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PROCESS TO PRODUCE LARGE GRANULES OF AMMONIUM NITRATE FROM PRILLS

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I - INTRODUCTION

The French production capacity in Ammonium Nitrate is about 4.2 millions tons per year as 33.5 % N. Most of the product is granulated by prilling towers.

A.Z.F., the most important French fertilizers producers, has of its own a yearly production capacity of some 1.6 millions tons ammonium nitrate.

Recently the market demand get oriented towards granules instead of prills. Clients generally ask for products having a granulation within the same range as the granulated NPK fertilizers, namely a d_{50} higher than 2.8 mm.

This granulation is impossible to obtain economically when using prilling towers.

Ourproblem was then to modify the production process to cope with the client's demand without having to stop all of the prilling towers production. As a matter of fact the erection of granulation plants would have needed high investments hardly justifiable by the depressed conditions of the market.

A.Z.F. decided then to use prills as an intermediate product, adding after the prilling tower some kind of swelling device.

Practical experiments have shown that, above the expected increase of the d_{50}, this process was giving granules of improved physical qualities and that the overall operating conditions of the plant were much easier than when operating a prilling tower alone.

II - ANALYSIS OF PRILLING PROCESSES

These processes are well known thus our analysis will be limited to the main drawbacks suffered in our production units.

2.1 - Characteristics of prills

- Under normal operating conditions the d_{50} for prills is about 2.5 to 2.6 mm and it is difficult, even impossible, to obtain industrially a $d_{50} > 2.7$ mm.

The granulation range, defined as the diameter change between d_{10} and d_{90}, is relatively low : 1 to 1.2 mm.

- In order to achieve a good Hansen Test the moisture content in prills must be very low (< 0,20 %) which gives some problems in concentration, specially when producing low grades needing some kind of calcareous filler.

- The physical characteristics of prills are often poor :
  - low hardness leading to caking tendancy
  - low erosion resistance leading to dust generation

  Even when using appropriate chemical additive and/or coating agents, bulk storage in clients' stores is often delicate.

2.2 - Prilling towers

- Due to the huge air flow needed (often 300 000 m3/h and more) air conditioning is impossible and the production rate of the towers can vary widely, according to the climatic conditions. Designing the towers for the worse case leads to so important an oversizing that the investments become unacceptable.

- In the special case of nitrate prilling (aerosols generation) difficult to solve pollution problems arise at the top of the tower. Any kind of really efficient dedusting system is extremely expensive.

- The production of high grade ammonium nitrate (34.5 % N) presents generally few problems. The situation is not the same when producing low grades (e.g. 26 % N) as a filler is needed which leads to frequent problems of plugging and/or erosion of the spraying system.

2.3 - Main characteristics of prills - prilling tower

Average values for 800 t/d - 33.5 % N

Prills :

Nitrogen content	: 33.5 %
Hardness	: 1.5 to 2.0 kg (diameters 2.5 - 3.15 mm)
Abrasion	: 18 %
H_2O	: 0.15 % to 0.2 %
Bulk density	: 0.9

Granulation (tower outlet)	: > 3.15 mm	12 %
	2.5 - 3.15	38 %
	2.0 - 2.5	40 %
($d_{(50}$ = 2.5 to 2.6)	1.6 - 2.0	5 %
	1.0 - 1.6	4 %
	< 1	1 %

Prilling tower

Prills temperature (tower outlet)	: 100°C
Air flow	: 10 000 m3/t

III - DESIGN FOR SWELLING

- The swelling should make it possible to obtain
  - a d_{50} = 2.8 to 3.2 mm
  - the production of low grade AMN (26 %) starting from high grade prills (34.3 - 34.5 % N)
- Above that the general characteristics should be at least as good as these of the prills.

IV - INDUSTRIAL TESTS

These tests were conducted in an old and vacant granulation unit. The granulation capacity for ammonium nitrate was supposed to be 300 t/d. In fact it was not a pilot plant but a true industrial unit.

4.1 - Prills

After some trials it appeared that it was interesting to work with small prills (average diameter 2.1 to 2.2 mm) obtained at a temperature of 90-100°C. The nitrogen content of the prills was adjusted to 34.5 % N.

