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PRODUCTION OF AMMONIUM PHOSPHATE IN A SPRAY TOWER

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Summary

A spray tower process for producing ammonium phosphate has been investigated in our laboratories in Landskrona. Phosphoric acid mixed with some sulphuric acid is sprayed into gaseous ammonia in a tower. The heat of reaction between the ammonia and the acid is sufficiently large to evaporate the water in the droplets while they are falling through the tower, and a dry powdery product is removed from the bottom. A stream of gas is withdrawn from the lower parts of the tower and passed to a condenser where the water vapour formed in the reaction is removed. The uncondensed gas is heated and recirculated back to the top of the tower and mixed with the ammonia feed.

The minimum strength of the acid that can be used is determined by the fact that the amount of heat evolved by the reaction must be sufficient to evaporate the water. Calculations of the heat balance in our plant show that for pure phosphoric acid a concentration of at least 45%  $P_2O_5$  will be necessary. If sulphuric acid is added, lower  $P_2O_5$  concentrations can be used. A formulation diagram has been constructed, showing the relationship between the  $P_2O_5$ - and  $H_2SO_4$ -concentrations of the feed acid for various amounts of heat added to or lost from the system.

The product obtained from an acid containing 38 - 39%  $P_2O_5$  and 7-8%  $H_2SO_4$  is a fine powder, 75% through ASTM 120, with an approximate composition of 19.5% N, 44.5%  $P_2O_5$  and 2.8% S. The molar ratio of the ammonium phosphate is 1.95. Its bulk density is as low as 0.5 g/cm<sup>3</sup>, but a sample has been successfully granulated to hard sharp-edged particles with a bulk density of 0.8 g/cm<sup>3</sup>.

Introduction

The spray tower process for producing ammonium phosphate from wet-process phosphoric acid has been investigated in our laboratories in Landskrona for some years. In this process the acid is sprayed into an ammonia atmosphere in a tower. The heat of reaction between the ammonia and the acid is sufficient to evaporate the water in the acid droplets, and the product can

be removed as a dry powder from the bottom of the tower. The spray process has been in use in Japan for many years, but it was decided to carry out this investigation to gain first-hand knowledge of the process.

The main advantages of the process are:

Dry ammonium phosphate with a high molar ratio  $N_2PO_4$  is obtained. The capital and operating costs for a spray<sup>4</sup> tower plant are lower than the costs for a conventional slurry process of equal capacity.

The disadvantages of the process are:

The product is a powder, which normally must be granulated. Further, to produce a pure diammonium phosphate one has to use a phosphoric acid with a minimum concentration of about 45%  $P_2O_5$ . A weaker acid contains more water than can be evaporated by the heat of reaction. As the heat of reaction between ammonia and sulphuric acid is somewhat higher than between ammonia and phosphoric acid, a weaker acid may be used if mixed with a proper amount of sulphuric acid. However, the product will then necessarily be a mixture of phosphate and sulphate.

### Apparatus

The pilot-plant is shown schematically in figure 1. The spray tower (1) consists of a welded cylindrical shell, 2 m. in diameter and 4 m. high, with a flat bottom and a dome-shaped roof. It is externally insulated with mineral wool, and the process may be observed during operation through a couple of windows. The tower is made of mild steel. A rotating disk sprayer (4) is positioned in the centre of the roof. It has a capacity of 200 - 250 kg/h, and all parts in contact with the acid are made of stainless steel.

