

Plant-Scale Production of Fertilizers in a Pan Granulator

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TVA is now producing granular high-analysis fertilizers in a demonstration-scale plant utilizing a pan-granulation process that was developed on pilot-plant scale. Products currently being made in the plant are 30-4.4-0 (30-10-0) and 25-10.9-0 (25-25-0) grades of ammonium phosphate nitrate and 30-0-0-5S ammonium nitrate sulfate. The plant was designed for a production rate of 480 tons per day; however, rates as high as 560 tons per day have been achieved. In this process, the acids are partially neutralized to pH 1.5 with ammonia, the resulting solution is concentrated by

evaporation, and neutralization is completed to pH of about 5.5 in a second-stage neutralizer. Granulation is accomplished by spraying the hot concentrated solution onto a cascading bed of fines in a 14-foot-diameter pan granulator. The material from the pan is dried, screened, cooled, and conditioned prior to bulk storage in controlled humidity. The granular products have good storage and handling characteristics when dried to a moisture content of 0.2 to 0.4% and coated with conventional conditioning agents.

Pan granulation of high-nitrogen fertilizers has been studied for the past several years, and specific processes have been developed utilizing this type of equipment (Young and McCamy, 1967). These processes are based on the use of an inclined rotary pan granulator to granulate materials containing high proportions of water-soluble fertilizer salts (such as ammonium nitrate or urea) that do not require ammoniation during granulation. Pilot-plant tests indicated that the pan granulator was superior to a rotary drum or pug mill for granulating products of this type because of the better classifying action in the pan and the lower recycle ratios required to control granulation. Based on the favorable results obtained from the pilot-plant operation, a demonstration-scale pan-granulation system was designed and constructed as part of the TVA granular combination fertilizer unit. The demonstration plant, designed to produce 20 tons per hour of high-nitrogen fertilizers, such as ammonium nitrate, 30-4.4-0 (30-10-0) and 25-10.9-0 (25-25-0) ammonium phosphate nitrate, and ammonium nitrate sulfate, has been in operation since November 1965. Raw materials used in the process include nitric acid, anhydrous gaseous ammonia, concentrated sulfuric acid, and electric-furnace phosphoric acid. TVA purchases the sulfuric acid and produces the other raw materials used in the plant. The use of wet-process phosphoric acid was satisfactory in the pilot-plant operation, but this material was not available for the plant operation. The pan-granulation system, which shares operating time and some equipment with two other processes in the plant, has now produced more than 100,000 tons of granular fertilizer. This paper describes the plant equipment and operation of the plant.

DESCRIPTION OF THE PROCESS

Granulation is accomplished by spraying a hot concentrated solution of the fertilizer salts onto a cascading bed

of recycle fines in the pan granulators. A sketch showing pan operation is shown in Figure 1. A general flowsheet of the pan-granulation system is shown in Figure 2. The granular material discharges from the pans to the predryer where sufficient moisture is removed to permit drying and provide effective recycle material. The undersize and oversize fractions are cooled in the recycle cooler; the

