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*In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).

INTENSIVE AMMONIATION IN THE PREPARATION OF GRANULATED FERTILIZERS

Вy

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1. INTRODUCTION

Up to now, the cheapest source of nitrogen for the manufacture of fertilizers has been anhydrous ammonia. The difficulty lies in achieving satisfactory absorption of the NH₃ by the rest of the materials in the formula, without recourse to expensive or complicated equipment. The process generally used is that developed by the TVA, in which NH₃ absorption takes place in a preneutraliser and the resultant pap is passed to the granulator. This involves a complicated set-up to prevent crystallisation and obstructions in the pipes.

This paper describes simple equipment for introducing the NH₃ directly into a conventional granulator and achieving a high standard of ammoniation with a satisfactory degree of absorption. Details are also given of the results obtained in industrial manufacturing with this equipment in the following products:

- Granulated ammonium sulphate and granulated sulphourea.
- Granulated ammonium phosphates.
- Granulated NPK fertilizers.

The equipment and its application to the processes indicated are protected by the Spanish patents (developed by CROS) shown under items 1, 2 and 3 of the bibliography.

2. DESCRIPTION OF THE EQUIPMENT

Figure 1 shows details of the equipment used, which comprises essentially:

a) Conventional granulator equipped with the specially-designed appliances for introducing anhydrous NH₃ into the mass of solids.

- b) Tubular reactor which produces in situ a pap which is poured on to the mass of solids. The reactor is continuously supplied with anhydrous NH₃ and sulphuric and/or phosphoric acid.
- c) NH₃ recovery equipment, consisting of a conventional spray scrubber.
- 2 (a) The basic aim of the design of this equipment is to ensure that there is a constant influx of NH₃ at a suitable speed, without causing obstructions in the outlets. For this reason the outlets have been suitably designed, or else the tubes for addition of ammonia are protected by a metal grille which is inserted deep into the bed of solids and to which are welded the NH₃ and acid tubes on the side opposite to the attack of the material. Thus, in conjunction with the special design of the bottom of the grille, the NH₃ and acid are caused to pour into and react in the furrow created by the grille, thereby preventing the solid material from reaching the tube outlets, and consequently preventing obstructions.

In the case of the cylindrical granulator, a series of grilles and tubes are fitted in line, their number depending on the size of the cylinder.

- 2 (b) Figure II is a diagram of the layout of the tubular reactor by means of which another portion of NH₃ is added in the form of pap with sulphuric and/or phosphoric acid.
 - (1) and (2) represent the NH₃ and sulphuric or phosphoric acid storage tanks. (4) and (4') are valves which permit the fluid to pass in one direction only. The NH₃ supply depends on the vapour pressure in the tank, whereas a pump (3) is needed for the acid to overcome the reactor inlet pressure. (5) and (5') are flowmeters to measure the flow. (7) is a system for the addition of water with its meter (5") to regulate the acid concentration.

Details of the reactor are shown in Figure III. It consists of a cylindrical chamber (2) in which the reaction takes place. The acid enters the chamber (1) via (5), is finely atomised by the nozzles (3), and then enters the chamber (2). The liquid NH₃ enters via the central tube (6) which is also fitted with nozzles (4) which reduce the liquid to fine droplets. This entry in a state of fine division facilitates the reaction and mixture of the products in the chamber (2). The acid should

be diluted to a sufficient concentration to form sufficient water vapour to facilitate the expulsion of the pap via the tube (7) and at the same time to control the reaction temperature.

The length and shape of the tube (7) and bends (8) vary according to the sal ammoniac and the characteristics of the pap it is desired to produce.

In Table 1, we show the results of some tests carried out on an industrial scale, by way of illustration of the possibilities of this reactor.