These prills were introduced at the inlet of a granulation drum at a rate of 4 t/h (about 100 t/d).

4.2 - Slurry

Some preliminary trials shown that it was possible to add about 8 t/h of slurry (200 t/d). The slurry is prepared by mixing a 97.5 % ammonium nitrate solution with a calcareous filler. The filler must be as unreactive as possible in order to avoid the production of calcium nitrate.

The filler amount is adjusted so as to obtain in the final product (prills + slurry) the correct nitrogen content. Practically it is possible to produce any grade between 34.5 % N and 26 % N. The mixing (ammonium nitrate solution + filler) is made in a stirred tank also receiving

- the washing solutions
- the solutions obtained by dissolution of fines

The water content of the slurry thus obtained can vary between 2 % and 6 % depending on the nature of the filler and the desired nitrogen content. The slurry temperature is kept around 150-160°C, here again depending on the filler (nature and amount).

4.3 - Final product structure

The basic principle of production : "coating" of a high grade prill by a slurry containing a filler could well lead to the production of heterogenous granules. More specifically there was a risk of non-adhesion between the prills and the coating and, when dry, the granules could fall apart in prills and nitrate shells.

Practically this phenomenon did not appear and adhesion between prills and slurry is excellent. As long as prills are entering the drum at a high enough temperature (90°C- 100°C) experience shows that the external area of the prills is remelted with the warm slurry (150° - 160°C). An intermediate area is thus created between the central prill and the external layer without any discontinuity. All together the final result is very much like a kind of welding. This is confirmed by hardness control and microscopy observations. The granules final structure is pratically the same as if the granules were obtained by direct granulation (fluidised bed, drum, pan). included photo (fig. 4 and fig. 5).

4.4 - General operating conditions

From the first results it appeared clear that it was possible

- starting from prills (d_{50} = 2.1 to 2.2 mm) to obtain bigger granules (d_{50} = 2.8 to 3.5 mm)
- to coat pure prills with a slurry containing a high amount of filler and obtain an AMN at the desired grade (between 34.5 % and 26 % N).

It was then necessary to find out the general operating conditions in order to have a stable granulation loop, a low recycle ratio, a limited number of workers.

Ammonium nitrate solution

The ammonium nitrate solution coming from the wet section contains 95 % nitrate. Concentration is made in two steps. After the first step a 97.5 % solution is sent to a mixing tank which receives also the filler, the fines to remelt and the washing solution (cf. fig. 1). The remaining solution is concentrated up to 99.8 % in a second concentration step and then sent to prilling. The nitrogen content is adjusted to 34.5 - 34.6 % by adding small quantities of sulfuric acid and ammonia.

Thus the prilling is never feeded with a solution containing a filler and there are no problems of plugging and/or erosion of the spraying system.

Prilling operations

It is apparent from fig. 1 that for a granules production of 100 the amount of prills needed is around 35. As an example, if we consider a plant producing 600 t/d as prills and if we wish to increase this production to 1000 t/d by using a swelling-drum it is enough to run the prilling tower at half capacity (300 t/d) to feed the drum.

Apart of that we have seen (◊ 4.1) that a convenient average diameter for prills is 2.1 to 2.2 mm to obtain large granules averaging a d_{50} = 3.2 to 3.3 mm.

Practically it is then possible to simultaneously operate the prilling at 50 % of its design capacity whilst producing much smaller prills (d_{50} = 2.1 mm instead of 2.5 mm). These two facts converge to significantly reduce the pollution problems linked with the prilling tower operations. The air flow through the tower can be reduced by about 50 % and the production of smaller prills results in avoiding almost completely the generation of satellite droplets which produces fines, dust and fumes.

Recycle (fig. 2)

The first tests made it clear that a good granulation was strongly depending on the recycle characteristics.

Recycling very fine granules should be avoided as their specific area is so important that they catch too much of the slurry. It is much better to remelt fines.

Recycling nearly on size product (e.g. 2.5 mm) is detrimental too as producing oversized granules which, by crushing, generate fines and dust.