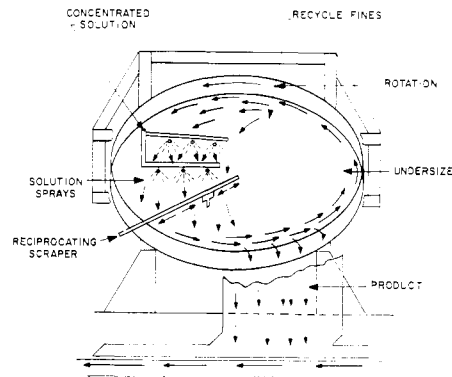


Figure 1. Sketch of pan operation

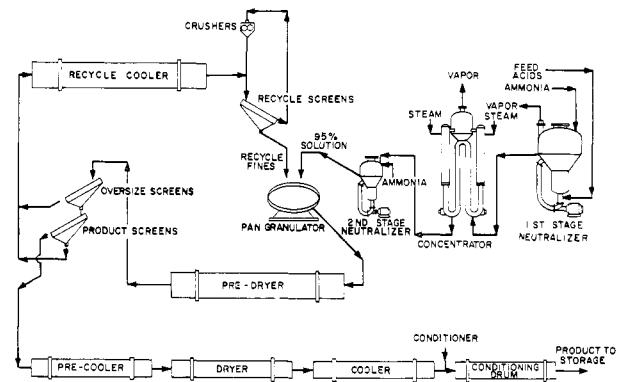


Figure 2. Flow diagram: pan-granulation process for the production of granular products containing ammonium nitrate

oversize fraction is crushed and combined with the under-size fraction for use as recycle material. From the product screens, the onsize fraction ($-7 + 9$ mesh) enters the product pre-cooler where the temperature is reduced to about 85°F ., which is slightly lower than one of the crystal phase transition points for ammonium nitrate. Ammonium nitrate dries rapidly when treated in this manner (Datin, 1945). Unpublished TVA data indicate that the drying of the other products containing large proportions of ammonium nitrate is substantially improved when the products are subjected to this treatment. Most of the product from the pre-cooler goes to the product dryer where final drying occurs; some of the cool product from the pre-cooler is diverted, as required, to the crushers to supply supplemental recycle material. The product from the final dryer enters the product cooler where it is cooled to about 110°F . The cooled product is then treated with diatomaceous earth in the conditioning drum and conveyed into the bulk fertilizer storage building. The fertilizer is kept in the storage building under controlled humidity conditions until ready for shipment.

The concentrated solutions or slurries used in the pan granulators are prepared by partially neutralizing appropriate acids with gaseous anhydrous ammonia, concentrating the resulting solution by evaporation, and further ammoniating the concentrated solution to the desired final pH. The flows of acid to the first-stage neutralizer are set at fixed rates, and the flows of ammonia to the first- and second-stage neutralizer are controlled automatically to maintain the pH of the solution within the desired range for each neutralizer. The pH control system used to control neutralization is unique in that the sample streams of solution or slurry are continuously diluted and cooled with water before coming in contact with the pH electrodes. Diluting the sample keeps crystals from salting out, and cooling it keeps the electrodes from deteriorating and malfunctioning as they would if the temperature remained high. The evaporation step is located between the two stages of neutralization to permit concentration of the solution at low pH and thereby minimize nitrogen losses during production of the ammonium phosphate nitrate and ammonium nitrate sulfate grades.

During production of ammonium nitrate, the nitric acid is completely neutralized before the evaporation step because of possible hazards involved in handling concentrated ammonium nitrate solution at high temperature and low pH.

PLANT

Instrumentation. Instrumentation for the plant is designed to meter raw materials, monitor and control process conditions in the various pieces of process equipment, and to meter the product leaving the plant. Almost all phases of the process can be operated by control instruments and process equipment switches located in the control room. The process variables monitored include temperature, pressure, flow rate, pH, liquid level, and density of liquids. Ratio control instruments are provided to proportion raw materials to the process and to control automatically the rate of conditioner added to give the desired proportion in the final product. Instru-

mentation is also provided for automatic safety shutdown of the solution system if the temperature in the process vessels exceed preset values.

Acid feeds into the process are metered with conventional magnetic flowmeters. Orifice-type meters are used for metering ammonia flows. Orifice meters and rotometers are provided for metering the scrubber solution and the solution used for dissolving cyclone fines. Pressures and liquid levels in process vessels are indicated by stainless steel diaphragm-type gages. Temperatures at various points in the plant are indicated by stainless steel gas or liquid bulbs. The flow of dry solids on belt conveyors is metered with nuclear density meters. The operation of this instrument is based on absorption of gamma rays by the material being weighed as it passes between a radiation source and the detector target. The absorption of the radiation is a function of the mass of material on the belt. Cesium-137 is used as a source of radiation for the meter.