TABLE 1
Industrial Tests with Tubular Reactor

Test	NH 3		H ₂ SO ₄	(67%)	Comp H ₂ O	' A '	products (NH ₄) ₂ SO ₄	NH ₃ losses	Reaction time
No.	kg/h		kg/h		*	**************************************		- -	. <u>s</u>
1	155		1,570		14.7	82.5	2.8	0.5	0.4
2	400		1,700		8.0	0.6	91.3	5.0	0.3
3	492		4,000		17.0	57.6	25.3	2.0	0.15
4	700		5,710		17.9	56.4	25.7	15.0	0.1
			H ₃ PO ₄ kg/h	50%	H ₂ O	N %	P ₂ O ₅		<u> </u>
5	400		2,400		8	14.7	54	l	0.25
6	400		2,600		12	14.1	51.5	1	0.25
	•	Н ₃ РО́ ₄ 5О%	H ₂ SO ₄ 67%	· · · · · · · · · · · · · · · · · · ·					·
7	570	2,600	500		8	15.5	44.4	2	0.15

The characteristics of the reactor used in these tests are:

Reaction tube diameter = 70 mm

Length = 1,500 mm

Reaction chamber capacity= 50 cm³

The advantages offered by this reactor in comparison with conventional preneutralization methods are:

- Lower cost. In the conventional systems, the residence time is 20-30 minutes, as compared with l sec in our reactor. This is accompanied by a considerable difference in the volume of the equipment.
- 2) Less power consumed, as mixers are not used. In our case the mixture is produced by the turbulence created by the water vapour formed.
- 3) In conventional preneutralizers, there is always a substantial residue in the reactor, which represents a disadvantage in the case of stoppages because of the danger of solidification, and makes cleaning essential when there are changes in the manufacturing cycle. In our reactor, the type of sal ammoniac can be changed without a stop.
- 4) The reactor is sufficiently small to be fitted inside the granulator, thus enabling production in situ of ammonium sulphates or phosphates which are mixed with the mass of solids without need of equipment to convey and pump the hot pap.

2.(c) Scrubber

The installation has a conventional gas scrubber consisting of a spray tower which is sprayed with sulphuric or phosphoric acid. The scrubbing water is conveyed to the granulator, thus maintaining a constant level of acidity to guarantee the absorptive capacity of the tower.

3. VARIABLES WHICH AFFECT AMMONISATION.

Apart from the need for properly designed equipment to ensure uniform addition of ammonium, the following variables have to be controlled and kept within certain limits:

- Size of solid particles on entry to granulator.
- Temperature of mass undergoing granulation.
- % of liquid phase.
- Residence time.

All these variables affect the granulation capacity, and condition the absorption of ammonia by the mass of solids in the reactor.

It is necessary, for each type of formula it is desired to produce, to have available the necessary data which interrelate all these variables, so as to obtain optimum conditions in which to carry out the process. These data are obtained from previous laboratory tests and pilot-plant tests, the necessary adjustments being subsequently made when industrial manufacturing commences.

Figure IV shows for example the curve which relates the variables temperature and % humidity in respect of a specified formula.

4. REDUCING COSTS TO A MINIMUM. PRODUCTION OF FORMULAS FOR MINIMUM COST.

It is possible, by means of a computer and linear programming techniques, to work out a raw-materials formula for each fertilizer so as to keep costs to a minimum.

To do this, it is necessary in each case to know the ratios which interlink the variables which affect the process of ammoniation and granulation. These data are obtained as explained in the previous paragraph.

Generally speaking, the method we used consists of the following phases:

- 1) Optimum granulation conditions are determined for each group of possible raw materials:
 - Granulator outlet temperature.
 - % H₂O at granulator outlet.
 - Recycling/product ratio
- H₂O in final product.
- 2) The mass balance equation is formulated for N, P_2O_5 and K_2O .
- 3) The ammoniation equation is formulated.

For the purpose of this equation, it is necessary to determine experimentally the degrees of ammoniation of the raw materials:

A requirement of this equation is that:

 $free NH_3 = 0$

Thus, once the consumption of NH_3 in respect of the raw materials is known, the acid needed to fulfil this requirement is calculated.

4) The heat balance is established.

To do this, it is necessary to know:

- Heat of reaction of the ammoniation reactions.
- Specific heats of the materials of the mix.
- Granulator inlet and outlet temperatures.
- Estimated system heat losses.

Should this balance be positive, the granulation temperature will be maintained by evaporation of water; should it be negative, the "necessary steam supply" to maintain this temperature has to be calculated.

5) The water balance is established.

Water entering with raw material + water supplied to the system = water evaporated + water leaving granulator with solids.

A requirement of this equation is that water supplied should equal 0, as otherwise the liquid phase of the process would be excessive and the process be uncontrollable.

6) The equation of costs is established.

On the basis of the foregoing, the linear system of equations for calculating the composition which gives the minimum cost is determined.

