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A new fluid bed granulation product.

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### SUMMARY

With the ever growing use of high-grade fertilizers like urea and DAP, the main crop nutrients are now applied through sulphur-free products instead of through AS and SSP for example. This can initialize sulphur deficiency in many areas with sometimes dramatic crop failures, since sulphur is an essential plant nutrient.

Therefore a specialized fertilizer providing both N and S without major grade loss has a large market potential.

Urea-ammonium sulphate (41 % N - 5 % S) is a typical product meeting this requirement.

It is efficiently produced with the fluid bed granulation process from a slurry of AS in aqueous urea, where the amount of undissolved AS is significantly lower than in the waterfree slurry that would be required in other granulation processes.

The product is free flowing in bulk storage, with excellent crushing strength and wide size flexibility.

### INTRODUCTION

Nederlandse Stikstof Maatschappij (Dutch Nitrogen Company) is a 55 years old major nitrogen fertilizer producing company, with worldwide export experience. As such, we have witnessed and frequently steered the shifts in product use.

Many years ago sulphate of ammonia was the most important straight nitrogenous fertilizer and single superphosphate was the most widely used phosphate fertilizer.

Sulphate of ammonia with its low nitrogen content of 21 % only and its very high sulphur content 24 % is a very acid fertilizer but, at the time that the fertilizer applications were low, it has proved to be an excellent fertilizer also under tropical conditions.

This fertilizer provided the crops with two nutrients-nitrogen and sulphur-indispensable in plant development. Both elements are the essential buildingstones in the production of plant proteins. However, in former times it was not only the sulphate of ammonia that supplied the crop with sulphur but this element was also supplied by single superphosphate containing 20 % P<sub>2</sub>O<sub>5</sub> and 12 % sulphur. Sulphate of ammonia and single superphosphate widely used in former days supplied the crops with more sulphur than was necessary to cover the needs of the crops.

In the course of the years the situation concerning the supply of sulphur in crop production changed dramatically, from a too high provision with sulphur to practically no sulphur applications at all.

Urea, nowadays by far the most important straight nitrogenous fertilizer under tropical conditions, containing 46 % nitrogen but not containing sulphur, replaced sulphate of ammonia, whereas the non sulphur containing ammoniumphosphate and the straight phosphate fertilizer triple superphosphate both with 46 %  $P_2O_5$ , replaced the sulphur containing single superphosphate.

With the harvest of the crops, almost as much sulphur as phosphorus is removed from the field whereas in addition sulphur is lost from the soil by leaching. It is clear that, as no sulphur is supplied, its content of the soil will decrease in the course of the years and the soil may become even deficient in sulphur.

In fact it is not amazing that nowadays the occurrence of sulphur deficiency in crops is reported from all over the world. Crop responses to sulphur have been reported already from 50 countries for more than 25 different crop species indicating the importance of sulphur as plant nutrient in crop production (Ref. 1).

Some crops have a greater need for sulphur than others. Cruciferae like oilseed rape, cabbage, cauliflowers, kale, turnips, radish and liliacae like onions, asparagus are high sulphur consumers and need 20-45 kg of sulphur per ha.

The commercial tropical crops such as cotton, coffee, sugarcane, oil palm and also crops like groundnuts, lucerne, maize, millet have a relative high requirement of sulphur : 15-35 kg per ha. Whereas grasses and cereals do use amounts of 10-15 kg sulphur per ha.

In order to satisfy the need for sulphur in crop production and to provide the farmer with the possibility to supply the indispensable nutrient sulphur to his crops in an efficient way, new high analysis nitrogenous fertilizers based on urea and containing sulphur, are being developed.

Sulphur content is variable depending on the ratio of ammonium sulphate and elementary sulphur chosen for manufacturing. When only ammonium sulphate is used, then the fertilizer contains 41 % N and 5 % S; for higher sulphur levels, elemental sulphur should be used with or without addition to ammonium sulphate.

It is interesting to mention the form in which S is available. Plants take up sulphur in the form of sulphate ( $SO_4^{2-}$ ). So the sulphate of the ammonium sulphate is immediately available for the plant while elemental sulphur needs to be transformed to sulphate ( $S \rightarrow SO_4^{2-}$ ), which process depends on the size of the sulphur particles used in the mixture.

In fact, due to the very slow oxidation rate, most forms of elemental sulphur have been shown to be inadequate suppliers of nutrient. This is especially valid in cold climates and dry soil conditions.

Studies conducted by the Saskatchewan Institute of Pedology have confirmed that the rate of oxidation of elemental sulphur is directly proportional to the specific area of the particles

(Ref. 2). Thus fine sulphur particles will be oxidized more rapidly than larger particles.

In many cases, only micron-sized (less than 10  $\mu\text{m}$ ) elemental sulphur can be an acceptable sulphur fertilizer, still underrating ammonium sulphate.

Urea-ammonium sulphate (UAS/41-0-0-5S) is a fertilizer that meets the main requirements of :

1. high-analysis nutrient.
2. limited acidity.
3. ratio of nitrogen to sulphur near to 10, well within the optimal plant uptake ratio when all sulphur is in sulphate form.
4. lower ammonia volatilization than urea alone (Ref. 3).

#### MANUFACTURE OF UAS

UAS has been test-produced by TVA in the past by pan-granulation and oil-prilling (Ref. 4). Tests also indicated that air-prilling would be feasible (Ref. 5).

All those techniques are somewhat hampered by the limited solubility of ammonium sulphate in the mandatory anhydrous urea feedstock (Fig. 1). They never reached large scale industrial production.

