by a 3.5-hp. motor. The freeboard was 25% in the ammoniation tanks and 50% in the carbonation tanks.

The first three tanks were used for continuous ammoniation of the acidulate to an ammonia-nitrate mole ratio of about 0.60; at this mole ratio most of the phosphorus pentoxide had been precipitated as calcium phosphate and substantially all of the fluorine had been precipitated as calcium fluoride. As in earlier work (6), distribution of ammonia to these three tanks was decreased stepwise to avoid loss of phosphorus pentoxide availability. The ammonia, measured with rotameters, was supplied at 20 pounds per square inch and was added to each ammoniator through a slotted tube placed horizontally just below the lower impeller.

Ammonia and carbon dioxide were added to the last two tanks simultaneously to convert the remaining calcium nitrate to ammonium nitrate and calcium carbonate. The amount of ammonia added was sufficient to increase the ammonia-nitrate mole ratio in the slurry from 0.60 to 0.95. One mole of carbon dioxide was added for each mole of ammonia. This amount of carbon dioxide was about twice that required to react with the ammonia that was added to form ammonium carbonate. About one fifth of the ammonia and carbon dioxide used was added to the first of these tanks and four fifths to the second. The ammonia was introduced through a 1/2-inch pipe near and just below the lower impeller. The carbon dioxide, which was contained in a waste gas from TVA's ammonia plant, was supplied at a pressure of 5 pounds per square inch. It was measured with Venturi meters and fed to each carbonator through a 1-inch pipe which also extended below and near the lower impeller and discharged about 90 degrees from the ammonia pipe.

About two thirds of the carbon dioxide passed through the slurry and was lost. Of the one third retained in the slurry, about 20% was lost in the first-stage dryer. The weight ratio of carbon dioxide added to phosphorus pentoxide in the slurry was 0.7. However, the weight ratio of carbon dioxide to phosphorus pentoxide in the dried product was only about 0.2. With this amount of carbon dioxide in the product, no calcium nitrate was detected there. Less carbon dioxide was retained by the slurry when a 12-12-12 grade product was made, and loss of carbon dioxide during first-stage drying was greater, so that the weight ratio of carbon dioxide to phosphorus pentoxide in the product was 0.02. However, no calcium nitrate was found in the product. Tests were not made to determine the minimum quantity of carbon dioxide required for eliminating calcium nitrate from the products.

In the pilot plant, an attempt first was

made to use only two tanks for ammoniation and two for ammoniation-carbonation. Sulfate was not used in these tests, and only 58% of the phosphorus pentoxide in the carbonated slurry was in an available form (citrate-soluble) when the phosphate feed rate was 100 pounds per hour. When an additional ammoniation tank was used, 90% of the phosphorus pentoxide in the carbonated slurry was available at the 100-pound feed rate. However, when the slurry being ammoniated contained a small amount of potassium sulfate, satisfactory operation and high phosphorus pentoxide availability (97%) were obtained at a phosphate feed rate of 150 pounds per hour, which was the rate used with the other nitric phosphate processes. The results of these tests are shown in Table I. In most tests, the availability of the phosphorus pentoxide in the final product after drying was slightly higher than in the carbonated slurry.

In initial work on the process, it was believed that the phosphorus pentoxide would precipitate during ammoniation as dicalcium phosphate. At the acidulation that was used, the addition of ammonia until the ammonia-nitrate mole ratio in the slurry was about 0.60 theoretically should have precipitated all the phosphorus pentoxide as dicalcium phosphate. However, when this ammonia-nitrate mole ratio had been reached, only 85 to 90% of the phosphorus pentoxide had been precipitated; essentially complete precipitation was not obtained until the ammonia-nitrate mole ratio was about 0.70, which usually occurred in the first carbonator. Moreover, petrographic examination of the final dried product disclosed only a minor quantity of dicalcium phosphate. Apparently the bulk of the phosphorus pentoxide was present as a calcium phosphate phase of apatite structure.