- 5. INDUSTRIAL APPLICATION OF THE EQUIPMENT.
- 5 (a) Production of granulated ammonium sulphate and ammonium sulphate - urea.

Using the equipment described in conjunction with a conventional granulation plant, it was possible to produce ammonium sulphate and mixtures of ammonium sulphate/urea in hard, stable grains of between 1 and 4mm. The process consists of adding dilute sulphuric acid to a recycling bed of under 1 mm, and adding liquid NH₃ into the mass, whilst keeping the pH at between 4 and 5. At the same time, an ammonium bisulphate pap was added to the mass of solids by means of the tubular reactor.

The addition of urea in differing proportions facilitates granulation, as does the addition of small quantities of superphosphate or phosphoric acid.

The tests were carried out initially in a pilot plant with an hourly capacity of between 200 and 400 kg. Subsequently, 1,000 tonnes were produced in a conventional granulating plant with an output of between 6 and 10 tonnes per hour.

Table 2 shows some of the results obtained.

To sum up, it is possible by using this process to produce, in a conventional granulation plant, granulated ammonium sulphate by direct reaction between liquid NH₃ and 68% sulphuric acid produced by a chamber process.

The conclusions reflected in Table 2 are:

- Granulation is better with an acid pH when the final product retains between 0.1 and 0.5% free H₂SO₄.
- The inclusion of small quantities of P₂O₅ (superphosphate, phosphoric acid, monoammonium phosphate) facilitates granulation and improves the physical properties of the finished product. It is sufficient to add about 0.3% of P₂O₅.
- The urea and ammonium sulphate in differing proportions display an excellent tendency to cogranulation.

TABLE 2
Granulation of ammonium sulphate by direct reaction

Test	Additive	Final pH	% H ₂ O final prod.	% H ₂ SO ₄ final prod.	Total	% N Ammon- iated.	Characteristics of finished product	
1	-	2	0.3	1.7	18.5	18.5	soft hygroscopic grain	
2		2.5	0.2	1.2	19.2	19.2	hard grain	
3		3	0.2	0.6	20.6	20.6	hard grain	
4		4.5	0.2	0.1	20.9	20.9	soft grain	
5	P ₂ O ₅	3	0.3	0.5	20.7	20.7	hard grain	
6	P ₂ O ₅ + urea	11	0.2	**	20.7	20.2	tt ff	
7	P ₂ O ₅ + urea	Ħ	0.3	11	25.0	18.0	very hard grain	
8	Urea	** 11	0.3	71	21.2	20.6	hard grain	
9	Urea	11	0.2	T†	30.0	13.5	very hard grain	

5 (b) Production of ammonium phosphates.

It is possible, in the same way as that described above, to produce monoammonium phosphate by direct reaction between phosphoric acid and ammonia on a recycling bed of fines, in a granulating drum. In this case the tubular reactor is supplied with 50% phosphoric acid and liquid NH₃, maintaining the molar ratio N:P = 1.4, which is the point of maximum solubility.

The success of the process depends in this case on the design of NH_3 inlet tube device in the bed, protected by grilles as already described.

All attempts made with other types of injectors failed because of the continual blockages which occurred.

The process is continuous, with a continuous recycle of fines entering the granulator. The pap from the tubular reactors is added to this bed, and the phosphoric acid is atomised together with the NH₃ recovery liquids of the scrubber. The liquid NH₃ is injected into this mass.

As usual, the variables which govern the process are:

- pH (determines the ratio NH3 : POuH3)
- % H₂O (affects granulation and ammoniation output rate).
- Temperature (has to be kept below 95°C).
- Recycling /finished product ratio.

Table 3 shows the process conditions.

TABLE 3

Production of granulated ammonium phosphates

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Granulator input	MAP	DAP	
50% phosphoric acid	10,500 kg/h	9,250 kg/h	
Anhydrous ammonia	1,640 "	2,250 "	
Recycled material	15 - 24 tonnes/h	10 - 20 tonnes/h	
Granulator output (on dry base)	27 - 36 tonnes/h	20 - 30 tonnes/h	
Granulation condition	15		
pH (10% sol.)	4 - 4.2	7 - 7.2	
Temperature	70 - 90°C	70 - 95°C	
% H ₂ O	4 - 8 %	3 - 7 %	
Production	12 tonnes/h	10 tonnes/h	
Product Composition			
N	11 - 11.5 %	17.8 - 18.3 %	
P ₂ O ₅	52 - 53 %	46 - 46.5 %	
H ₂ O	1 - 1.5 %	1 - 1.5 %	
% between 1 - 4 mm	over 95%	over 95%	

5 (c) Production of NP binaries with urea

A binary granule (28-28-0) was also produced continuously at the pilot plant at rates of 300 - 400 kg/h.