The solubility of ammonium sulphate can however be significantly enhanced by the presence of water in the urea feedstock (Fig. 2).

This prompted NSM to test the production of UAS in its patented fluid bed granulation process of urea, where feedstock does not have to be nearly anhydrous urea but is preferably a 95-96 % urea solution.

The development of the new UAS product followed the same company policy as previously applied with urea :

1. initial laboratory screening to evaluate the process and product prospects in batch granulation test plans.
2. short continuous laboratory runs to further define the optimal process conditions.
3. initial extrapolation at semi-industrial scale to evaluate plant operability and process stability.
4. continuous production at this semi-industrial scale of 150 MTPD for further long term reliability assessment, including bulk storage, handling and marketing of large tonnages of test product in the main consumption areas.

Up to mid-1984, the new UAS material had already been shipped in major lots to the following countries, besides numerous ton-size test quantities :

Brazil	4 600 t.
Guatemala	4 600 t.
Ireland	15 300 t.

#### FLUID BED GRANULATION PROCESS

The general description of the fluid bed granulation process is done on the basis of agricultural grade urea, now industrially in operation in several plants with a total capacity of ca. 3500 MTPD, with another 5500 MTPD in final construction.

#### PROCESS DESCRIPTION - UREA GRANULATION

Fig. 3 illustrates the NSM urea granulation process. The feedstock is urea solution concentrated to approximately 95 percent, which is obtained through one evaporation step using low pressure steam from the urea synthesis plant.

In the granulator, the unit at the heart of the NSM process, the solution is sprayed onto granular urea seed material held in fluidized suspension (Fig. 4) Low pressure compressed air fed to the urea solution injection system is preheated to slightly above the solution's crystallization temperature, consequently avoiding plugging of the solution spray nozzles.

Since fluidization air is fed at ambient conditions, no dehumidification is required even where the climate is hot and wet. The air is distributed under the fluidized layer and, together with the injection air, is extracted from the top of the granulator. Basically, the rectangular granulator vessel consists of a perforated plate, lower casing, upper casing, and feed solution injection headers. The perforated plate acts as a distributor to evenly spread incoming fluidization air. The plate also acts as a grid to support the granule bed when it is at rest.

The lower casing supports the perforated plate, houses the solution injection piping, and is the plenum chamber which receives fluidization air. The injection headers in the lower casing deliver feed solution to the nozzles from which the solution is sprayed onto seed particles.

The upper casing is partitioned into chambers by baffles that prevent backmixing. The fluidized layer is confined to the upper casing where the seed materials grow. In the "disengaging" space above the fluidized layer, coarse product dust is separated from the upflowing air and is retained within the granulator, circulating back to the fluidized layer.

Initially, seed material is introduced to the granulator. The seed is continuously coated by urea droplets from the spray nozzles. The average diameter of the droplets is much smaller than the final diameter of the granules, and thousands of drops are required to form a single granule.

Known as slow accretion, this mode of growth gives each granule a homogeneous structure, considerable hardness and low residual moisture, even though the feed solution contains about five percent water. Accretion differs appreciably from agglomeration, which is the operative mechanism of most other granulation processes. In accretion, each granule develops from a single seed, whereas agglomeration produces granules that incorporate several seed particles. Average buildup time for a granule via accretion is several minutes.

With the NSM granulator's specially designed spray nozzles, the urea solution is atomized at low air pressure. The nozzles are

located within the fluidized layer, and their spray cones are completely immersed. Urea droplets are therefore prevented from entering the disengaging space and being carried along with the exhaust air. Keeping the spray cones submerged in the fluidized layer also prevents urea buildup on the walls of the granulator.

The solid particles in the fluidized layer of the NSM process vary considerably in terms of size. Average size is well above that characteristic of most other fluid bed systems. This creates an irregular, turbulent fluidization pattern that causes all granules to move repeatedly through the spray cones. While the largest granules tend to settle on the perforated plate, they are laterally thrust to the next chamber by aerodynamic forces arising from the design of the perforations. The granules follow this pattern of movement until they flow out of the granulator at the bottom. In contrast to overflow bleeding utilized by other fluidization processes, bottom bleeding effectively avoids the accumulation and further buildup of agglomerates.

Another important principle operative in the NSM design concerns the balance of heat within the granulator. For processes which granulate from a melt feed of 99+ percent urea, either a very large amount of air or a very high solid recycle ratio to the granulator is needed to dissipate the heat released by urea solidification. However, since the NSM feed is a 95 percent urea solution, heat generated during urea solidification is transferred to the accreting granules, vaporizing the water contained in the atomized feed solution.

Product extracted from the granulator flows to a standard fluidized bed cooler that uses air as the fluidizing and cooling medium. If the ambient air is sufficiently cold, further dehumidification and cooling are not necessary. For plants in tropical and sub-tropical areas, refrigeration is usually provided to chill and dehumidify that portion of the air to the cooler which will be in contact with the granules at the lower temperature. In large capacity plants this is more efficiently achieved in a second cooler which treats only the screened end product. Warm air extracted from the coolers contains a much smaller amount of dust than the air from the granulator.

After cooling, the granules are screened and separated into three fractions. Appropriately sized product granules are sent to storage after final cooling. Undersized and coarse granules are recycled to the first chamber of the granulator as seed material.

Adjustment of the roll mill crusher controls the amount of seed available per unit of granulator feed solution. By adjusting the crusher and air flow, an NSM granulation plant can produce either agricultural urea or larger bead size granules. Average particle diameter for the first type ranges from 2 mm to 3 mm, while product can be up to bead size 8 mm in diameter. Under normal operating conditions, the solid recycle ratio between recycle and product is very stable and low. It is 0.5 : 1 as opposed to a higher, less efficient ratio of 2 : 1 or even more for other urea granulation processes.

