International Fertilizer Industry Association

IFA Technical Conference

Kallithea, Greece

5-7 October 1982

THE NSM PROCESS FOR GRANULAR AMMONIUM NITRATE

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INTRODUCTION : NSM IN A FEW FIGURES

Nederlandse Stikstof Maatschappij (NSM) was established in 1929 to produce synthetic ammonia and nitrogenous products for agriculture and industry.

The manufacturing plant is located in the Southern part of The Netherlands at Sluiskil, whereas the Company's head offices are in Brussels, Belgium. The plant, with its own harbour facilities on the Ghent-Terneuzen Canal, has direct access to both the North Sea and the European system of inland waterways.

The current NSM production capacities are :

Ammonia	: 700,000 MTPY*
Urea solution	: 750,000 MTPY
Nitric acid (as 100 %)	: 700,000 MTPY
Ammonium nitrate solution (as 32.5 % N)	: 880,000 MTPY.

The urea solution can be diverted alternatively to the manufacture of finished products in facilities with capacities of :

46 % N prilled urea	: 700,000 MTPY
46 % N granulated urea	: 275,000 MTPY
32 % N liquid fertilizer	: 800,000 MTPY.

The ammonium nitrate solution produced can be diverted to the manufacture of the following products :

26 % N prilled calcium ammonium nitrate	: 800,000 MTPY
35.5 % N prilled ammonium nitrate	: 400,000 MTPY
32 % N liquid fertilizer	: 800,000 MTPY.

NSM's R AND D ACTIVITY

To ensure that the quality of its products ranks among the best, NSM allocates considerable resources to the Research and Development function.

* Metric tons per year

NSM's research activity is centered on applied research in the field of its own end products : urea and ammonium nitrate.

For example, in the urea field, NSM developed in the sixties, a formaldehyde based coating process for prills that imparts them free flowability and absence of dust during storage and handling. This process is widely used throughout the world.

In the early seventies, NSM began to develop its own fluid bed granulation process aimed at applying it to both ammonium nitrate and urea manufacture. On the basis of the data from a bench scale unit built in 1973, NSM started up a semi-industrial pilot plant that came on stream in 1976. That plant produced urea granules from 1976 to 1979, achieving a daily output of 150 t.

Market launch of the product brought to NSM feedback information necessary to improve the plant and to fine tune the process parameters until the obtention of a wide product acceptance.

The 800 MTD industrial urea granulation plant went into operation in 1979.

Simultaneously, the semi-industrial pilot plant was converted to ammonium nitrate production. Between 1979 and 1982, it produced all grades of ammonium nitrate and calcium ammonium nitrate.

Pilot plant operation clearly demonstrated that the fluid bed granulation process is an attractive way to make high quality AN granules in large single train units. A 1 500 MTD unit is now in the detailed design phase and is expected to be built at NSM's own site for completion in late 1983.

In 1975, NSM planned to expand its AN solution facilities by adding one single neutralizer featuring low energy consumption and low pollution ; since there was no such process available in the market at the time, NSM decided to develop an original design of the well known neutralization process. This 1 600 MTD unit was started up in 1977.

In the meantime NSM also felt the need to have all its AN prills coated in order to make bulk storage and shipment feasible : the NSM's AN prills coating process minimizes dust formation during handling and prevents caking during bulk storage. The same type of coating process is applied to granular AN.

Through its newly created subsidiary NSM Licensing (NSM-L), NSM's technology is available to fertilizer producers all over the world.

THE NSM PROCESS FOR GRANULAR AMMONIUM NITRATE

The process starts from raw materials, i.e. nitric acid and ammonia (liquid or gas) and ends up with granular product ready for bulk storage or shipment.

It fulfils the following requirements :

- a large unit capacity : one single unit is capable to fully utilize all the acid produced in a nitric acid unit whatever its size may be ;
- a high reliability and reduced maintenance ;
- the lowest possible energy consumption (steam, electrical energy);
- a pollution level that complies with all the existing legislations ;
- an effective treatment of the process water which can be entirely recovered ;
- outstanding physical characteristics of the product.

