

# IFA Technical Conference

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## 1ST WORKING SESSION

TA/84/1 New developments in the dual pipe reactor process by P. Chinal & Y. Cotonea, Cdf Chimie AZF, France (Rapporteur J. Cariou, COFAZ SA, France)

Q - Mr. H.G. JENNEKENS, UKF, Netherlands

Is it possible to by-pass the pre-neutraliser in DAP-production after plant modification as proposed by you?

A - Yes, we can revamp a DAP plant with the use of a pipe reactor without preneutraliser. We have adapted the internal equipment of the granulator.

Q - Mr. K.L. PARKS, Agrico, USA

Would you indicate the iron, aluminium and magnesium content of the 52% P2O5 acid used in DAP manufacture and forecast, if actual data not available, what DAP analysis you would expect for 52% P2O5 acid containing about 1.7% Fe2O3, 1.4% Al2O3 and 0.8% MgO.

A - Analysis of P2O5: (ex Togo) P2O5 : 49, Al2O3: 1.6, Fe2O3: 2.0, MgO: 0.36 ; Analysis of product: 17.8 - 46.25.

Q - What is the maximum capacity you would project for DAP in a single train?

A - Maximum capacity: 100 t/h without any problem.

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands

Concerning application of a pipe reactor in the drier, I presume there is a potential danger of build-up by solidification of pipe reactor-slurry on the wall of the drier. Does it occur? Can you prevent it? Is there a certain minimum drier diameter?

A - We don't meet problems of build-up in our dryers. We avoid it by the use of classical hammers on the first part of the dryer. Minimum drum diameter is 2.5 m.

Q - Mr. K.L. PARKS, Agrico, USA

Can you expand on the statement, "The problems of blocking encountered in the early industrial plants were quickly overcome"? What are a) pipe cleaning methods, b) periods between cleaning, c) nature of scale, d) influence of acid composition on scaling rate, e) reasons for this improved operation?

A - Pipe cleaning method - by use of steam.

Q - Mr. R. MONALDI, Fertimont SpA, Italy

The installation of the pipe reactor in the dryer has provoked several questions during the IFA Conference held in Greece centred almost on the problems connected with the production and recycling of the MAP dust in the DAP production.

I should like to consider this under a different point of view. The circulation in the dryer practically consists in the blending of products that have a different grade of ammoniation.

I should like to know what is the variation, possibly in statistical terms, of the content of the N and of the P2O5 in the finished product.

A - The maximum content of MAP in the final product is 4% (several analysis).

Q - Have you ever used the pipe reactor in the ammoniation of the slurry which comes from nitric attack of the rock?

If so, what are the operating problems during ammoniation?

A - The pipe reactor is not used in a nitric reaction of phosphate, but can neutralize  $\text{HNO}_3 + \text{H}_3\text{PO}_4$  with  $\text{NH}_3$ .

Q - Mr. N. LOUIZOS, SAHPEC, Greece

What is the estimated life of a pipe reactor especially when sulphuric acid is used? What is the optimum material of construction?

A - The life time of the reactor, especially with  $\text{H}_2\text{SO}_4$ , is 2 years. Material of construction, stainless steel to produce MAP, and teflon with  $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$ .

Q - Mr. E. SEUNA, Kemira Oy, Finland

How do you measure the sphericity of the granules? How do you control the granulation when manufacturing different grades with different granulating properties? How does the capacity change when producing different grades?

A - The sphericity is measured by one test in which the granules are made to slide on an inclined plane and the number of granules are counted at the bottom. Capacity range is of the same order as in conventional processes.

Q - Mr. I.K. WATSON, UKF Fertilizers Ltd, United Kingdom

We also operate pipe reactor but in conjunction with a blunger. We have also noticed that a much higher moisture content is possible for products made with the pipe reactor, and in particular 17-17-17 (see page 1-9). Do the authors have an opinion or knowledge to explain this phenomenon?

A - AZF experience is that it is necessary to maintain a low humidity to ensure good storage properties.

Additional questions put in writing

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

Norsk Hydro Fertilizers experience is that MAP is difficult to ammoniate in the solid phase. The situation must be easier if the

MAP produced in the dryer is finely divided. This goes against efficient cyclone operation:

- 1. Would Mr. Cotonea care to comment.
- 2. Is there any control over MAP particle size produced by the dryer pipe reactor. What is the optimum mean particle size?
- 3. On the converted SAEPA plant, how does dryer dust loading and cyclone efficiency compare before and after the conversion?

A - The size of MAP crystals does not affect the efficiency of cyclones. When we revamp a DAP plant, we check the good running conditions of valves of the cyclones.

For example in SAEPA, the content of dust in the scrubbing system was not changed before and after the start-up of the pipe reactor in the dryer.

Q - Mr. R. SCHOEMAKER, UKF, Netherlands

In one of your schemes you show the pipe reactor at the outlet of the granulation-drum. If this is a correct image, what function has the granulator since the major part only serves for transportation of the bulk of the recycle plus the solid raw materials?

A - Yes the pipe reactor is at the outlet of the granulation drum. The slurry is sprayed down to the bottom of the drum. And the efficiency of the granulator is the same as with a slurry system.

Q - Mr. H. BOUCHENY, Sté J. Boucheny et Cie, France

Why do you use a Venturi type scrubber? Is it because of the problems associated with the pipe reactor or of the formation of ammon'um nitrate mist in the dryer?

What is the loss of pressure in the Venturi scrubber and its efficiency?

A - The Venturi scrubber ensures a good contact between gas and liquid and a good dust collection.

TA/84/2 Start-up in the USSR of two of the world's three most powerful NPK granular fertilizer plants by P. Chinal, CdF Chimie AZF, France (Rapporteur J. Cariou, COFAZ SA, France)

Q - Mr. R. MONALDI, Fertimont SpA, Italy

From the tables 2 and 3 it is not easy to evaluate the quantity of the NH3 that enters in the scrubber. I should like to know the quantity of the NH3 that escapes from the granulator.

A - When the plant is running in DAP the total amount of NH3 through the scrubbing system is 12% of the NH3 in the product.

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

Which material is used to heat 62% HNO<sub>3</sub> to 60°C?

A - Normally with 56-58% acid, 25/20 low carbon stainless steel. No experience with 62% acid, but it should not raise any difficulty.

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

Please give more details on how liquid effluent is avoided?

A - The acid and the scrubber liquor discharged from the gas scrubber are introduced in the pipe reactor and the water is found as steam in the drier and in the granulator.

