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MODIFIED DIAMMONIUM PHOSPHATE PRODUCTION IN A TVA TYPE GRANULATION PLANT

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The purpose of this paper is to describe the process used by International Minerals and Chemical Corporation to produce a modified dammonium phosphate having a guaranteed analysis of 18% nitrogen and 46% P_2O_5 (water soluble plus citrate soluble).

The process, identified as the "TVA Diammonium Phosphate" process, was selected after a thorough and comprehensive evaluation of the various processes available. The elements which IMC felt were most desirable in plant performance were as follows:

1. Finished product must meet high chemical and physical quality standards.

2. Plant must be capable of producing N-P-K fertilizers and granular triple superphosphate as well as diammonium phosphate.

3. Recovery of ammonia and phosphoric acid must exceed 98%.

4. Process must provide a low recycle ratio in order to limit equipment size.

5. Maximum standardization and duplication of equipment and parts being utilized in other IMC operations in Florida.

6. Plant must be capable of meeting Florida fume and particulate matter emission standards.

7. Plant must operate with a minimum number of operating, supervisory and maintenance personnel.

8. Capital investment must be at a minimum cost per ton of product.

After evaluations were made of the proposals by Engineering Contractors, the D.M. Weatherly Company of Atlanta, Georgia was selected to build DAP plant number one in 1961 and DAP plant number two in 1965.

PROCESS DESCRIPTION

The process is shown schematically in Figure 1 and is described as follows:

1. REACTOR (Figure 2)

Each DAP plant is equipped with a rubber lined reactor tank. The reactor for DAP plant number one is 12 feet in diameter by 20 feet high and for DAP plant number two, it is 12 feet in diameter by 25 feet high. Recycle acid from the scrubber sump and phosphoric acid containing 52-54% P_2O_5 are fed in metered amounts to the reactor. Anhydrous ammonia is sparged under the liquid level. The major chemical reactions taking place are:

(1) $NH_3 + H_3PO_4$ -------- $NH_4H_2PO_4$ + Heat

(2) $NH_3 + NH_4H_2PO_4$ -------- $(NH_4)_2HPO_4$ + Heat

The reaction represented by equation (1) goes to completion in the reactor. Since the reactor is normally controlled at an ammonia to phosphoric acid mole ratio of 1.4-1.5, the second reaction also takes place to some degree.

Phosphoric acid at 52-54% P_2O_5 is fed directly from a shift tank to the reactor. The flow is measured by a teflon lined Electromagnetic Flowmeter (EMF) with platinum electrodes, and is controlled by an air operated, rubber lined, flow control valve with a teflon diaphragm. The feed rate is held constant by a flow recording controller. Approximately 60% of the P_2O_5 is fed as 52-54% P_2O_5 acid.

The remainder of the P_2O_5 requirements for production is fed as 30% P_2O_5 acid. The acid comes directly from the phosphoric acid units to the 30% shift tanks at the DAP plant. This acid is pumped from the shift tanks to the scrubber sump tanks. The feed rate is measured and controlled by the same methods used with the 52-54% P_2O_5 acid.

The 30% acid is recirculated continuously from the sump tank through the scrubbers and back to the sump. Make-up water and/or dilution water is added continuously to the sump through a rotometer. The recycle acid is withdrawn continuously and fed to the reactor at a constant rate using an EMF meter.

Liquid anhydrous ammonia is fed to the DAP unit from storage tanks. All of the flow goes through the tubes of the ammonia cooler to remove any vapours which would cause any inaccuracies in metering. Leaving the cooler, the ammonia is split into two streams with approximately 70% going to the reactor to maintain the 1.4-1.5 mole ratio; the other 30% goes to the ammoniator to give the final mole ratio of 1.9 to 2.0.

The ammonia flow to the reactor is measured by a Rotometer and controlled by a Flow Recording Controller and

Flow Control Valve. The ammonia temperature drops, passing through the Rotometer and valves so that it can be used on the outside of the tubes of the cooler to cool the total incoming ammonia feed. The ammonia may be fed directly to the reactor spargers as a liquid or passed through the vaporizer and then to the reactor spargers as a gas.

Sulphuric acid is added to the process to maintain the grade at 18:46:0. This acid is added to the reactor in the number two plant and to the scrubber sump in the number one plant.

The slurry is pumped from the reactor to the ammoniator at a constant rate, and is metered by an EMF meter.

2. AMMONIATOR

Each DAP plant is equipped with a rotary ammoniator-granulator. The one for DAP number one is 10 feet in diameter by 20 feet long; the one for DAP number two is 11 feet in diameter by 25 feet long. The slurry from the reactor is distributed through spray nozzles above the rolling bed of solids in the drum. Anhydrous ammonia is distributed under the rolling bed through a distributor pipe.

Granulation is controlled primarily by the rate of recycle; other factors affecting granulation are:

1) Mole ratio in drum.

2) Evaporation rate in drum.

3) Particle size, moisture content and temperature of recycle.

4) Slurry composition and quantity.

5) Temperature.

The rotary ammoniator has a rubber lining which flexes and allows build-up to fall off. Retainer rings at each end of the drum regulate bed depth.

The normal mole ratio of the ammoniator product is 1.9-2.0 or sufficient to produce the desired 18:46:0 product. As the mole ratio increases the ammonia loss to the scrubber increases.

Granulated material flows from the ammoniator to the rotary dryer for moisture removal.

3. DRYING

a. Rotary Dryer Each unit is provided with a rotary dryer equipped with lifting flights. The dryer at DAP number one is 10 feet in diameter by 60 feet long; the one at DAP number two is 11 feet in diameter by 60 feet long. Hot gases from the combustion chamber enter the dryer co-currently with the ammoniator granulated material.

b. Combustion Chamber Burner The burner is a John Zink Model YE-22 combination oil and gas unit with John Zink, Jr. pilot assembly and Burner Block Model YB-7-22. The unit is rated at 30,000,000 BTU/hr. at a pressure drop of one inch water gauge.

The burner has instrumentation to meet Factory Mutual and Federal Insurance Association approval. Included in this instrumentation is a Honeywell safeguard unit with Honeywell Ultraviolet Flame Detector, alarm lights and horn, automatic start-stop circuitry and gas-oil changeover selector switch.

Primary air is automatically controlled from the burner air blower by a butterfly valve in one of the two air discharge lines. Steam atomization is used when operating on fuel oil. The unit is designed for gas or No. 2 fuel oil and no provisions are made for oil temperature control.

4. SCREENS

Four, 4 feet by 15 feet, double-deck, type 38 Tyler Hummer vibrating screens are used to separate oversize and fines from the product. The screens are equipped with dust hoods and are connected to the exhaust system for dust removal. Each screen is equipped with a Model 618D Tyler Thermionic power unit and three Model V-50 vibrators. Oversize from the screens is discharged to two 44 inch Stedman Cage Mills.

