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SAVINGS ON ENERGY IN GRANULATION OF FERTILISERS BY A NEW METHOD OF USING PIPE REACTORS

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1) INTRODUCTION

Ever since granulated fertilisers have been made from a slurry containing fertilising salts in solution and suspension, a technique introduced by the company Dorr-Oliver in the nineteen-thirties, a constant concern of the manufacturers has been to reduce the quantity of recycled solid product required to act as support for the liquid phase and to regulate the granulation. The recycling rate was considerable in the initial plants, often more than 10 times the production rate. In the case of NP/NPK fertilisers based on ammonium phosphate and a salt contributing the additional nitrogen, the replacement of ammonium sulphate by ammonium nitrate or urea, which are far more soluble than the sulphate at the temperature of the granulator, has only increased the difficulty.

The efforts made during the last twenty years have led to considerable progress in reducing this recycling rate.
According to the NP/NPK formula manufactured, this rate now generally lies between 3 and 6 times the production rate, starting from raw materials which are liquid for the most part, i.e. phosphoric acid, ammonia, concentrated solutions of ammonium nitrate and urea. So long as the desired recycling rate was determined by the quantity of liquid phase, however, and not by the granulation yield, it was conceivable that an even better figure could be achieved. The considerable increase in the cost of energy, plus the high price of investments, was an added incentive to pursue this line of investigation.

Profiting from the experience acquired in previous years by the operation of 8 pipe reactors for the neutralization of phosphoric and sulphuric acids in its various plants, Générale des Engrais perfected in 1979, on an industrial scale, an improved process for the granulation of high-content fertilisers based on substantially liquid raw materials. Thanks to a new concept in the use of pipe reactors, this process enables the recycling rate to be reduced considerably, at the same time improving the water balance and the ammonia yield. A substantial saving on energy is achieved, both in electricity and in fuel, and a significant diminution in the cost of investments for a given production capacity.

Before describing this process, we shall begin by recalling briefly the prior state of the art.

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(1) Currently SICNG, Greece.

2) USUAL MEANS FOR REDUCING THE RECYCLING RATE

In the case of the traditional plants comprising a separate granulator (mixer, revolving drum, or inclined rotary disc) and dryer, three means are currently used:

- Diminution of the water content of the slurry. This diminution is possible, without excessive increase in viscosity, by suitable adjustment of the molecular ratio $NH_3 : H_3PO_4$, or else by introducing all or part of the ammonium nitrate liquor into the phosphoric acid neutralization tank. Co-neutralization of the nitric and phosphoric acids is also possible, enabling slurries with less than 3% water to be obtained.

- Cooling of the recycled product, by means of a cooling drum placed downstream of the dryer, before screening.

- Introduction of all or part of the ammonium salts in solid form.

Monobasic ammonium phosphate (MAP) in powder form is generally used for this purpose, manufactured in a separate plant, e.g. by means of a pipe reactor. Granulated dibasic ammonium phosphate (DAP) can also be employed, or prills of ammonium nitrate. The introduction of urea in solid form is also current practice.

These three means, used separately or in combination, have enabled considerable progress to be made. They nevertheless have certain disadvantages:

- In the case of highly concentrated, not to say almost anhydrous, slurries, grain quality is often unsatisfactory, and the recycling quantity required is still determined by the amount of the liquid phase, which increases with the temperature of the product in the granulator, itself a function of the thermal balance.

- The cooling of the total product at the dryer outlet increases the investment, consumes electrical energy, and constitutes a thermal loss, which increases the fuel consumption considerably.

- The use of solid MAP, highly effective in reducing the recycling rate, requires for its preparation additional investment, plus manufacturing and servicing expenses, and entails an increased fuel consumption, since the heat of crystallization and the sensible heat of the MAP are lost.

Another quite different route, patented by the company SIAPE in 1953 (1), then in 1955 by C and I GIRDLER under the name of "Spherodizer", (2, 3,4,5), consists in preparing a liquid slurry containing all the liquid and solid raw materials, including potassium chloride and the dust particles of the cyclones, and then combining the granulation and the drying by pulverizing the slurry inside a single drum, which serves both as granulator and dryer.

This process permits operation with a low recycling rate (1 to 2 times the production rate), but has four main disadvantages:

- High water content of the slurry to be pulverized, entailing a high fuel consumption.

- Production capacity limited to about 700 t/day by "Spherodizer".

- Impossibility of manufacturing DAP, or DAP-based fertilisers, by this process, in the absence of an ammoniator-granulator to effect a super-ammoniation in the solid phase. The additional ammonia contained in the DAP, relative to MAP, must therefore be replaced by another nitrogenous material (sulphate, nitrate, urea), which is more expensive.

- The process is not versatile. For example, unlike the traditional plants, it does not permit the granulation of PK fertilisers based on superphosphates.

This was the situation when the neutralization pipe reactor technique made its appearance. Because of the low water content of the slurries which are obtainable with it, the prospect of fresh progress was opened up. Before studying its application to granulation, we consider a short history of its precursors in this field to be useful.

3) NEUTRALIZATION PIPE REACTORS, SHORT HISTORY

It may appear surprising that one had to wait until the seventies for the use of pipe reactors to be developed in the fertiliser industry.

For nothing is simpler or more obvious than the idea of mixing and reacting two liquids, or else a liquid and a gas, by introducing them into the same pipe. No doubt the human mind is naturally inclined to complicate matters, and the simplest solutions are often those which take the most time to discover and put into practice.