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

Considering a production capacity of the ammonium nitrate plant reported in the material balance of table 1, a pressure reactor with steam recovery can reduce to zero the steam consumption and export 6-7 t/hr of pure steam at 6 bar. Why you have not considered to introduce this energy saving in such a huge plant?

A - Yes, but a lot of steam was available on the plant.

Q - Mr. V. SCHUMACHER, BASF, Germany

Would you please give some more technical details about the "motor driven ammonia distributors" and the "automatic self-cleaning chute"?

A - At the beginning a mobile chute was built in to avoid scaling on the ammonia pipes in the granulator. Recently AZF developed a chute which avoids internal bearings. It is driven by a shaft and bears the pipe reactor.

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands

Is it more economic to achieve a very large production capacity, as in USSR, in one line, or in two lines with half the capacity in parallel?

A - It would take too long to discuss all the economic parameters, but we favour more and more very big units (for instance in USSR in the last 15 years).

Q - Mr. S. BOUCHERAT, SCFA, France

Does the very great length of the drier (40 m) not result in the breaking of granules and the increase of the recycle ratio?

A - No, numerous measurements were made in the AZF driers in the USSR and on NPK plants, both conventional (slurry process) and with pipe reactors.

TA/84/3 The use of a pressure neutralizer for slurry granulation by D.M. Ivell & N.D. Ward, Norsk Hydro Fertilizers Ltd, United Kingdom (Rapporteurs J. Cariou, COFAZ SA, France, and S. Swanström, Kemira Oy, Finland)

Q - Mr. K.L. PARKS, Agrico, USA

What is the composition of the acid stream used: a) to scrub the reactor and granulator exit gases and b) as the final gas scrubbing liquid (page 3, § 2 & 3 resp.)?

A - The scrubbing liquor is generally controlled at a mole ratio of 0.3 to 0.4:1. For DAP production, P2O5 concentration in the scrubbing liquor would be 40-45%.

Q - Mr. K. BELL, ICI PLC, United Kingdom

Do you experience any choking problems at the liquid outlet standpipe or ammonia inlet point and if so how do you clear the chokes? Do you experience any variations in flow due to foaming in the reactor?

A - There are certainly no significant problems with choking at either the liquid outlet or the ammonia inlet. We do however have automatic steam purges on all feed lines to and from the reactor. The feeds and steam purges are activated by multiposition switches. On the odd occasion the ammonia flow or the solution outlet flow can begin to drop off due to some small deposits. These deposits can be very easily cleared by selection of the appropriate steam purge for a few seconds. The plant would continue to run during this operation.

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

When you use phosphoric acid high in impurity, have you problems of building-up inside the reactor? If yes, how do you operate? What is the frequency of the reactor cleaning?

A - We have used sludge acid containing up to 15% solids in the pressure reactor without suffering from build-up problems or line blockages. We never have to drain the reactor for cleaning purposes.

Q - Mr. N. LOUIZOS, SAHPEC, Greece

What are the advantages/disadvantages of DTR compared to the pipe reactor? Could you convince a potential user to employ a DTR rather than a pipe reactor? What are normal ammonia losses with DTR?

A - We are happy to discuss the advantages of DTR compared to the pipe reactor and will leave others to discuss any perceived disadvantages.

The DTR has as its basis the same basic principle as a pipe reactor - that of operation at elevated pressure and temperature to produce a low water content solution and hence reduce recycle

ratio and fuel consumption in the solids section of the process. As with a pipe reactor agitation and slurry pumping are dispensed with.

The main difference between the DTR and a pipe reactor relates to the buffer capacity of around 20 minutes which the DTR has. This enables changes in flow or composition of the feeds to be smoothed out before the ammonium phosphate is sprayed into the granulator. This ensures that the condition of the ammonium phosphate solution - mole ratio, temperature and water content - is consistently maintained. Problems of blockages in the reactor (well documented for pipe reactors) or in downstream equipment are avoided. A consistent slurry quality ensures that granulation is stable and easily controlled and results in minimum recycle ratio and therefore maximum production rates.

The buffer capacity in the reactor, in combination with the efficient mixing afforded by the draught tube, ensures a highly efficient reaction - ammonia loss closely approaches that due to the ammonia vapour pressure.

The vapour in the reactor is easily controlled so as to maintain slurry conditions of temperature and water content independent of throughput. The reaction steam is disengaged from the slurry in a controlled manner prior to spraying in the granulator. In this way the water and energy balance in the granulator is improved and controlled and the moisture carrying burden of the granulator is drastically reduced.

In summary, we believe that the DTR combines the advantages offered by pipe reactors with the traditional virtues of conventional atmospheric reaction processes. We believe that the inherent problems with pipe reactors are sufficiently serious, particularly for DAP production, to convince many potential users to employ a DTR in preference. The new plant in India and the revamped plant, referred to in the paper, are recent examples of this.

Q - Mr. A. SARKKA, Kemira Oy, Finland

Is a pressure neutraliser suitable for ammoniation of digestion liquor (calcium nitrate, phosphoric and nitric acid mixture)?

A - We have no direct experience of using the pressure neutraliser for ammoniation of nitrophosphate digestion liquor. We do have experience however of ammoniation of sulphuric/phosphoric acid mixtures as well as ammonium nitrate/phosphoric acid mixtures. In principle we see no problem with the use of the pressure neutraliser for this duty.

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands

Why do you limit the pressure in the reactor to 1 kg/cm<sup>2</sup> g? If you would go up in pressure the water content would be even lower and recycle further reduced.

A - We have used pressures of up to 2.1 kg/cm<sup>2</sup> g for certain MAP based grades. The problem is, in general however, one of ammonia

vapour pressure. Whilst increasing pressure above 1 kg/cm<sup>2</sup> g certainly allows us to reduce water content, we can only do so by increasing the temperature.

Q - Mr. J. LE PAGE, ICS, Senegal

Is the NH<sub>3</sub> used in the pressure reactor and in the granulator in liquid or gaseous form?

A - Liquid or gaseous ammonia can and have been used in both the reactor and the granulator. In general liquid ammonia is used in the granulator whilst in the reactor the decision would depend on acid strength: if acid of 48-49% or stronger is available for DAP production then liquid ammonia is used.

Q - Mr. E. SEUNA, Kemira Oy, Finland

How do you start the DTR? How long does it take? What is the minimum strength of H<sub>3</sub>PO<sub>4</sub> to have any advantage of a pressurized neutralization? What is the difference of ammonia content in off-gases between atmosphere and pressurized reactor?