Unfortunately, a large loss of phosphorus pentoxide availability usually occurred while the mole ratio was being increased from 0.60 to 0.70. In an ef-

Table I. Effect of Number of Stages, Production Rate, and Potassium Sulfate on P₂O₅ Availability

Test	Phosphate Rock Feed Rate, Lb./Hour	Ammoniation Stage			Carbonation Stage		Bagged Product
		1	2	3	1	2	
A	100 (no sulfate)						
	NH ₃ :NO ₃ mole ratio	0.46	0.56	...	0.77	0.98	...
	P ₂ O ₅ availability	84	78	...	68	58	...
B	100 (no sulfate)						
	NH ₃ :NO ₃ mole ratio	0.37	0.55	0.60	0.71	0.98	0.96
	P ₂ O ₅ availability	97	98	97	96	90	94
C	150 (no sulfate)						
	NH ₃ :NO ₃ mole ratio	0.42	0.54	0.58	0.67	0.97	0.95
	P ₂ O ₅ availability	99	98	97	82	83	83
D	150 (plus 11.9 lb. K ₂ SO ₄)						
	NH ₃ :NO ₃ mole ratio	0.40	0.51	0.59	0.68	0.92	0.87
	P ₂ O ₅ availability	99	99	99	99	97	98

fort to avoid these losses, carbon dioxide was added to the fourth tank in the ammoniation-carbonation system, since the change in the mole ratio usually occurred in this tank. Though very little carbon dioxide was retained in the slurry in this tank, reversion of phosphorus pentoxide was decreased but not sufficiently to make possible operation of the pilot plant at the capacity attained with other nitric phosphates.

In small scale tests it was found that the addition of a small amount of soluble sulfate or of calcium sulfate during acidulation or in an early stage of ammoniation prevented the loss of phosphorus pentoxide availability that usually accompanied the precipitation of the last portion of the phosphorus pentoxide. The minimum amount of sulfate required to prevent reversion was that equivalent to a sulfate-phosphorus pentoxide weight ratio of 0.12. Sodium, ammonium, potassium, magnesium, and calcium sulfates and langbeinite (a potassium - magnesium sulfate) were about equally effective in preventing reversion. Sulfuric acid was effective also. Potassium and magnesium chlorides, dolomite, sodium nitrate, and borax were of little or no value. When a high-alumina phosphate matrix (7% Al_2O_3) was used as the source of phosphorus pentoxide, the alumina content did not prevent reversion of phosphorus pentoxide. The reason for the beneficial

effect of the sulfates has not been determined yet.

It was postulated and later shown in the pilot plant that once the phosphorus pentoxide was precipitated carbon dioxide and ammonia could be added to convert the remaining calcium nitrate to calcium carbonate and ammonium nitrate without affecting phosphorus pentoxide availability.

In the pilot plant in the production of 14-11-11 fertilizer, potassium sulfate was used to prevent reversion. This salt was selected because it would supply not only sulfate but some potash also.

Addition of Potash and Drying

The carbonated slurry, which contained about 35% water, was mixed with potassium chloride before it was dried. This practice was possible because drying was carried out at relatively low temperatures, which eliminated the danger of initiating the self-propagating decomposition of ammonium nitrate-potassium chloride mixtures that had been found to occur when the temperature of the mixture exceeded 380° F. (3).

After the addition of potassium chloride, the water content of the carbonated slurry was 31%. The mixture was maintained at a temperature of about 130° F. to prevent precipitation of potassium nitrate and other salts in the line leading to the first-stage dryer.

Drying was carried out in two stages.

The procedure consisted in (1) partially drying the slurry to a water content of about 10% in a rotary dryer; (2) mixing the partially dried slurry, which was discharged from the rotary dryer as a fluid, with recycled fines and oversize from the final dried product in a double-shaft paddle mixer; and (3) drying the mixture, which contained about 4% water, in a Roto-Louvre dryer to a water content of 1 to 2%.

In the first stage a rotary dryer 10 feet long and 1.5 feet in diameter was used. It contained eight stainless steel radial flights and was operated with cocurrent feed and drying gas at 20 r.p.m. and a slope of 0.2 inch per foot. The inlet temperature of the drying gas was about 700° F. and the exit gas temperature was about 300° F. The temperature of the product as discharged was about 190° F. The feed rate was about 700 pounds of slurry per hour.

The paddle mixer in which granulation of the product occurred was 6 feet long, 10 inches deep, and 15 inches wide.

The Roto-Louvre dryer was 10 feet long and 2 feet 7 inches in outside diameter. It was operated with inlet drying gas at 370° F. and exit gas at 140° F. The temperature of the product was about 165° F. About one third of the product from the Roto-Louvre dryer was in the form of granules of the desired size, minus 6 plus 50 mesh.

Demonstration Operation

After methods of control were developed and satisfactory operating conditions were defined, the pilot plant was operated to demonstrate the process and to obtain data for evaluation and design purposes. Average data for 4-day demonstration runs to produce 14-11-11 and 12-12-12 grade products are presented in Table II.