Fines flow by gravity to a 48 inch recycle conveyor.

5. CAGE MILLS

The system is equipped with two 44 inch Stedman Cage Mills for particle size reduction. The grinding action of the mill is produced by two rows of steel bars, one inside the other, rotating in opposite directions. Each mill is equipped with a 40 HP and 60 HP motor and is capable of handling 40-45 TPH of plus 8 mesh material.

Milled product is discharged to a 48 inch recycle conveyor.

6. PRODUCT SURGE HOPPER

Product size material from the screens is discharged to a surge hopper. The required recycle is transferred to a 48 inch recycle conveyor by the Product Recycle Feeder. Excess material is discharged through the hopper overflow and flows to the rotary cooler.

7. PRODUCT RECYCLE FEEDERS

The product recycle feeder is a Carrier Model FD 2480 Amplitrol Feeder rated at 150 TPH with 60 pounds per cubic foot material.

8. ROTARY COOLERS

Each DAP unit is equipped with an 8 feet by 50 feet Rotary Cooler. Product from the cooler is discharged to the product elevator and is transferred to storage by belt conveyors. A product weigh-bridge with totalizer is provided on the product belt conveyor.

Air from the cooler is exhausted through a cyclonic dust collector and a wet scrubber prior to being exhausted to the atmosphere.

9. EXHAUST SYSTEM (Figure 3)

a. Dry Dust Collectors The rotary dryer, rotary cooler, and mill and screen exhaust systems are equipped with dry cyclonic dust collectors for first stage dust removal. Dust from the cyclones discharges to the recycle conveyor. The discharge point of the chute is equipped with a flapper valve or rotary gate valve to prevent air leakage.

The cyclones are equipped with internal swinging chains to prevent solids build-up in the bottom discharge.

The exhaust gases are discharged from the top of the cyclone and flow to the wet gas scrubbers.

b. Doyle Scrubbers (Figure 4) The exhaust gases from the three cyclone systems are fed to a Doyle Scrubber. The dust-laden air enters through two round ducts at the top at one end of the scrubber. These ducts extend to just below the liquid level. Concentric cones in the discharge end of each duct greatly increase the velocity of the inlet air. Recycle liquor or slurry enters the inlet duct through a spray pipe above the cone and conditions the air by wetting the dust particles and lowering the air temperature. The high air velocity as it enters the thin slurry causes the dust particles to penetrate into the slurry giving a high dust recovery efficiency. The baffles cause the gas to reverse directions three times before exhausting to the fan and stack. Entrained liquid droplets are separated from the gas stream by these changes in direction. The liquid level is controlled by an overflow weir.

Phosphoric acid at 30% P_2O_5 is recycled from the scrubber sump to the Doyle Scrubbers and back.

c. Cyclonic Spray Tower The ammoniator and reactor tank are equipped with a cyclonic spray tower and exhaust fan. The gases from the reactor, primarily water vapour, and the gases from the ammoniator (air, water vapour and ammonia vapour) are combined and flow to the cyclonic spray tower where they are scrubbed with a phosphoric acid solution. The spray tower is equipped with block valves for control of the recirculating acid flow to the scrubber sprays. Acid flows from the bottom of the scrubber to the sump tank. Gases from the spray tower are exhausted to the atmosphere by the ammoniator exhaust fan.

10. STORAGE AND SHIPPING FACILITIES (Figure 5)

Diammonium phosphate is transferred to the storage building by belt conveyors. The product is distributed by means of a movable, reversible belt (shuttle) conveyor. The storage building has a capacity of 5000 tons of product, which is considered the minimum on-site capacity for satisfactory operation.

For shipping, the DAP is reclaimed from the storage building by front end loaders. The loader dumps the DAP on a floor grid where it flows to an adjustable speed apron conveyor. A 30-inch product belt conveys the product to the feed chute of the product shipping elevator.

The shipping elevator discharges the product to four Rotex screens. The screens are double-deck, dividing the feed into oversize, product, and fines.

Oversize from the screens is crushed in a Stedman Cage Mill. The cage mill discharges to the product shipping elevator for recycling to the screens.

The fines from the screens are transferred to the storage building and are returned as recycle when required.

Product from the screens goes to the shipping product bin. From there it flows directly to railroad hopper cars, trucks, or through a slinger (swivel loader) to box cars for shipping. Railroad cars and trucks are loaded on scales.

The shipping system is equipped with dust collection facilities. Each material transfer point or dust point is designed with an exhaust hood or similar device. The Rotex screens, shipping elevator, and cage mill are also hooded. The exhaust air streams are combined and passed through a Doyle wet gas scrubber.

RAW MATERIALS

The raw materials used in producing 18:46:0 are:

(1) 30% P_2O_5 phosphoric acid from the Prayon filter;

(2) 52-54% P_2O_5 phosphoric acid from the acid clarifying tank.

(3) anhydrous ammonia as either vapour or liquid; and

(4) 93% H_2SO_4.

A typical analysis of the 54% P_2O_5 acid is shown in Table 1.

TABLE 1

COMPONENT	PERCENTAGE
P_2O_5	53.4
CaO	0.3
SO_3	3.2
Fe_2O_3	1.4
Al_2O_3	2.0
K_2O	0.014
F	1.35
Na_2O	0.05
MgO	0.45
Solids	2.7

MATERIAL FLOWS

When the number one plant is operating at 30 tons per hour, the material flows in the system are as follows:

TABLE 2

Product, total	30 TPH
P_2O_5 @ 46.8	14.04 TPH
N @ 18.2%	5.46 TPH
Recycle	150-180 TPH
Ammonia to ammoniator	2.06 TPH
N	1.69 TPH
Sp. Gr. @ 90°F	0.59
GPM	13.9
Ammonia to reactor	4.73 TPH
N	3.89 TPH
Sp. Gr. @ 90°F	0.59
GPM	31.9
Phosphoric acid to reactor, 52% P_2O_5	16.54 TPH
P_2O_5	8.60 TPH
Sp. Gr.	1.80
GPM	36.7
Phosphoric acid to reactor, 30% P_2O_5	19.10 TPH
P_2O_5	5.73 TPH
SP. Gr.	1.29
GPM	59.2

OPERATING CONDITIONS

Table 3, shows various operating conditions (moisture, temperature, recycle, etc.) encountered in DAP plant two.