A - a) From cold the DTR is started by first filling the tube with phosphoric acid. The acid is then ammoniated to the required mole ratio depending on grade. Once this is accomplished spraying can begin and the reactor put into continuous operation. Start-up time depends on reactor mole ratio and characteristics of the acid but around 45 minutes is typical.

Note however that during short shut-downs (which can be up to several days) there is no need to empty the reactor. Shut-down is automatic and the reactor contents are maintained at the required temperature and pressure by closing all inlet and outlet valves. Small steam purges to the ammonia and phosphoric acid lines can be used if necessary to compensate for any leakage through the pressure control valve. Starting from this situation is achieved within five minutes.

b) Acid strength required depends on the grade being produced. For DAP production 40-42% P<sub>2</sub>O<sub>5</sub> acid can be used although to take full advantage of pressure neutralisation 43-44% is preferred.

c) Reactor ammonia loss is only slightly in excess of that due to the vapour pressure of ammonia. Because the reactor operates at some 25°C higher than a conventional atmospheric reactor the ammonia partial pressure will be higher. One would expect therefore that the DTR would lose 1 or 2% more ammonia than an atmospheric reactor when producing DAP. This of course assumes that the atmospheric reactor and its agitator gives optimum mixing efficiency. However after scrubbing to recover ammonia the differences would not be detectable and therefore the overall ammonia efficiency of the atmospheric and pressure process would be the same, that is 99%.

Q - Mr. H.J. BOHTE, UKF, Netherlands

What is the recycle temperature to the granulator in DAP



production when the recycle ratio is 3.9:1?

A - Recycle and granulation temperatures can vary depending on the layout of the plant. In the particular case referred to in the paper recycle temperature was around 60°C.

Q - Mr. S.K. MUKHERJEE, FAI, India

a) What is the experience of build-up of solids in pressure neutralizer?

b) How often do you need to clean the neutralizer? How much production time is lost due to cleaning?

A - a) We have used a wide range of acids in pressure reactor including sludge acid and have not experienced build-up or scaling problems within the reactor.

b) The reactor is only drained for long shut-downs (say in excess of 72 hours). The reactor is never drained for cleaning purposes.

Q - Mr. M. SIPILA, Kemira Oy, Finland

a) How much are the ammonia discharges leaving the draught tube reactor and the granulator to the ammonia scrubber and what is the efficiency of this type of scrubber?

b) What is the amount of gaseous ammonia leaving the drier?

c) What is the most preferable design of the rotary granulator?

d) Is it possible to use a blunger in this particular case?

A - a) The ammonia losses from the pressure reactor, granulator and drier depend on the grade of final product being produced. Losses are greatest during DAP production.

b) We have found that losses from the reactor are only slightly in excess of those due to the ammonia vapour pressure. In the case of DAP this is usually just over 5% of the ammonia fed to the reactor.

Losses from the granulator are of the order of 15% plus of the ammonia fed to the granulator.

Losses from the drier are of the order of 1-2% of the total ammonia fed.

The void tower scrubbers have an efficiency of around 90%. The gases from the reactor and granulator are scrubbed first in the ammonia scrubber and then in the gas scrubber thus ensuring an overall ammonia efficiency of 99%.

b) The detailed design of the rotary granulator has been developed over many years by Norsk Hydro Fertilizers and its predecessor Fisons Fertilizers.

For the production of DAP or related grades the most salient features of the design are:

- ammonium phosphate solution is sprayed under pressure through floodjet nozzles onto the bed of recycle solids.
  - ammonia is added via an under-bed sparge pipe of special design.
  - an outlet weir plate maintains a deep bed within the drum.
  - the drum is air-swept to remove evaporated moisture, and ammonia for recovery in the scrubber system.
  - flexible rubber panels are used to keep the inside of the drum free from build-up.
- c) It is possible to use a blunger in combination with the DTR. We prefer however to use a rotary granulator as this enables recycle ratio to be minimised.

As the pressure is released through the spray nozzle to atmospheric pressure some flash evaporation takes place. The heat generated from the ammoniation in the granulator causes further evaporation. Efficient removal of evaporated water from the granulation device improves the water balance and thus minimises recycle ratio. This is best achieved in an air-swept rotary granulator where a large surface area of material is presented.

Additional questions put in writing

Q - Mr. M. LAURENS, Krebs, France

On the flowsheet of page 3-2, there are:

- a prescreening of fines before cooling: Why?
- a fluidized bed cooler. Is it not difficult to operate this equipment with a wide granule size range product?

A - The scheme in figure 1 shows the undersize and a proportion of product sized material recycled hot. Oversize and the remaining product is cooled. This arrangement is considered the optimum for absolute minimum recycle ratio. It is however not essential and, for example, both the new DAP plant for Hindustan Lever and the pressure reactor revamp, referred to in the paper, feature a total hot recycle.

The object of the screening arrangement shown - i.e. hot fines, cold oversize - is to give the optimum recycle temperature for minimum recycle ratio. (You should note that the secondary fines screening is a polishing screen only, the vast majority is removed hot).

A carefully designed fluidised bed cooler can cope with a combined product and oversize stream. Large lumps are crushed by a crusher situated in the drier outlet hood. Some further scalping is carried out in the screen feeder to remove any

remaining lumps which then bypass the cooler.

Q - Mr. J. DEMEY, CNO, Belgium

1. How do you control the level?
2. How do you control the N-P ratio?

A - 1. The feeds to and from the reactor are both under flow control. The level is allowed to float, to a limited extent, although fluctuations are slow and small due to the buffer capacity of the reactor. The reactor is also fitted with a standpipe as a simple back-up level control.

2. The N-P ratio is controlled by making occasional fine adjustments to the ammonia flow as a result of periodic pH measurements on samples from the reactor. Once again fluctuations are slow and small due to the reactors' buffer capacity.

Q - Mr. M. BARLOY, OTP, Togo

Could you provide typical data for DAP production:

- P2O5 concentration of the phosacid
- Fuel consumption?

Which is governing the recycle ratio (or the production rate for a given plant); the amount of merchant product available in the dryer or the liquid phase absorption in the granulator?

In my opinion, the percent of final product between 1 and 4 mm is not representative of the size distribution quality since most of the product could be between 1 and 2 mm. It should be associated with the percent of products between 2 and 4. Do you agree?

A - For DAP production 43-44% P2O5 acid is required to realise the full benefit of pressure reaction. Fuel consumption is typically 3 kg/t.