In operation to produce 14-11-11 nitric phosphate, Florida pebble phosphate ground to pass a 35-mesh screen was fed to the extraction section through a screw feeder at a rate of 150 pounds per hour. Forty-two per cent nitric acid was fed simultaneously with the rock at a rate that gave a nitric acid-calcium oxide mole ratio of 1.87. Solubilization of phosphorus pentoxide was substantially complete. Potassium sulfate was added at a rate of 11.9 pounds per hour, and the acidulate was then treated with anhydrous ammonia to give an ammonium nitrate mole ratio of 0.59. The ammonia was added in the first three tanks in proportions of 68, 19, and 13%, respectively. In the last two tanks, additional ammonia was added simultaneously with carbon dioxide. About 27% of this ammonia was added to the first of these tanks, and 73% to the last. The ammonium-nitrate mole ratio was 0.92.

The distribution of carbon dioxide to the two tanks was the same as that of the

Table II. Summary of Operating Data

Product grade (N-P ₂ O ₅ -K ₂ O)	14-11-11	12-12-12
Nominal		
Actual	14.7-11.6-11.0	12.0-12.4-12.9
Acidulation		
Phosphate rock, lb./hour	150	150
P ₂ O ₅ content, %	34.6	34.3
CaO content, %	49.5	48.7
Nitric acid (41.7% HNO ₃), lb./hour	374	294
Sulfuric acid (93% H ₂ SO ₄), lb./hour	...	33.6
Ammoniation		
Water added, lb./hour	...	33.0
Potassium sulfate (52.1% SO ₄), lb./hour	11.9	...
Ammonia, lb./hour	24.9	23.0
Carbonation		
Ammonia, lb./hour	13.8	5.2
Carbon dioxide, lb./hour	34.4	31.2
Carbonated slurry output, lb./hour	557	475
pH	6.6	6.2
P ₂ O ₅ availability, %	99	99
Addition of potassium chloride (61% K ₂ O), lb./hour	72	88
First-stage drying (rotary dryer)		
Slurry feed, lb./hour	712	1090
Moisture content of feed, %	31.3	26.8
Moisture content of product, %	10.2	11.5
Carbon dioxide loss, %	20	50
Heat requirement, B.t.u./ton product × 10 ⁶	3.18	2.40
Paddle mixer operation		
Recycled dried product, lb./hour	1990	1950
Output, lb./hour	2535	2850
Second-stage drying (Roto-Louvre dryer)		
Moisture content of feed, %	3.8	4.9
Moisture content of product, %	1.8	1.9
Carbon dioxide loss, %	Negligible	Negligible
Heat requirement, B.t.u./ton product × 10 ⁶	0.89	0.92