The process is similar to the foregoing, except that urea is added, either with recycling solids or in conjunction with the phosphoric acid in the form of phosphate of urea dissolved in the phosphoric acid. This suspension is atomised on the bed of recycled solids, and anhydrous NH₃ is injected into the mass with the appliance described.

In this case, granulation conditions are as follows:

pH = 6.5 - 7% $H_2O = 2 - 4$ % Temperature = $50 - 80^{\circ}C$ Recycling ratio = 1/2 to 1/3

5 (d) Production of NPK granules

The appliance described is also used for the addition of NH_3 in conventional ternary formulations.

In order to optimise all the formulations manufactured by our company (some 40 - 45) and in accordance with the system described in section 4, the following tests were carried out to deduce experimentally the parameters necessary for the mathematical treatment: (4)

- 1) The following granulation conditions are deduced for each group of raw materials by laboratory and pilot-plant tests: pH, temperature, % H₂O, recycling ratio.
- 2) Using the injection appliances, the degrees of ammoniation were determined experimentally for:
 - Normal and triple superphosphate (18% and 45% P_2O_5)
 - Monoammonium phosphate (powder) (11 52 0) for separate products and for mixtures with other constituents such as urea, potassium chloride, potassium sulphate, inerts, etc. The average values noted are:

Super 18% = 0.05 kg NH₃/kg Super 18 Monoammonium phosphate = 0.10 kg NH₃/kg MAP Super 45% = 0.09 kg NH₃/kg Super 45.

3) The heats of reactions of ammoniation and specific heats were taken from those available in literature.

Using these data and the method indicated in section 4, the optimised formulas of all the NPK manufactured by the company were calculated.

These calculated formulae were subsequently tested in the industrial plant. The results obtained tallied with those predicted in the majority of cases, and only in about 5% of the calculated cases was it necessary to make minor adjustments to granulating conditions.

Bibliography

- 1. Process for producing ammonium sulphate in rotary reactor: Spanish Patent No. 385 735.
- 2. Method and appliances for the production of ammonium phosphates: Spanish Patent No. 385 827.
- 3. Process for producing nitrophosphatised and diammonium fertilizers: Spanish Patent No. 385 734.
- 4. "Formulation for a TVA-type Ammoniator-Granulator by computer": J. L. Nevins, F.P. Achorn. Proc. of the 19 An. Me(e)t. Fert. Ind. (1969).

DISCUSSION

MR. R. LOSTE (S.A. Cros, Spain) Our paper describes basic equipment employed for the direct use in fertiliser manufacture of anhydrous ammonia, preferably in the liquid state and without the prior use of conventional preneutralisers. Once the equipment had been designed and perfected, a test programme was initiated to obtain nitrogenous fertilisers by the direct reaction of ammonia with the appropriate acid, using as reaction medium a bed of fine particles. The NH₃ was introduced in two ways:

- Through an ammoniator unit inserted into the bed of solids in the reactor/granulator.
- Through a tubular reactor, as the product of an acid slurry poured over the bed of solids.

The equipment was supplemented by a suitable scrubber. On average at least 90% of the NH3 was fixed in the granulator.

The industrial applications of the equipment are listed in the report, and relate primarily to the manufacture of:

Granulated ammonium sulphate Granulated sulpho-urea Granulated ammonium phosphates Manufacture of NPK

The ammoniation equipment was tested for the purpose of determining the ammoniation rates of materials used for the manufacture of NPK, such as:

Normal and concentrated superphosphate Monoammonium phosphate in powder form Sulphuric and phosphoric acids

both individually and mixed with other normal ingredients:

Potassium and magnesium salts Urea Inerts, etc.