For DAP production the liquid phase absorption in the granulator governs recycle ratio. For certain NPK grades which can be made at very low recycle ratios then the amount of product available ex dryer could determine recycle ratio.

Since generally recycle ratio is determined by liquid phase absorption rather than granulation efficiency, a very narrow size range can easily be obtained if required (e.g. 90-95% between 2 and 4 mm). The data reported on page 7 came from different places where local regulations allowed product of 1 to 4 mm and 1 to 5 mm respectively. No real attempt was therefore made to improve on the figures quoted.

TA/84/4 Granulation of NPK compound fertilizers at the new fertilizer complex of INA-Petrokemija, Yugoslavia by J.A. Hallsworth & W.F. Fortescue, Davy McKee, United Kingdom, D. Fresl, INA-Petrokemija, Yugoslavia (Rapporteur S. Swanström, Kemira Oy, Finland)

Q - Mr. J.H. MARKHAM, ICI PLC, United Kingdom

Do you anticipate increased use of triple superphosphate for production of low nitrogen formulations and, if so, will corrosion be a problem due to liberation of hydrochloric acid gas?

A - The major grades that are produced at the moment in Yugoslavia tend to be high PK products with low nitrogen. These have been produced consistently on the plant in the 18 months of operation and we have also produced similar grades on other plants for very long periods. Clearly there will be a presence of HCl gases in these overhead gases but we have not experienced any significant corrosion. This has been firmly established over many years.

Q - Mr. J. LE PAGE, ICS, Senegal

Why is MAP from Tunisia used?

A - When the plant was initially started up INA purchased some granular TSP from Tunisia at a low price. This was partly because the price was so beneficial and also as material to the initial dry runs on the plant.

Q - The transit of MAP solution from the reactor to the tower is it by pumping or by the pressure of the reactor?

A - It is a pressure reactor. It is of the type that I was describing in a previous paper except that the reactor has an agitator because it is in fact an old design.

Q - The plastic curtain, is it located inside the casing of the tower?

A - There is no shell to the spray tower. The curtain is a self-standing curtain which is just strapped to structural steelwork. So there is no solid shell surrounding or inside the curtain.

Q - Mr. G. CALIS, UKF, Netherlands

What is the water content of the MAP solution before spray drying?

A - In the case of spraying to the MAP tower it is about 10% and for spraying into the granulator it is 12-15%.

Q - Mr. R. SCHOEMAKER, UKF, Netherlands

Your scrubbing scheme shows acid scrubbing for the cooler gases and water scrubbing for the dryer gases in which I expect to be

more ammonia than in the cooler gases. Is this correct and, if so, can you comment on it?

A - The reason that we have acid scrubbing on the cooler gases is in fact because the gases from the ammonia scrubber are passing through the cooler scrubber. When we are operating on the solids route there is no acid scrubbing of the cooler gases. It is only because of the influence of the gases from the granulator that we have a final acid wash in the cooler scrubber.

Q - Mr. V. SCHUMACHER, BASF, Germany

What is the temperature of the fines recycled from the fines hopper?

A - This depends a little on which product is being made but it is generally about 60-65°C, it can be up to 70 for certain products. It depends of course to some extent on the drying temperature. For the high urea compounds we are drying at a lower temperature which ultimately affects the recycle.

Q - Mr. M. MIYAMOTO, Nissan Chemical Industries, Japan

At a designed recycle ratio of 3.5:1 for slurry route, what specific consumption figure of urea can the process accommodate?

A - We have produced products such as 26-13-0 and 27-27-0 which contain about 40-45% urea. So this is a figure that we would consider to be a reasonable maximum.

Q - Why does hot fines recycle to granulator assist in lowering the recycle ratio? And to what extent?

A - Generally for solids route products the higher the recycle temperature the better, because this assists in the actual granulation agglomeration process. For slurry route products again high temperature is beneficial because it increases the evaporation rate. But for slurry route products there is a limit and really the higher the granulator temperature the higher the ammonia losses, so it does depend to some extent on the products. But in principle we screen in two stages as mentioned keeping the recycle temperature pretty high.

Q - In solid route case, how is the optimum pH achieved to obtain maximum product size granulation?

A - This is done by fine adjustments of the granulator conditions of the various liquid feeds and the actual figure of pH is fixed based on experience. We generally aim for around about 6-6.2 in the granulator but this has to be adjusted a little in practice, depending on the grade of the MAP and the actual conditions.

Q - Mr. P. ORPHANIDES, PFI, Greece

Size of granulator?  
Size of main dryer?  
Size of screens?  
Forms of ammonia sparger?

A - The granulator is 3.6 m in diameter and 7.6 m long.

The dryer is 4.2 m diameter and 36 m long.

I do not remember the actual area of the screens but we had a total of 6 screens on the plant, 4 screens in the hot screening system and 2 in the cold screening.

The internal parts are similar to those found in any other plant.

Q - Mr. S.K. MUKHERJEE, FAI, India

With solid feed, what has been the experience with the homogeneity of the finished product granules?

A - With any solids agglomeration process the raw materials are stuck together in the granule and so they do not have the product form that you would get with a slurry route. In other words it is not an onion skin type of granulation. But the properties are good, the handling properties are good, storage is good, very little caking. It is a procedure that we have used very widely and it is perfectly satisfactory.

Q - With urea grades, what is the hardness of the granules (crushing strength) with solid feed relative to similar formulas with urea through slurry route?

A - We have not made much product at all by the slurry route incorporating urea on this plant. In principle the hardness with the slurry route product will be somewhat higher, by the scales that we use probably about double the hardness.

TA/84/5 An improved process for the production of calcined North Carolina phosphate rock by K.L. Parks, Agrico Chemical Company, USA, H. Beisswenger, Lurgi GmbH, Germany & H. Barner, Lurgi Corporation, USA (Rapporteur S. Swanström, Kemira Oy, Finland)

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

Was calcination the only technoeconomic process route chosen and tried for North Carolina rock?

A - Yes

Q - What is the relative total energy consumption for single stage and the two-stage calcining processes?

A - When using the same starting material and calcining to a fixed final temperature, the energy differences are a function of heat recovery. Therefore, the processing heat requirements are identical.

Q - Mr. M. BARLOY, Office Togolais des Phosphates, Togo

What was the main target of your calcination: to produce a light green SPA, or others?

A - Acid color was never a consideration. The main objective was to produce a calcine that could be used to produce phosphoric acid at a filter production rate and recovery level equivalent to Moroccan 70/72 BPL rock.