TABLE 3

Temperature of slurry to granulator	$235^{\circ}F$
Temperature of recycle to granulator	$155^{\circ}F$
Temperature of material discharged from granulator	$195^{\circ}F$
Temperature of dryer inlet gas	500°-$700^{\circ}F$.
Temperature of dryer exit gas	$185^{\circ}F$.
Temperature of product exit dryer	155°-$180^{\circ}F$
Moisture content of slurry to granulator	20%
Moisture content of recycle to granulator	1-2%
Moisture content of dryer feed material	5-7%
Moisture content of material discharged From dryer	1-2%
Recycle ratio	6:1

MANPOWER REQUIREMENTS

Two operators per eight-hour shift, and one-fifth of a supervisor's time are required to run each plant. Maintenance is supplied, when required, from a central maintenance force.

CHEMICAL ANALYSIS OF 18:46:0

The chemical analysis of 18:46:0 varies slightly due to variations in the impurities in the phosphoric acid. A typical analysis of product made during 1967 is shown below.

TABLE 4

COMPONENT	PERCENTAGE
Total nitrogen	18.2
Total P_2O_5	46.8
Citrate soluble P_2O_5	46.3
Water soluble P_2O_5	41.7
Calcium	1.0
Fe_2O_3	1.65
Al_2O_3	0.88
Fluorine	2.1
Sulphur	1.6

SCREEN ANALYSIS

Table 5 shows the typical screen analysis of 18:46:0 as it is loaded for shipment.

TABLE 5

TYLER STANDARD SIEVE SIZE	CUMULATIVE WEIGHT % RETAINED
+6	1.5
+8	20
+10	70
+14	96
+20	98

CONCLUSION

Both DAP plants have operated consistently above design capacity and the operating factor (operating hours per year) has been 10-15% higher than design. Plant maintenance has been well within the budgeted maintenance and is considered low for a granulation unit of this complexity.

SUMMARY

The diammonium phosphate plants described in this paper combine anhydrous ammonia and wet process phosphoric acid to produce a granular high analysis fertilizer. The units are designed to produce 18:46:0 at a rate of 200,000 TPY for plant number one and 260,000 TPY for plant number two. The product has a minimum chemical analysis of 18% nitrogen and 46% citrate soluble P_2O_5; the screen analysis is 97% passing through an 8 mesh, Tyler screen and retained on a 14 mesh screen. The unit is designed to recover 98% of the ammonia and 99% of the P_2O_5 fed to the process. These recoveries do not include shrinkage losses in warehousing and shipping.

In the process, wet process phosphoric acid (52-54% P_2O_5) is metered and pumped to a rubber lined reactor tank. Phosphoric acid containing 30% P_2O_5 is metered and pumped to the scrubbers to remove and recover ammonia vapour and dust from the reactor, ammoniator, cooler, dryer and screens-and-mills exhaust. The 30% phosphoric acid is then pumped at a controlled rate to the reactor.

In the reactor tank anhydrous ammonia is sparged below the slurry. The slurry consists of a mixture of mon-ammonium phosphate, diammonium phosphate and phosphoric acid. The distribution of these materials is such that the ammonia to phosphoric acid mole ratio of the mixture is maintained at 1.4 to 1.5. The partially neutralized slurry is pumped at a controlled rate to the ammoniator-granulator.

The rotary ammoniator contains a rolling bed of solids. Slurry from the reactor is distributed above this rolling bed, and anhydrous ammonia is added beneath the bed through a distributor pipe. The motion of the bed mixes the ammonia and slurry and aids in agglomerating particles. Ammoniation in the rotary drum increases the mole ratio of the material to 1.9-2.0 Solids from the granulator are discharged to a rotary dryer where the moisture content of the material is reduced to approximately 1.5%

Material from the dryer is fed by elevator to double-deck Tyler Hummer screens for size classification.

Oversize from the Tyler screens is discharged to Stedman Cage Mills for size reduction and returned to the recycle system. Fines from the Tyler screens flow by gravity to the recycle conveyor.

Product from the screens flows by gravity to a product surge hopper. The product surge hopper bottom discharge feeder conveys a controlled amount of material to the recycle conveyor. Overflow from the product surge hopper flows by gravity to a rotary cooler.

Cooler discharge material is conveyed to the boot of the product elevator where the material is elevated to a product belt conveyor for transfer to storage.

Material deposited on the recycle conveyor is transferred to the recycle elevator which elevates the material to the ammoniator feed inlet. This recycle material provides the rotary drum rolling bed. The quantity of recycle is controlled by regulating the amount of material transferred from the product surge hopper to the recycle conveyor.

The ammoniator, dryer, cooler, screens and cage mills are equipped with dry cyclonic dust collectors and/or wet scrubbing systems for removal of fumes and dust from the working areas and for recovery of dust entrained and fumes evolved in the process unit. Fines collected in the dry cyclones are returned as recycle. Phosphoric acid (30% P_2O_5) is used as the wet scrubber recirculating liquid.

Product conveyed to storage is discharged to the pile with a motor-propelled belt conveyor tripper.

DAP is reclaimed from the storage building by front end loader. The loader dumps the DAP on a floor grid and from there it is conveyed to the product shipping elevator.

The shipping elevator discharges to four Rotex screens. The screens are double-deck, thereby dividing the feed into oversize, product and fines.

Oversize material is crushed in a Stedman Cage Mill and recycled back to the screens. The fines are collected and returned to the granulation plant to be used as recycle.

Product size material from the screens flows to a shipping product bin where it can be discharged to railroad hopper cars, box cars or trucks.

The TVA diammonium phosphate process was selected after a thorough and exhaustive evaluation of the various processes available. It was felt that this process offered the following advantages to IMC:

1. Produces a product meeting high quality standards.

2. Flexibility of end products available.

3. High recovery of ammonia and phosphoric acid.

4. Low recycle ratio compared to other processes.

5. Standardization and duplication of equipment and parts being utilized in other IMC operations in Florida.

6. Ability to meet State fume and particulate matter emission standards.

7. Minimum operation personnel required.

8. Minimum capital cost per ton of product.

Both plants have operated consistently above design capacity and the operating factor (operating hours per year) has been 10-15% higher than design. Plant maintenance had been well within the budgeted maintenance and is considered low for a granulation unit of this complexity.