It must not be imagined, however, that the idea of pipe reactors was thought of yesterday; "There is nothing new under the sun" (Ecclesiastes 1,9). The earliest description which we have found of a device whose principle is similar to that of the pipe reactor dates back to 1927, in a patent of IG Farben Ind. (32). This involves a sort of atomiser placed on top of a tower, into which the mixture of inorganic acids (nitric, sulphuric, phosphoric) is introduced axially and atomised by means of ammonia, or of a mixture of air and ammonia, which arrives concentrically. The product is obtained directly in the form of a dry powder.

The period from 1947 to 1965 saw the filing of numerous patents describing the use of a pipe reactor for the neutralization of sulphuric, phosphoric and sometimes nitric acid, separately or in a mixture, by anhydrous liquid ammonia, gaseous ammonia, or ammonia in solution.

We have listed these various patents in the following table 1, classifying them according to the main use for which the pipe reactor was intended.

TABLE 1 - Pipe reactors - Patents applied for between 1947 and 1965

Use of the pipe reactor	Company	1st date	Ref.
Direct feeding of a granulator-ammoniator mixing drum	Swift Continental Oil	25.01.50 3.02.65	6 7
Feeding of a granulator-ammoniator drum via a cyclone separator	Swift Consolidated Mining Dorr-Oliver	3.11.52 30.12.60 30.10.62	8 9 10
Direct obtaining of solid ammonium salt, in particular MAP powder, by prilling	Kerley Swift Sonic Eng.	27.01.55 1.04.65 17.08.65	11 12 13
Liquid fertiliser based on ammonium orthophosphate	Barnard Monsanto Barnard Sonic Eng.	28.11.56 21.01.63 30.08.60 17.08.65	14 15 16 13
Ammonium polyphosphate solid or in solution (liquid fertiliser 10.34.0 or 11.33.0), from orthophosphoric or superphosphoric acid	Collier Carbon Dorr-Oliver W.R. Grace Swift W.R. Grace	22.08.60 18.03.64 30.03.64 18.08.65 30.08.65	17 18 19 20 21
Manufacture of ammonium nitrate (Stengel packed pipe reactor)	Commercial Solvents	3.05.47	22

During the period considered, 1947 to 1965, it seems that industrial embodiments remained very rare. An article which appeared at the beginning of 1956 (23) described a Stauffer granulation shop, in Los Angeles, the granulator drum of which is fed by a pipe reactor, inclined at 45°, called an "ammoniator" or "bazooka". The anhydrous ammonia enters axially, while the sulphuric or phosphoric acid is introduced tangentially.

Towards the end of the fifties, the company W.R. GRACE (30) was using a concentric pipe prereactor for feeding with liquid raw materials the double arm kneader of its Davison granulation shops. The mixture of phosphoric and sulphuric acids arrived by an internal axial pipe into the T-shaped reactor, while the anhydrous ammonia or the nitrogenous solution arrived in the annular space through the lateral branch, an opposite arrangement to that of the T-shaped reactor of the T.V.A.

The Stengel packed pipe reactor, for the manufacture of ammonium nitrate, was used as long ago as 1953 (24), but although the principle is similar, the design differs notably from that of the ammonium sulphate or phosphate reactors.

All in all, subject to omission on our part, it seems that in the patent field the precursors have been:

- Swift for the use of pipe reactors in granulation.

- Kerley for the manufacture of monobasic ammonium phosphate in powder form.

- Dorr Oliver for the manufacture of ammonium polyphosphate from orthophosphoric acid containing 50-58% of P_2O_5.

As far as industrial embodiments are concerned, it seems that the precursors are:

- Stauffer for the feeding of a granulator by a pipe reactor.

- Swift for the manufacture of MAP powder (25) and that of ammonium polyphosphate from orthophosphoric acid (26).

From 1966 onwards, patent filings and publications on the design and use of pipe reactors have multiplied, and it would be impossible to cite them all here. Mention will nevertheless be made of the numerous publications of the TVA and the company Cros, whose work has made a significant contribution in this field. Attention should also be drawn to Montedison's pipe reactor with forced recirculation (27).

If the pipe reactor is intended specifically for the neutralization of sulphuric acid, as is described in a Gardinier patent (28), instead of calling the apparatus a "bazooka", as in the case of Stauffer, it would seem even better to use the equally descriptive term "sulphator".

4) PIPE REACTOR FEEDING THE GRANULATOR

The neutralization of the phosphoric and/or sulphuric acid in a pipe reactor which runs directly into the granulator is now traditional, and has been the subject of numerous industrial embodiments.

In the plants of our company, the first pipe reactors came on stream in 1974, first of all in the existing shops, then in two new large-capacity shops. The pipe reactor was fed, as the case required, with liquid or gaseous ammonia, with dilute and/or concentrated phosphoric acid, and in some cases with 93% sulphuric acid.

In the manufacture of medium-content fertilisers, containing superphosphate and/or ammonium sulphate, the results have been highly satisfactory. The pipe reactor replaced with advantage the neutralization tank, enabling the slurry to be atomized effectively into the granulator at constant speed with neither slurry pump nor slurry flow indicator.

Conversely, in the manufacture of high-content fertilisers based on ammonium phosphate and ammonium nitrate, such as 17.17.17, 18.22.12, 23.23.0, it was found necessary to maintain a recycling rate just as high as, if not even higher than, when using a neutralization tank, despite the fact that a slurry with a low water content was obtained.