Q - Have you produced SPA and what was the color of the acid?

A - We have not produced SPA. At 54% P2O5 the acid is green.

Q - Is it possible to get a green acid when using coal as a fuel?

A - Yes, it is.

Q - What is the maximum value of organic carbon for getting a green acid?

A - Filter acid (28% P2O5) contains about 25 mg C per liter of acid and is light green in color.

Q - What are the costs of natural gas and coal in the United States?

A - Natural gas prices vary from less than to more than twice the price of coal on a BTU basis. As gas cannot be used in the proposed process due to the low operating temperature the relative cost of gas is not pertinent.

Q - Mr. D.W. LEYSHON, Jacobs Engineering, USA

Can the North Carolina rock be treated without calcination and, if so, what are the advantages to using calcined rock which requires extra cost, assuming a green acid is not required?

A - Uncalcined North Carolina rock can be processed into phosphoric acid provided the phosphoric acid plant is capable of handling the voluminous and stable foam (foam control reagents can do this at a cost, if available) and the filter design is such that insoluble and non-dispersed organics can be prevented from blinding the filter cake surface or filter media.

In the absence of such a plant capability, calcined rock is more compatible with existing facilities.

Fuel costs for calcining are not appreciably higher than fuel costs for drying and the savings in reduced foam control reagent use, transportation of a higher grade rock (70 BPL vs. 66 BPL) and plant modifications are positive vs. uncalcined rock use at a non-mine site.

Production rates may be higher with calcined rock which is also a plus for calcining.

Q - The performance of the rock during acidulation is stated for a ground rock. How much improvement in gypsum filtrability is due

to grinding or, put another way, what is the effect of grinding? Normally, North Carolina rock is treated as unground rock.

A - The main reason for using ground rock in the pilot scale tests was to reduce IPA losses and to effect better sulfate control when evaluating test calcines during the development of the calcining process. As the products improved in performance the evaluation method was not changed.

We have recently conducted tests with unground rock and note a very uniform performance. The IPA losses are higher for unground rock and we attribute much of this to the configuration and limitations of our test reactor.

Q - What is the oxidation-reduction potential during acidulation of the rock? This is perhaps the best indication of relative corrosion.

A - We routinely monitor the Fe(II) content of No. 1 filtrate during acidulation runs. Due to the very uniform composition of N.C. rock we can estimate the Fe(III)/Fe(II) ratio in the slurry by simply titrating the FeII and calculating the ratio which should be above 0.9 and preferably above 1.0.

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

What is the P2O5 efficiency in production of phosphoric acid from calcined N.C. rock?

A - Reaction and filtration losses in our test reactor are less than 3% for ground rock and less than 4.5% for unground rock.

Q - What would be the overall economics of use of North Carolina rock for phosacid production after calcination?

A - The overall economics of the use of North Carolina calcined rock for producing phosphoric acid are as good as any other commonly available rock source, and more favourable than many. Pilot plant tests on this rock indicate recovery, filtration rate, other operating cost factors such as no defoamer usage and high reactivity without grinding, as well as the quality of both phosphoric acid and gypsum produced from it to be as good as the better quality rocks now being mined. Although relatively high in its sulfuric acid requirement compared to some, it is comparable in this respect to a great many, including Moroccan rock.

Since the income of any particular producing plant depends on its individual configuration and location, specific overall economics are only relative, but our forecast shows North Carolina rock to be as competitive as any.

Q - If calcine is done at pit-head what would the cost of calcined rock be in relation to its recoverable P2O5?

A - The cost of calcined North Carolina rock via the process we have developed and described in this paper is expected to be competitive on a recoverable P2O5 basis for any conventional phosphate fertilizer production.



Q - Mr. S. ORMBERG, Norsk Hydro, Norway

As North Carolina could be an important supplier of phosphate rock in the future, it would be of interest to know if it is possible to reduce the rather high cadmium content, for instance by calcining at a higher temperature in the second step. For some fertilizer processes the surface area and the filtration rate are of less importance.

A - There is always the possibility that a method can be developed to reduce the cadmium content of North Carolina rock and we have some preliminary indications that this might be accomplished.

The use of a high temperature process such as used for Nauru rock is not thought to be practical as North Carolina rock has a softening temperature of about 775°C.

Your question defines an area for further development work.

Q - Mr. K. BELL, ICI PLC, United Kingdom

Can you indicate whether the fluid bed cooler is a deep bed or shallow bed and describe the heat exchange internals for cooling water and gas preheating.

A - The calciner cooler is a multi-chambered shallow bed fluid bed cooler. Fluidising air in the cooler is heated up, dedusted and then introduced as secondary combustion air into the first-stage furnace.

Primary combustion air for both CFB furnaces is heated in separate coils immersed in the cooler's chambers. Coil elements are constructed of heat-resistant steel. The cooler shell is refractory lined.

Process water for the beneficiation plant is heated in an additional separate casing on the cold end of the cooler.

Q - Mr. K. DALY, Compagnie des Phosphates de Gafsa, Tunisia

Effect of the residence time and the temperature on the specific surface, size distribution, the P<sub>2</sub>O<sub>4</sub>, CaO and MgO contents and the rate of decarbonization.

A - The loss of specific surface and inorganic C(CO<sub>2</sub>) is quite rapid at the operating temperature. The elemental analysis change from uncalcined to calcined can be calculated from changes in LOI.

Q - Minimum content of organic matter at thermal equilibrium.

A - The total organic matter, as C, in the final product is 0.10 to 0.12 wt.%.

Q - Mr. P. BECKER, COFAZ SA, France

What is the biggest unit capacity you would be able to build today? Calcined capacity.

A - If this plant is designed to operate and produce 150 mt/h and this for the moment seems to be a feasible unit, we have not reached the upper limit.

TA/84/6 Effects of impurities on production of diammonium phosphate by M.M. HANDLEY, IM & C, USA (Rapporteur S. Swanström, Kemira Oy, Finland)

Q - Mr. M. MIYAMOTO, Nissan Chemical Industries, Japan

In table 1, do you have an analysis of sodium oxide Na<sub>2</sub>O?

A - The baseline acid contained 0.07% Na<sub>2</sub>O.

Q - Mr. K.L. PARKS, Agrico, USA

How is the DAP moisture determined and were the samples ground or unground?

A - The samples were determined immediately upon catching them from the test runs, they were unground samples.

Q - Mr. N. HUMMADI, Jordan Fertilizer Industry, Jordan

What is the water soluble P<sub>2</sub>O<sub>5</sub> in the final product (in % of total)?