The results were assembled in groups for which granulation conditions were similar and for each group we determined the optimum conditions of simultaneous ammoniation and granulation which decided the output of the equipment. These were, in order of importance:

Humidity as a percentage of the reacting mass

Temperature

pН

Granulometry of the bed of solids.

Once these results had been obtained in a pilot scale laboratory test with hourly production rates of 400 Kgs., the transition was finally made to industrial plant, and a drum-type granulator and another of the plate type were fitted with the NH3 injection systems described. The results were very satisfactory and the hourly ammoniation rates in the production of NPK were increased by more than 100%, with resulting reductions in costs, as large quantities of solid ammonium sulphate or urea were replaced by liquid NH which was neutralised in situ with residual sulphuric acids such as those deriving from gas scrubbing circuits in contact equipment or acids from other sources which were not suitable for sale, and by the solid components with neutralising capacity, i.e. single or triple superphosphate and monoammonium phosphate.

Using a plate of 4m in diameter with a suitable depth of bed, we managed to introduce up to 2,000 kgs/hour with the help of supplementary tubular reactors.

In a conventional drum of 5m in length and 2m in diameter, fitted with a unit with 6 injector grilles and two tubular reactors, it was possible to fix up to 2,500 kgs/hour of NH3 with losses at the drum of less than 10% of the NH3 introduced. A scrubber spraying the acid to be used in the granulator is essential in the case of these intensive ammoniation processes. These modifications have been gradually extended to all the Company's NPK plants.

Even if this does not represent any innovation, we give the basic formula used to obtain, with a computer and linear programming techniques, the items to be used in each of the company's plants, bearing in mind the available raw materials price for each centre, and the exact chemical and physical data.

The system is based on a previous laboratory and pilot-scale test, used to determine the optimum granulation conditions of groups of possible raw materials, with the granulation curves being fixed in terms of the Liquid phase present.

We give one in the paper, as an illustration. The formulae calculated were later tested in industrial plants. In only about 5% was it necessary to make adjustments to the composition to improve the granulation conditions. This occurred where the heat balance was theoretically very tight for supply to the mass of the right temperature and thus the percentage of liquid phase.

The designs for injection of ammonia direct to the granulator under the moving solid bed have provided an excellent distribution of the NH3 and acid liquid, preventing local concentrations of NH3 which might give rise to reversion reactions. The results obtained may be summarised as follows:

- a) Formulations based on ordinary superphosphate as source of P2O5.

 Final pH NPK = (10 gr/100 ml) = 5

 Degree of ammoniation = 0.05 kgs NH₃/kg

 super 18%

 P2O5: assimilable (soluble water + neutral citrate)

 = 97% P₂O₅ total

 P2O5 assimilable (80% water soluble

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 (20% soluble in neutral ammonium citrate)
- b) Formulations based on M.A.P. and phosphoric as source of P2O5

 Final pH NPK = 6

 Degree of ammoniation M.A.P. = 0.09/0.10 kgs

 NH3/kg M.A.P.

 P2O5 assimilable (soluble water + neutral citrate)

 = 98%

 P2O5 assimilable (95% water soluble

 (5% soluble in neutral ammonium citrate)

MR. Y.F. BERQUIN, (Azote et Produits Chimiques, France.) I should like to thank the Cros Company for its contribution to our industry, and for informing us of the improvements it has made in the preparation of granulated fertilisers. I should also like to congratulate the authors on the excellent paper which has just been presented to us.

Their merits in my view are many:

- 1) First, they have perfected a general method by which not only different fertiliser formulations but also different types of formulations based on different raw materials can be made without distinction. This is an excellent response to the need we all experience of having to put forward, despite our efforts at simplification, lists of formulations which are unfortunately exorbitant in number: the Cros Company for its part puts forward 40 to 45!
- 2) They have used simple, standard equipment which is reliable, but which has modifications or additions which considerably improve its performance.
- 3) They have succeeded in reconciling theoretical considerations and calculations, backed by modern data processing methods, with the strict conditions of presentation of experimental results and with the care of the practical operator aware of the difficulties of a user who is often confronted with an apparatus ideally calculated theoretically but difficult to handle in practice if no account is taken of the lessons drawn from daily experience.

I feel therefore that I am interpreting the wishes of all in requesting clarification of certain points in order more clearly to define the possibilities offered by the results of this study.