This disappointing result must no doubt be attributed to the fact that the pipe reactor injects into the granulator a slurry and some steam at high temperature, which heats the contents and increases the solubility and the liquid phase. If a preneutralization tank is used, the steam resulting from the heat of neutralization is released from the slurry before its introduction into the granulator, whereas in the case of the pipe reactor the heat of reaction occurs wholly in the granulator.

This disadvantage is confirmed by the experience of other producers. Thus the new NPK shop of UKF at PERNIS presented at the last ISMA Technical Conference in Vienna (29) comprises a rotating cooler after drying, which is used to control the granulation with a low recycling rate in the case of high-content fertilisers, for example 14.28.14.

In the case of our own shops, the problem has been resolved provisionally by introducing solid monobasic ammonium phosphate in place of part of the phosphoric acid. But as we have seen earlier, this solution cannot be regarded as really satisfactory. Another route therefore had to be sought.

5) PIPE REACTOR OPENING INTO THE DRYER

Since the need for a high recycling rate is the consequence of an excess of liquid phase and heat in the granulator, the simplest solution is to divert this excess into another unit of the granulation system, where the crystallization of the salts and the evacuation of the steam can take place almost instantaneously.

Without major addition to the existing equipment, this unit can be none other than the dryer, hence the idea of installing a pipe reactor at the top of the dryer, as we envisaged as long ago as 1975.

The opportunity to build such a plant presented itself in 1979, at Grand-Quevilly near Rouen, in the company's biggest granulation unit with a capacity of 1500-2000 t/day of high-content NP/NPK fertilisers. The neutralization tank of this unit, designed to function with the molecular ratio NH_3 : H_3PO_4 of 0.7 suitable for the manufacture of fertilisers based on monobasic ammonium phosphate, became too small when it was desired to manufacture fertilisers based on dibasic ammonium phosphate, from a slurry with a molecular ratio of 1.4, i.e. a doubling of the ammonia flow rate. Instead of complementing the tank with a pipe reactor opening into the granulator, it was decided to install this pipe reactor at the top of the dryer.

Ever since this unit came on stream in May 1979, the results obtained have exceeded expectations: reduction of 40 to 60% in the recycling rate, increase in the production capacity of 50 to 60% (limited by the final product cooler), improvement in the ammonia yield, reduction of 30 to 50% in fuel consumption. The same installation was used later to manufacture dibasic ammonium phosphate 18.46.0.

At a later stage, four other units equipped with a pipe reactor opening into the granulator have been fitted with a second pipe reactor running into the dryer, with equal success. By regulating suitably the distribution of the phosphoric acid between the two pipe reactors, it becomes possible to control at will the recycling rate, instead of being at its mercy as in the prior art.

We shall now give some details of the installations and the operating conditions in certain manufacturing plants.

6) DESCRIPTION OF A TYPICAL INSTALLATION

6.1 GRANULATION SYSTEM (Table No. 1)

The process utilizes a traditional granulation system; the reactions of preneutralization of the phosphoric acid by ammonia take place in two pipe reactors, one in the dryer tube, the other in the granulator tube.

- The pipe reactor of the dryer atomizes a concentrated slurry of ammonium phosphate at the top of the rotating tube.

The reactor is of simple and proven design. Its position and orientation are adapted to the internal equipment of the dryer.

In our Rouen unit, its production is 30 t/h of ammonium phosphate. It is fed with liquid ammonia and with concentrated, non-clarified phosphoric acid. The concentration of the acid (about 50-52% of P_2O_5) depends on the origin of the acid and its solid matter content. In our installations we have experience of acids from Togo, Morocco, Tunisia, Florida, and up to 15% solid matter content.

The reactor functions with the atomic ratio N/P of 1.05; its ammonia yield is thus more than 98%. The flows of the two fluids are linked by a proportionnator, thus preventing the arrival of acid alone in the dryer tube.
The back pressure can fluctuate between 3 and 5 bar.

- The pipe reactor of the granulator is designed to receive all the other fluids making up the formula.

  - liquid or gaseous ammonia (if it is wished to make use of the heat of vaporization to treat the air of the cooler).
  - non-clarified phosphoric acid of varying concentration.
  - sulphuric acid 93%.
  - scrubbing liquids from the gas processing plants.
  - possibly concentrated solutions of ammonium nitrate or urea in the downstream part of the reactor, thus guaranteeing their atomization. These concentrated solutions can also be introduced directly into the granulator.

The ammoniation ratio can fluctuate between 0.6 and 1.4 according to the formulas. The yield will then fluctuate between 99 and 90%.

- The complementary ammoniation is ensured by fitting an injector into the granulator which is adapted to the siting of the reactor in order to avoid accumulation of product on this equipment.

6.2 TREATMENT OF GASES

The increasingly strict requirements of the legislation on the environment and the fact that this installation comprises a reactor able to carry out up to 20% of the fixing of the ammonia have led us to design of our own accord an installation capable of resolving the problems linked with fluorine and ammonia, and of eliminating any liquid effluent, thus providing guaranteed yields of 99.5%.