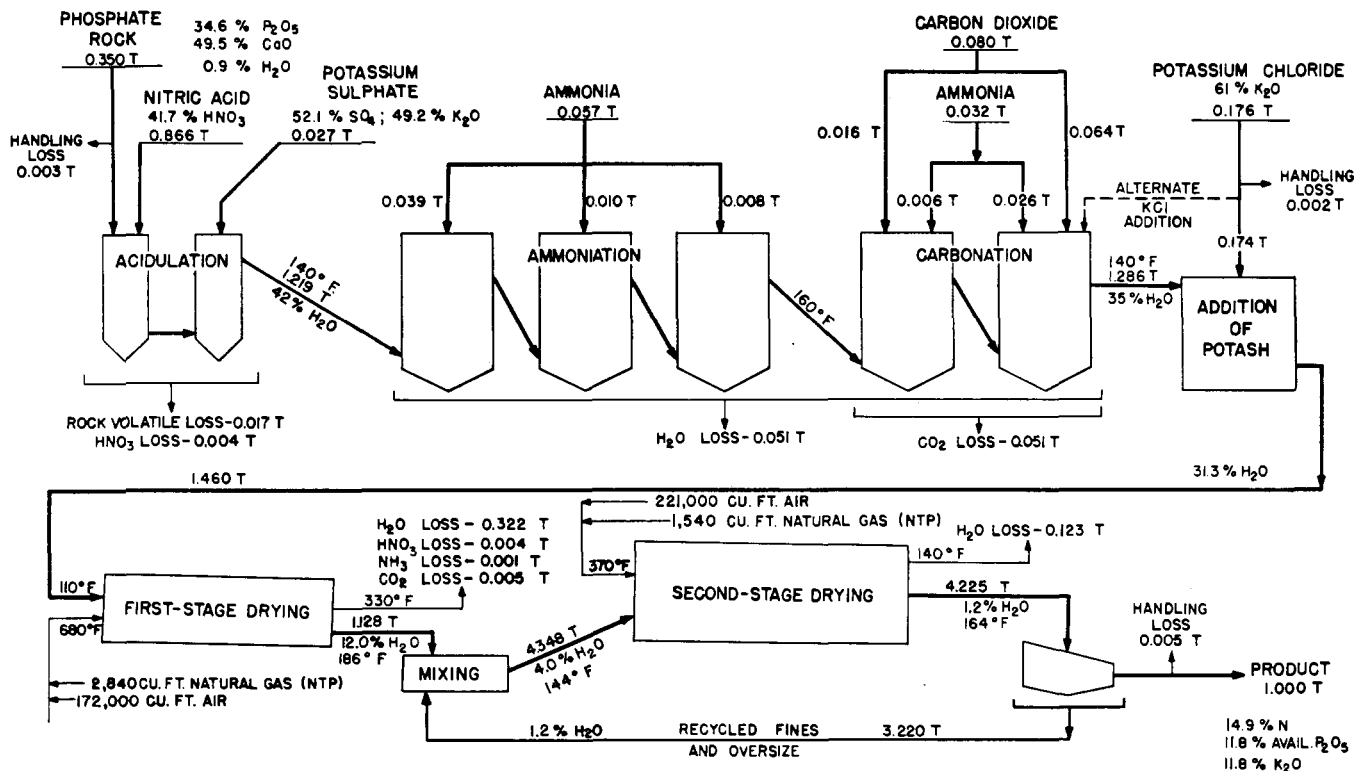


Figure 1. Quantitative flow diagram for production of 14-11-11 nitric phosphate using carbon dioxide

Quantities are in tons per ton of production

ammonia. The weight ratio of carbon dioxide to phosphorus pentoxide was 0.7. The ammoniated, carbonated slurry contained about 35% water, and 99% of the phosphorus pentoxide present was in an available form. The pH values of the slurry in the last four tanks were 1.4, 2.8, 5.4, and 6.6, respectively.

The potassium chloride required to give a product having a potassium oxide-available phosphorus pentoxide weight ratio of 1 was mixed with the slurry in a surge tank prior to feeding the slurry to the first-stage dryer. However, in a large-plant operation, provision of a surge tank would not be necessary, and potassium chloride could be added in the last carbonating tank. Tests showed that the addition of potassium chloride at this point had no undesirable effects.

The capacity of the drying equipment was somewhat greater than the rate at which carbonated slurry was produced, so that the drying section could be operated independently at slightly higher rates than the ammoniation and carbonation section when sufficient carbonated slurry was available. The temperatures maintained during the drying operation were the same as those shown in Figure 1. As stated above, about one third of the discharge from the Roto-Louvre dryer was in the size range desired for bagged product. However, the screening operation was inefficient and enough product-size material was recycled so that the ratio of recycled dry product to partially dried slurry was about 4. Tests made during other

periods of operation indicated that a recycle ratio of about 3 was sufficient to provide a satisfactory feed for the Roto-Louvre dryer; it is possible that a lower ratio would be adequate. The bagged product (-6 +50 mesh) of the Roto-Louvre dryer contained 14.7% nitrogen, 11.6% available phosphorus pentoxide, and 11.0% potassium oxide. About 99% of the total phosphorus pentoxide content was in an available form, and about 1% was water-soluble.

Over-all nitrogen loss was about 4% of the nitrogen entering the process; most of this loss occurred in the acidulation step. During most of the pilot plant operation about 99% of the nitric acid fed was recovered in the acidulated slurry, which indicated that the loss incurred during the demonstration operation was not inherent in the process. It appeared that an over-all nitrogen loss of about 1.5% could be expected in large scale operation.

A quantitative flow diagram showing how the process would be operated to produce a 14-11-11 fertilizer in a large-scale plant is given in Figure 1.

In a short test in which no sulfate was used, 300 pounds of 8-16-16 grade product were made by filtering ammonium nitrate from the carbonated slurry before the addition of potash; the potash, as potassium chloride, was mixed with the filter cake. This appeared to be a feasible means of preparing a fertilizer with a N:P₂O₅:K₂O weight ratio of 1:2:2 while simultaneously producing a solution of ammonium nitrate that could be

used in the preparation of ammoniating solution. This test indicated the flexibility of the process.