- I) If it is not indiscreet, I should like to have some additional details about the particular features of the injector used. We are indeed all aware that the risks of obstruction of ammonia injectors are a frequent cause of difficulty in manufacture, and the experience of Cros in this field would certainly be of value to us. Moreover, what is the maximum unit capacity of an injector?
- II) The reactor where the neutralisation of the acids by NH₃ takes place seems very ingenious.

Several questions, however, arise:

- a) What are the dimensions of the appliance, for the report gives a few indications which are in fact difficult to interpret: in particular the volume (50 cubic centimetres) in the light of the diameter (70 millimetres).
- b) What are the functions, dimensions and form of the details given at 7 (tube) and 8 (bends)?
- c) Is there not considerable corrosion of the reactor and its outlet, in particular by fluorine, when using phosphoric acid at 50%? Otherwise, how has it been possible to prevent such corrosion?
- d) We have noted, from the experimental tables, that this apparatus can deliver in the region of 500 kg/hour of ammonia without major losses, and at least keeping the medium sufficiently acid. This is already an excellent result in view of the size of the apparatus.

In the case of production on a larger scale, we note that the authors proposed to use several appliances in parallel.

However, there will certainly be many cases where a larger unit production would be desirable. Have the authors already some ideas about the possible extrapolation of their apparatus to production figures representing for instance 2 or 3 tonnes per hour of injected ammonia?

- III) In the case of preparation of diammonium phosphate, where the temperature may reach 95°C, what is the fraction of ammonia to be recycled after recovery in the scrubbers?
- IV) The use of urea in granulation is tricky, as the losses by dissociation are often high.

What are these losses

- on granulation properly so called?
- 2 on drying? and how is the drying carried out?

What is the pH of the fertiliser on granulation after the final drying?

V) Elsewhere it is stated that in the case of a sulphate-urea mixture a 1 mm. bed was used. This seems too little to prevent incrustation on the wall: is this perhaps a printing error?

These are the few observations this paper suggested to me, but in view of its importance I believe that other members are eager to take part in the discussion. I shall therefore hand over to them.

Once again, our congratulations and thanks.

MR. LOSTE:

- I) The ammonia injection equipment was in fact planned to prevent the difficulties caused by obstruction which we experienced with the conventional type. In essence our system completely prevented this by:
- Suitable design of the orifices to achieve high velocities of the liquid NH3
- mechanical design which causes the injector to be withdrawn from the bed during shutdown periods
- design of the bottom of the grille, to which the NH3 pipe is attached; it forms a furrow into which the NH3 pours, so that the solid material cannot reach the NH3 outlet:

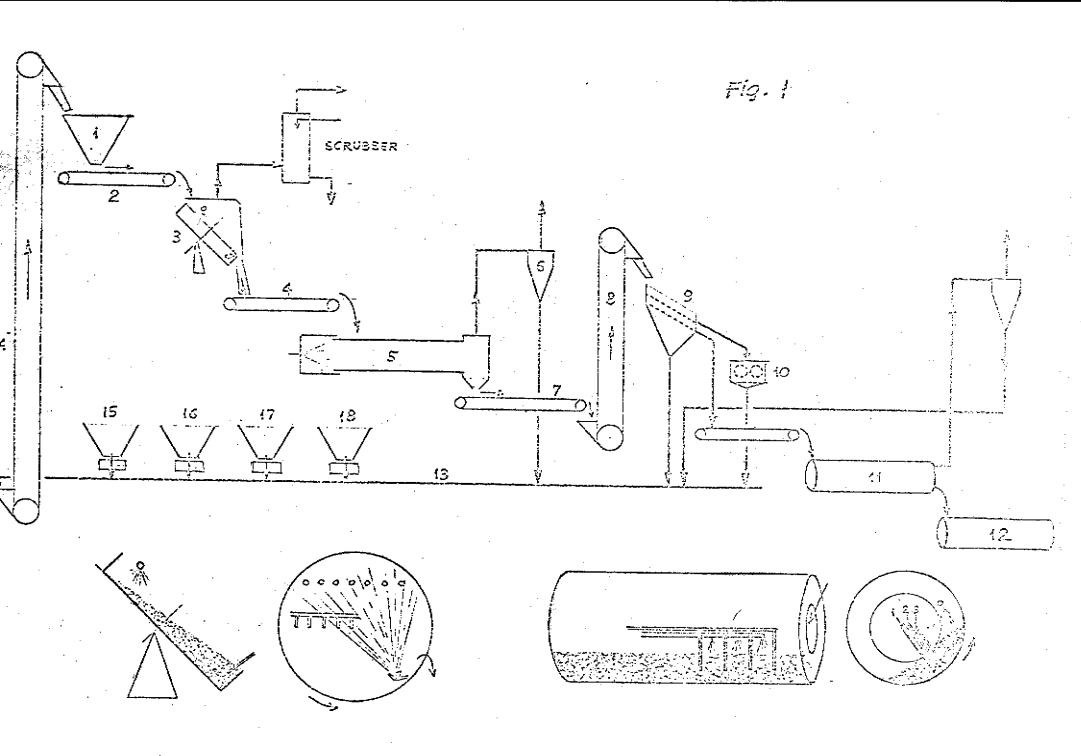
The unit capacity of each injector varies between 300/350 kg NH3/hour.