Both problems have been solved by using a screening in 3 steps :

1 cloth for oversized (4 mm x 2.5 mm void)
1 cloth for on-size (2.5 mm x 10 mm void)
1 cloth for fines (1.5 mm x 25 mm void)

This kind of screening system is not often used in granulation, the current practice beeing to use double-deck-screens merely putting apart oversized (> 4 mm) and fines (< 1.6 mm).Working in 3 steps has a number of advantages :

a - Recycling only product with a given granulation range allows to reduce the production of oversized at a very low level (less than 3 %). The remaining oversized products consist mainly in fragments coming off from the walls (drum and chutes) and from agglomerated granules, unavoidable in a granulation process.

b - The recycle fed to the drum is very homogeneous as consisting in

- prills d_{50} = 2.1 to 2.2 mm
- fines d_{50} = 2.1 to 2.2 mm

Under these conditions the amount of fines evolved from the swelling-drum is low (< 20 %) most of the product beeing on size (> 75 %). As a result the recycle ratio can be very low.

c - When the void of the screen-cloths is judiciously selected it is possible to adjust the flows of various granules so that the granulation range keeps in line with a given value.

Above that it is possible to decide of the amount of fines to be remelted and the amount of product recycled to the drum.

Under these conditions no scalping screen is needed before storage.

d - Screening in 3 steps makes it possible a screening on the warm granules without problems (such as plugging of the cloths). The final cooling is made only on on-size product going to storage (2.5 mm to 4 mm). This product contains practically no dust and there is no need for a dedusting device on the cooler.

<u>The swelling drum</u>

The first patent dealing with the characteristics of a swelling drum is dated sept. 1975 (B.F. :2324355). The equipment consists in a rotary drum, with built-in flights. A convenient slurry is sprayed onto a curtain developed by the rotation of the drum.

The granules go along the drum in a parallel-air flow. Granulation and drying are simultaneously achieved, the heat needed for drying is evolved by the cristallisation heat of the slurry. In the last part of the drum some cooling of the dry granules is performed.

Ambient, non conditioned, air is entering the drum at about 20°C. At the drum outlet air is at some 80-85°C and nitrate at 95-100°C.

In order to achieve a good granulation process it is necessary to maintain all along the drum a well defined temperature gradient in order to avoid any localized overheating (which could slacken the cristallization rate and clog the drum) and to secure a progressive water evaporation.

A convenient design of the flights allows to reach most of these goals. Fine tuning is achieved, when needed, varying

- the flow of air
- the granulation and temperature of prills
- the moisture content and temperature of the slurry
- the spraying characteristics

Mastering simultaneously all of these parameters is necessary to obtain a smooth running and a good granulation.

4.5 - Conclusions from industrial tests

The general operating conditions summarized in the here above ◊ 4.4 show how interesting it may be to connect a prilling tower with a swelling drum.
Besides, the physical characteristics of the product are good too as stated below :

Nitrogen content	: at will between 34.5 and 26 %
Hardness	: 2.5 to 3.5 kg (diameter 2.5 to 3.15mm)
Abrasion	: 0.5 %
H_2O	: 0.15 % to 0.2 %
Bulk density	: 0.9 to 1.00 (depending on the filler)

Granulation	: > 3.15 mm	44 %
(drum outlet)	2.5 - 3.15	34 %
	2.0 - 2.5	11 %
(d_{50} = 3.0 to 3.3):	1.6 - 2.0	8 %
	1.0 - 1.6	2.5 %
	< 1	0.5 %

Considering the overall results obtained during the tests it has been decided to use this process in our plant in Toulouse.

V - INDUSTRIAL REVAMPING

We had in Toulouse an ammonium nitrate plant producing 600 t/d by prilling. By addding a swelling drum after the prilling tower we expected to produce 900 t/d of ammonium nitrate granules (d_{50} = 2.8 to 3.3 mm).

5.1 - Production diagram

The production diagram is given by fig. 3.

We have been using equipments existing in an old NPK plant located near the prilling tower. This use of recovered equipments introduced some constraints in the general lay-out of the plant, as well as in the flow sheet. Nevertheless the overall design is as near as possible of the ideal design deducted from the industrial tests.