Most of the variables measured in the plant are indicated or recorded in the control room. Transmitters are used at the point of measurement to convert the process conditions, such as temperature, pressure, liquid level, flow rate, etc., into air signals that are transmitted through stainless steel tubing to the control room. These signals operate indicating or recording instruments calibrated in terms of the variable involved. These signals also actuate indicating or recording control instruments that automatically control the variable at any desired value. Control valves in the plant are operated by air pressure that is controlled by signals from the automatic control stations; the control stations may also be operated manually.

Solution System. The solution preparation system consists of a first-stage neutralizer, surge tank, concentrator, and second-stage neutralizer. Mechanical agitators or forced circulation systems are provided for all vessels in the solution system because of the large amount of crystals present during production of ammonium nitrate sulfate. All the solution preparation equipment is constructed of Type 316L stainless steel. The sulfuric acid sparger in the first-stage neutralizer is made of Carpenter 20 alloy. Corrosion of the equipment has not been a major problem.

The first- and second-stage neutralizers are of the same general design and utilize a circulating system for keeping crystals in suspension. The design of the neutralizers is based on an ammonia loading of 15 to 35 pounds per hour per cubic foot of pool volume and an evaporation rate of 60 to 180 pounds of water vapor per hour per square foot of pool surface.

Details of the first-stage neutralizer are shown in Figure 3. The neutralizer is designed to keep the cross sectional area of the reaction zone relatively small and that of the evaporation zone relatively large. The bottom section of the vessel is 6 feet in diameter and the top section is flared out to a diameter of 12 feet. Positive circulation is provided with a 2000-g.p.m. recirculation pump. Feed acids are introduced in the recirculating stream of solution just before it enters the bottom of the neutralizer. Ammonia is introduced through drilled-pipe spargers located near the bottom of the neutralizer. Internal thermal circulation is provided by a circular baffle installed between the ammonia sparger and the wall of the neutralizer.

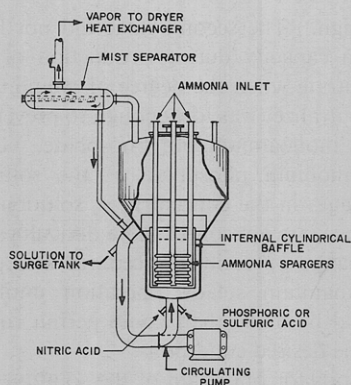


Figure 3. Sketch of first-stage neutralizer

The neutralizer is designed to operate at 5.3 p.s.i.g. pressure. Vapors from the neutralizer pass through a mist separator and are piped to heat exchangers for heating the solution which dissolves fines, and for preheating air to the dryers. A water-cooled shell and tube heat exchanger is provided for condensing surplus vapor from the neutralizer.

A cone-bottom surge tank receives solution from the first-stage neutralizer and feeds the concentrator. This tank is 8 feet in diameter; its vertical section is 7.5 feet high, and its conical section is 4.2 feet high. It is covered and is equipped with a 10-hp. mechanical agitator. The tank is equipped with an ammonia sparger for adjusting the pH of the solution in the unit and a steam coil for adding heat. The vapors from the surge tank are vented to a scrubber used for dissolving fertilizer dust in the air stream from the product cooler.

A two-stage, forced circulation, steam-heated vacuum concentrator is used to evaporate the solution before the final neutralization stage. The unit has a single vapor chamber that serves two separate systems for heat exchange and solution circulation. The bottom section of the vapor chamber is divided with a baffle to separate the two solution systems. Each heat exchanger has a tube surface area of 1000 square feet and the axial-flow circulation pumps have a rated capacity of 4000 g.p.m. at a 15-foot head. The vapors from the concentrator are condensed with water in a barometric condenser and non-condensable gases are removed with a steam ejector.