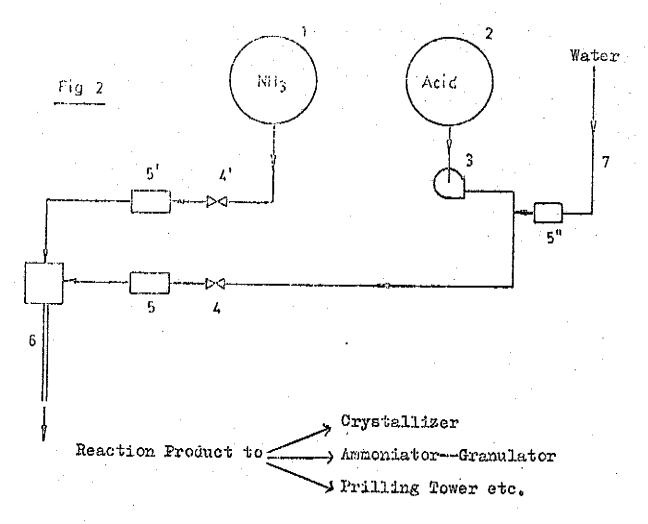
II) 2a)- 2b) The volume of the reaction chamber in which the mixture of liquid NH3 and acid takes place is 50cm³. Both are in a state of fine division, i.e. fine droplets.

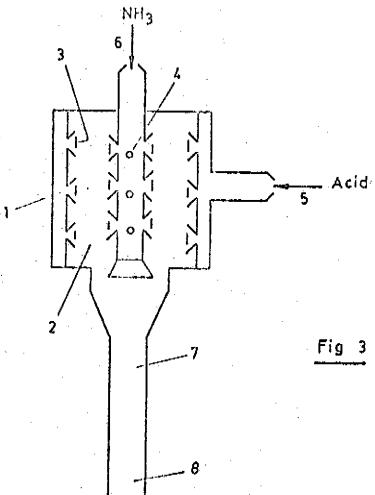
This chamber leads into a pipe the length and shape of which vary - it also incorporates bends - and whose exact characteristics we cannot give for obvious reasons of technological secrecy. The length of the tube referred to in Table 1 was 1,500 mm. and its diameter 70 mm.

- c) The material of which it is made is ordinary steel lined with teflon. The acid is diluted, so that the heat of reaction is eliminated by evaporation of water. The working temperature fluctuates between 125/155°C.
- d) To secure better distribution over the bed of solids in the granulator, the use of several units in parallel is recommended. However, we are planning units capable of neutralising 750 kg/hour/unit. After testing, we shall consider the feasibility of scaling up to twice or three times this amount.
- III) With D.A.P. it is advisable not to exceed 85°C in the granulator by controlling the recycling rate and acid dilution. We consider that when the temperature rises to 95°C the losses of NH3 may range between 10/12% of the total injected.
- IV) 1. If the temperature does not exceed 70° and with suitable control of the humidity of the mass at 4/5% and pH between 5/6, no losses are to be expected in the granulator.
 - 2. It is of fundamental importance not to exceed 70/75°C in the mass. However, N_2 balances have enabled us to estimate that 2% losses may be normal. The pH of the final product ranges between 5/5.5.
- V) In answer to this question, we would explain that the text should read "a recycling bed consisting of granules of 1 mm. in diameter." This was a typographical error.

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