Because of the moderate air flow involved, the level of urea dust in the granulator air stream amounts to only four to five percent of plant capacity. Air extracted from the top of the granulator is scrubbed. The air is washed with process condensate from the urea synthesis plant, and the resulting dilute urea solution is used for scrubber circulation.

Virtually all of the dust from the granulator is removed in the scrubber. Dust recovered from both stages forms a 40-50 percent urea solution which is recycled to the evaporation section of the urea synthesis plant. The quantity of dust vented from the scrubber falls easily within limits specified by the most stringent air pollution requirement.

#### KEY FEATURES OF NSM FLUID BED GRANULATION

The NSM fluid bed granulation process for urea has a variety of features that greatly enhance technical efficiency and cost effectiveness. Since the feedstock solution is concentrated to about 95 percent through the application of crystallization heat released in the granulator, the evaporation section of the synthesis unit can be a single stage. This also reduces the load on the condensate treatment section of the synthesis unit. The result is lower expenditures for equipment as well as decreased steam and cooling water requirements.

As a direct consequence of the thermal balance in the granulator, less fluidization air is needed and the fluid bed granulator can be physically smaller than drum or pan units. In turn, this means smaller investment and operating costs for the system's blowers and heat exchangers. Additionally, minimal dust loading in the exhaust stream entering the scrubber contributes to a low solution recycle ratio to the synthesis unit, which further reduces the duties of the synthesis evaporation and condensate treatment sections, thereby achieving even greater operational and investment economy.

Along with a low solution recycle ratio, the NSM granulation process has a very favorable solid recycle ratio. This is due to continual "disengagement" of dust particles from the air stream moving through the granulator and retention of particles in the granulator until they grow to product size. A low solid recycle ratio eliminates the need for a large capacity recycle loop, which affects the size and cost of units such as conveyors, screens and crushers.

Formation of biuret, an undesirable by-product of urea manufacture, is practically zero due to the low processing temperature and the 95 percent concentration of the urea granulator's feedstock solution. The amount of biuret formed in the course of the entire granulation process is only 0.03 percent. Such a figure is negligible compared to the increase in the level of biuret during urea synthesis 0.5 percent with crystallization and 0.8 percent with evaporation.

In the design of an NSM granulator, the number of spray nozzles per unit of fluid bed area remains constant regardless of plant capacity. Only the overall area of the fluid bed and the total number of spray nozzles will vary with capacity. Since other design parameters such as nozzle size, air velocities, fluid bed depth and residence time are constant despite plant size, the NSM granulator is well suited for large, very economical single-train designs.

### HIGH RELIABILITY, SIMPLIFIED OPERATION

Having no moving parts, the NSM granulator is essentially free of maintenance. Since only the fans and blowers require scheduled maintenance under normal conditions, a typical plant can be kept on stream for at least 345 days a year.

Operation of an NSM granulation plant is flexible and uncomplicated. Throughput is adjusted by opening or closing valves which deliver urea solution to the injection subheaders and by control of the solution pressure set point. Turndown to about 10 percent of design load is possible, and full load can be regained in less than two hours.

Product size is regulated by the screen opening selected and the size of seed particles as determined by the setting of the roll crusher. Plants with two 50 percent capacity screens and proper conveyor systems can be switched from production of 2.0 mm granules to granules as large as 8 mm without shutting down operations. The only factors which affect the stability of granulator operation are the inlet temperature of the fluidization air and the concentration of the feed solution. Any variation in these parameters tends to occur slowly and can be compensated for very readily.

The final NSM granular product has very high crushing strength as well as low moisture and biuret content. Of these qualities, the most important process advantage is hardness - some five times that of prills. This helps maintain proper granule structure and markedly reduces dust formation during storage, loading and unloading.

### ENVIRONMENTAL ADVANTAGES

The NSM granulation process has a number of inherent advantages with regard to the control of potential environmental problems. As mentioned earlier, the amount of air required is substantially less than for other processes, which reduces dust loading. Further, since the air space above the granulator's fluid bed is under a slight vacuum, dust cannot leak from the granulator into the surrounding work area.

In sum, the NSM fluid bed granulation process offers a significant number of advantages over other granulation techniques. These include lower capital requirements, reduced energy consumption, less maintenance and superior product qualities.

### ADDITIONAL FEATURES FOR UAS GRANULATION

The main flow-sheet loops are similar to their design for urea granulation, in such a way that a dual-purpose plant can be conceived (Fig. 5).

The additional features required involve :

1. Injection of a solution/suspension of ammonium sulphate crystals in the urea feedstock solution; the modified granulation mechanism also allows some extra flexibility in urea concentration.

The biuret content is "frozen" as soon as the sulphate is

added to the urea solution.

2. Clogging-free header design and erosion-resistant spray-nozzle specification.
3. The presence of ammonium sulphate crystals in the injected feedstock results in a slightly different product buildup, where some internal seeding influences the seed balance in such a way that less oversize crushing is required without sacrificing the product size flexibility.
4. The scrubbing can be completed with sulfuric acid injection to catch ammonia present in the urea feed solution.
5. The end product is coated with a proprietary liquid agent to avoid further moisture uptake during bulk storage and handling in wet climatic conditions.

### UAS PRODUCT QUALITY

Addition of ammonium sulphate provides an overall improvement of the physico-chemical properties of granules as compared with urea.

Since the physical outlook of UAS granules is very similar to urea granules, NSM adds a yellow dye to the feedstock for identification.

This dye is then symbol of the typical brimstone color.