The NSM-L plants are designed as "single stream" units of large capacity and were thought out by producers for producers who specially wanted production units capable to face, from their very conception, all those demands. These plants offer a combination of three NSM's technological developments : the highly efficient neutralizer, the fluid bed granulation and the coating process.

Those units allow the production of all the agricultural grades of granulated ammonium nitrate fertilizers : the 34.5 % N grade, the 33.5 % N grade, the dilute grade (26 or 28 % N), the dilute grade with high thermal stability for hot climates.

NSM-L installations are designed and planned in such a way that they can switch from one grade to another one in the same unit.

In the NSM process, the granulation unit and the neutralization system make a whole integrated package of which the performances are remarkable. The concentration of the nitrate solution matches the need of the granulator ; the neutralization reaction supplies the heat necessary to concentrate the solution and to close the thermal balance of the granulator and of its ancillaries.

For the sake of simplicity, the process steps will be described and commented on separately later on..

THE NEUTRALIZER (see flow sheet no.1)

In this type of neutralizer, the reaction loop includes a small reactor in which the exothermal reaction between nitric acid and ammonia takes place, a separator in which process steam is separated from AN solution, an AN circulation pump, and a boiler in which a large flow of AN solution is cooled, thereby producing steam.

The reactor is a simple and small vessel operating at a maximum temperature of 180°C ; since reactor is small, the best corrosion resistant material can be selected with little leaving on the cost.

Part of the reaction heat evolved in the reactor is converted directly into process steam whereas the remainder is indirectly extracted to generate steam in the boiler.

The heat load of the boiler is controlled by AN forced circulation flow adjustment, thus ensuring very stable operation of the reactor loop.

Process steam from the separator is purified in a wash column and in turn used as heat source in the medium vacuum concentration stage where the nitrate solution from the reaction loop is concentrated to 97 %.

Steam produced in the boiler is mostly utilized in the granulation section and partly exported. The neutralizer section exports a net amount of 150 kg/t AN of 5 bar steam.

Safety considerations set the maximum temperature in the reactor loop at the ceiling value of 180°C, which is accepted all over the world as safe, provided there is an accurate pH control in the reactor loop.

Water separated from the nitrate solution, which is basically water contained in the nitric acid feed can be divided in two streams : on the one hand, the purified process water coming out of the wash tower may be utilized as raw material for boiler feed water ; on the other hand, the non purified process water, evolved in the vacuum concentration stage, may be used as absorption water in the nitric acid unit, and for make up to the scrubbers of the granulation section.

Doing so, the entire plant does not release any liquid effluent. Purified process water contains as little as 50 ppm AN and 50 ppm NH_3 ; it is suitable for deionization in order to yield BF make up water.

All the aforementioned features are used in the 1600 MTD plant NSM put into operation in 1977

THE FLUID BED GRANULATOR

In case pure high density ammonium nitrate 34.5 % is made (see flow sheet 2), the vacuum concentration unit already described is designed to yield 97 % AN solution containing approx. 1.6 % magnesium nitrate as a stabilizer.

Magnesium nitrate is produced in a small side unit by direct reaction between nitric acid and magnesium oxide.

The 97 % ammonium nitrate solution is fed to the granulator in which it is atomized into the midst of a fluidized layer of granules (see fig. 1).

Fluidization is obtained by blowing air through a horizontal perforated plate acting as a distributor. The fluidization air is preheated to set the fluidized bed temperature at its optimal value. The grid is supporting spray heads that spray upwardly into the fluidized bed.

The spraying is assisted by atomization air delivered by a blower.

Granule growth proceeds by continuous depositing of fine solution droplets from which the water evaporates in a continuous way throughout the solidification.

Thanks to this mechanism the NSM granulator possesses an outstanding capability of water stripping.

In such a way, one can easily obtain high density granules from mother solution containing several percent of water ; this characteristic of NSM granulator does away with the film evaporator which is normally present in the vast majority of prilling or granulation units.