5.2 - Swelling drum

Due to the special design of the drum it has been necessary to use new equipment

diameter : 4.2 m
length : 15 m

5.3 - Screening - gas washing

The recovered material being not suitable we had to erect a new gas washing section and to add new screens

5.4 - General results

Results are identical to those obtained during the industrial tests. It eventually resulted possible to produce 1000 t/d of ammonium nitrate of various qualities ranging from 34.5 % N to 26 % N.

The main characteristics of the products are in close agreement with the data here above given (◊ 4.5).

Above that it has been confirmed that :

- an important operating factor is to conduct granulation with a constant recycle : temperature and flow.
- the plant is much easier to start up than a classical granulation unit as hot prills are immediately available. A preliminary warming up of the granulation loop is not necessary.
- the amount of remelted fines is about 10 % as for a prilling system.

5.5 - Revamping cost

The total cost Studies + Equipment + Modification + Erection was about 30 millions francs.

VI - CONCLUSIONS

With a relatively low investment cost it has been possible :

- to increase the production capacity from 600 t/d to 1000 t/d (The prilling is running at half capacity 300 t/j).

- to obtain granules of the desired size (d_{50}=2.8 to 3.3 mm)

- to obtain granules with much better physical qualities than prills. In fact these granules are very similar to products obtained by direct granulation.

- to reduce the level of pollution of the plant

Globally it is a simple and efficient process to revamping an existing prilling tower while increasing the production capacity and improving the product qualities.

A.Z.F. is planning a progressive revamping of all its prilling towers with this swelling process.