A - It varied somewhat on each test effort.

Q - What was the F content in the DAP produced, any limit of F in DAP imposed by regulations in USA?

A - There are no regulations on the amount of F in DAP. The DAP reported stoichiometrically to the product.

Q - Any foaming problems in the preneutralizer?

A - We had no foaming problems in the preneutralizer, in the scrubber there were some.

Q - Mr. M. BARLOY, Office Togolais des Phosphates, Togo

What is in your opinion the influence, if any, of Al, Fe and Mg on:

- the plant output, related to the granulation rate
- the caking tendency in the bulk storage.

A - My conclusions are that each of these chemicals basically has effect but on a molecular weight basis they are essentially equal. I would expect from our tests on the high MgO product that at some percentage of MgO we would start having caking problems with final product upon storage. However it is probably higher than the MgO we reached in our tests.

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

Hemihydrate process produces phosphoric acid with much lower aluminium level than concentrated dihydrate process acid. Would you speculate on the effect on DAP production using hemihydrate acid?

A - I do not think I can speculate on that, I do not know enough about the acid, I have never run DAP from acid produced by the hemi process.

Q - Mr. K. DALY, Compagnie des Phosphates de Gafsa, Tunisia

Do the impurities  $Fe_2O_3$ ,  $Al_2O_3$  and  $MgO$  have an influence on the particle size of the product coming out of the granulator?

A - Yes. In all cases with all the test efforts we ran with all three impurities, we had granulation problems all the time: I think we solved probably the most effect on granulation of  $MgO$ , next with iron and least with aluminium.

Q - Have you tested the effect on the recycle rate?

A - No. The idea was originally to test a chemical, so we tried to maintain constant plant effort all throughout.

Q - Mr. Y. COTONEA, CdF Chimie AZF, France, Mr. G.H.M. CALIS, UKF, Netherlands

Could you confirm that the viscosity of the slurry is better with the high content of magnesium?

A - With the high magnesium runs we had lower viscosity than we did with high aluminium and high iron. But I did not put this on a mole basis, so I am not sure that I can confirm that.

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

In the high Al acid tests no more ammonia was added to the granulator than in the other tests. Could not nitrogen grade have been made at the expense of a little higher ammonia loss in the scrubber?

A - No, we actually ran an extra test running much more stoichiometric, trying to get much more ammonia into the preneutralizer and we were unable to. We also overammoniated the granulator. No success.

Q - Mr. J.A. BENES, Uhde GmbH, Germany

Were you, as a result of the tests described here, able to reduce the citrate-insoluble P205 in DAP product from your new plant in New Wales? If yes, how did you do it?

A - The main thing we found out which reinforced the work we had done was that the temperature and the retention time were the two critical factors along with the fluorine levels, if you have

control of that in the preneutralizer. By reducing retention time and holding temperatures as low as possible you still keep the fluidity, we were able to reduce the CI's.

Q - Mr. J.C. ABRAHAMSE, UKF, Netherlands

Your tests were carried out with magnesium fluosilicate and magnesium sulfate. Did you notice an effect from silicates on production and on product's properties?

A - No we did not. As to the runs that we repeated with magnesium sulfate versus the fluosilicate, we did not see a great deal of difference.

Q - Mr. P. BECKER, COFAZ SA, France

Do you have an explanation why aluminium reduces the granule's porosity?

A - I do not have. I am sure it has to do with the viscosity and the fact that we were really making a MAP along with the DAP with the aluminium runs.

2ND WORKING SESSION

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

b) Please give more details about the spray nozzles in the fluidised bed. How do they differ from NSM process?

A - a) There is no theoretical problem for granulating a NPK slurry. Hot air must be used. Lab tests have proven it possible. But some caking is likely to happen, mainly with fertilizers containing calcium sulphate. We have not experimented the process long enough to have a clear opinion.

b) Spray nozzles are very simple and inexpensive. No air addition. We have been using "Lechler" or "Spray System" nozzles.

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

For urea granulation, what percentage of material would be between 2 and 4 mm in the product, when operating at a recycle rate between 2.2-2.5?

A - The percentage of material between 2 and 4 mm can be adjusted in a wide range by modifying the operating conditions (e.g.: screen cloth).

As recycle is needed for bed cooling no special effort was made to increase the onsize product amount.

Typical values are:

4 mm:	5%
2 to 4 mm:	35 to 55%
2 mm:	60 to 40%

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

What is the essential difference between Perlomatic process and other fluidised bed granulation techniques? Has there been any necessity felt in your process for the addition of formaldehyde to improve the storage properties of urea granules and to reduce dust formation? Comparative steam and power consumption figures per tonne of urea production may be given.

A - The essential difference between Perlomatic and other fluidised bed process is that:

- there is no perforated plate to support the bed
- there is only one spray nozzle.

There is no necessity to add formaldehyde for the granulation step itself (no dust formation). But formaldehyde is needed to improve the storage properties when a 98% slurry is sprayed.

The steam consumption per ton of urea is about 50 kg. This value may vary according to the dedusting system (bags or washing tower).

The power consumption for the granulation step itself is about 22 kwh per ton. Some power will be needed for the dedusting step; depending on the process it may vary from 8 to 15 kwh/ton. The total power consumption should then be between 30 and 35 kwh/ton.

Q - Mr. R. MONALDI, Fertimont SpA, Italy

In your paper it is stated that the perlomatic itself is 1.8 m diameter and the rate of granulated urea is 5 t/h coming from the slurry with water content 1-2%. This production rate seems low for a spouted bed having the above size as the operating conditions demonstrate.

In fact the amount of air, 8000 kg/t of product, is bigger than in all other processes (NSM, MTC, TVA, Fertimont). The increase of the water in the slurry, for example up to 5%, could give an increase in production of about 40%; but that increment, in accordance with our experience, should give notable difficulty in the spouted bed during the drying up of the urea granules because the amount of air passing through the annulus is too small.

I should like to know how you think to solve this problem.

A - The given value of 1.8 m refers to the body of the Perlomatic, not to the cone which is the working item. The upper diameter of the cone is about 0.9 m and the lower diameter about 0.2 m.

The amount of air (8000 kg/t) may vary widely depending on the air temperature at the Perlomatic inlet, and at the cooler inlet. It depends too on the temperature of the urea after cooling and the recycle rate. Values of 7000 kg/t could be reached.