The granulator comprises three granulation chambers, in which the solution is sprayed onto the granules, followed by a cooling section (3 chambers) in which complete solidification, as well as some cooling is achieved. Chambers are separated by transverse baffles that prevent backmixing.

The product discharged from the granulator is screened off to the final required size ; the undersize and the oversize fractions, the latter one after crushing, are recycled to the granulator, where they are used as seed material to initiate granulation. The solids recycle ratio is 0.4 : 1.

Average granule diameter is set to the requested value by the crusher adjustment and by the right selection of the mesh size of the sieves. The operator can vary the granule diameter without interrupting the production.

The granulator, which does not feature any moving part, is highly reliable and requires no maintenance.

Air exhausted from the top of the granulator is treated in a wet scrubber for dust removal and then discharged to a stack. AN dust is recovered in the scrubber as 50 % AN solution ; the quantity of dust is approximately 30 kg/MT.

PRODUCT CONDITIONING

It has always been NSM's policy to lay great emphasis on the product quality at each stage be it of production, storage or handling. In that instance, it is very important that product remains free flowing in the storehouse until reclamation, to prevent dust generation while moving it out for shipping.

To achieve that objective, NSM considers essential to treat the end product in the following way :

- cooling to a temperature low enough to minimize moisture migration during storage, thus minimizing the formation of salt bridges and the consequent caking.

- coating with a liquid coating agent in order to cut down dust formation during handling, and to reduce the negative effect of moisture absorption by bulk product after shipping.

The conditioning stage includes (see flow sheet no.3) :

- a fluid bed cooler with a wet scrubber ;
- a coating drum in which end product is sprayed with approx. 0.3 ‰ of a liquid containing oil and amines.

PRODUCTION OF DILUTE AN FERTILIZER (CAN)

Since production, transportation and/or use of pure AN is limited by law in most countries, a diluent has to be incorporated into the melt (or to the concentrated solution) prior to granulation. This diluent is generally ground limestone or ground dolomite.

With the NSM process, all grades between 26 % N (CAN) and 34.5 % N can be produced, by adjusting the diluent to AN ratio. This is also valid for the 33.5 % N agricultural grade AN, that is favoured in several European countries.

To make dilute AN, a mixing tank is necessary between the vacuum concentration unit and the granulator. In this tank, all the "ingredients" requested for the correct final composition are mixed and heated to reach the temperature level required for granulation.

SPECIFIC CONSUMPTION FIGURES

Table 1 gives specific consumption figures for the following grades :

- 34.5 % N high density AN, stabilized with magnesium nitrate ;
- 33.5 % N " " " " " " " ; and dolomite added
- 26 % N CAN (dolomite added).

SPECIFIC CONSUMPTIONS (table 1)
(per metric ton of end product)

	HD AN 34.5 % N	HD AN 33.5 % N	HD AN (dilute) 26 % N
Nitric acid 100% (Concentration 60%) (t)	0.787	0.761	0.584
Liquid ammonia (t)	0.210	0.203	0.159
Inerte (dolomite) (t)	-	0.034	0.257
Additive (MgO) (kg)	4.3	4.3	-
Coating agent (kg)	0.5	0.5	0.5
Electrical energy (kWh)	36	36	36
Imported steam (13 bar) (t)	0.04	0.04	0.05
Exported steam (5 bar) (t)	- 0.08	- 0.07	- 0.04
Cooling water (T = 10°C) (m^3)	7.4	4.5	3.6
Process water to nitric acid (m^3)	- 0.14	- 0.13	- 0.06
Purified process water exported (m^3)	- 0.20	- 0.19	- 0.15

POLLUTION CONTROL

As previously mentioned the plant does not release any liquid effluent. Atmospheric emission of AN dust can be controlled much more efficiently than in prilling towers thanks to the fact that the air flow (granulator exhaust) is approximately 3 times lower than in a prill tower. Furthermore the scrubber is installed at ground level so that air ducts are short.

Air exhausted from the granulator is treated in a wet washer of high efficiency that NSM considers more reliable than the dry collecting system (cyclones). The nitrate is recovered in a form of a 50 % solution which after evaporation is eventually fed back to the granulator.