Figure I TVA TYPE GRANULAR DIAMMONIUM PHOSPHATE PLANT

ATELIER DE GRANULATION DE PHOSPHATE D'AMMONIAQUE DU TYPE TVA

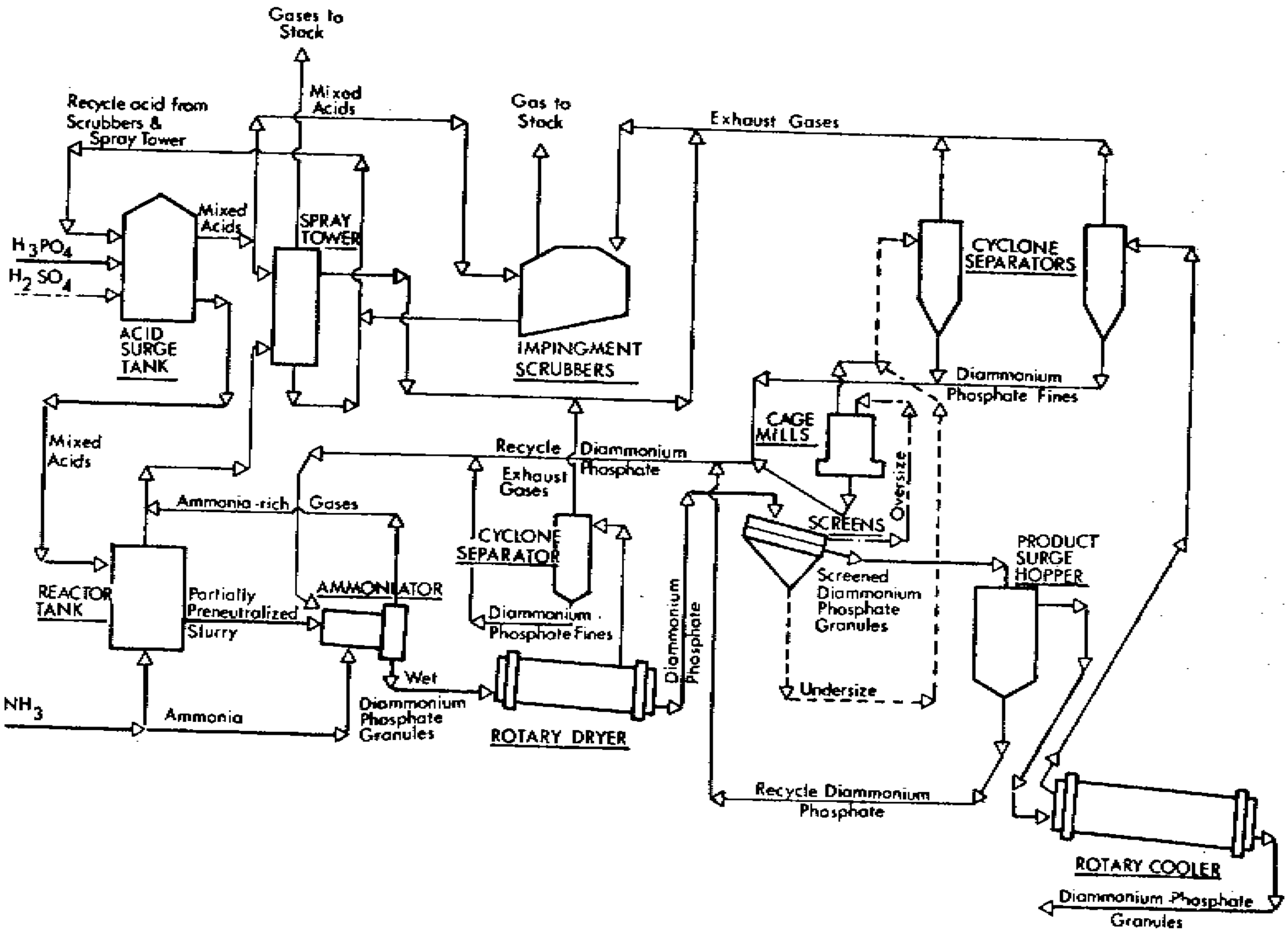


FIGURE 2 PRENEUTRALIZER APPAREIL DE PRE-NEUTRALISATION

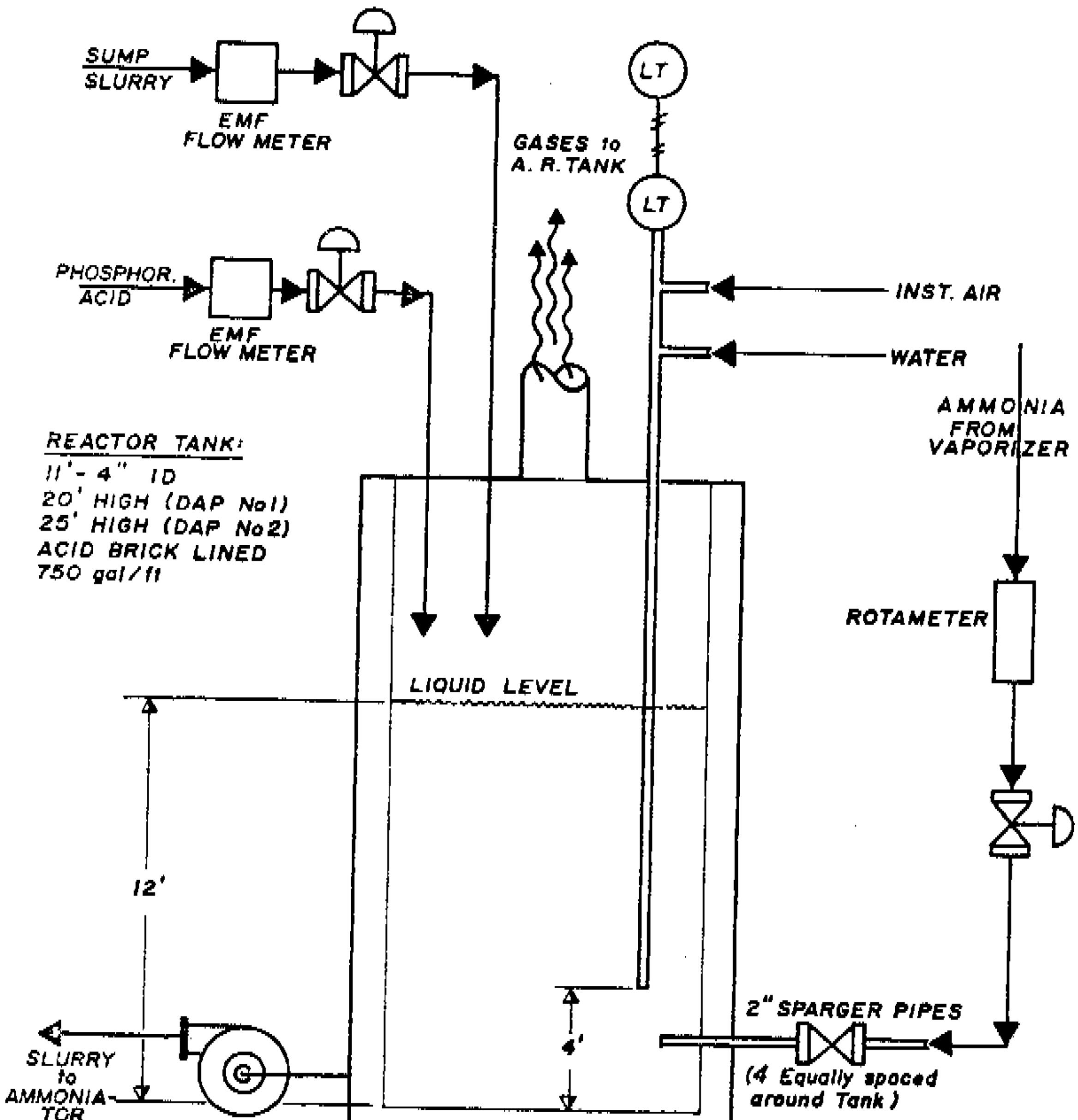


FIGURE 3 EXHAUST SYSTEM SYSTEME D'EVACUATION

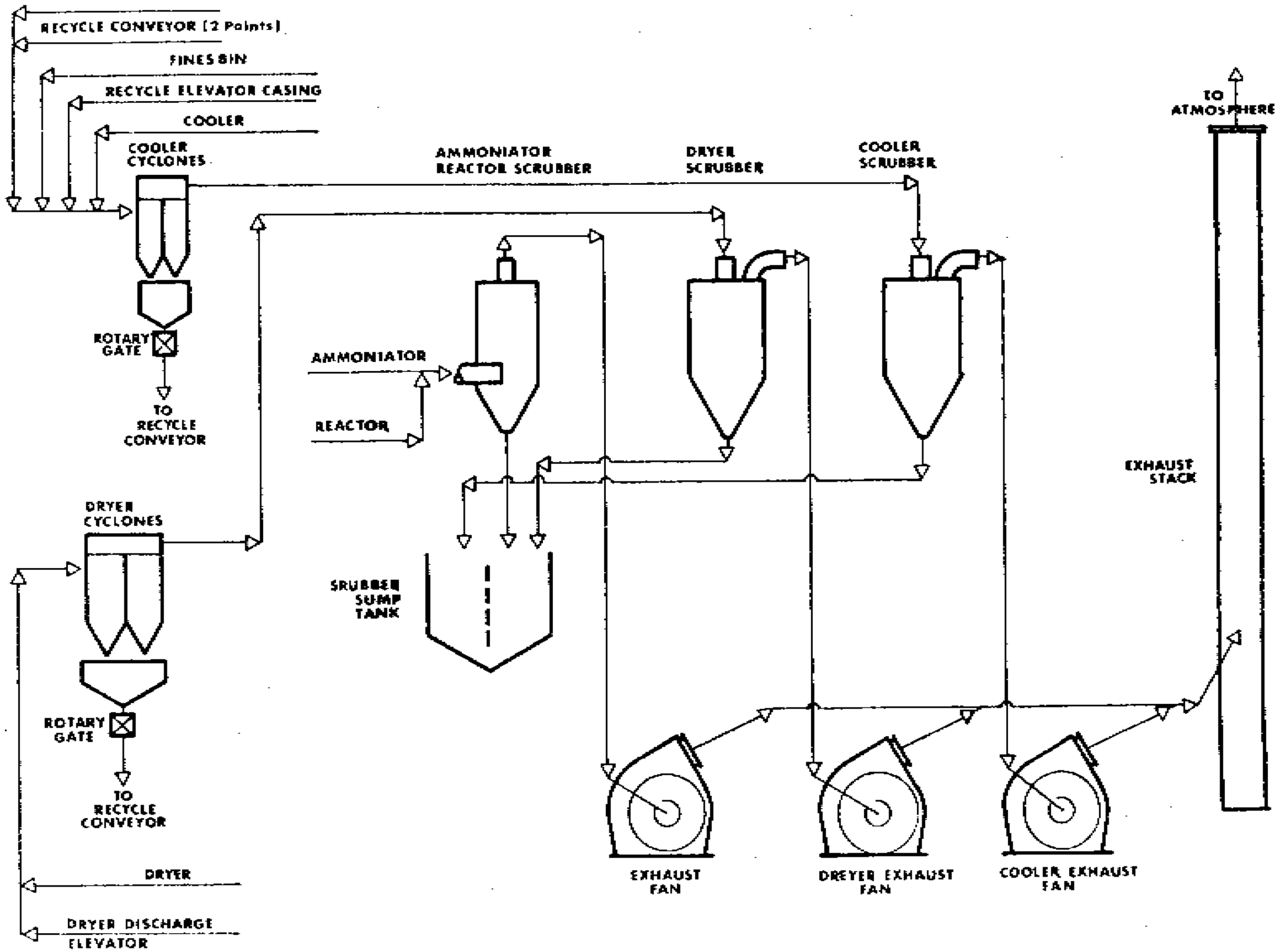


FIGURE 4 CUTAWAY VIEW OF DOYLE SCRUBBER
COUPE DU LAVEUR DOYLE

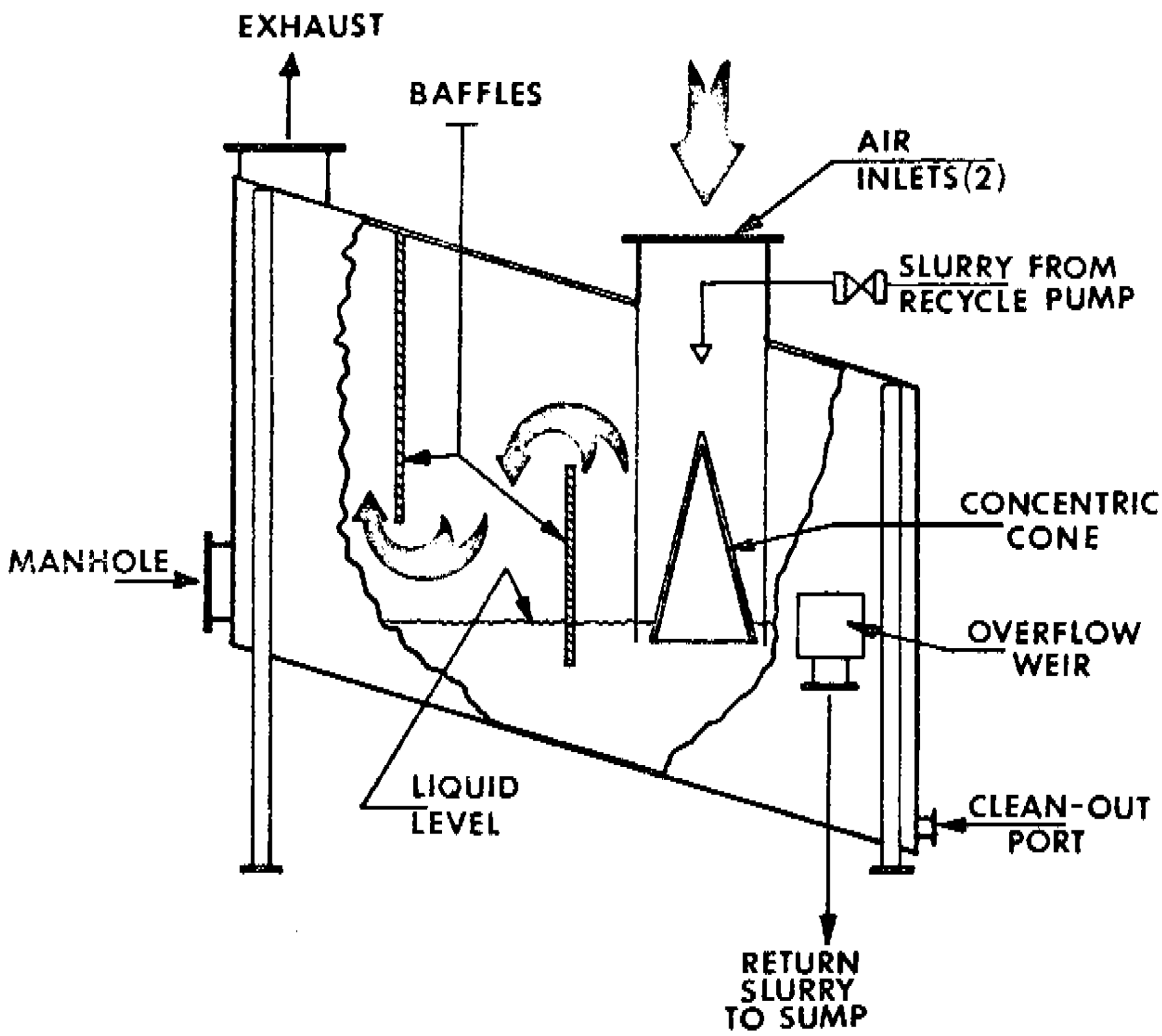
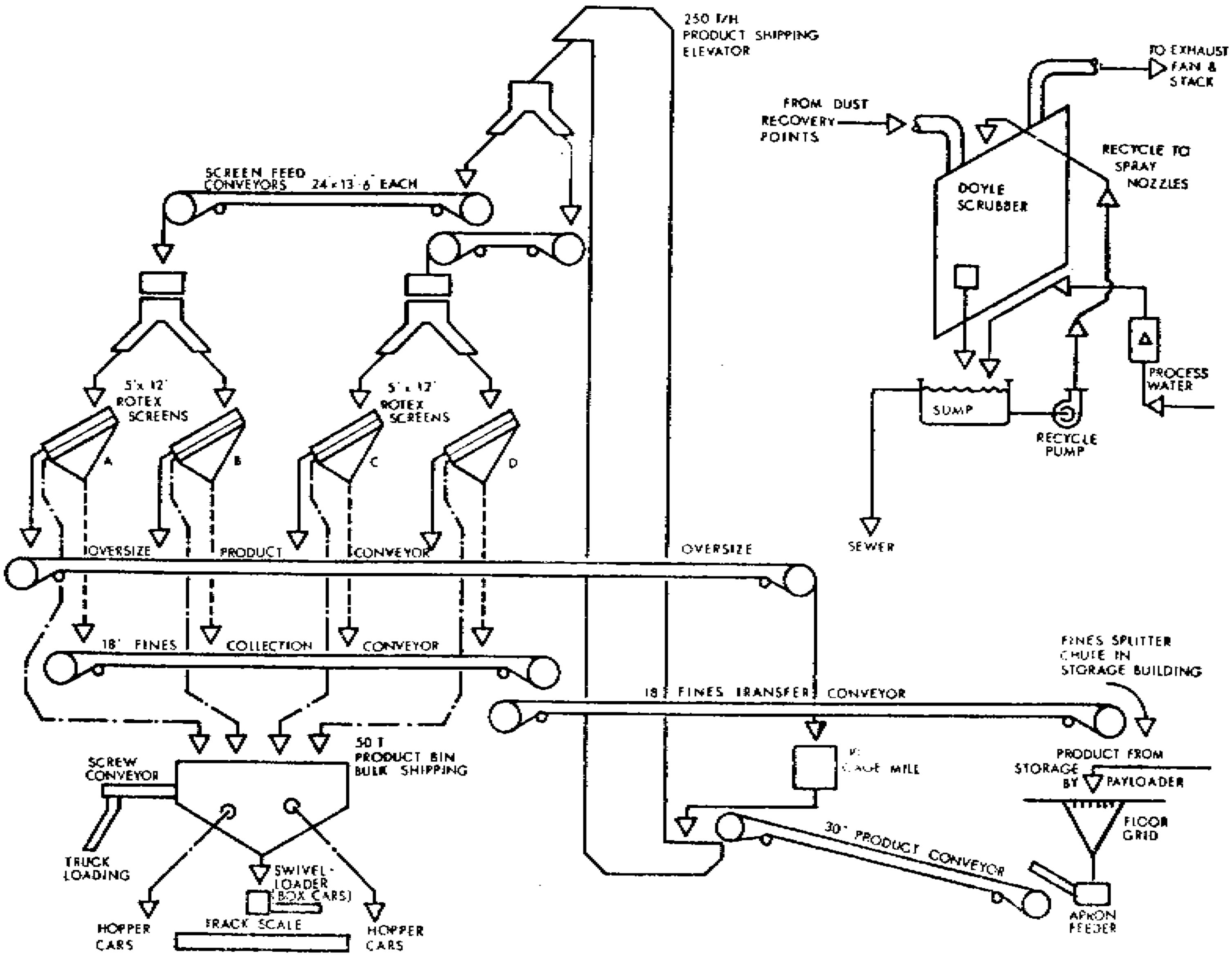


FIGURE 5 STORAGE AND SHIPPING FACILITIES

INSTALLATIONS DE STOCKAGE ET D'EXPEDITION



DISCUSSION

DR. J.H. ZOELLNER (Int. Minerals & Chem. Corpn.): presented the paper on behalf of Dr. Heck and read the summary.

DR. T.K. VAHERVUORI (Rikkihappo Oy, Finland): I have the pleasure to congratulate Mr. Heck and his colleauges on this most realistic and detailed paper.

The DAP plant has obviously been designed most scrupulously and you have been able to take into consideration the experience gained from the first unit when building the second unit.

There are some questions I would like to put to the authors. Firstly, the authors mentioned that the plant must also be able to produce N P K fertilizers and granular superphosphate. It is not clear from the paper whether these have been produced, but if so, what kind of experience have you gained? Have you any experience of what happens in the Doyle's scrubber when potassium chloride containing dust enters if the scrubbing solution contains phosphoric acid? Is there any corrosion damage to be expected? It would also be interesting to hear if any special arrangement or change of process is made when you produce triple superphosphate.