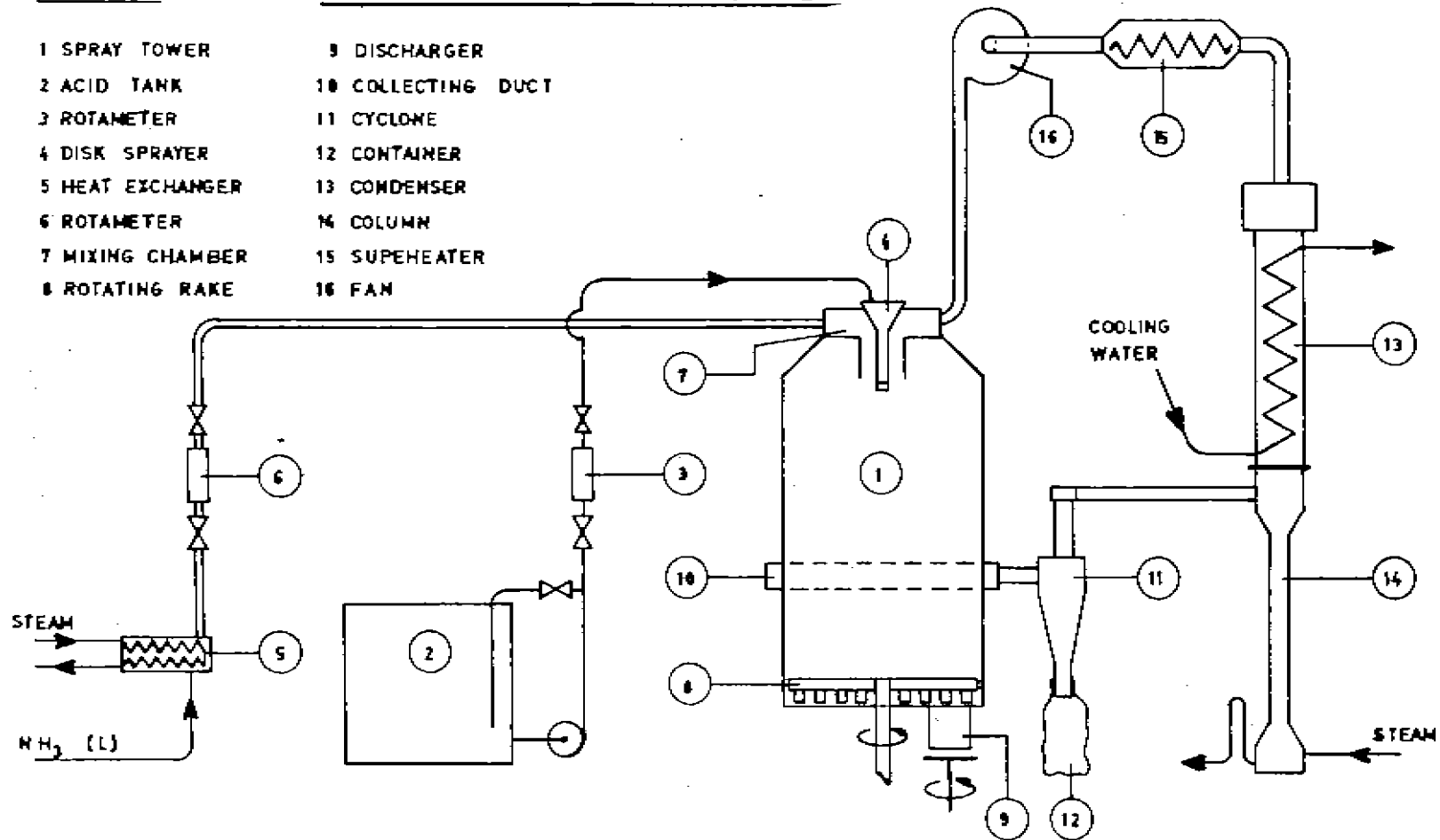
The acid is pumped from a 2 m<sup>3</sup> tank (2) through a rotameter (3) to the sprayer. Liquid ammonia from a mobile tank (capacity 1.5 ton) is evaporated by steam in a shell-and-tube heat exchanger (5). The vapour passes through a rotameter (6) to an annular chamber (7) surrounding the upper part of the sprayer unit, where it is mixed with recirculated gas. This chamber is extended downwards by a tube, arranged concentrically around the sprayer, and ending just above the rotating disk. By this arrangement the paths of the droplets emitted from the sprayer are bent downwards by the issuing gas, and the amount of unreacted acid reaching the walls of the tower is reduced.

The dry product is continuously removed from the bottom of the tower. A rotating rake (8) pushes the powder down through a vertical tube to a rotating disk discharger (9). The vertical tube is kept filled with powder to prevent gas leakage from the tower.