Air from the fluid bed cooler is similarly treated in a low pressure drop scrubber.

Dust emission to the atmosphere can be as low as 30 mg/Nm^3 ; this is low enough to comply with the most stringent regulations specially with the opacity test imposed by certain countries. The wet washers recommended by NSM-L to obtain that result, are simple and sturdy pieces of equipment and require little atttention.

PRODUCT QUALITY

Product quality is a paramount characteristic which is kept in mind throughout the entire development of the project. This quality is confirmed by the fruitful commercialization experienced by NSM in exporting large amounts of bulk product.

In the case of the high concentration ammonium nitrate, the physical characeristics criteria and the safety criteria during storage and transport are closely related. Schematically, one can say that to obtain a product non sensitive to detonation, it must be a high density, low porosity product and also of stable dimensions.

Magnesium nitrate utilized by NSM as an additive modifies the ammonium nitrate transition mechanism between the crystallographic phases 3 and 4 in a way that the product does not expand and does not desintegrate when undergoing 32°C transition stage : it is therefore dimensionally stable. It will stand the severe official tests required in various countries such as detonation and oil retention tests.

NSM recommends the application of a liquid coating onto the high concentration nitrate when it is stored and transported in bulk. Such a coating is hydrophobic ; it considerably reduces caking and prevents the dust formation during handling.

Diluted ammonium nitrate (below 28 % nitrogen) must not comply with the same stringent safety requirements ; it is therefore not necessary to incorporate magnesium nitrate to it. However, product deprived from magnesium nitrate does not withstand the thermal cycling around the 32°C point ; in certain cases, for example, night/day fluctuation in subtropical weather it may be necessary to stabilize the dilute nitrate in incorporating also magnesium nitrate.

The NSM granulator enables to obtain regular shape granules of which the diameter can be easily adjusted between 2 and 6 mm, into the same unit, to match the market demand. It is so possible to adapt the dimensions of the granules to those of other components of the bulk blends, or to produce 6 mm granules utilized in aerial spreading for forest culture.

Product characteristics are summarized in table 2.

PRODUCT CHARACTERISTICS (table 2)

		Nitrate 34.5 % N	Nitrate 33.5 %N
Chemical composition			
Nitrogen	% N	34.5	33.5
Moisture	%	0.25	0.25
Magnesium nitrate	%	1.6	1.6
pH (10 % solution)		6.5	6.5
Physical properties			
Average diameter	mm	from 2 to 6	from 2 to 6
Crushing strength (product diam. 2.5 mm)	kg	2	2
Apparent density (average product diam. 2.9 mm)	g/l		
- tamped product		1,030	1,030
- loose product		980	980
Oil retention	%	<1	<1