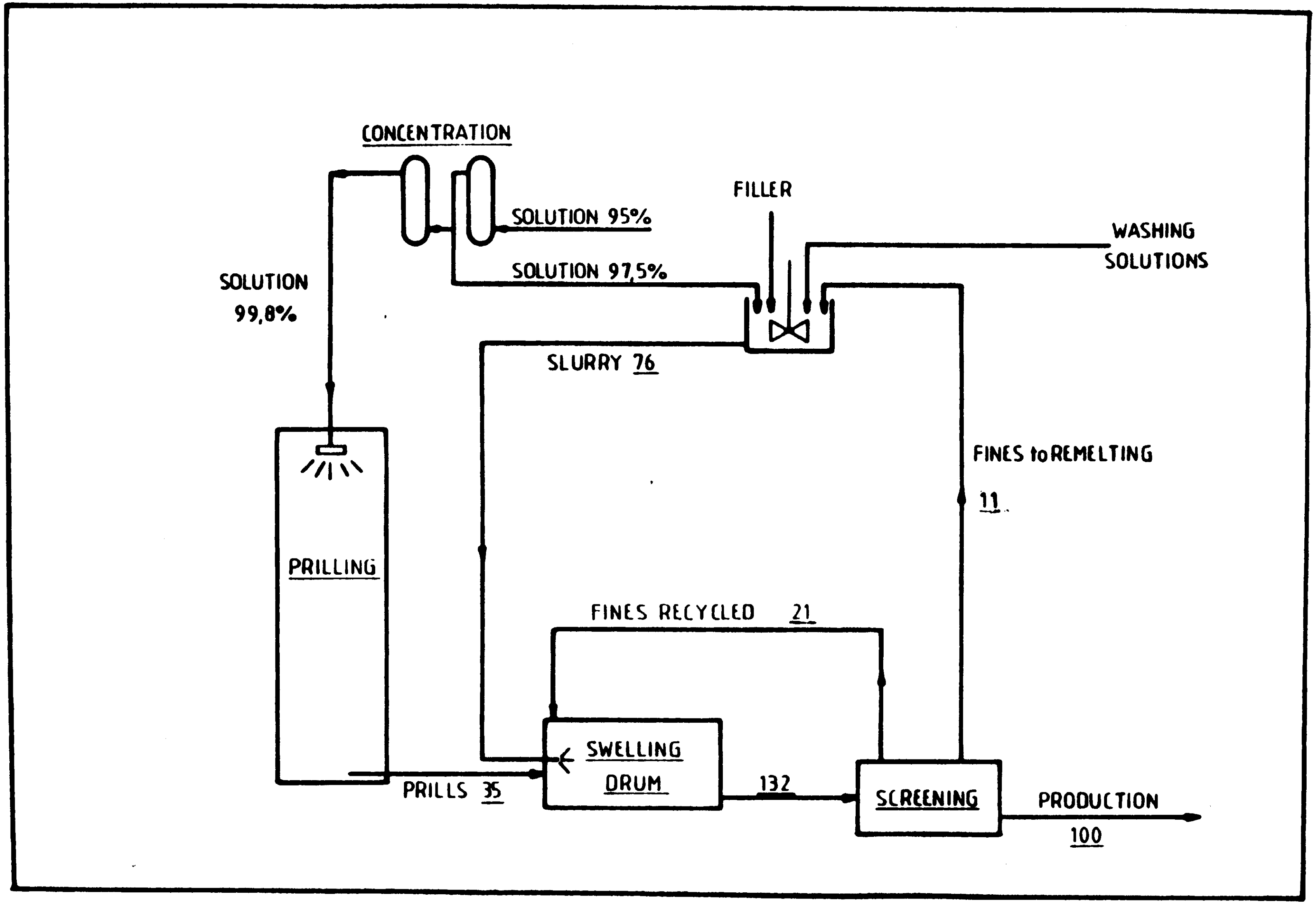

Fig.1 BASIC FLOW SHEET

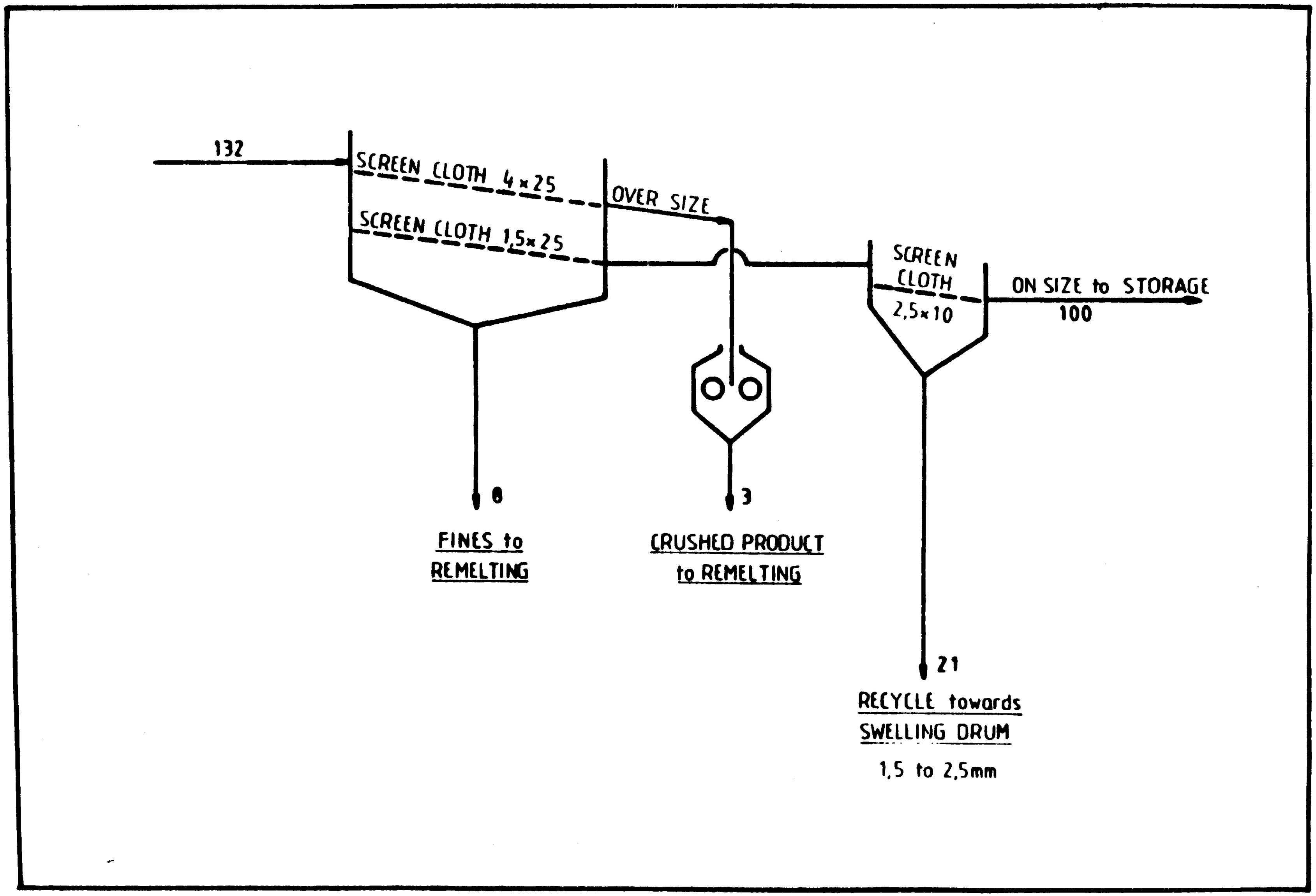

Fig. 2 SCREENING DIAGRAM