FIGURE 1.

SPRAYTOWER AND AUXILIARY EQUIPMENT

- |                  |                    |
|------------------|--------------------|
| 1 SPRAY TOWER    | 9 DISCHARGER       |
| 2 ACID TANK      | 10 COLLECTING DUCT |
| 3 ROTAMETER      | 11 CYCLONE         |
| 4 DISK SPRAYER   | 12 CONTAINER       |
| 5 HEAT EXCHANGER | 13 CONDENSER       |
| 6 ROTAMETER      | 14 COLUMN          |
| 7 MIXING CHAMBER | 15 SUPHEATER       |
| 8 ROTATING RAKE  | 16 FAN             |



The gas, mainly ammonia and water vapour, and some air, is withdrawn from the tower through a number of openings in the shell positioned along a circumference one metre above the bottom. These outlets are equipped with steel sheet "eyelids" to avoid excessive entrainments of solids. The gas streams are collected in a duct (10) encircling the tower, and led through a mineral wool insulated cyclone (11) to a water-cooled vertical shell-and-tube condenser (13) where most of the water is condensed. The condensate runs down through a column (14) filled with Raschig rings, where dissolved ammonia is stripped off by direct steam. The gas from the top of the condenser passes through a gas fired superheater (15) to the circulation fan (16) and back to the mixing chamber (7).

During the investigations certain parts of the apparatus have been changed. At first the acid was atomized by means of a pressure nozzle, with the ammonia issuing from an annular slit of adjustable width around the nozzle. The recirculated gas entered the tower through ports placed one metre above the bottom and was taken out at the top. Breakdowns occurred regularly because the nozzle became plugged with solid impurities in the acid, and it was decided to change to the present rotating disk sprayer. At the same time, on advice from the manufacturer of the new sprayer, the recirculation of the gas was changed to the co-current system used now, with the gas entering around the sprayer. This was done to prevent the atomized droplets from reaching the upper parts of the tower, as mentioned earlier. (It is, however, possible that better results could be obtained with countercurrent recirculation of the gas in a tower of sufficiently large diameter, as the more fully reacted droplets would then be in contact with relatively dry gas).

#### The heat balance

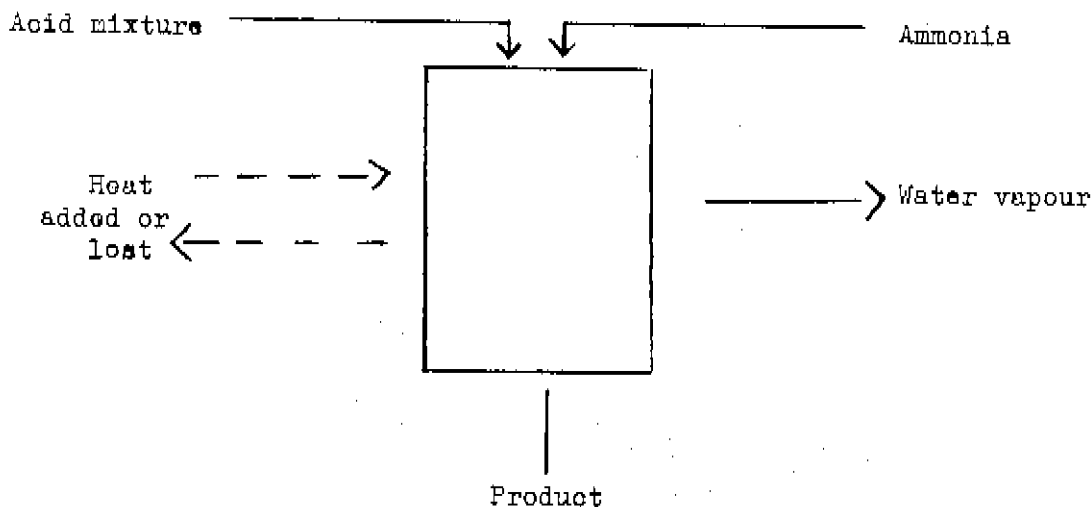
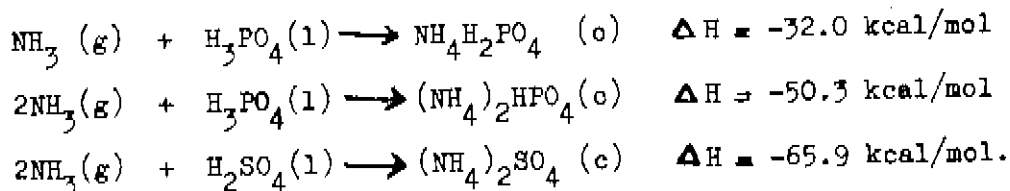