The original second-stage neutralizer was a vertical cone bottom tank about 6 feet in diameter and about 7 feet high. It was equipped with an ammonia sparger, steam coil, and a 5-hp. mechanical agitator. This unit was later replaced with a new second-stage neutralizer of the same design as the first-stage neutralizer, because the original unit did not have enough ammoniation capacity for modified operating conditions that were desired. The bottom section of the new neutralizer is 4 feet in diameter, and the top section is 8 feet in diameter. The recirculation pump is identical to that used in the first-stage neutralizer, and the ammonia sparger is of the same design. The second-stage neutralizer operates at atmospheric pressure, and the vapors are vented to a scrubber used for removing fines from the air stream leaving the recycle cooler. The neutral

concentrated solution from the second-stage neutralizer is pumped to the solution sprays at the pan granulators. The flows of solution to the granulator are metered with magnetic flowmeters and controlled with automatic control valves.

Pan Granulators. Granulation is carried out in two pan granulators which may be operated individually or in parallel. The granulators are 14 feet in diameter and 20 inches deep and constructed of mild steel. These units are equipped with variable speed drives, reciprocating scrapers, and provisions for varying the slope or angle of tilt. Figure 4 shows one of the pans in operation.

Rotary Equipment. The rotary dryers and coolers are standard countercurrent flow units equipped with lifting flights and cyclone-type dust collectors. Dust collected in the cyclones is dissolved in warm, dilute process liquor in pots beneath the cyclones and returned to the solution system for reprocessing. Dust in the exhaust air from the cyclones is recovered in wet scrubbers, and the air from the scrubbers is exhausted to the atmosphere. Condensed vapors from the first-stage neutralizer are used as makeup liquor for the scrubbers. Vapor from the first-stage neutralizer is used for preheating the air for the dryers; supplemental heat, as required, is supplied by high-pressure steam. A refrigeration system is provided for cooling the air for the product pre-cooler.

Material Handling. Granular solids are handled with belt conveyors and bucket elevators. A pneumatic system is provided for conveying and handling the conditioning dust.

Screens. Electrically vibrated single-deck screens are used in the product and recycle fines screening system. Slotted-type wire mesh with 7- and 9-mesh openings is used for the product screens. The recycle fines screens are equipped with slotted-type wire mesh with 10-mesh openings.

Crushers. Parallel double-shaft chain mills are used for crushing oversize and product-size material used for recycle fines. The chain mills are operated in a closed circuit with the recycle screens to ensure complete crushing and prevent oversize material from entering the recycle stream.



Figure 4. View of pan granulator in operation

PLANT OPERATING EXPERIENCE

After almost 2 years of operation, production in the plant has become fairly routine and does not require a great deal of operator attention. Process and equipment changes have been made from time to time. As a result of these changes, the plant can now produce more with one pan granulator in operation than it was designed to produce with two. The plant experience has demonstrated the effectiveness of the pan granulator as a means of producing granular high-nitrogen fertilizer from concentrated solutions. The process for products based on ammonium nitrate would be more attractive if the drying step could be eliminated by the use of melts or more highly concentrated solutions in the pan. Production of granular urea without the use of a drying step has been demonstrated in pilot-plant tests (Young and McCamy, 1967), and other tests are planned to study this procedure with the grades that are produced in the demonstration plant.

A large number of difficulties and problems of the usual type were experienced during preliminary operation of the plant. Some of the difficulties were corrected with relatively minor process or equipment changes; others required more extensive changes. The initial operation indicated that some process conditions that were favorable for good operation in the pilot-plant tests could not be attained in the plant operation because of differences in design and size of the equipment. Flows of solution through the plant system were more difficult to maintain and control under conditions similar to those used in the pilot plant because of longer lines and more elaborate control systems in the large plant. The pilot-plant solution system was operated at atmospheric pressure and used gravity flow. The solution flow through the plant system is regulated by level control valves instead of gravity overflow, because the first-stage neutralizer operates under pressure, and the concentrator operates under vacuum. Operation of the concentrator under vacuum also provides less superheat in the solution than was obtained during operation of the pilot-plant evaporator at atmospheric pressure. Bed action in plant pan granulators was not so good during initial operation as it had been in the pilot-plant pans. The plant units would not operate at sufficient speed to give good classifying action when the pans were tilted to the slope necessary to obtain a good cascading bed. The equation (Young and McCamy, 1967) for calculating the optimum rotational speed for a pan granulator