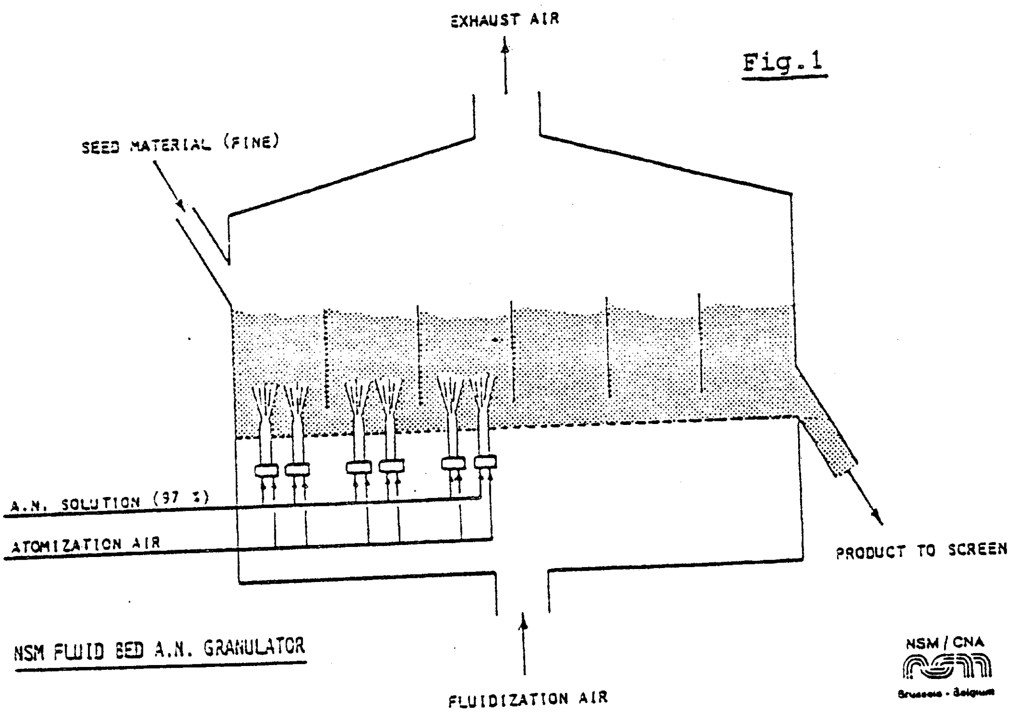
INTEGRATION WITH THE NITRIC ACID UNIT

Considering the integrated nitric acid/ammonium nitrate plant as a plant that converts anhydrous ammonia into solid ammonium nitrate, it is interesting to note that this plant is a net energy exporter, unlike a urea plant which, even with the modern stripping processes, is a large energy consumer.

In the integrated NA/AN plant, the main raw material is ammonia of which one half is used in the nitric acid unit and the other one in the ammonium nitrate unit. The end product is the granulated nitrate ; high pressure steam is exported as by-product whereas the low pressure steam generated in the neutralization loop is utilized in the nitric acid unit.

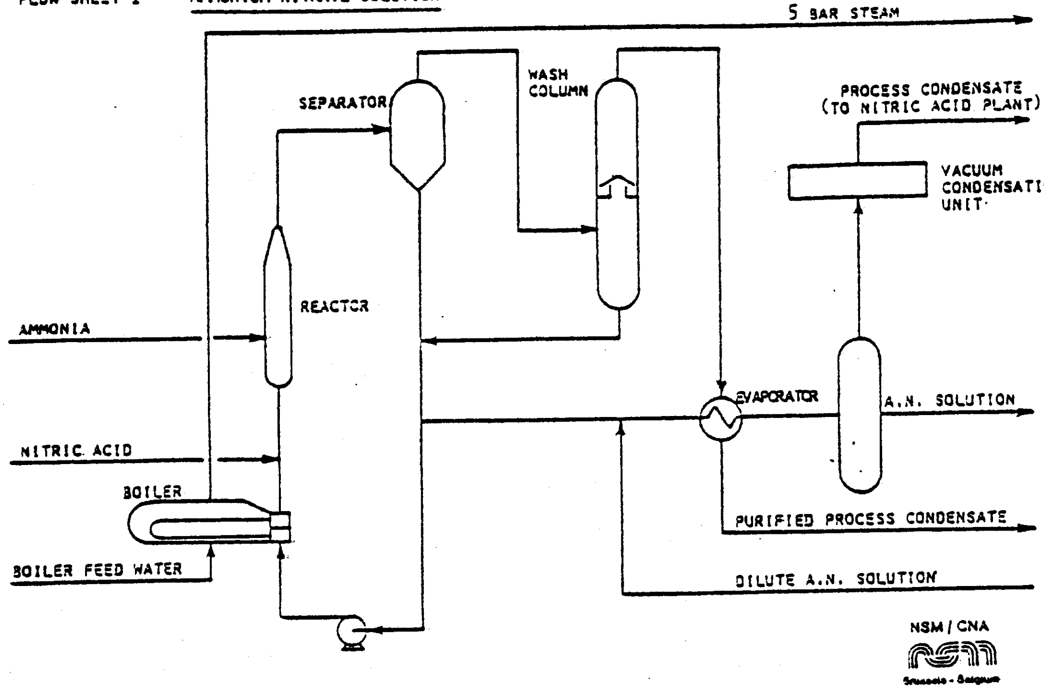
Although the granulation unit is fitted with pollution abatement devices and with a final cooler which requires considerable energy, the global unit is a net energy exporter.