The installation comprises venturis and a cyclone column. The first scrubbing stage receives the moist and dusty gases from the granulator and the dry and dusty gases from the dryer. The gas-liquid contact is ensured in venturis with small pressure drop, sprayed with a recycled solution of ammonium phosphate, ammonium sulphate or ammonium sulphophosphate with a pH of between 4 and 5 permitting good absorption of the fluorine. The two gas flows pass through a cyclone column sprinkled with an acid solution (pH 2 to 3) before escaping through the single stack of the installation. The liquid effluent of the second stage is introduced into the first stage. The bleed of the first stage is sent to the pipe reactor of the granulator. The concentration of the recycled liquid is controlled by topping up with fresh water.

6.3 OPERATING CONDITIONS

- For a given formula, the pipe reactor of the dryer is fed in such a way as to obtain in the granulator an optimum liquid/solid ratio for the granulation. Thus the proportion of phosphoric acid introduced into the reactor of the dryer can fluctuate between 50 and 100%.

- The feed of acids to the scrubbing unit is controlled by the measurement of the pH of the liquids in circulation.

- The pipe reactor of the granulator is set to the chosen N/P ratio. In the case of the sulpho-phosphates, the admission of the liquids coming from the scrubbers enables the reaction temperature to be controlled.

- The final N/P ammoniation rate is checked at the granulator outlet and set by the flow rate of ammonia into the injector embedded in the solid product.

- The recycling rates are maintained constant in the system at a value of between 1.7 and 3 times the production rate according to the formula manufactured.

- The procedures for stopping and starting the installation are very rapid and normal operation of the shop is practically immediate.

- The simplicity of the circuits and the instrumentation makes the process very reliable. The service life of the reactors is about 2 years.

7) MANUFACTURE OF DIAMMONIUM PHOSPHATE

At first we wondered whether it would be possible to manufacture dibasic ammonium phosphate by this process. Would not the injection of monobasic ammonium phosphate into the dryer reduce excessively the molecular ratio $NH_3 : H_3PO_4$ in the final product?

A simple calculation shows that if one adopts a recycling rate equal to 3 times the production rate, and a molecular ratio of 1.97 in the product leaving the granulator, by introducing half of the phosphoric acid into the pipe reactor of the granulator, and the other half into the pipe reactor of the dryer set at the molecular ratio 1.1, the mean molecular ratio in the whole of the product leaving the dryer, or four times the production, is equal to 1.86, which remains satisfactory.

The experimental results are far more favourable.

It is found that the monobasic ammonium phosphate injected into the dryer is again present for the most part in the fines after screening and the dust particles of the cyclones. The following molecular ratios have been measured at different points of the circuit:

	Outlet granulator	Final product 2-4 mm	Fines after screening	Dust particles of the cyclones
Mol. ratio $NH_3 . H_3PO_4$	1.90	1.87	1.60	1.30

In other words, a large part of the fine droplets of molten monobasic ammonium phosphate crystallizes in flight in the dryer or is fixed preferentially to the fine particles, which are separated by screening and return to the granulator with the dust particles. Their divided state is eminently suited to the complementary absorption of ammonia in this apparatus, up to the ratio 1.9-1.95. Everything takes place as if this monobasic ammonium phosphate had been introduced into the granulator in the hot powdery solid state and been superammoniated in this apparatus.

Apart from the reduction in recycling rate, another advantage of the process is that the heats of reaction and crystallization are fully utilized for the drying of the product. The superheated steam which leaves the pipe reactor of the dryer at about 140° is evacuated from the latter apparatus at about 100°, mixed with air. It therefore contributes by its sensible heat to the heating of the air and the drying of the product.

In addition, the amount of water which has to be introduced into the gas scrubbing system, in the form of acid containing 28-30% P_2O_5, is far less than in the traditional process, as the ammonia losses at the outlet of the two pipe reactors are far smaller than those of a neutralization tank: about 10% for the reactor of the granulator functioning with the molecular ratio 1.4 and 2% for the reactor of the dryer with the ratio 1.0-1.1.

There is therefore both less water to be evaporated than in the traditional process and more heat available. As a result the process becomes auto-thermal. By limiting to the value absolutely necessary the air flow through the dryer, heating of this air becomes superfluous if the mean concentration of the phosphoric acid is about 43%.

OPERATING CONDITIONS

Table No. 2 attached shows the operating conditions for the manufacture of DAP. The distribution of the phosphoric acid and the ammonia between reactors, granulator and scrubber is as follows:

	Pipe reactor dryer	Pipe reactor granulator	Granulator	Scrubber
Phosphoric acid	50%	37%		13%
Ammonia	28%	29%	43%	(18%) *

* The 18% represents the losses coming mainly from the granulator.

8) MANUFACTURE OF HIGH-CONTENT GRANULATED NP/NPK FERTILISERS

We possess today five units equipped with a pipe reactor opening into the dryer. Table No. 3 gives the operating results obtained for some high-content formulas. It seems that the best results are obtained by introducing into the reactor of the dryer almost the whole of the phosphoric acid, thus achieving the optimum recovery of the heat of the reaction. Under these conditions, auto-thermal operation is achieved for very numerous formulas (by using a concentrated phosphoric acid containing 50-52% P_2O_5 and a 92-94% ammonium nitrate liquor).

Under these operating conditions, the work-load of the gas scrubbers is much reduced and the ammonia which passes through the scrubbers represents less than 5% of the total introduced.

9) COMMENTS

. FLEXIBILITY OF OPERATION

The possibility of working with a single reactor, either that of the granulator or that of the dryer, has been verified in our installations on numerous occasions. It contributes to a substantial improvement in the rate of operation of our shops and enables the production shop to be maintained at reduced working if one of the circuits breaks down.