Figure 2. Simplified spray tower system for the heat balance calculations.

When producing ammonium phosphate and ammonium sulphate by the spray process, the heat balance is of special significance. If the heat of reaction is not sufficient to evaporate the water in the acid and also compensate for any losses from the tower, additional heat must be supplied. To obtain a relationship between the necessary concentrations of  $H_2SO_4$  and  $P_2O_5$  in the feed acid, the heat balance has been calculated for the system shown schematically in figure 2. For the calculations the following three reactions have been considered.

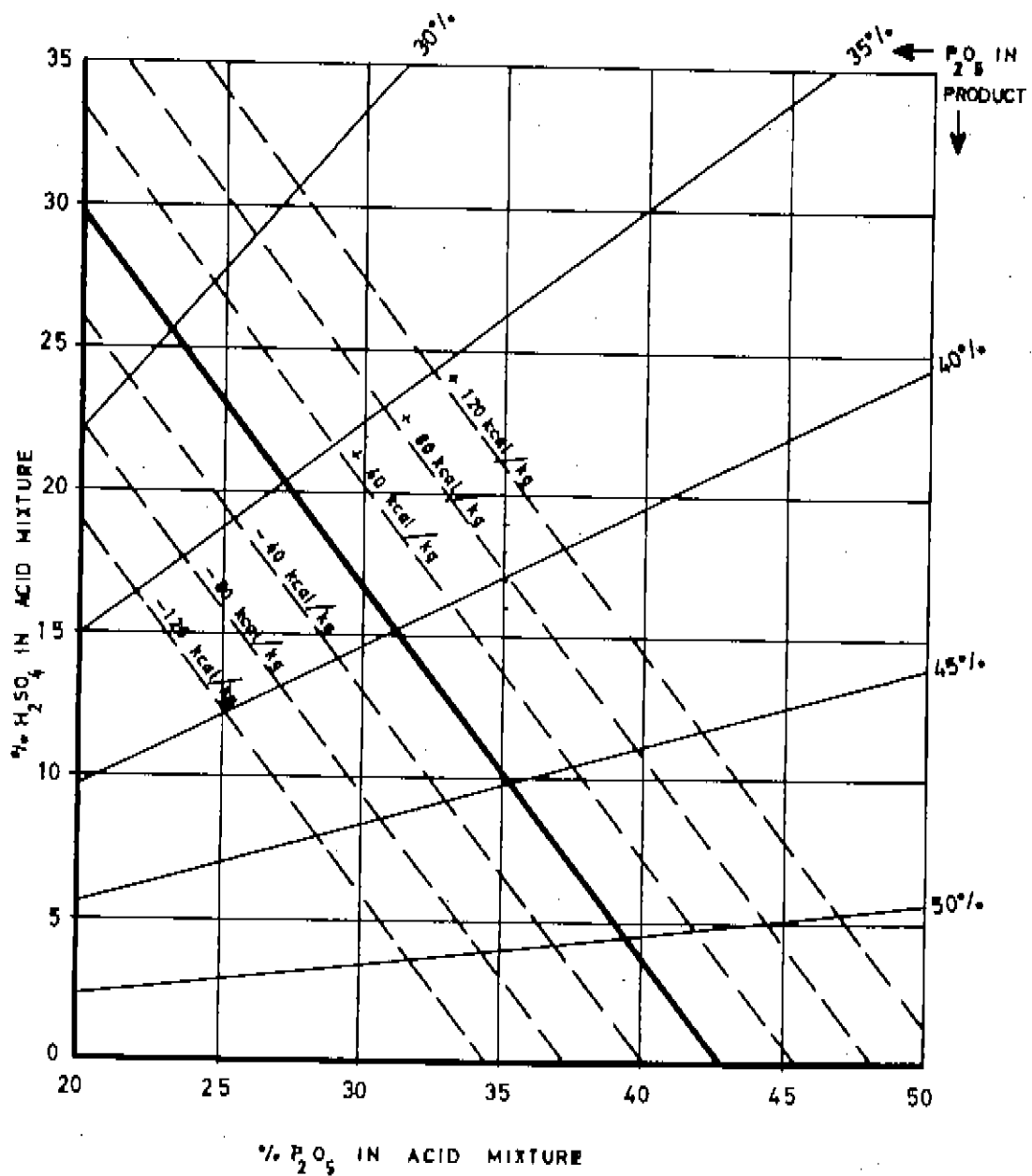


Based on experience gained from the pilot plant, the following assumptions concerning the operation variables have been made. The temperature of both the acid and the ammonia (gas at slightly above atmospheric pressure) entering the process is supposed to be  $15^\circ C$ . The product is a crystalline salt mixture leaving the tower at a temperature of  $110^\circ C$ . Its specific heat has been taken as  $0.3 \text{ kcal/kg } ^\circ C$ . Water vapour is withdrawn from the system at  $110^\circ C$ . The heats of dilution for the phosphoric and sulphuric acids, given in the literature and confirmed by laboratory tests, are taken as constant within the concentration limits. This approximation has only a slight effect on the result. Finally a value of 1.95 has been set for the molar ratio  $N:PO_4$  in the product.

The results of these calculations are shown graphically in figure 3. As can be seen, a linear relationship between the  $H_2SO_4$  and  $P_2O_5$  concentrations is obtained. The thick unbroken line represents the case when the heat of reaction is just enough to evaporate all the water and bring the temperature of the product and the water vapour up to  $110^\circ C$ . Parallel to this line are the lines for constant deficit and excess heat in the system. In practice there will always be a certain heat loss from the tower, so that an acid giving an excess heat of reaction must be