### Typical Physical Characteristics of Granules

	<u>UAS Granules</u>	<u>Urea Granules</u>
Moisture content	0.15 %	0.20 %
Biuret content	0.60 %	0.80 %
Total nitrogen content	41.00 %	46.00 %
Sulphur content	5.00 %	-
Crushing strength (2.5 mm)	3.6 kg	3.0 kg
Average size	between 2.0 and 3.5 mm	
Bulk density loose	780-800 g/l	730-750 g/l
Bulk density tamped	820-840 g/l	770-790 g/l
Coating	liquid 1 %	none

### CONCLUSION

Urea-ammonium sulphate (UAS 41-0-0-5S) is an excellent fertilizer formulation to counter the rising sulphur deficiency detected in numerous countries for many crops.

Fluid bed granulation is an efficient technology for large capacity granulation of this product.

The outstanding physical properties of the product match the most severe bulk distribution requirements, even to tropical countries.

Fluid bed granulation plants can be designed to produce alternately straight urea and UAS with minor additional equipment.

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LIST OF FIGURESFig. 1

Phase diagram of urea-ammonium sulphate mixtures.

Fig. 2

Water diluted entectic of urea-ammonium sulphate.

Fig. 3

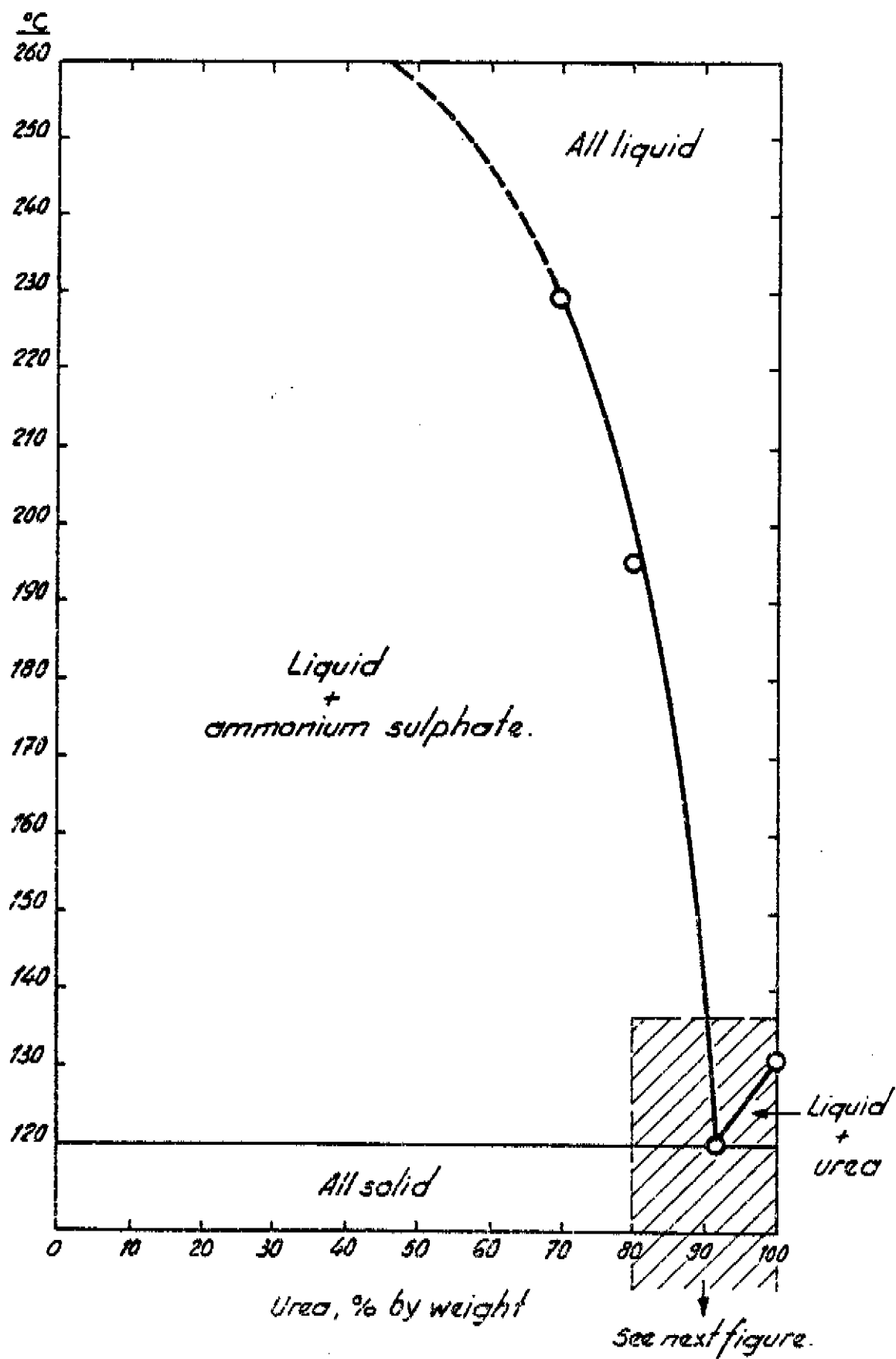
Flowsheet of urea granulation process.

Fig. 4

NSM fluid bed granulator.

Fig. 5

Flowsheet of UAS granulation process.