Fig.1

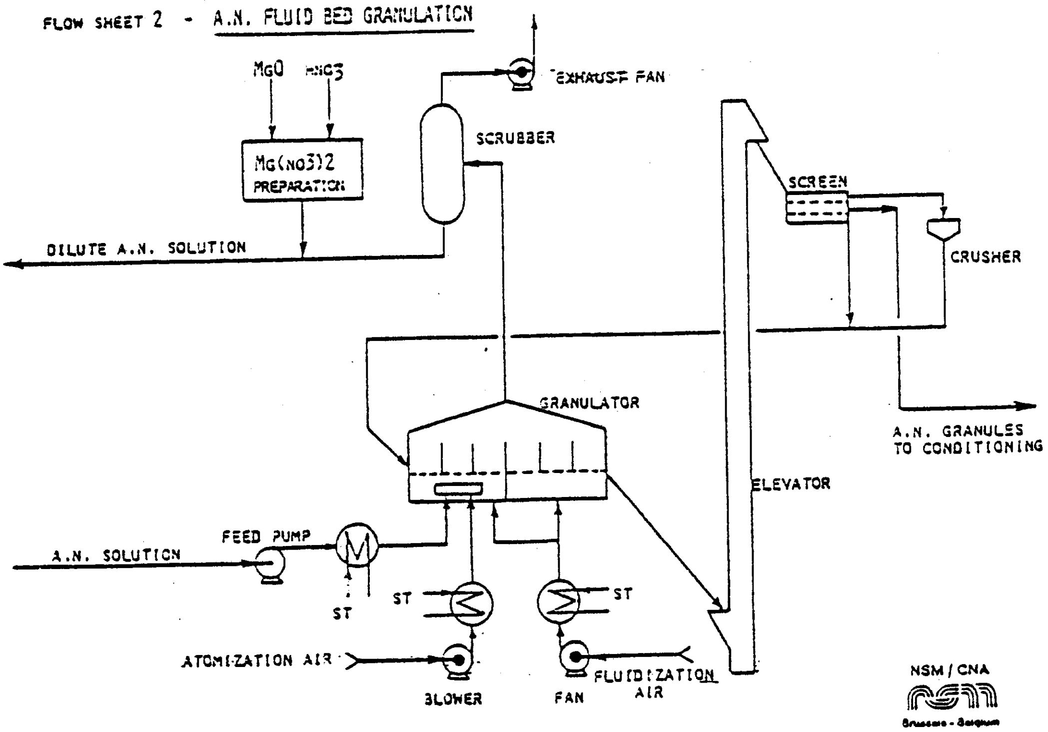


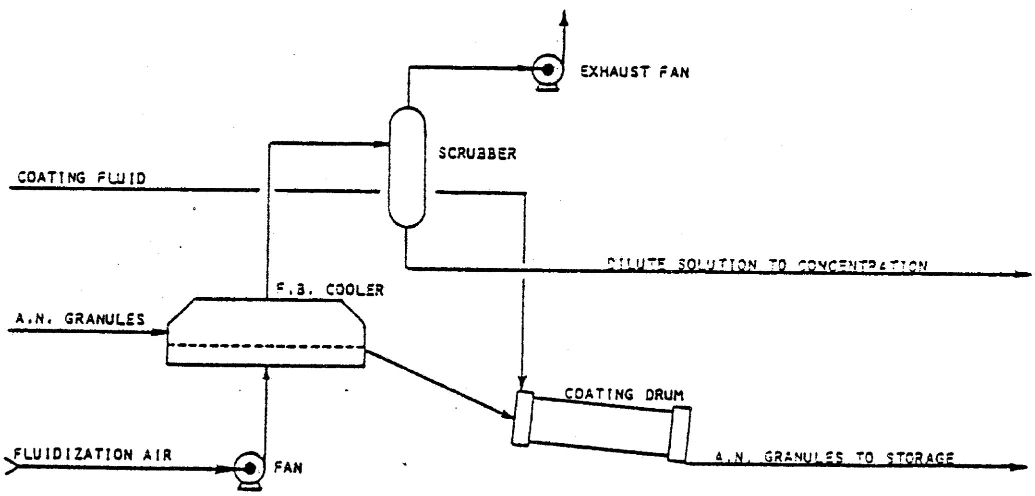
NSM FLUID BED A.N. GRANULATOR

FLOW SHEET 1 - AMMONIUM NITRATE SOLUTION



FLOW SHEET 2 - A.N. FLUID BED GRANULATION





FLOW SHEET 3 - A.N. GRANULES CONDITIONING

NSM / CNA
Bruxelles - Belgium

TA/82/12 The NSM process for granular ammonium nitrate, by V. BIZZOTTO, CNA, Netherlands

DISCUSSION : (Rapporteur Mr. T. HEGGEBOE, Norsk Hydro, Norway)

Q - Mr. W. VAN DEN HEUVEL, BASF AG, Germany

Can you give some information about the washing solution in your wash column washing?

A - Washing is performed in two stages: firstly with dilute nitric acid and secondly with water.

Q - Mr. R. LUDWIG, Chemie Linz AG, Austria

What is the circulation channel of the product inside the granulator?

A - The product circulates continuously without internal recirculation from the recycle inlet to the outlet. When moving through the equipment, it is regularly sprayed with the solution.

Q - Mr. A.C. VAN KLEEF, UKF, Netherlands

a) Do you have experience with other additives than magnesium nitrate?

b) Would non-soluble additives like nuclo-add not be abrasive in the nozzles and cause excessive wear?

A - a) We tested several other additives but we rejected them because they do not confirm the requested thermal stability.

b) Non-soluble additives like nuclo-add do not cause abrasion in the spray nozzles because they are less abrasive than dolomite. Nuclo-add was one of the products we tested and rejected.

Q - Mr. J. LOGEMANN, DSM, Netherlands

On page 8 you state that magnesium nitrate addition to ammonium nitrate results in no expansion when passing the 3 to 4 transition temperature of 32° C. Can you comment on the way the 3 to 4 transition was induced, say time-temperature relation; and the method by which the 3 to 4 transition was actually observed?