used. There are, however, cases when the sulphur content of the product must be low and this may prohibit the use of enough sulphuric acid to fulfill the heat requirements. An extreme example of this would be the production of pure ammonium phosphate from an acid containing less than about 43%  $P_2O_5$ . Extra heat must then be supplied to the system, for instance by heating the circulating gas.

Figure 3 also shows the resulting  $P_2O_5$  content of the product for a given composition of the acid. As the product is a mixture of diammonium phosphate and ammonium sulphate, the nitrogen content will be constant at 21% N, since both the ingredients have the same nitrogen content. For instance, an acid mixture containing 35%  $P_2O_5$  and 17%  $H_2SO_4$  will give an

FIGURE 3. HEAT BALANCE IN THE SPRAYTOWER BASED ON THEORETICAL VALUES.



THE BROKEN LINES IN THE DIAGRAM GIVE THE EXCESS OR DEFICIT HEAT OF THE SYSTEM IN  $\text{kcal/kg}$  ACID MIXTURE.

excess amount of heat of nearly 80 kcal/kg acid mixture and a dry product containing 21% N and 40%  $P_2O_5$ .

In the pilot plant we have used an acid with a concentration of 38 - 39%  $P_2O_5$  and 7 - 8%  $H_2SO_4$ . According to figure 3 this acid gives an excess heat of about 20 kcal/kg acid mixture. This is correct only for a chemically pure acid. Normally wet-process phosphoric acid contains a great deal of impurities, which will reduce the water content of the acid and thereby reduce the amount of heat needed to evaporate the water. In a heat balance diagram for ordinary wet-process phosphoric acid, the balance line would be somewhat displaced towards lower acid concentrations.

### Operation

During normal operation of the plant, using an acid mixture containing 38 - 39%  $P_2O_5$  and 7 - 8%  $H_2SO_4$ , the production rate has been 170 - 200 kg dry product per hour. About 20% of this is taken out through the cyclone. It should be possible to raise the production capacity of the plant considerably with the same tower dimensions, but in that case a sprayer of greater capacity would be needed.