The increase of water in the slurry gives no difficulty at all in the spouted bed with slurry containing 3.5 to 4% water. Problems arise with the residual water content in the urea granules, which gives poor storage properties when too high (above 0.3%) regardless of the formaldehyde amount. It is clear that, in order to work with a high water content, it will be necessary to improve the spray nozzle operating conditions.

Q - Mr. S.K. MUKHERJEE, FAI, India

a) Crushing strength given in the paper is 0.38 kg/mm<sup>2</sup> for d = 2 mm. Is this correct?

b) Is the screening of urea effective when operating under conditions of between 72-76% relative humidity?

A - a) The crushing strength is normally 0.5 to 0.7 kg/mm<sup>2</sup> for d = 2 mm.

b) The screening of urea was made on a product at a temperature of about 50°C; under our climatic conditions the relative humidity is normally 55% to 65% and no special problem arose. If the R.H. is higher (75-80%) some problems may happen if the urea is not warm enough.



TA/84/9 Experimentation on the use of urea-ammonium sulphate (UAS) liquid fertilizer in Kuwait, by M.F. Abd El-Hameed, Petrochemical Industries Company, Kuwait (Rapporteur N.D. Ward, Norsk Hydro Fertilizers Ltd, United Kingdom)

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

Same results could possibly be achieved by balanced application of urea and SSP. Has the economics of application of fertilizers in various forms been worked out and how does it compare with UAS solution?

- A - 1. Table 1 shows the laboratory experiments to obtain a solution with the maximum nitrogen content (that is for transportation reasons) and the lowest solidification temperature to prevent the solidification of the solution during winter time.
2. Solution n° 4 (table 1) is the most suitable one according to our needs.
3. We could not fix the sulphate contents and change the urea content of the solution, because we do not know the behaviour of the mixture and the urea itself has very high solidification temperature as shown from curve n° 1, so we used different concentrations of ammonium sulphate and urea until we get table 1 and from which we get curve n° 2.

Q - Mr. G.H.M. CALIS, UKF, Netherlands

In constructing figure 2, solutions with different  $(\text{NH}_4)_2\text{SO}_4$  content have been used (see table 1). We believe that only solutions with the same sulphate content should be compared. Can you comment on that? In table 2, you compare NPK with NS fertilizer. Conclusions based on this comparison could be doubtful. Would it not be possibly better to compare UAS with straight N or NS fertilizer?

Q - Mr. A. EL-HOUARI, OCP, Morocco

For field trials, was UAS used alone or with the addition of equivalent amounts of P205 and K2O contained in NPK fertilizers used as references. If so, in which form were P205 and K2O added. If not, are the results obtained not affected by the absence of these two elements?

A - The (UAS) was compared against NPK (12+12+17+2) for the following:

- a) The NPK (12+12+17+2) is the most commonly used fertilizer in Kuwait.
- b) The comparison was only for the nitrogen and sulphur source, so the NPK were used according to the normal methods of agriculture with the quantity required for each plant.

For the (UAS) we used a dilute solution containing the same nitrogen % (12%) as the NPK for drip irrigation, the other

elements (P&K) were mixed with the soil with the same percent as they are found in the NPK, so as to make all the other factors (except the nitrogen and sulphur sources) stable.

Q - Mr. C.H.M. VINKE, Windmill Holland, Netherlands

I have a question on distribution and storage. Can you indicate the distance between the point of production and the point of application of this fertilizer?

A - Our factories are very near to the agricultural area in Kuwait, the distance is 5-10 km. We use, in our field scale experiments, tanks to transport this solution to the agricultural area. The solution is still only produced and used on an experimental scale. Until now we have not produced it on a large scale.

Q - Do you have any idea of the maximum distance you will be able to cover by this method of fertilizer supply.

A - I think we can transport it for any distance because we already transfer ammonia solution from Kuwait to the outside world which requires more precautions than UAS solution.

TA/84/10 Prilling or granulation of urea - Some comparative criteria of recently developed granulation technologies and improved air-prilling by F. Kars, Stamicarbon, Netherlands (Rapporteur N.D. Ward, Norsk Hydro Fertilizers Ltd, United Kingdom)

Q - Mr. K. BELL, ICI PLC, United Kingdom

In your pilot-plant dust scrubbing trials which types of wet scrubbers did you test and which gave the highest removal efficiency?

A - System A: dynamic jet scrubber, developed by Stamicarbon. Use is made of a waterjet and the condensation effect of low pressure steam.

System B: Multex multiventuri dust collector, provided with polypropylene internals.

Q - Mr. S. ORMBERG, Norsk Hydro, Norway

a) Your paper gives a detailed description of all recent urea granulation processes, but not of your "special prilling design of the eighties".

Could you please describe briefly the highlights of this new prilling technique and could you also provide a list of industrial references among the 150 towers presently in operation?

b) How is the extra investment of nearly one million US\$, shown in fig. 10, justified by additional equipment items?

A - a) The rotating prilling bucket design was improved to narrow the size range of the product and subsequently enabled us to increase the average prill diameter which also depends on tower height and/or the possibility to install an additional cooler. This design was incorporated in some 20-25 towers. In the mid seventies C.N.C. in Augusta (Ga), USA, requested us to design a tower for an average prill diameter of some 2.2 mm in order to meet the bulkblenders' requirements. After that Stamicarbon never received such a request, though in some prilling towers the average prill diameter is somewhat larger than it used to be in the past.

b) The difference of nearly one million US\$ between a prilling system for standard grade and coarse grade product is caused by additional tower height and/or an additional product cooler.

Q - Mr. S.K. MUKHERJEE, FAI, India

What are the special features of "prilling" - if any - against conventional Stamicarbon "prilling process" (direct prilling) on which many commercial plants have been built and with which we are familiar?

A - In both cases direct air prilling is used, however the present rotating bucket design is improved (re my answer to Mr. Ormberg's question): up to some 0.2% formaldehyde is used, seeding is applied to prevent subcooling of the droplets, the prill capacity per m<sup>2</sup> crosssectional tower area is increased, air velocities are increased, the product temperature exit tower and/or cooler is decreased to some 50-60°C. In some plants dust abatement systems are installed.

Q - Mr. A. MARCHESI, ANIC SpA, Italy

In your paper and in your speech you clearly exposed the advantages and disadvantages of each process and a definitive choice between prills and granules cannot be made. I believe we must give the market what the market really needs and this is the question: What product do you think the market will demand in the near future?