The second question concerns the reactor: when explaining the construction of the reactor you mentioned that it is rubber lined. In Figure 2 it is mentioned that it is acid brick lined. If the reactor is merely rubber lined will it continually be able to stand up to high drawing temperatures? On the other hand, if it is acid brick lined the high price of this kind of construction comes into one's mind. Is it not possible to obtain a more economical construction by using a suitable acid proof material? Your reactor is quite big and therefore the retention time is relatively high, maybe nearly 2 hours. There is no mechanical mixing here. Would it be possible to make the reactor smaller by arranging mixing?

The third question concerns the feeding and distributing of ammonia. Which do you think more profitable to use, liquid or gaseous ammonia? Doesn't a sudden evaporation of liquid ammonia in the reactor fix much of the reaction slurry unnecessarily? Is there any difference in the quantities of water evaporating from the reactor if you use liquid or gaseous ammonia?

Fourthly, in the scrubber and the cyclonic spray tower, obviously the same phosphoric acid 30% P_2O_5 is circulated in both scrubbers, when the total you gave in your paper was about 40% of total P_2O_5. It would be interesting to hear how P_2O_5 used in scrubbing is divided between these two scrubber types. Furthermore, I would like to ask you what the temperature in the cyclonic spray tower is? How much of the ammonia fed into the system evaporates and is recovered in the scrubbers? I should also like to know how much fluorine from the phosphoric acid evaporates from the reactor and how much of it goes through the scrubbers into the air.

The fifth question concerns the Stedman cage mills. What is the percentage of oversize granules in the material coming out of the ammoniator and what is the fineness of it after grinding? The whole system pre-supposes using two different concentrations of phosphoric acid.

The last question I would like to know is whether you have made comparisons for the possibility that all the acid used would be of the same concentration. You would naturally have to concentrate a larger amount of acid, but perhaps a concentration of about 45% would be sufficient.

DR. J.H. ZOELLNER: Firstly, I must point out that there was an error in the manuscript as submitted to ISMA. The reading should have been 6 mesh instead of 8 mesh when I reported the 97% smaller than 6 mesh and greater than 14.

The first question concerned the flexibility of the plant, in other words its ability to produce either DAP, granular triple or NPK fertilizers. When the DAP plant No. 1 was built, it was considered that we might produce NPK granular fertilizers, but the demand for the 18-46-0 was so great that we had to build a second plant and as a result, we have never operated in the plant on NPK. In between building the DAP No. 1 and No. 2 we built another granulation plant of the same size to produce granular triple superphosphate. So there are actually three of these units operating at the Bonnie complex. So the answer is that we have not gained any experience in producing NPK. If we were to produce NPK the intent was in 1961 that the Doyle's scrubber would be converted and, instead of using the 23-26% acid, which was started up with 30% diluted, we would probably have used water to scrub the gases which came from the dry cyclones because of the potential KCl content. We have gained considerable experience in scrubbing the off gases from NP granulation plants and have facilities located away from Bonnie. Mr. Heck will describe these studies and the scrubbers that we are at present using. My point here is that I believe we would draw upon this experience if it were ever decided to convert a DAP unit at Bonnie to NPK.

Part of this question also had to do with the production of granular triple superphosphate in the DAP unit. I mentioned that we have built a third unit primarily for the purpose of producing triple. This granular TSP plant had also been called upon to produce ammonium phosphates. However if we were to convert a DAP plant for the production of granular triple I think the basic exchange or modification that would be required would be to take out a part of the rubber panels which we are using to keep the inside of the DAP shell from building up. We would want to replace the first 10 feet of rubber panelling with a stainless steel liner because of the heat involved if we are to derive part of our P_2O_5 from the reaction of phosphate rock and phosphoric acid and the balance coming from run of pile triple.

The second question concerns the reactor. You are quite correct: it is not only rubber lined. We have 2 courses of acid brick in the bottom and on the sides of the reactor, each course measuring 2.5 inches for a total of 5 inches of brick lining on top of the rubber lining. When we built the second DAP plant, drawing upon the experience gained from the No. 1 unit we chose to use a similar construction for the reactor. We needed the assurance that we would be able to maintain a large throughput, a high tonnage and a high operating factor. We did not feel justified in experimenting with a different material of construction when the rubber lining and brick lining proved quite satisfactory. The reactors are quite large. I do not know from experience whether the size could be reduced through the use of mechanical agitation. Since the second DAP plant has been completed we have installed agitators in the reactor of both No. 1 and No. 2 plants. The prime purpose for installing the agitators at that time was to increase the ammonia recovery. The agitators are of 25 HP each and turning at 68 r.p.m.

The third question concerns the feeding and distribution of the ammonia. It was not very clear in the text but we will try to clarify it now. We use gaseous ammonia in the reactor in both the No. 1 and the No. 2 plants. The variation comes in the ammoniator where 30% of the ammonia is added. In DAP plant No. 1 we are geared up to use gaseous and liquid ammonia. The DAP No. 2 plant can only handle liquid ammonia. The No. 1 plant is presently running with gaseous ammonia and the No. 2 on liquid. We have never attempted to introduce liquid ammonia into the reactor proper, because of bumping as well as the heat requirements that will be taken away from being able to evaporate additional water. The main point here is the fluidity of the scrubber liquor, and if we lose potential evaporating water in the scrubber, there is less water to add to the scrubber sump and therefore the viscosity in the Doyle's scrubber and the cyclonic spray tower would be higher. The design of the ammonia sparger in the bottom of the reactor tank is merely 4 open ended pieces of pipe spaced at 90° to each other.

The fourth question concerns the scrubbers. You asked what the distribution of the scrubber liquor was to the Doyle and to the cyclonic tower. Again for standardisation we are using the same pumps for each purpose. They are 5 x 4 inch Wolfleys. One Wolfley supplies the scrubber liquor to the Doyle exclusively, while the second pump pumps the scrubber liquor to the cyclonic tower as well as furnishes the feed acid to the reactor, so that the stream is split. If we assume the pressure drop is the same for the two scrubbing towers, it would be somewhat less than half of the flow to the cyclonic and then half to the Doyle with the difference being the feed to the reactor tank. The temperature in the cyclonic spray tower ranges between 140 and 150° F. The lower the temperature that we can maintain, the higher the scrubbing efficiency. Almost all of the ammonia reaching the scrubber comes from the ammoniator granulator. The designed material balance is 10% of the ammonia or nitrogen fed to the ammoniator granulator. When the system gets out of balance it can easily go to 15% and I understand it has even reached 20%. However the cyclonic tower is very efficient and I don't think we would have achieved 8% recovery if the feed consisted of 20% to the scrubber. The fluorine and the phosphoric acid fed to the reactor is tied up as hydrofluosilicic acid which when reacted with ammonia would produce ammonium fluosilicates which would go out with the DAP as a solid end product, so I would not anticipate any considerable amount being released and going to the scrubbers.