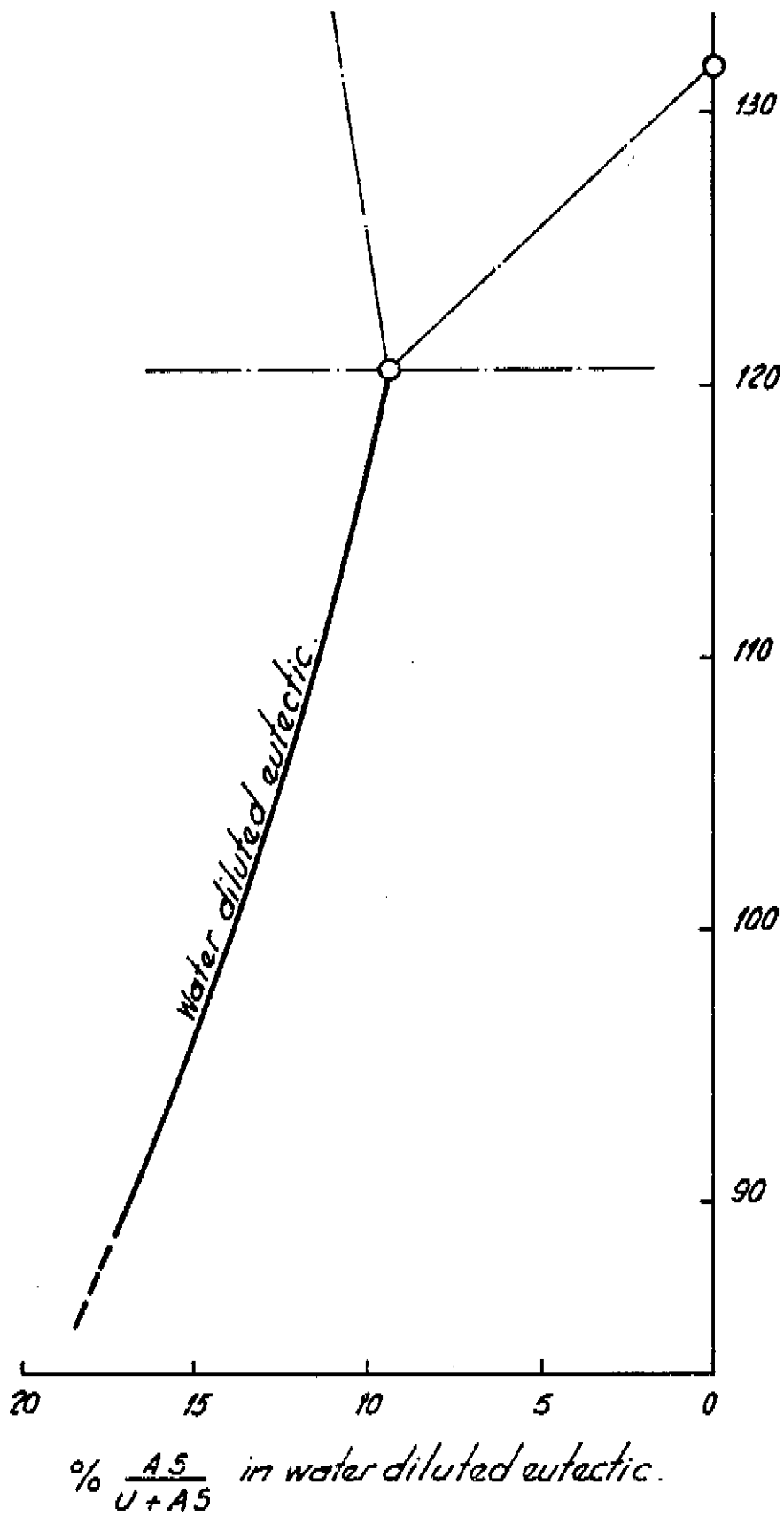
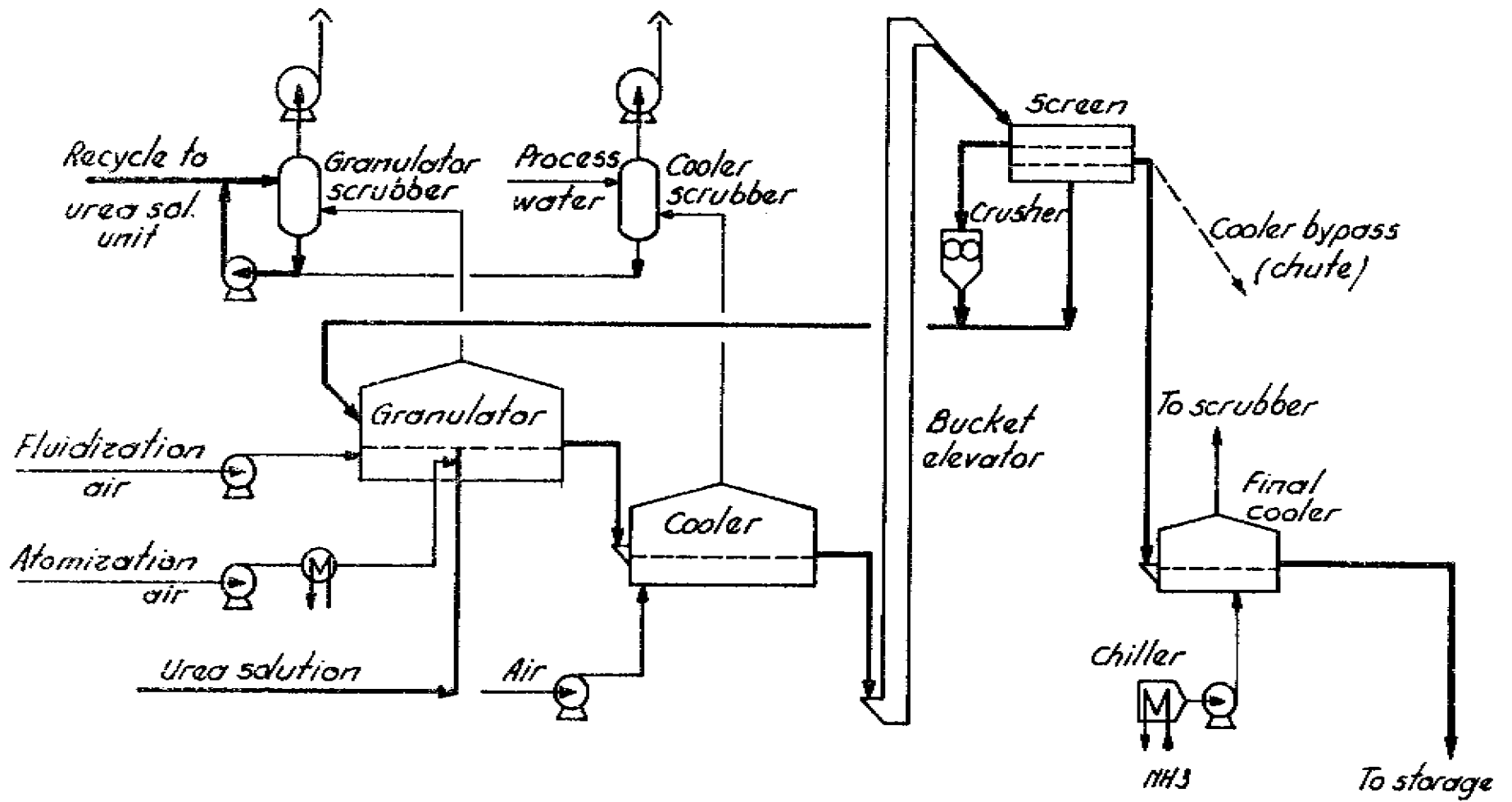