A - You could find detailed literature on the way transition points are modified by magnesium nitrate for instance in papers written by C. Sjolin. Without entering into theoretical details, we can say that we observe no swelling when product that contains magnesium nitrate is repeatedly heated and cooled between 25° and 50° C. We did not perform any theoretical study on transition points.

Q - Mr. W.J. KELLY, Albright & Wilson Ltd, United Kingdom

Magnesium oxide produced from magnesite or from sea water contains impurities such as iron and aluminium oxides and silica. These impurities can cause scaling in evaporators and in spraying systems.

1. What type or grade of magnesium oxide is used in your process?
2. Is it necessary to remove the impurities from the magnesium nitrate solution and if so,
3. Can you give some information on the method you use to do so?

A - The impurities contained in magnesium oxide do not cause scaling in heat exchangers and our spray nozzles. Any type of magnesium oxide is suitable. It is not necessary to remove the impurities. The only limitation is that the oxide should not contain either heavy metals or chlorine in quantities that would be unacceptable for safety reasons. See for instance EEC Rules on ammonium nitrate purity.

Q - Mr. L.K. RASMUSSEN, Superfos, Denmark

In production of CAN you use dolomite as filler. If limestone is used, would you then recommend adding of a stabilizer to have a good quality? If yes, what kind of stabilizer?

A - Limestone can be used instead of dolomite but it has to be tested on reactivity. Depending on reactivity, ammonium sulphate can be added to the melt.

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

1. What is the mechanism of mixing of ammonium nitrate/nitric acid and ammonia in the reactor? What material of construction is used at this point?