The product obtained from the above mentioned acid has the composition of approximately 19.5% N, 44.5%  $P_2O_5$  and 2.8% S. According to figure 3 the theoretical N- and  $P_2O_5$ -contents in the product should be about 21% resp. 47%. This discrepancy is due to impurities in the wet-process acid used. The impurities amount to 5 - 6% of the dry product.

As we did not have access to phosphoric acid of higher concentration, no attempts were made to produce pure ammonium phosphate. However, a certain amount of sulphur in a fertilizer is no disadvantage, as sulphur is an important plant nutrient in itself.

As the droplets fall through the tower, ammonia is absorbed from the gas, and water is evaporated into the gas. To achieve a high rate of reaction the concentration of ammonia should be high, and the water should be removed from the gas as fast as possible. This means that the rate of gas circulation must be above a certain minimum. During the trials a constant circulation rate of about 800 Nm<sup>3</sup>/h was maintained. The temperature in the tower should be at least 105°C and preferably 110°C. The temperature of the gas leaving the condenser was about 60°C, corresponding to a water vapour content of 20 - 25%.

In order to avoid a blanketing effect around the droplets, the concentration of air in the gas must be kept low. As the product outlet in the bottom of the tower is not quite gas-tight, a small overpressure of about 20 mm of water is maintained in the tower. Also, care is taken to prevent air leakage into



the circulation gas, which is at below-atmospheric pressure between the outlet of the tower and the circulation fan.

As mentioned earlier, the circulating gas enters the tower through an annular concentric tube around the sprayer. With the type of sprayer used, a flow area of this tube giving a gas velocity of 10 m/s at the outlet was found to be suitable to efficiently deflect the atomized droplets from the roof and the upper part of the wall of the tower.

In the pilot plant, acid is fed to the sprayer at a constant rate during a run, while the ammonia feed rate is regulated by hand to give a constant pressure in the tower. This mode of operation seems to be the easiest way of controlling the process, and should be applicable in a commercial scale plant on an automatic basis.

The main causes for loss of ammonia in the process are leakage through the product outlet from the tower, entrainment in the product and inefficiency of the stripping column. The first two of these causes appear to be rather insignificant. In most of the trials carried out until now, a stripping column was used which was not designed for this kind of service, and losses as high as 5% were measured. A new properly designed column has now been installed, but no measurement of its efficiency has yet been made. However, the losses of ammonia are estimated to be below 1%. In addition, product particles which are not recovered in the cyclone will dissolve in the condensate and leave the process through the stripping column. Determinations of  $P_2O_5$  and  $SO_4$  in the condensate during the experiments showed a loss of 2% of the production due to this effect. The old cyclone has now been replaced by a more efficient one, but no quantitative measurements have been made so far.

#### The product. Characteristics and uses.

From an acid containing about 39%  $P_2O_5$  and 7%  $H_2SO_4$  a product containing 19.5% N, 44.5%  $P_2O_5$ , 2.8% S and 0.2% moisture was obtained. The impurities amounted to about 6%. The product was very fine-sized, 75% through ASTM 120 (i.e. 75% with an effective diameter less than 0.125 mm). The molar ratio  $N:PO_4$  was 1.95, which is rather high compared with that of ammonium phosphate produced by the slurry process.

Ordinarily, the product as withdrawn from the spray tower will not be marketable in that form. To increase its bulk density and in general make it easier to handle, a granulation step has to be added. Wet granulation with subsequent drying will usually not be suitable, as this leads to losses of ammonia. (However, if a high  $N:PO_4$  molar ratio is not essential, the product may be sprinkled with a small amount of phosphoric acid before or during granulation). A better method

seems to be the so-called "compacting process". According to this method, the powder is continuously passed between two heavy rolls, which are pressed together. The powder is compressed and appears as flakes which are broken and screened to give hard, sharp-edged particles of desired size range. The powder usually has to be heated before it is compacted in this way, and this again increases the danger of ammonia losses. By adding urea to the powder as supplementary nitrogen, the heating can probably be omitted. This method of compacting has been tried on the pure product NP 19.5-44.5 and on the product mixed with ammonium sulphate and potassium chloride to give NPK 15-15-15, with satisfactory results. The bulk density of the pure product was increased from  $0.5 \text{ g/cm}^3$  as powder to  $0.8 \text{ g/m}^3$  after compacting.