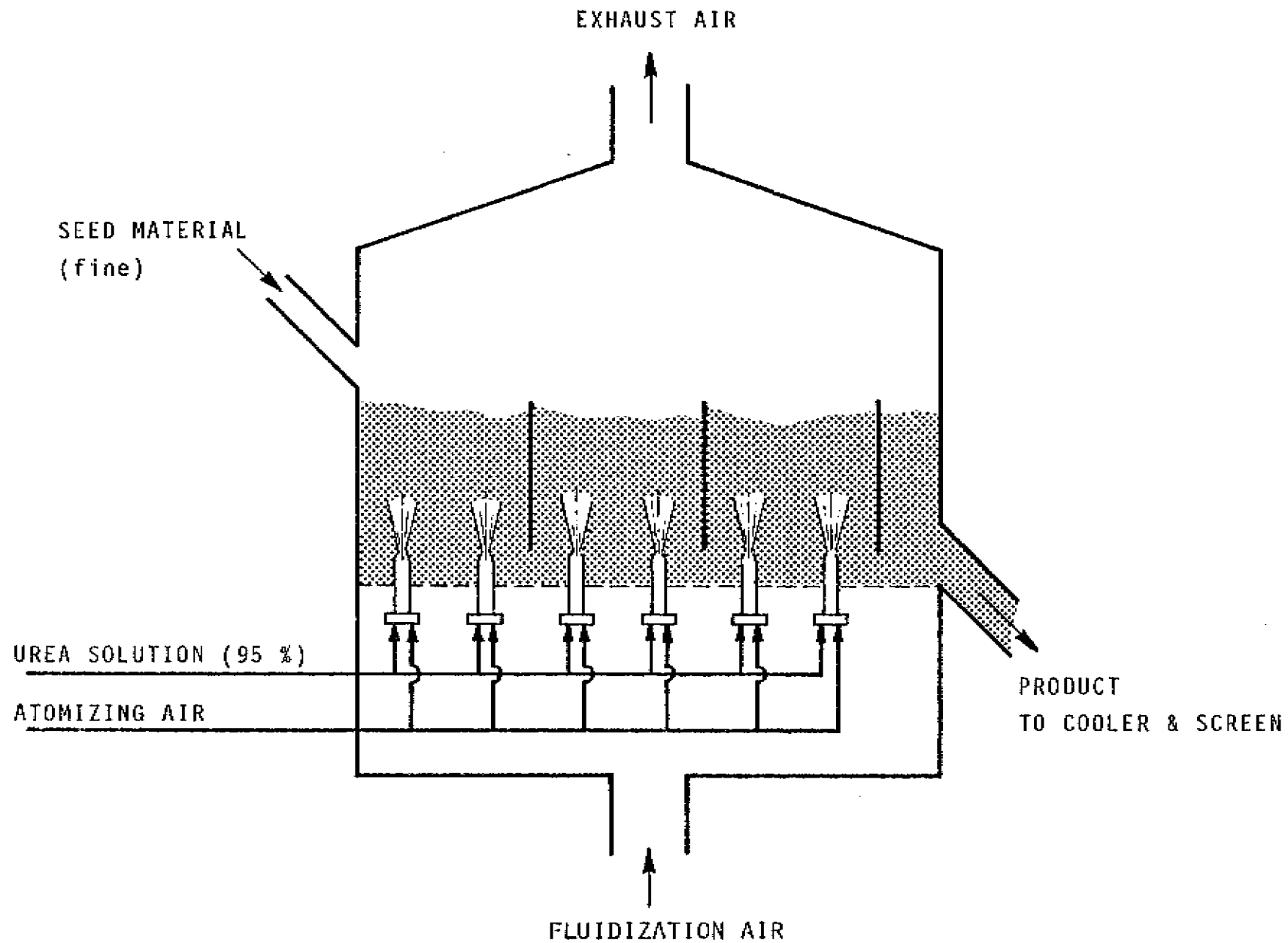