$$\text{Speed (r.p.m.)} = 1 - \cos \theta \frac{76.5}{\sqrt{D}}$$

does not contain a pan depth factor; the 20-inch depth of the plant pans probably is too great to give good results at the design speeds of 11 to 14 r.p.m. The plant products were not so smooth and well rounded as those obtained in the pilot-plant operation because of the low speed of the pans and the lower temperature of the solution used in the plant units. The speed of the pans was later increased stepwise to fixed speeds of 16 and 19 r.p.m. The fixed-speed drives were used because excessive maintenance was required for the variable speed drives. The original reciprocating scrapers on the plant pan granulators did not function properly and had to be replaced with scrapers of a

different design. The second stage did not have enough ammoniation capacity during production of ammonium phosphate nitrate when the degree of ammoniation in the first-stage neutralizer was low enough to prevent formation of insoluble monoammonium phosphate. The presence of monoammonium phosphate in the solution caused severe stoppages in the concentrator, solution piping, and control valves. As a result of these difficulties, it was necessary to change operating procedures and process conditions to maintain steady operation until equipment changes could be made that would permit routine operation under the desired conditions.

Two methods for operation of the solution system were used after the preliminary phase of operation. In the operation before March 1967, essentially all of the ammonia required for neutralization was added to the first-stage neutralizer, and only enough ammonia was added to the second-stage neutralizer to make up for losses in the system. This method of operation essentially prevented stoppages due to monoammonium phosphate in the system, but ammonia losses from the concentrator were high. A new second-stage neutralizer and auxiliary equipment were installed, and other equipment changes were made in February 1967. These changes permitted plant operation under conditions similar to those used in the pilot-plant work. Operation of the first-stage neutralizer at a slightly lower degree of ammoniation than was used in the pilot plant eliminated the formation of troublesome amounts of solids in the system. Ammonia losses from the concentrator were decreased to very low levels after the operation of the solution system was changed to maintain a low pH prior to concentration. Also, the steam requirement for concentration was decreased substantially since the first-stage neutralizer could be operated with solution of higher concentration for feeding to the concentrator. The larger second-stage neutralizer was adequate for completion of the ammoniation.

The refrigeration system for cooling the air to the product precooler has not been activated, because it was not required for satisfactory drying of the granular products. The benefits derived from the precooling step in drying have been demonstrated during periods when the temperature of the ambient air was low enough to cool the precooler product to less than the 89° F. crystal phase transition point for ammonium nitrate. During production of 30-4.4-0 (30-10-0) ammonium phosphate, the moisture content of the product averaged 0.16% during a day when the temperature of the precooler product using ambient air averaged 65° F.; with other plant conditions about the same, the moisture content of the product increased to 0.40% when the temperature of the precooler product increased to 93° F. These data confirm the benefits of precooling the product before the final drying step to obtain products of very low moisture content.

Ammonium Nitrate. Granular ammonium nitrate has been more difficult to make in the new plant than any of the other products. However, only a few hundred tons of this product were made during preliminary operation of the plant before its production was discontinued, and no attempt has been made to produce it since the plant changes improved operation for the other products. During initial production of ammonium nitrate, the granular product

was weak and considerable degradation occurred in the dryer. Operation at lower pan speeds and the use of solutions having less superheat than had been used in the pilot plant contributed to the production of the weak granules. Granules of adequate strength were produced when sulfuric acid was fed to the first-stage neutralizer at rates sufficient to give about 4% of ammonium sulfate in the product. The use of sulfuric acid had not been required for the production of strong granules in the pilot plant operation. No additional production of ammonium nitrate is planned.