The experiments with the spray tower process have given satisfactory results and a larger plant may be built based on the experience gained. Considering its low bulk density, the product seems suitable for use in connection with some other type of NPK-process or as a raw material for producing mixed NPK-fertilisers granulated by the compacting process.

DISCUSSION

Mr. L. G. NILSSON (A.B. Förenade Superfosfatfabriker, Sweden) :

In recent years there has been a world-wide trend towards more highly concentrated fertilisers. Much of this concentration has been achieved by using ammonium phosphate instead of superphosphate as the source of phosphorus. A large number of processes for producing both pure ammonium phosphate and NPK fertilisers based on ammonium phosphates have been developed. In our laboratories at Landskrona, the spray-tower process for producing ammonium phosphates has been investigated and is described in this paper. The advantages of this process are that a dry product with a high molar ratio is obtained and that the capital and operating costs of the process are comparatively low.

The principle of the spray-tower process is that the acid - in our case a mixture of phosphoric and sulphuric acids - is sprayed into an ammonia atmosphere. The heat of reaction evaporates the water in the acid, and the product is extracted from the bottom of the tower as a dry powder.

In our pilot plant, we have used an acid with a concentration of 19%  $P_2O_5$  and 7% sulphuric acid. The acid is fed to a rotating disc sprayer placed in the top of the tower. The ammonia, together with circulating gas, enters the tower through a tube arranged concentrically around the sprayer. The gas containing the evolved water vapour is led from the tower through a cyclone, where entrained particles are collected. The gas is led to a condenser, where most of the water vapour is removed. Dissolved ammonia in the condensate is stripped off by direct steam in a packed column. The gas from the condenser is superheated and recirculated to the tower. Ammonia is mixed with the circulating gas at the inlet of the tower.

The spray tower operates at about  $110^{\circ}C$  and is held at a slight over pressure to avoid inert gas - in this case, air - entering the system.

Two main types of losses of material are encountered in this process. Ammonia is lost, owing to leakage in the system; the amount is difficult to estimate but probably does not exceed 2% of the  $NH_3$  input. Salt dust carried over to the condenser dissolves in the condensate and is lost in the stripping column. The amount of product lost in this way depends on the efficiency of the cyclone, but it has been found to be less than 1%.

When an acid of 39%  $P_2O_5$  and 7%  $H_2SO_4$  is used, one obtains a product containing 19.5% N, 44.5%  $P_2O_5$  and 0.2% moisture. The average particle size is about 0.1 mm. A granulated 15-15-15 NPK fertiliser, containing the spray-tower product as the sole source of phosphorus, has successfully been made by the compacting process. The product can also be used as an ingredient when producing NPK fertilisers by some other process or in another plant.

Mr. J.P.A. MACDONALD (Scottish Agricultural Industries Ltd., U.K.) :

The process described in this interesting paper follows the present trend towards processes which are autothermic. By that I mean that the heat of reaction in the process provides the total heat requirement for moisture removal, heat losses, etc. The many and varied methods which are being studied at present will lead to a more general understanding of the principles involved, and this paper is a useful addition to our fund of knowledge.

On page 5, information is given on the method of calculating heat balance. From this it appears that the product and water vapour leaving the system at 110°C are not taken into account. In addition, heat is required for the superheater (item 15) and the ammonia distillation column (item 14). Can the author please indicate the overall thermal efficiency of the process when these factors are taken into account?

Again on page 5 and elsewhere, it is explained that the product which is a finely divided powder leaves the process at 110°C. Does any caking occur in storage, or does the product have to be cooled or treated in any way to improve its keeping qualities. The most important consideration relates to the methods of use referred to on pages 8 and 9. As stated, a low-density fine powder is not likely to be found suitable for wet granulation, and so compaction with urea and potassium chloride is suggested to give products such as NPK 15-15-15. Does the author have any information on the overall economics of producing such NPK products by spray-drying followed by compaction in comparison with the conventional slurry processes?