Fig. 2

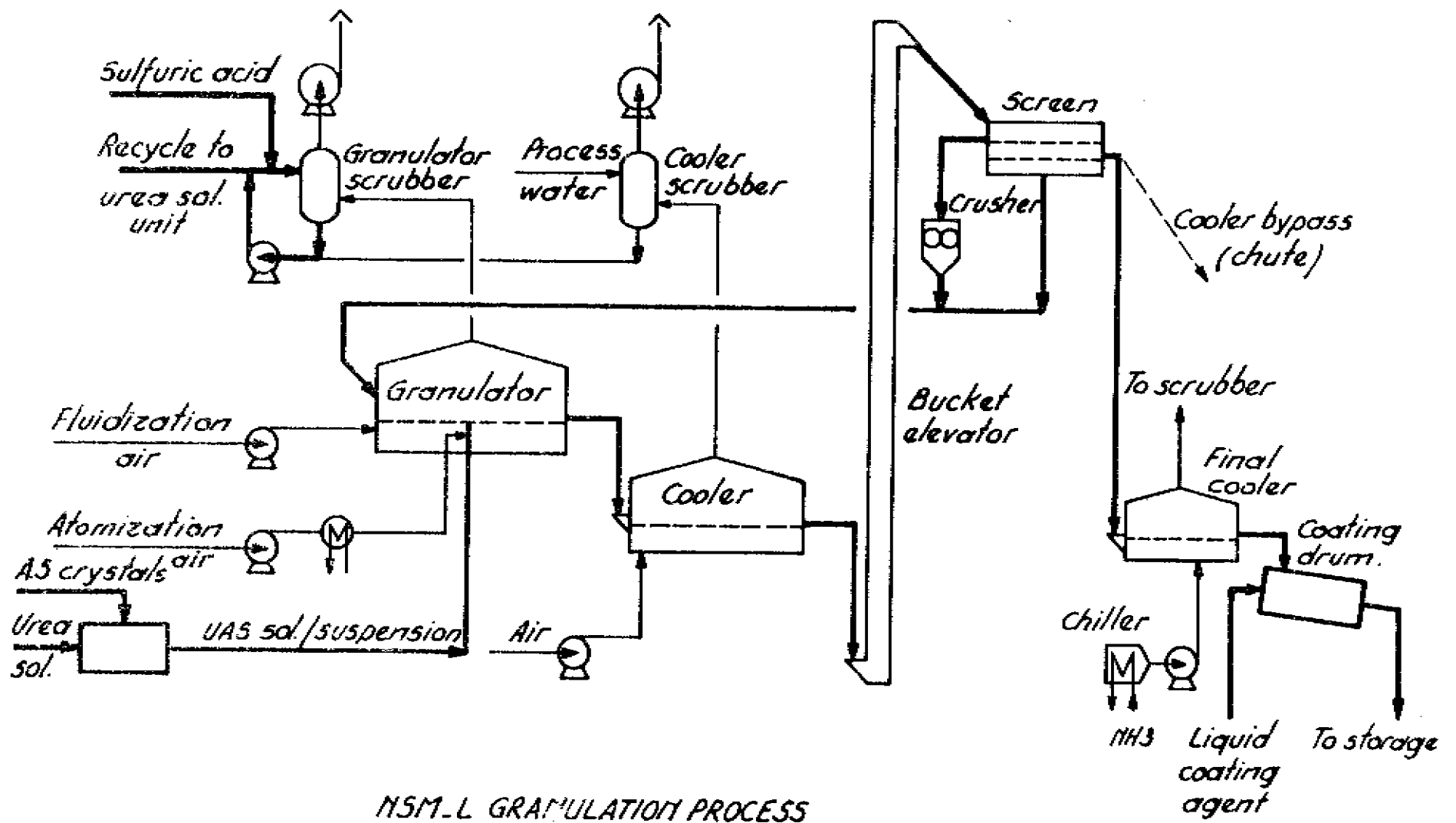


15M.L GRANULATION PROCESS



NSM-L FLUID BED UREA GRANULATOR

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TA/84/7 Granular urea-ammonium sulphate - A new fluid bed granulation product by V. Bizzotto, NSM, Netherlands

DISCUSSION: Rapporteur N.D. WARD, Norsk Hydro Fertilizers Ltd, United Kingdom

Q - Mr. T.P. HIGNETT, IFDC, USA

Do you add formaldehyde or any compound made with formaldehyde to granular urea or to granular urea-ammonium sulfate? If so, could you state the amount per ton of each product?

A - Several potential additives for UAS granulation have been identified, both organic and inorganic. The selection of their amount is part of the semi-industrial evaluation and therefore confidential.

Formaldehyde is the standard patented additive for NSM granulation of urea, although other additives have also been patented.

Q - Mr. B.K. JAIN, FAI, India

1. It is mentioned that there is lower ammonia volatilization in UAS (41-0-0-5S) than urea alone. How would this particular aspects compare with 5% sulphur coated urea?
2. The biuret content is stated to be "frozen" as soon as sulphate is added to the urea solution. What exactly happens? Is there any explanation for this happening?
3. Is UAS hygroscopic such as to warrant coating for handling in moist climate conditions? What is the critical relative humidity (CRH) of UAS?