. INCREASE IN PRODUCTION OF AN EXISTING UNIT

The siting of a pipe reactor in the dryer tube complements perfectly any existing granulation unit.

This conversion has been carried out on several NPK units and recently on some DAP shops.

- The fitting of the reactor does not necessitate any special shut-down of the shop (no modification to the gas scrubbing plant, although the fitting of a reactor in the granulator requires the diameter of the suction sleeves to be increased).
- The time required for the conversion is very short.
- The operating conditions in the rest of the system remain unchanged;

the feed rate to the neutralization tank can be diminished, and the work-load on the corresponding gas scrubber reduced.

- The training of the operators is very rapid.
- The technical investment is reduced.
- The gains in capacity (between 30 and 50%)
  in yields (the work-load on the tank scrubber is reduced)
  in fuel consumption (of 30 to 75%)
  have been verified in our shops.

The table below gives data before and after conversion of the units:

	17.17.17		18.46.0	
	without P.R.	with P.R.	without P.R.	with P.R.
Production in t/day	1500	2200 + 47%	1150	1500 + 30%
Consumption of fuel oil or gas	8 kg/t	1 kg/t	7 Nm^3/t	4.9 Nm^3/t
Yield NH_3	99%	99%	93%	95%

- In the case of high hourly productions, the distribution of the ammonia becomes particularly advantageous. Beyond an ammonia flow rate of about 6 t/h, the fitting of a second reactor becomes necessary.

For an identical investment, its siting in the dryer tube presents all the advantages mentioned above.

- The manufacture of granulated MAP (11.54.0) can be achieved in excellent conditions with this process, in auto-thermal operation if the phosphoric acid has a concentration of 50-52% of P_2O_5, and according to one of the following three alternatives:

The pipe reactor of the dryer is set at the ratio N/P = 1.

That of the granulator can

either work with the ratio N/P = 1,

or with the ratio N/P = 0.6 with complementary ammonia in the granulator,

or with the ratio N/P = 1.2 with complementary phosphoric acid in the granulator.

- Pipe reactors, albeit of simple design, call for long experience for their configuration, their dimensioning, and the choice of materials.

Générale des Engrais has in its own works 13 pipe reactors fed with phosphoric acid and/or sulphuric acid, of which 5 are installed at the top of the dryer.

- Although certain formulas are auto-thermal in normal operating conditions, it seems preferable to us to maintain the possibility of reheating the air of the dryer for the following reasons:

- The start-up of the installation.

- The rapid restoration of good operating conditions in the event of the water balance of the gas scrubbers being disturbed.

- The possibility of manufacturing certain formulas whose granulation requires a quantity of water higher than that which the heats of reaction allow to evaporate.

- Versatility of the installation, permitting working with solid MAP or a feed of more highly diluted phosphoric acid.

Finally, it seems to us today that no manufacturer would risk erecting a granulated fertiliser shop deprived of any apparatus for heating the gases of the dryer, even if the latter worked as a cooler.

10) CONCLUSIONS

It seems that this process comprising a neutralization pipe reactor at the top of the dryer tube has very numerous advantages:

- simplicity of equipment and operation.
- flexibility of the settings.
- increased productivity.
- saving on energy.
- improved ammonia yields.
- reduction in investment and maintenance costs.
- absence of pollution.
- versatility of the installation.

- Compared with a traditional process, the fitting of the pipe reactor at the top of the dryer leads to a rise of 30 to 50% in the production capacity of the granulation system. Unit capacities on a system amounting to 3 to 4 000 t/day of NP/NPK fertilisers can be envisaged today.

- The saving on energy applies not only to the quantity of fuel, as we have mentioned above, but also to the kWh expenditure, which varies for a given system in inverse proportion to the hourly production.

- This process seems to provide a definitive answer to the question: "What is the best phosphate intermediate for the manufacture of granulated NP/NPK fertilisers?", a question which has long been debated without a clear conclusion being reached (Ref. 31). It can now be affirmed that with consistent MAP and phosphoric acid prices, the use of phosphoric acid is more advantageous than that of MAP powder, since it permits a capacity which is at least equivalent, together with a notable saving on fuel and a gain on servicing.

- Another advantage of the process is the versatility of the installation. Besides ammonium phosphates and NP/NPK fertilisers, it allows the manufacture of granulated superphosphate, or granulated P.K. fertilisers, or even granulated ammonium nitrates.

- Applications for the patenting of the process have been filed in numerous countries. The European patent application was published on 18.11.1981 under No. E.P. 40.122.