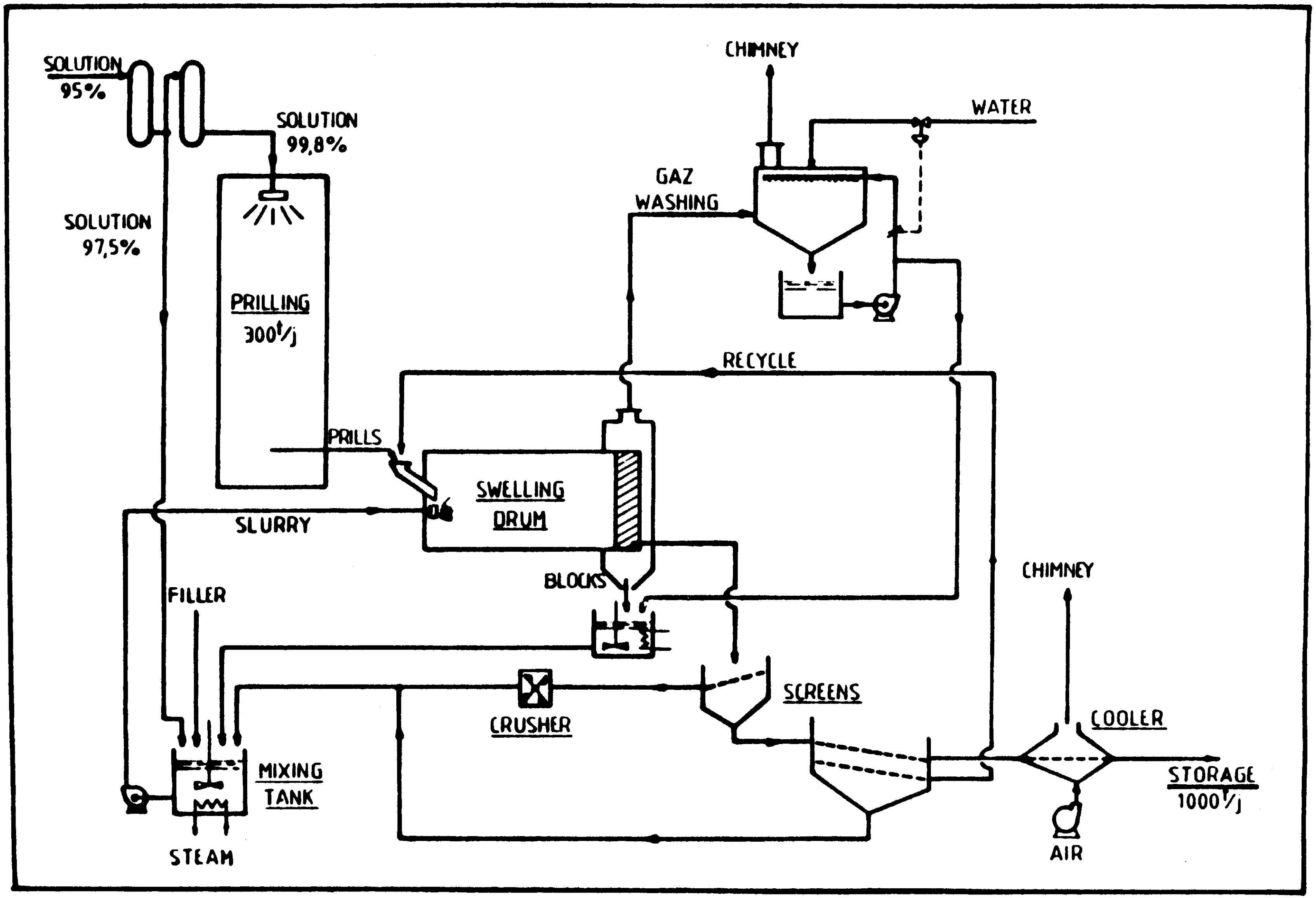

_Fig.3_PRODUCTION DIAGRAM
1000t/D. AMN.34,5%.26%

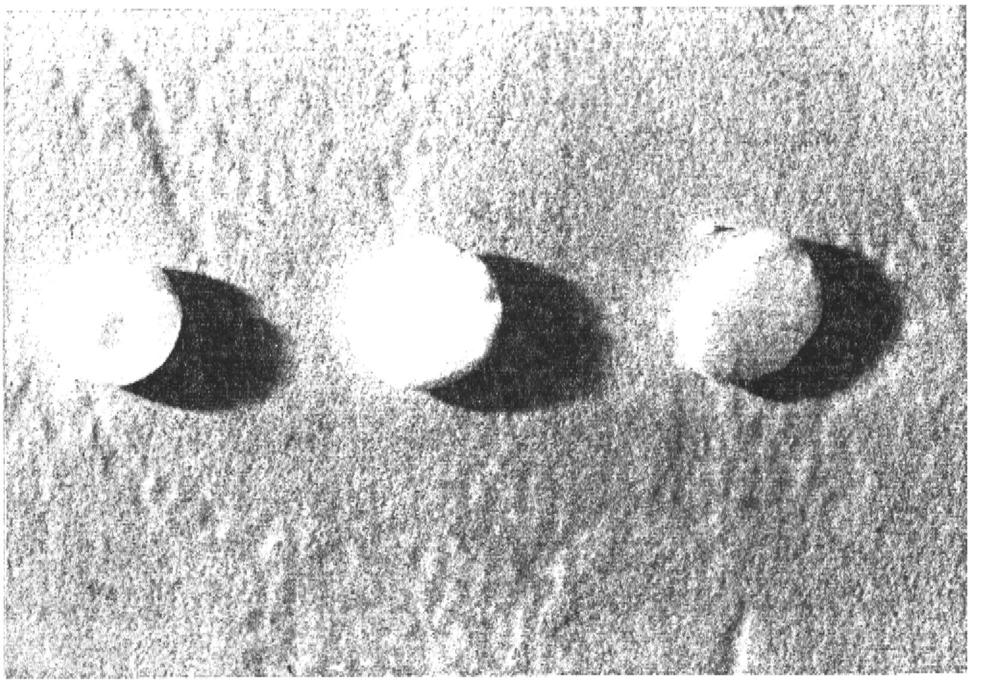

Figure 4 : Cross-section photos - left : prill,
center : AZF Process swelled prill,
right : AZF Process granules.