Ammonium Nitrate Sulfate. Ammonium nitrate sulfate (30-0-0-5S) granulated satisfactorily when 2 to 3 pounds of recycle were used per pound of feed. The chief difficulty encountered in making this product was stoppages in level control valves, slurry lines, and spray nozzles. These stoppages were more persistent in the large plant than in the pilot plant, because the slurry lines were shorter in the pilot plant. Also, the gravity overflow slurry system used in the pilot plant had allowed removal of lumps by passing the slurry through a basket strainer before it was pumped to the sprays. Stoppages in the plant system were substantially reduced by operating the first-stage neutralizer at a pH of 6.0 instead of a pH of 2.0 used earlier and operating the concentrator to produce 91 to 93% slurry instead of the usual 95%. The plant operated at these conditions until March 1967 when the new second-stage neutralizer system was installed. Typical data for this operation are shown in Table I. Ammonia losses from the concentrator were fairly high during the operation at high pH in the first-stage neutralizer. The temperature in the first-stage neutralizer was kept down to about 265° F. to decrease the amount of ammonia leaving with the vapors.

Since March 1967, the first-stage neutralizer has been operated at a pH of about 1.2 (NH₃ to H₂SO₄ mole ratio of about 1.4) and at a temperature of 300° F. to maintain maximum fluidity of the slurry in the first-stage neutralizer and concentrator. Operation of the first-stage neutralizer at low pH and high temperature gave a solution of higher concentration than was provided in previous operation. Ammonia losses from the concentrator and the amount of steam required for the evaporation step were greatly decreased by the low pH method of operation. Typical data for the preferred low pH method of operation are given in Table I. The production capability of the plant for ammonium nitrate sulfate is limited by the flow capacity of the slurry piping. The maximum production of ammonium nitrate sulfate obtained during a 24-hour period was 472 tons. The granular product is of good appearance and has excellent physical properties.

Ammonium Phosphate Nitrates. Both 30-4.4-0 (30-10-0) and 25-10.9-0 (25-25-0) grades of ammonium phosphate nitrate granulated satisfactorily when 2 to 3 pounds of recycle were used per pound of feed solution to the pan. Granulation was satisfactory with one pan at rates up to 25 tons per hour after the speed of the granulator was increased to 19 r.p.m. and the slope increased to about 70 degrees. A recycle ratio of about 4 to 1 was required to cover satisfactorily the bottom of the pan after the speed and slope were increased.

Both the low- and high-pH methods of operating the solution system were used. At first, essentially all the

Table I. Typical Operating Data for Plant-Scale Production of High-Nitrogen Fertilizers by the Pan-Granulation Process

	30-4.4-0 (30-10-0), Ammonium Phosphate Nitrate		30-0-0-5S (30-0-0-5S), Ammonium Nitrate Sulfate	
	Method of Operation of First-Stage Neutralizer			
	High pH	Low pH	High pH	Low pH
Operating time, hour	302	251	123	268
Production rate, tons/hour	17.6	21.3	17.5	18.2
Speed of pan, r.p.m.	13	19	13	19
Slope of pan, degrees from horizontal	65	70	65	70
Approximate recycle ratio	3 to 1	4.5 to 1	3 to 1	4.5 to 1
Operating temperature, ° F.				
First-stage neutralizer	257	300	256	290
Concentrator	315	289	313	315
Second-stage neutralizer	316	338	300	325
Recycle fines	143	162	133	134
Granulator product	189	197	184	186
Predryer product	172	189	166	168
Precooler product	73	99	74	86
Dryer product	173	199	124	197
Cooler product	90	108	79	109
Concentration of solution, %				
First-stage neutralizer	67.7	84.7	64.9	79.5
Concentrator	93.8	94.2	92.4	90.4
pH				
First-stage neutralizer	5.6	1.6	6.1	1.3
Second-stage neutralizer	5.2	5.1	6.7	6.3
Granulator product	5.1	5.0	5.9	5.5
Chemical analysis of conditioned product, %				
Nitrogen	30.4	30.6	30.7	30.9
P ₂ O ₅	10.3	10.0
Sulfur	5.1	5.2
Moisture	0.27	0.34	0.17	0.15
Conditioner	2.6	1.9	2.8	2.2
Screen analysis of conditioned product (Tyler), cumulative, %				
+5 mesh	0.0	0.0	0.0	0.0
+6 mesh	0.0	0.0	1.0	0.0
+8 mesh	23.0	15.0	34.0	20.0
+12 mesh	95.0	94.0	99.0	97.0
+16 mesh	99.0	99.0	99.0	99.0
-16 mesh	1.0	1.0	1.0	1.0