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MANUFACTURE OF DAP-NPK - GRANULATION SYSTEM

GENERALE DES ENGRAIS
PROCESS

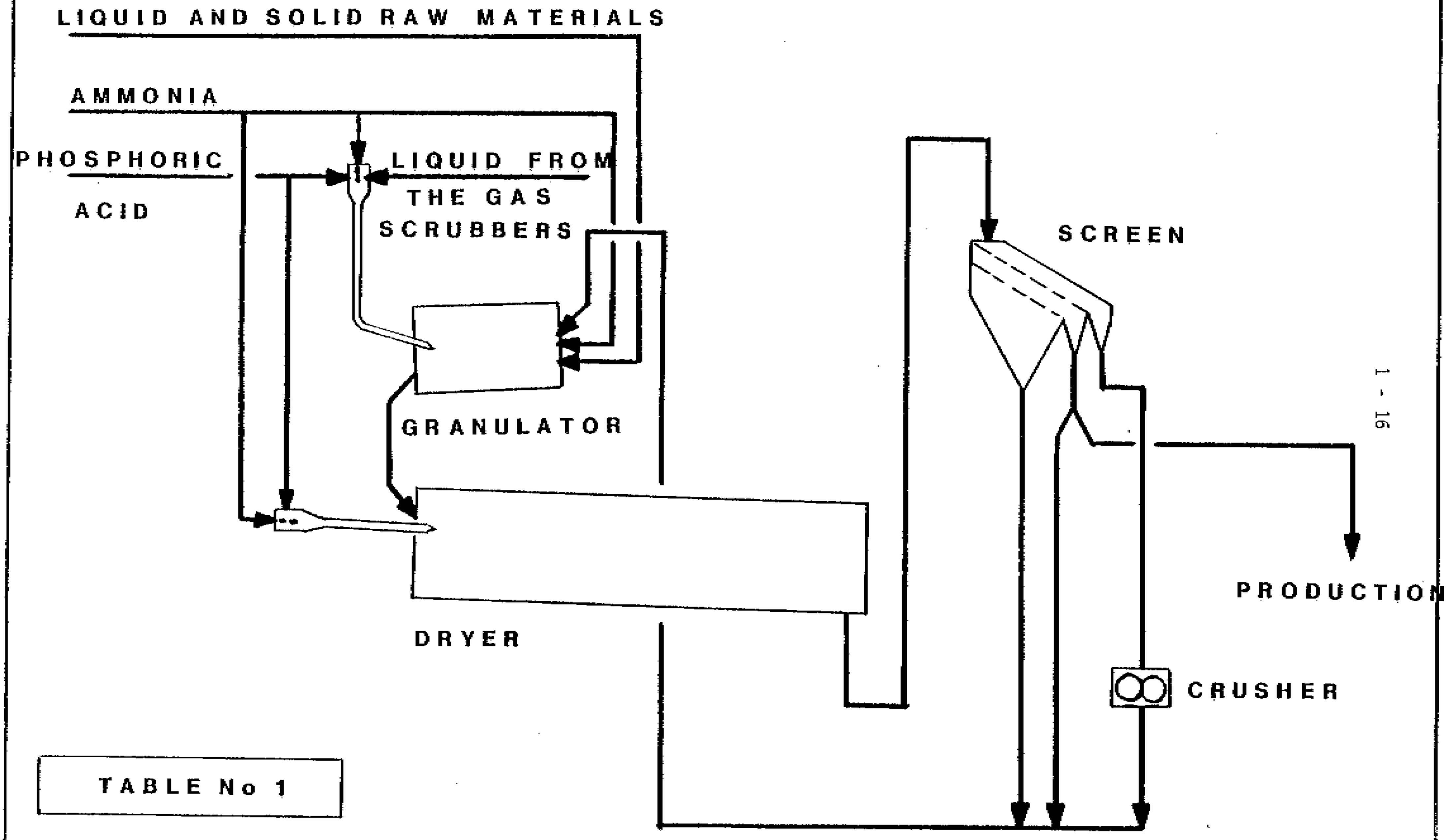

TABLE No 1

MANUFACTURE OF DAP 18.46.00

BALANCE FOR 1 TON OF PRODUCTION

GENERALE DES ENGRAIS PROCESS

EXPRESSED IN KG OF 100 % P2O5 AND 100 % H2SO4 EXCLUDING LOSSES

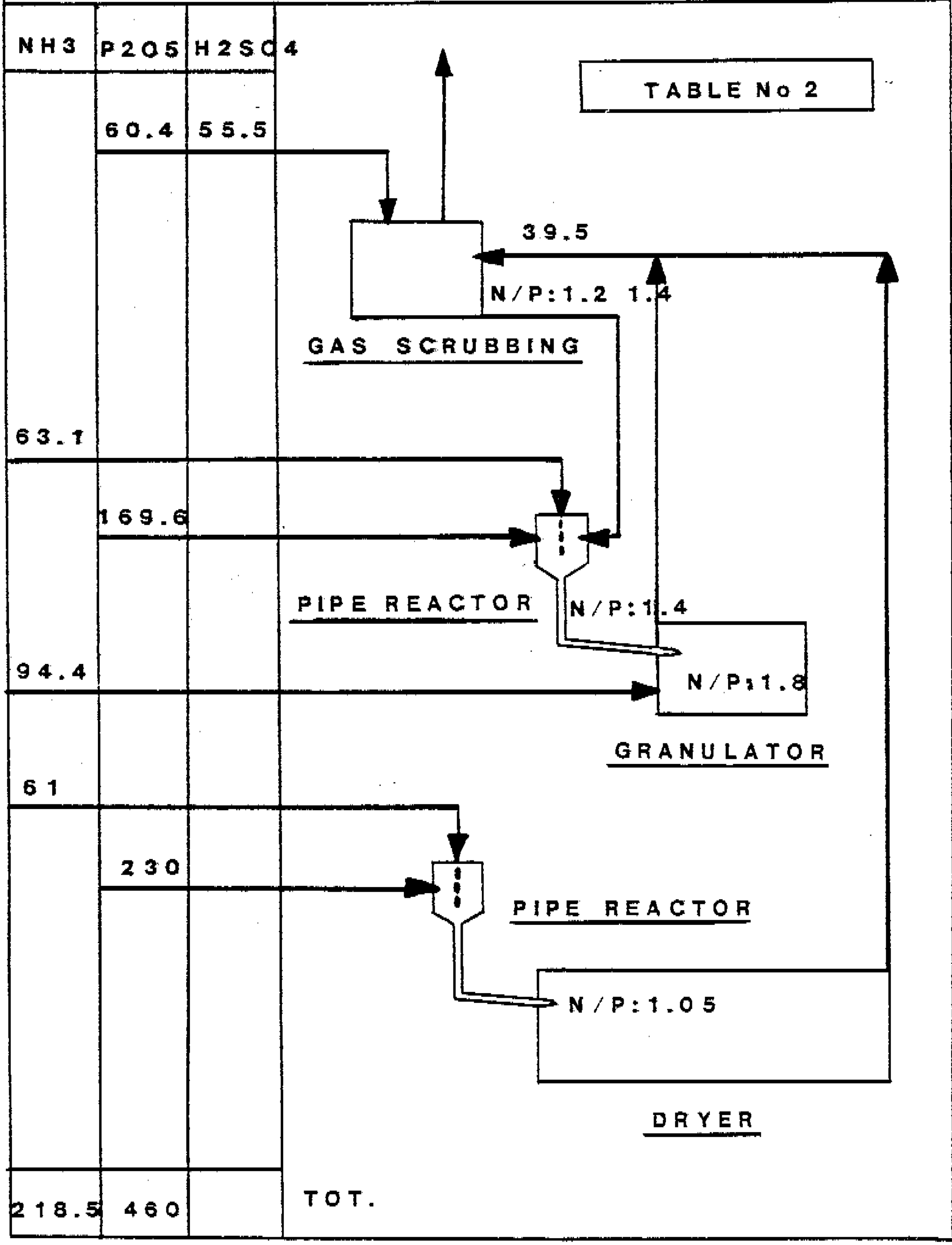

TABLE No 2

TABLE No 3

Distribution of liquid raw materials between the granulator and the dryer and other operating conditions

Formula	17.17.17	17.17.17	15.20.20	18.22.12	23.23.00
Distribution of raw materials					
Phosphoric acid					
% to granulator	50	0	25	20	0
% to dryer	50	100	75	80	100
All raw materials					
% in granulator	76	51	55	55.4	53.5
% in dryer	24	49	45	44.6	46.5
Ratio $\frac{\text{liquid materials}}{\text{solid materials}}$ entering the granulator	0.16	0.14	0.14	0.15	0.19
Atomic ratio N/P					
- outlet pipe reactor of granulator	1.40	-	1.36	1.39	-
- outlet granulator	1.73	1.61	1.80	1.78	1.00
- outlet pipe reactor of dryer	1.04	1.10	1.10	1.09	1.00
- outlet of dryer (final product)	1.66	1.49	1.67	1.67	1.00
Rate of recycling	3.2	2.5	2.5	3.0	3.0
Hourly production in t	92	92	95	85	80
Consumption of fuel oil in kg per t of final product	4	1	0	0	0
As comparison:					
Operating conditions without using pipe reactor of dryer					
Rate of recycling	6	6	4.5	5.3	6
Hourly production in t	60	60	70	65	55
Consumption of fuel oil in kg per t of final product	8	8	7	7	6

TA/82/1 Savings on energy in granulation of fertilisers by a new method of using pipe reactors, by Ph. MORAILLON[*], Y. COTONEA (Générale des Engrais S.A., France)

DISCUSSION : Rapporteur P. BECKER, COFAZ SA, France

QUESTIONS PUT TO THE AUTHOR

Q - Mr. P. NIEHUES, Uhde GmbH, Germany

1. What are the sectional area and the specific volume of the pipe reactor, referred to energy production or used NH_3 quantity?
2. Are there scaling or blockage problems in the reactor especially when reactor runs with a mole ratio of 1.4?