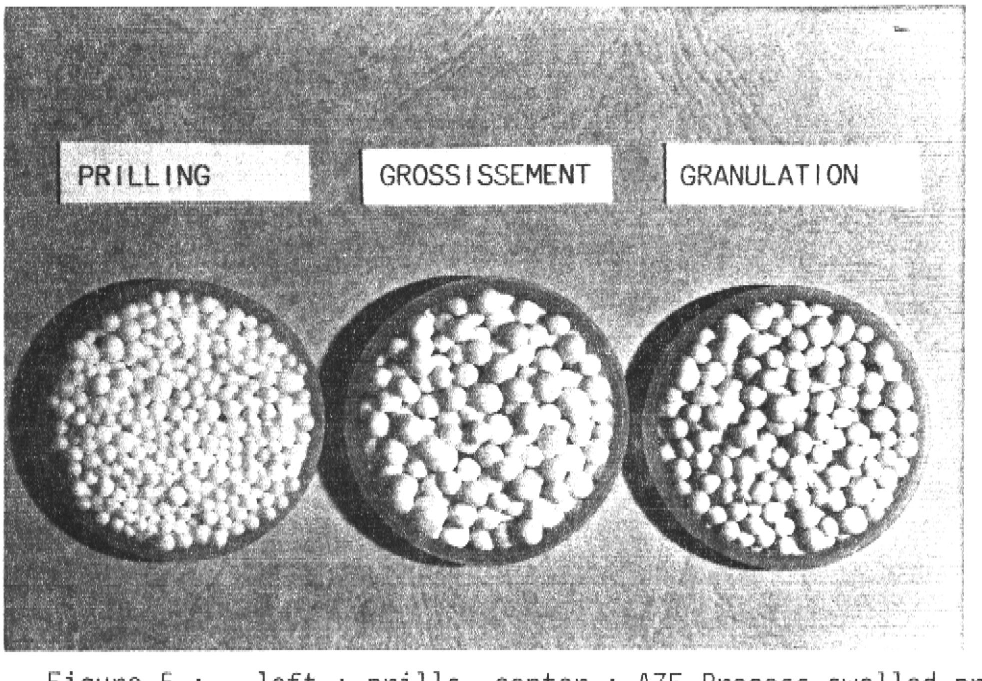

Figure 5 : - left : prills, center : AZF Process swelled prills,
right : AZF Drum Process granules.

TA/86/8 Process to produce large granules of ammonium nitrate from prills by P. Chinal, C. Debayeux, H. Lacroix & J.B. Peudpiece, CdF Chimie AZF, France

DISCUSSION : (Rapporteurs Mr A. Constantinidis, SICNG, Greece & Mr V. Bizzotto, NSM, Netherlands)

Q - Mr P.I.J. SUPPANEN, Kemira Oy, Finland

In your paper you have mentioned the prilled product to be minimum 35%
What is the maximum amount of prilled product you can use in your process?

A - In our paper we indicated that the amount of prills is about 35% of the finished product. This amount corresponds to an optimum in particular for:

- the growth ratio
- the increased capacity

But, if a lower growth ratio is wanted or if an overlapping of granule size ranges of the prilled product or of the thickened product is accepted, the proportion of prills can be greatly increased.

Another important aspect is: All the filler introduced to reduce the prill grade from 35% to 33.5 or 26% is carried by the slurry fed to the thickener. The slurry needs to be pumped and sprayed. The viscosity depends on the proportion and the quality of the filler.

In some cases, the physical properties of the slurry themselves can determine the maximum amount of prills which can be introduced in the thickener.

Q - Mr P. ORPHANIDES, DUETAG, France

The figure you indicate in your paper to revamp your plant to produce 900 t/d AN 34% is rather high: 30 million FF for the modification mainly in the dry part of the plant.

I believe the cost for design + equipment + erection for a low recycle AN process, for instance Pan Granulator, to produce 900 t/d 34% AN is not higher than the figure you indicated.

Furthermore the operation of a low recycle granulation plant is more simple than the revamped prilling + granulation plant. Can you please comment this remark?

A - The three purposes of the revamping of our prilling tower at Toulouse by ammonium nitrate swelling process were:

- to make a product of a high enough quality for storage in bags and in bulk.
- to produce, in the same plant, medium and high grade ammonium nitrates.
- to have as low as possible an investment cost.

No granulation process (including pan granulation) could fill these three conditions with an investment cost lower than or equal to the proposed one. I would also like to emphasize the easy operation of this process in which granulation is done with a constant recycling in terms of granule size and temperature.

Q - Mr B. DROCOURT, SPIE Batignolles, France

Which type of screen did you use for separating the various granule size fractions?

Did you find clogging problems in the screen and how did you solve them?

A - The various granule size fractions were separated in a three stage screening system involving two consecutive screens (3 cloths)

The screens used are of the giroplan type with ball anticlogging. We did not find any problem of cloth clogging.