The fifth question concerns the Stedman cage mills. You asked for the percentage of oversize granules in the material coming out of the ammoniator and what is the fineness of it after grinding or crushing. I have a screen analysis of the product coming out of the dryer rather than out of the ammoniator which should be similar. This is a simple sample rather than a composite sample, +4 mesh Tyler was 1.3%, +6 mesh 5.8%. A typical specimen of the product is 1.5% + 6. Obviously this sample was taken during a very good operation, the plant must have been in a perfect equilibrium, because it would only be necessary to crush approximately 4.5

or 5% of the total tonnage coming out of the dryer. When the system gets out of equilibrium, possibly due to a very wet bed in the ammoniator, the design of the plant is such that the cage mills are capable of handling 45 tons hour, which would be roughly 20% of the total tonnage discharged from the dryer. So there is quite a range in the design, and flexibility is there to get us out of trouble if the system got out of balance. I have not checked the screen analysis of the cage mill discharge in the DAP plant. We have checked it in our off size NPK plants many times and we would achieve 60 to 70% of the discharge of the cage mill as product size. It does not produce such a lot of pulverized dust as, for example, a roll crusher.

Your last question concerns the concentration of the phosphoric acid feed to the DAP plants. In the very early days of the DAP No. 1 they did attempt to run with a single acid source, which was 40 to 42%. We had continuous problems with the scrubbers plugging due to solids build-up and also to the fluidity of the acid, and ammonia recovery was particularly poor. So we promptly switched to the present two-acid system. We mentioned a 30% acid which is taken directly from the filters and diluted to approximately 23 - 26%. There are also advantages to be gained other than in the operation of the scrubbers. This relates back to the evaporators and the acid clarification circuit. We have less tons of acid to be evaporated, so we can run at lower throughputs and gain P_2O_5 efficiently at this point, and we achieve increased acid clarification due to the longer retention time.

MR. W. VAN BENTEN (V.K.F. Mekog-Albatros N.V., Holland): In Europe, we consider diammonium phosphate as a fertilizer with an analysis given in ammoniacal nitrogen and the phosphate as water soluble phosphate. In your paper, you give the analysis as nitrogen and the phosphate as citrate soluble phosphate. Why do you state this differently from our normal system of guaranteed analysis?

Secondly, can you indicate what percentage of your phosphate in this fertilizer is water soluble?

DR. J.H. ZOELLNER: In the United States, P_2O_5 is sold on an available P_2O_5 basis, the available being defined as water soluble plus citrate soluble. Most of our customers would not know the relationship if we reported it as water soluble. I do realize that in many countries in Europe you are on a water soluble basis. We quote 41.7% P_2O_5 as the water soluble content and if I recall correctly, the guarantee on this material is that 90 or 93% of the available P_2O_5 as we defined it, is also guaranteed as water soluble.

MR. P. MORAILLON (Péchiney St. Gobain, France): I should like to raise a point about the calorie consumption indicated on page 4. This consumption appears to be very high. Is there a single combustion chamber for both units or does each unit have a combustion chamber? How are these combustion chambers arranged with regard to the introduction of additional air? Is this air added at the entry of the combustion chamber, or at the exit, to obtain the fairly low temperature of 260-360° F which is indicated?

Secondly, could we have details of the design of the two ammonia distribution systems in each of the units? They must be different, because the first unit is designed for liquid ammonia as well as for gaseous ammonia.

Thirdly, it is stated that the gaseous effluents corresponded to the state pollution standards, although the gases are not washed in water scrubbers.

Could we know what is the fluorine content of these flue gases emerging from the cyclone scrubber and from the Doyle scrubber?

DR. J.H. ZOELLNER: There is a separate combustion chamber for each of the DAP dryers, as the plants are in separate buildings. From memory I do not believe that I could describe the details of the design of the Doyle scrubber. The primary and secondary air is admitted at the front of the combustion chamber and the bulk of the air is pulled in just prior to the hot gases entering the dryer. So therefore the exit gas temperature of the combustion chamber would be considerably higher than reported, but because of all the diluting air at the inlet to the dryer we can achieve the lower temperatures reported.

The ammonia distributor pipes in the two plants vary only as to the diameter of the pipe below the rolling bed in the ammoniator-granulator. The configuration and drawing of the holes in the pipe is similar except in size. We would calculate the number of holes and the diameter of the holes primarily on the volume flow, which obviously would be quite a bit lower for the liquid ammonia.

Mr. Heck has a paper this afternoon on effluent control which touches upon the different fluorine emissions. Fluorine emissions from the DAP plants are extremely low. As I previously mentioned, the fluorine in the phosphoric acid is converted to ammonium silico-fluoride which stays with the solid DAP product. The Bonnie complex has an overall fluorine limit imposed by the State of Florida. We are allowed 0.4 g/m3/ton of P_2O_5 produced in the plant. The bulk of the fluorine is emitted from the phosphoric acid plant, the triple superphosphate plant and the evaporators. The amount from the DAP plant is extremely low.

DR. A. BROWNLIE (S.A.I. Ltd., United Kingdom): In table 3, the moisture content of slurry to granulator is quoted as 20% and the recycle ratio as 6: 1. Are these average figures or minimum figures? If they are not minimum figures, could the author give some idea of the variation?

DR. J.H. ZOELLNER: The moisture content of the slurry to the granulator is an average of 20%. The range is 15-25%. The water recycle ratio varies from as low as 5:1 to probably as high as 6:1. Again I would say that the typical average would be just about in the middle: 5.5:1.

MR. RAEMAKERS (Ugine Kuhlmann, Belgium): Table 1 on page 7 gives the analysis of 54% phosphoric acid. On the same page there is a mention of 52-54% P_2O_5 phosphoric acid from the acid clarifying tank. I should like to ask firstly whether the 2.7% of solids shown in the analysis originates in the clarifying tank and, if so, what is the initial content? Secondly, if the CaO content is 0.3%, what do the solids consist of? Thirdly, with regard to the fluorine content of 1.35%, is this figure derived from filtered acid?

DR. J.H. ZOELLNER: The suspended solids in the No. 1 filtrate, as most of you probably know, can vary considerably depending upon the condition of the filter media. I don't believe that I even have a decent range that I could report. Again it would be dependent on the rate of production. I think we have approximately 24 hours of setting time at normal production rates. When we run the two DAP plants at above design rates, obviously the retention time would drop considerably. Again the figure reported in table 1 is an average figure taken over a period of probably one year. The underflow from the clarification circuit would consist primarily of gypsum and iron and aluminium phosphates. This material is fed to the triple superphosphate plant to recover the P_2O_5 values. Again all of the figures in table 1 are an annual average and this would be typical of what is in the acid feed to the reactor in the DAP plant which, in other words, is after the clarification step. The 52-54% acid is clarified while the 30% acid fed to the scrubber is direct from the filter with no additional clarification.