3. Are there scaling problems in the drying drum and in the vent pipe of the drying drum?

Q - Mr. F.P. ACHORN, TVA, United States

1. In your written paper you state one of the pipe reactors discharges into a dryer and it is operated at an N:P mole ratio of 1.1. This indicates probably some MAP is in product from the process. Have you been able to make the conventional 18.46.0 grade of DAP when phosphoric acid produced from Florida or North Carolina phosphate rock is used?
2. You report that the fine-sized material from the dryer is monoammonium phosphate and it is returned to the granulator as a solid material. Is it your opinion that these solids are converted directly to diammonium phosphate in the granulator or do you believe the solids must first go in solution and then recrystalize as DAP in the product?
3. If there is suitable mixing in both the preneutralizer and the pipe reactors, the losses of ammonia would be dependent upon the partial pressure of the ammonia above the saturated solution from the pipe reactor as well as that of the saturated solution in the preneutralizer. The question is, in your tests comparing operations with a preneutralizer and with a pipe reactor, was the temperature of the material in the reactor the same as the temperature of the slurry in the preneutralizer?

Q - Mr. M. GAURON, COFAZ SA, France

1. Is the temperature of the slurry at the discharge of the pipe reactor a critical factor?
2. What are the limits of variation of the temperature in the reactor of the granulator which receives scrubbing liquors?
3. The pipe reactor of the dryer produces MAP dust. Did you reinforce the systems for dedusting and dryer gas scrubbing?

Q - Mr. G. BRUSASCO, Fertimont SpA, Italy

You have found that monobasic ammonium phosphate injected into the dryer is again present for the most part in the fines after screening and in the dust particles of the cyclones.