- A -
1. The presence of ammonium sulphate ensures a lower soil pH than with standard urea. This in turn results in lower ammonia volatilization. Sulphur coated urea on the contrary does not involve a chemical link but just a physical barrier for urea dissolution. The elemental sulphur oxidizes only very slowly to sulphate, without instantaneous effect on soil pH.
  2. The ammonium ion in ammonium sulphate displaces the equilibrium of the dimerization of urea to biuret, so that no further increase in biuret concentration is noted as soon as both components are mixed as a solution/slurry.
  3. UAS is no exception to the rule that mixed salts are more hygroscopic than their components. Therefore, a proprietary coating has been developed. The critical relative humidity of UAS is about 40% at 20°C.

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

1. Is it necessary that AS is fully soluble in urea solution before granulation. Do AS crystals affect product quality?

2. What is the coating system used for UAS?
3. The author has mentioned "large market potential" for UAS. Where are the markets? Do you get a price for sulphur?
4. What is the capacity flexibility of NSM process?

- A -
1. A mixture containing 20% AS is not fully soluble. As illustrated in figure 2, the eutectic solubility is 9% in a waterfree melt, gradually increasing with the concentration of solution water. Control of AS crystal growth is an important process parameter.
  2. The coating system for UAS is based on a proprietary liquid coating agent.
  3. The market potential for UAS covers all sulphur deficient areas where urea is presently dominant. Initial sales areas of the semi-industrial production are listed on page 7-3. The extra sulphur content is used as a marketing argument for the main urea component.
  4. The capacity flexibility for UAS is identical to that for granular urea, from 20 to 120%.

Q - Mr. A.G. ROBERTS, ICI PLC, United Kingdom

Is it possible to produce products of nitrogen content other than 41% N. If so what is the range of nitrogen concentrations which have been produced?

- A - The UAS production 41-0-0-5S is considered to be close to most agronomical requirements, when all sulphur is in sulphate form.
- For higher levels of S, we feel that micro-sized elemental sulphur should be used in the granulation mix.

Q - Mr. D. IVELL, Norsk Hydro Fertilizers Ltd, United Kingdom

1. What percentage of material is greater than 4 mm in the stream from the fluid bed when producing average product size of 2.9 mm?
2. How can fluid bed granulator and cooler be designed to produce different product sizes?
3. With 10% water in UAS slurry what is product moisture?

- A -
1. Presently UAS is produced with size distribution similar to standard bulk blending grade granular urea, where the percentage greater than 4 mm is 5% for average product size of 2.9 mm.
  2. In the size range covered by granular urea, only marginal corrections of fluidization air flows are required and design for largest size is therefore satisfactory.

3. The UAS slurry contains the same amount of water as for standard urea granulation, that means 4-5% with residual moisture content of 0.2% in the end product.

Q - Mr. P. BAEKEN, Coppée Rust, Belgium

What do you think of the production of UAS using phosphogypsum as a sulphur source?

A - The sulphur source in UAS is ammonium sulphate. The use of gypsum as sulphur source leads to another product, with much lower nitrogen content 36-0-0-5S instead of 41-0-0-5S and probably other agronomic characteristics.

It could however be a valuable outlet for phosphogypsum to be further explored.

Q - Mr. H. COUTINHO, Petrofertil, Brazil

1. Could you compare dust vented in NSM process x others (amount)?

2. Inclusion of second cooler (tropical countries) doesn't affect economic feasibility?

3. Could you tell, roughly, type and components of proprietary liquid agent, when operating on UAS basis?

A - 1. Dust vented from the NSM granulator is directly sent to a very efficient scrubber. Primary dry dust collection has been discarded for reliability purposes and also because the small load of about 4% of production does not economically justify it.

Other granulation processes exceed 10% dust load, even after some form of dry collection.

2. For large plants in tropical countries, addition of a second cooler improves the economics since it:

a) avoids deep cooling of the solids recycle stream (remember the unsensitivity of the granulation heat balance for solids recycle amount/temperature).

b) decreases the total cooling air flow requirement and its fraction that requires chilling.

3. The proprietary additives for UAS granulation are part of the semi-industrial evaluation and therefore still confidential.

Q - Mr. K. HELLAMAKI, Kemira Oy, Finland

Have you tried to granulate other products like AN, CAN, ammonium phosphates?

A - Fluid bed granulation has presently been developed by NSM up to industrial scale for products fitting in the plant production line, which include urea, AN, CAN and now UAS.

It is a company policy to only offer fully industrially proven processes.

Q - Dr. B.K. BHATTACHARYA, FAI, India

1. How would capital and operating costs of NSM fluid bed urea granulation compare with conventional prilling?
2. What percentage of erected capital cost of the fluid bed urea granulation plant would be the software component viz licence, know-how fees and basic plus detailed engineering?
3. What is the present outlook of commercialisation of this process?

A - 1. Capital and operating cost comparison of NSM granulation with prilling has been published at the 4th Conference on Fertilizer Technology by British Sulphur Corporation in London, January 1981.

The comparison basis was a 1000 mtpd prilling plant incorporating:

- 1 - equivalent dust abatement,
- 2 - equivalent process water purification,
- 3 - product with urea-formaldehyde for improved (although not equivalent) storage/handling properties,
- 4 - product cooling to an identical and constant storage temperature.

Under those conditions - valid 1981 - granulation features 1.300.000 US\$ investment advantage and about 1 US \$/t product cost saving.

2. The software component for granulation (including licence, know-how, basic engineering, training/site assistance) is within the usual range of erected capital cost for similar plants. Its overall impact can however be magnified by local taxation.
3. UAS produced by fluid bed granulation is now in the semi-industrial production phase (150 mtpd).

Fluid bed granular urea however has already a significant world market share with 3500 mtpd already in production on a total order package of more than 9000 mtpd.