The feasibility of producing a 12-12-12 nitric phosphate by substituting sulfuric acid for a small proportion of the nitric acid used in the acidulation of phosphate rock also was demonstrated in a 4-day run. The nitric-sulfuric acid weight ratio was 3.9. The concentration of the sulfuric acid used was 93%. When no additional water was used, the slurry was thicker than that resulting from the use of nitric acid alone. When water was added in proportion so that the equivalent concentration of the sulfuric acid was 46%, the slurry was fluid enough for easy handling. No other operating problems were caused by the use of sulfuric acid. The quantity of sulfuric acid added was only about half that required in the production of an 11-11-11 nitric phosphate with a mixture of nitric and sulfuric acids as described by Striplin (9) and about 30% of that required to solubilize an equal amount of phosphorus pentoxide in the production of ordinary superphosphate. More than enough sulfate was present in the slurry to prevent reversion of phosphorus pentoxide during ammoniation.

The compositions of typical products made during this investigation are shown in Table III.

Product Characteristics

Field tests of 14-11-11 and 12-12-12 grade products are in progress in several

Table III. Chemical Compositions of Bagged Minus 6- Plus 50-Mesh Product

Nominal Grade	Nitrogen, %			P ₂ O ₅ , %								
	Total	HN ₃	NO ₃	Total	Citrate-insoluble	Water-soluble	Availability	K ₂ O, %	CO ₂ , %	SO ₃ , %	CaO, %	H ₂ O, %
14-11-11	14.7	6.8	7.9	11.7	0.1	<0.1	99	11.0	2.5	1.4	16.6	1.8
12-12-12	12.0	5.9	6.1	12.5	0.1	0.1	99	12.9	0.3	6.0	17.8	1.9
17-13-0 ^a	17.7	8.4	9.3	14.3	0.8	0.0	94 ^b	...	1.7	...	20.8	...
8-16-16	8.6	4.2	4.4	17.7	1.5	...	92 ^b	15.8	0.7

^a Made in bench-scale apparatus.

^b Made without addition of sulfate.

states. A limited number of greenhouse tests indicated that the 17-13-0 product was as effective as concentrated superphosphate and ammonium nitrate when these materials were used to supply equivalent amounts of phosphorus pentoxide and nitrogen and when limestone was not added. When limestone was added, the first crop response of 17-13-0 was less than that of the standard materials, but the second crop response was about equal.

The products were in satisfactory condition after warehouse storage for 6 months in six-ply paper bags having two asphalt-laminated plies; it is believed that bags with only one asphalt-laminated ply, or equivalent, would be adequate.

Tests with a John Blue No. 30 screw-type fertilizer distributor showed the products to have satisfactory drilling

characteristics before storage and after storage for 6 months.

Economics

Estimates indicate that the costs of producing 12-12-12 and 14-11-11 grade nitric phosphates by the carbon dioxide process should compare favorably with the costs of producing nitric phosphates by the other processes developed by TVA (3, 6, 8, 9). The process should be especially attractive to fertilizer manufacturers who produce their own ammonia, since carbon dioxide is available in abundance as a waste gas in most ammonia plants. The process should be economically attractive even if it is necessary to manufacture carbon dioxide.

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NITRIC PHOSPHATES

Economics of the Nitric Phosphate Fertilizer Processes

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Processes in which nitric acid is used to convert phosphate rock to forms suitable for fertilizer use (nitric phosphate processes) are being studied by the Tennessee Valley Authority. Much of the work has been carried through the pilot plant stage. This paper evaluates the economics of the nitric-phosphate processes under current market conditions based on the results of TVA pilot plant work. The evaluation involved preparation of estimates of investments, production costs, and wholesale selling prices in a given market area for the nitric phosphates and for mixed fertilizer of comparable grade, and comparison of the values obtained. The estimates indicated that, under current market conditions, the economic positions of the nitric-phosphate processes are favorable when compared with the conventional mixed fertilizer process for an annual volume of sales of 50,000 tons of product and that the position would be improved for larger volumes of sales.

NITRIC ACID has been used commercially in Europe for about 20 years to convert phosphate rock to forms suitable for fertilizer use. The processes, carried on principally in Norway, Hol-

land, Germany, and France, were described by Hignett (2) and others (7, 5, 8).

Several of these nitric phosphate processes are being studied by the Tennessee Valley Authority. The study by TVA

was undertaken because very little detailed information was available with regard to methods, equipment, and suitability of types of phosphate rock available in this country and because it