It can be supposed that more dust than usual is entrained by the air going out.

From cyclones to the venturi scrubbers:

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[*] Currently SICNG, Greece

- Did you measure or calculate the entrained dust flow?
- Had you troubles for solid deposits in the first scrubbing stage due to entrained powders or supersaturation of the circulating solution?
- In the circulating solution you are controlling only the pH or also density?

Q - Mr. J.E. LEONARD, Grassland Fertilizers Limited, Ireland

I have 4 questions in connection with operating conditions in relation to the dryer:

1. The use of the pipe reactor in the dryer necessitates the material flow being split into 3 ways. Does this give rise to any difficulty in getting the plant into balance- or, is the process particularly suited to long runs of a fixed product grade?

2. Does the slow speed of the dryer net result in poor granulation?

3. How the stream from the pipe reactor not cause any build-up in the dryer? If so, how is it eliminated?

4. Finally, what is the residual free moisture content in the finished product, for example DAP? This question should be of interest to bulk blenders where production of moisture can be a critical factor?

Q - Mr. K.J. BARNETT, Norsk Hydro Fertilizers Ltd, United Kingdom

My question and remarks are in connection with the scrubbing system described in the paper:

1. The possibility of working with a single pipe reactor has been indicated. When operating without the granulator reactor, how are the scrubber liquors re-introduced?

2. If and when the scrubber system is temporarily shut down, because of a plant problem, how long can it be left before the MAP liquors need to be drained from the system?

3. Can the author comment on the stability of the scrubber system and, in this connection, what is the buffer capacity and residence time of the venturi scrubber?

4. Reference is made to good fluorine absorption in the scrubbers. However, the use of around 15% weak acid involves the introduction of significant quantities of volatile fluorine. When operating with weak acid added to the scrubbers, which level of fluorine emissions are typical of the process?

   Can the author comment on how, unlike the conventional scrubber system, the volatile fluorine is apparently fixed?

Q - Mr. D.W. LEYSHON, Jacobs Engineering, United States

On page 8, a 99.5% ammonia recovery is discussed, but the table on page 11 lists 95% recovery for 18-46-0.
On page 10 you indicate only 13% of the P_2O_5 is fed to the scrubber.

In our experience, your 95% figure is optimistic with so little acid and in adding the scrubbers experience severe scaling. Can you clarify?

In your paper, you only refer to mole ratio. Can you tell us what pH is equivalent to say 2.0 mole ratio? Various new points and techniques are used so that mole ratio reporting is not always consistent.

ANSWERS

Dimensions: We have the experience with pipe reactors fed with 1 to 10 t/hr NH_3. The diameters vary from 40 to 150 mm.

Operations at 1.4 mole ratio: We have no blocking or scaling problem at that ratio. The temperature should be controlled at about 140° C by adjusting the flow of washing liquors or water addition.

Influence on the dryer scrubbing systems

The additional dust load produced by the pipe reactor in the dryer remains at a very reasonable level for the usual large size cyclones included in the drying circuits. We never found any excessive MAP entrainments to the scrubbers in our plants. Wash liquor densities are kept at the usual values of 1.3-1.4.

We estimate that the proportion of MAP passing through the dryer scrubbing system is 30%.

Operational conditions

The fact of splitting the feedstock inlets does not raise any plant adjustment difficulty, on the contrary. Indeed, for a given formulation, the pipe reactor of the dryer is set in a stable way and the problem is only to use solid MAP in the loop. The operation of the pipe reactor of a granulator is much easier than that of a tank.

The process is specially adapted to frequent changes of formulations (low circulation load, no retention in the reactors - thermal balance in the loop very quickly reached).

The steam evolved in the pipe reactor of the dryer contributes to a small increase of the dew point temperature of the gases.

When the pipe reactor of the dryer runs alone, it is then possible either to introduce the washing liquors in that pipe reactor or to spray them in the granulating drum.

18-46-0 can easily be produced by this process, since the coating of granules ranging from 1 to 5 mm in the dryer with MAP is not a predominant factor.

So far our experience concerned Togo, Morocco, Florida, North Carolina phosphates.

Concerning the superammoniation of the product returned to the granulator, in a pilot plant we checked the special ability of the product formed in the dryer to fix ammonia, without reverting to the slurry phase.

Gas scrubbing

Gas scrubbing equipment is usually quite large in relation to the dryer. Difficulties arise in the granulator. In the GESA process, this disadvantage is minimized:

1. By the efficient NH_3 conversion in our reactors
2. By the splitting of the reagents between the drying and granulation circuits.

As already mentioned, the excess dust load of the plant is not a problem if the battery of cyclones on the drying loop is operational.

The plant works at a pH of 4 to 5 in the first stage which facilitates the absorption of F- and avoids the stripping of F- present in the 30% phosphoric acid.

The final stage works in more acid conditions to fix the remaining ammonia.

In the operational conditions of our pipe reactors, fluorine emission is quite negligible.

The density of the washing liquors going to the granulation loop is kept below about 1.4, make up water being added to keep that density.

Improvement of the efficiency of the existing plants

The table page 11 mentioned 95% efficiency for 18-46-0. In that example, the introduction of a pipe reactor in the drying tube resulted in an increase in the previous efficiency, which was about 93%, without altering the existing plant and with an increased capacity.

When 13% phosphoric acid added in the washing circuit is mentioned, one should take account of the sulphuric acid (e.g. 55 kg/t).