2. What plot area is required for a 1600 tpd neutralization plant.

A - 1. In our neutralizer there is a very high circulation rate of AN solution. Nitric acid and ammonia are injected through spargers in the circulating stream. The ammonia sparger is in stainless steel; the nitric acid sparger is in titanium.

2. The approximate plot area is 10 x 15 m.

Q - Mr. P. MORAILLON, SICNG, Greece

In the ammonium nitrate neutralizer loop, how does the boiler withstand corrosion? What happens in case of low pH of the liquor?

A - The boiler is not subject to corrosion. Low pH is avoided for safety reasons. In case pH was low, it would be only for a short time and it would not cause any corrosion. The boiler of our prilling plant is in operation since 1977 and no corrosion has occurred.

Q - Mr. G. JUIF, Kaltenbach Thuring, France

a) Your flow diagram shows that nitric acid injection takes place on the recirculation line. What are the consequences for:

1. The stability of a nitrate solution with free acid at 180° C?
2. The corrosion of reactor?

b) What is the design principle of your washing tower?

A - a) There is no free acid in the neutralizer loop. It is very important for safety to avoid the presence of free acid.

b) Corrosion of the reactor is negligible.

c) The washing tower consists of two sections of packing. In the first section, process steam is washed by low concentration nitric acid; in the second section, by water.

Q - Mr. J.E. REYNOLDS, Grace W.R. & Co, USA

Reference is made to the need for great emphasis on product quality at each stage of production, storage and handling.

1. Can you further identify the liquid coating agent and procedure used for coating the ammonium nitrate and is the same procedure used for urea?

2. What is the product retention time in the fluid bed granulator-cooler?

A - 1. The liquid coating agent can be either any of the commercially available products or an NSM proprietary coating liquid reducing moisture uptake. For urea prills, we use our own coating process in which warm prills are coated with urea formaldehyde condensate.

2. The retention time in the granulator is approximately 15 minutes.

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

Has the fluid bed granulation technology been applied to commercial production of urea and/or DAP?
What are the limitations and prospects of using fluid bed granulator for DAP production particularly in conjunction with pipe cross reactor?

A - The fluid bed granulation technology is presently applied to commercial production of urea. Preliminary tests we recently performed show the granulation feasilibity of urea - DAP compounds and of MAP. The use of a pipe cross reactor can be considered but ammoniation in the fluid bed is not feasable.

Q - Mr. J. CARIOU, Gardinier SA, France

What is the nature of MgO additive for which it is indicated a consumption of 4.3 kg/t (oxyde or carbonate)?

A - The magnesium oxide consumption which is indicated, namely 4.3 kg/t, is expressed by means of 100% of oxide, which corresponds to the real nature of this additive.

Q - Mr. P. ORPHANIDES, PFI, Greece

1. Is in table 1 the steam consumption included for concentration of the 50% AN scrubber liquor? How much is this consumption?

2. What are the means for controlling the size distribution of granules in the F.B.G.?

3. In case of CAN (26%) production where is the limestone introduced and what is the fineness of the L.S.?

4. In case of CAN (26%) production what are you doing with the scrubber liquors?

5. Quantity and quality (oil free, dry, what pressure) of the atomizing air?

6. The indicated recycle ratio = 1 product: 0.4 recycle includes the quantity of recycled scrubber liquor?

7. Quantity of fines collected in the scrubber when producing AN (33.5%) and CAN (26%)?

A - 1. The steam consumption indicated in table 1 includes the quantity requested for concentration of the scrubber liquor, amounting to 50 kg/t.

2. Size distribution of the granules is controlled by proper selection of the screen gauzes and by an adjustment of the distance between crusher rolls.

3. In case of CAN production, the dolomite is introduced in a mixer where it is mixed with the concentrated solution. The size distribution of dolomite powder is unimportant as long as particle size is not larger than 0.3 mm.

4. When producing CAN, scrubber liquor is reconcentrated.

5. Atomizing air does not require drying or oil free compression. Pressure is approximately 0.5 bar.

6 & 7. The indicated recycle ratio of 0.4 is the solid recycle ratio. It does not include liquid recycle. The liquid recycle ratio is less than 0.05.