ammonia required for neutralization was added to the first-stage neutralizer and only enough ammonia was added in the second stage to make up for losses in the system. The first-stage neutralizer was operated at a pH of 5.2 and a temperature of about 280° F. Ammonia losses were very high in the concentration step. The low-pH operation of the solution system was started in March 1967. The rates at which ammonia was fed and mother liquor returned to the first-stage neutralizer were adjusted to maintain the pH at about 1.6 (NH₃ to H₃PO₄ mole ratio of about 0.65) and the temperature at about 300° F. in that unit. The concentrator was operated at low-pH, and enough ammonia was added in the second-stage neutralizer to bring the pH in the final solution to the final range of 5.0 to 5.3. The concentration of the solution fed to the pan was about 95% and the temperature was in the range 335° to 340° F. The entire solution system

Table II. Typical Screen Analysis of Materials in Solids System
[Grab samples from 30-4.4-0 (30-10-0) production at low pH]

	Screen Analysis, Mesh % (Tyler)								
	+6	-6 +8	-8 +10	-10 +12	-12 +14	-14 +16	-16 +20	-20	
Recycle fines	2	6	26	27	16	12	5	6	
Product from granulator	4	11	32	22	11	11	4	5	
Product from predryer	2	10	31	26	10	12	2	7	
Product from precooler	0	15	64	17	2	2	0	0	
Product from dryer	0	14	66	16	2	2	0	0	
Product from cooler	0	14	66	15	3	1	1	0	
Conditioned product	0	16	64	15	4	1	0	0	

operated satisfactorily after changing to the low-pH method of operation. Nitrogen losses from the solution system dropped from about 6000 pounds per day during operation at high pH to the range of 300 to 500 pounds per day when operating the first-stage neutralizer at low pH. Operation at low pH and the higher temperature in the first-stage neutralizer substantially decreased the amount of steam required for evaporation. Typical operating data for the two methods of operation during production of the 30-4.4-0 (30-10-0) grade are shown in Table I. Screen analyses for the 30-4.4-0 (30-10-0) product are shown in Table II. The size and chemical composition of the granular products have been satisfactory, but generally the granules have not been so round and smooth as the products made in the pilot plant. The maximum production obtained from the plant during a calendar day was 566 tons of 30-4.4-0 (30-10-0) ammonium phosphate nitrate.

Pollution Control and Recovery of Process Losses. Process losses from the plant during preliminary operation were considerably higher than expected. These losses were in the form of dust and vapor evolved from process equipment and aqueous wastes from the floor sump and the concentrator hot well. Changing to the low-pH method of operation essentially eliminated ammonia losses

from the concentrator. Steady progress has been made in reducing the amount of dust and fume evolved from the equipment. A new hood and scrubber system has been installed for recovery of fume and dust from the pan granulators. The principal remaining source of process losses from the pan-granulation system is the aqueous wastes from the floor sump. This material is presently being pumped to a holding pond, because it is contaminated with lubricants spilled on the floor and cannot be returned directly to the process without an oil removal step. Methods for removing oils and other contaminants from the liquor collected in the floor sump are being studied. An infrared spectrophotometer was purchased for determining the amount of oil in the aqueous wastes and for evaluating methods of removing the lubricants from the aqueous wastes.

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