Mr. NILSSON : The heat loss from the product and water vapour leaving the tower at 110°C is taken into account when calculating the heat balance, but the superheater and the distillation column are not taken into account. The overall thermal efficiency is difficult to calculate exactly, because we have a gas-fired superheater, and the only way open to us is to measure the temperature of the circulating gas before and after the superheater. The temperature difference in this case is about 30°C. As far as the distillation column is concerned, we use direct steam at the rate of about 35 kg. of steam for a production of 180 kg/hour.

Our product is free flowing and we have not met with any serious caking problem.

We have not carried out any exact comparison between a slurry process and the process using a spray-tower and a compacting plant, but I think it is obvious that the capital cost of the latter process must be much lower than for a slurry process plant.

Mr. M. DETUNCO (Péchiney-St.Gobain, France) : Is it not beneficial to feed hot phosphoric acid into the spray tower, e.g. at 80 ~ 100°C, which would permit a reduction in its concentration for the same proportion of sulphuric acid?

Does the condenser become rapidly blocked up? How do you clean it?

What is the overall steam consumption of this plant, and, in particular, what is the steam consumption of the superheater?

Mr. NILSSON : No attempt has been made to use hot phosphoric acid in our pilot plant, but I think it could be used without any difficulty, if the right construction materials were used. An increase in the temperature of the acid from 15° to 80-100° would decrease the required concentration of pure phosphoric acid from 43% to about 40%  $P_2O_5$ . The heat balance shown in Fig. 3 would be displaced to a deficit heat of -40 kcal/kg.

The condenser we have is a tube heat exchanger, in which the gas passes inside the tubes. The amount of dust entering the condenser depends on the efficiency of the cyclone. We previously had a cyclone with a relatively low efficiency, but not even in this case did we have any serious trouble with blocking in the condenser. This depends, I think, on the condensed water, with which we have washed the condenser continuously. If we want to clean the condenser, we can easily take away the column, and the condenser tubes can then be cleaned with a brush on a long shaft.

Steam is used for two purposes in the plant : firstly, as direct steam for distillation of the condensate, and secondly in a heat exchanger for vaporisation of ammonia. As I mentioned before, we use about 35 kg steam per hour for distillation at 2 atm. overpressure for a production rate of 180 kg/hour. For evaporation of ammonia, 55 kg steam per hour at the same pressure and production rate is used. In total, we use about 80 kg. steam per hour.

Mr. P. MORAILLON (Péchiney-St. Gobain, France) : Have trials been made without any addition of sulphuric acid?

Can you give any information on the flow properties of the product obtained? Can it be stored in a silo without difficulty?

Do you think that by using higher towers it would be possible to obtain a product with a larger average particle size, thus avoiding subsequent granulation?

With regard to the heats of reaction quoted on page 5, they are given with liquid phosphoric and sulphuric acids and are valid for 100% acids. Where a solution is used - and this is the case in practice - the heats of reaction are lower. Is it not necessary to subtract the dilution heat of the sulphuric or phosphoric acid?

Mr. NILSSON : We have made no trials without sulphuric acid, because we have no evaporated acid in Landskrona. The highest concentration we had was the semi-hydrate acid referred to in Mr. Hakansson's paper, i.e. 42%  $P_2O_5$ , and this is not sufficient.

In my opinion, it is very difficult to store a fertiliser in a

silo, and I do not think this ammonium phosphate can be stored in this way.

It is, indeed, possible to achieve larger particles, but it is difficult to say whether this could be achieved by means of a higher tower. I think the circulation of a diluted slurry to the sprayer could give larger grains in the product.

The reaction heats on page 5 are, indeed, for 100% acids, but in the heat balance diagram the dilution heats are taken into account. On page 5, I write : "The heats of dilution for the phosphoric and sulphuric acids given in the literature and confirmed by laboratory tests are taken as constant within the concentration limits." But, to be exact, we would not have straight lines in the balance diagram just because of the dilution heats.

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