A - The paper identifies some selection criteria and my recommendation is to use these criteria in making a choice between (Stamicarbon) prilling and granulation. However, I also strongly advise to obtain the most recent information (may be partly as guarantees) from the process licensors or from their contractors with respect to these criteria. Local conditions etc. may have an important impact on e.g. investment costs, on stream time, etc... At the end the market demand will determine the product which has to be produced in the plant and I cannot predict that market demand. As my personal opinion I expect a slowly increasing demand for coarse agricultural grade product depending upon the demand for bulkblending and recognising that, for wider spreading of the fertilizers, a large product size is more attractive. I do not expect a strong market growth for feed grade urea, forestry grade urea or super granules.

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

The cost advantage of some \$ 4 per tonne for prilled urea (vs. granulated urea). Since agronomic tests show even a 10% improvement in the efficiency of nitrogen uptake in actual field application, granulated urea might yet be the answer for the future. Has the author any comments?

A - I have collected some information on improvement of nitrogen efficiency of urea when used for wetland rice. I understand that three possibilities exist:

- a. deep placement of urea
- b. using a coated urea, such as sulphur coated urea (SCU)
- c. using inhibitors.

A - a) Tests have been made with supergranules (e.g. IFDC) by placing one supergranule some 10 cm in the soil in between 4 rice plants, and usually the nitrogen efficiency was found to improve. Unfortunately supergranules are more expensive, labour costs for deep placement are high and to obtain farmers acceptance is a problem. Almost the same result was obtained with deep placed urea prills which are cheaper but share the other problems with supergranules. Obviously the secret is deep placement. Though applicators for deep placement of supergranules and prills have been developed, they still seem to be far from ideal.

b) With spread SCU the same nitrogen efficiency improvement was obtained. However again SCU is very expensive. It was hoped that the product costs could be reduced by coating larger granules with less sulphur, but so far only marginal improvements have been found.

c) In many laboratories work is done on inhibitors, however to the best of my knowledge so far without tremendous results. On the other hand when an attractive inhibitor is found, I expect that it can be used for every particle size.

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

Your paper concludes that prilling requires the lowest investments and yields product at the lowest operating and maintenance cost.

a. Can you explain why the latest plants have been based mainly on granulation and old prilling plants are converted to granulation?

b. Have you tested the bulk-blending behaviour of the prilled product coming out from the patented process in comparison with granular products in order to compare the segregation phenomenas?

A - a. I am only aware of one -non Stamicarbon - prilling plant in Indonesia where, because of poor product quality, a TVA pan granulator has been installed. I have also observed an increasing demand for granulation in new projects, though also

prilling is still requested. I like to emphasize that I compared Stamicarbon prilling with granulation and not other prilling technologies.

b. CNC Augusta (Ga), USA sells its coarse agricultural grade product to US bulkblenders and segregation has been found neither in the blends nor during spreading in the field. As long as the average particle diameters and size ranges of the blending materials match, no segregation will occur (re table 4 and TVA studies).

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

Does power consumption in table 2 include dust abatement system B? If not what would be the total kWh/tonne for air prilling?

A - Power consumption figures in table 2 include dust abatement system B.

Q - Mr. E. AASUM, Norsk Hydro, Norway

a. Page 14 refers to a prilling tower air exit temperature of 80°C. Is any plant operating at this temperature level and, if not, what considerations have to be taken in design and operation?

b. What would a resulting prilling tower height be for standard agricultural grade?

c. Compared to your patent literature in table 6 you have reduced the amount of air to almost 1/3. How is this reduction obtained?

A - a. In our prilling tower design we limit ourselves to max 80°C exit air temperature and a number of towers are operating at this temperature.

b. The tower height for standard agricultural grade urea with 25°C inlet air temperature, 80°C exit air temperature and a design product temperature of 50°C is about 50 m.

c. The air requirements in table 6 are based on the above design data; it indicates that we optimized our tower design quite considerably.

Q - Mr. F. MOSTAD, Supra, Sweden

Please clarify some figures in table 10.

a. Why is the steam consumption of the TVA evaporative drum granulation process lower than the base case although it starts from a highly concentrated melt (99% - see table 9) similar to the MT spouted bed process?

b. Why is ammonia-evaporation charged to the NSM granulation process, while air drying is not required for this granulation process?

c. Has Stamicarbon established the investment figures for the granulation processes through theoretical cost evaluation or by feed back of effective construction costs?

A - a. In the base case the urea melt is concentrated to 99.7%, for TVA 99% is sufficient. For 99.7% concentration a more sophisticated vacuum system is required in the evaporation steps, which is not required for 99% concentration or lower. It should further be recognized that the data in table 10 are calculated for the integrated situation, not only taking into account feedstock concentration but also dust recycled as a solution.

b. We have not included air conditioning for NSM or others. The ammonia is present in the feedstock. Though a low concentrated feedstock (95% for NSM) reduces the steam consumption as compared to feedstock concentrations of 99% or higher, unfortunately it contains more ammonia than the more concentrated feedstocks. This ammonia is assumed to be lost and not recovered in the scrubbing systems, which can hardly be expected. At the cost of additional investments and utilities the ammonia figure can be lowered, if desired.

c. Our investment figures were estimated by DSM's Estimating Department and later correlated with the estimates made by an engineering contractor.

Q - Mr. K.L. PARKS, Agrico Chemical Company, USA

a. What about fines and lumps? Surely there must be a way to recover this material.

b. What is the influence of the "seed particles" stream on biuret formation, i.e. what is the biuret content of the product with and without "seed particles"?

c. Prilling towers have a bad reputation as dust generators. Can you give comparative results for your process vs prior prilling tower dust emissions?

d. Can the positive effects of formaldehyde, relative to caking, be ignored?

e. Were biuret values calculated or analytically determined?

f. Our experience, prior to 1975, regarding maintenance and operation (bucket plugging, wall coating, dislodged chunks of material falling to tower base, difficulty of rate change, etc...) does not coincide with the author's observations. What operational or design changes have been made to decrease these old problems?

A - a. Very seldom a screening system is installed after the tower. As in most storage sheds, usually screening is installed in the reclaiming system from the storage before shipment, where about 2% of the shipped capacity are recovered and dissolved when feasible in a urea solution before recycling to the evaporation unit.

- b. Seed particles do not affect the biuret content of the product.
- c. Dust emissions from our prilling towers have been measured also in the past and a figure of 1-1.5 kg/M.T. urea was also found.
- d. Formaldehyde certainly has a positive effect relative to caking, and I never ignored that. The amount of formaldehyde required is however depending on the shaping technology, moisture content of final product etc... For prills 0.2% formaldehyde is sufficient.
- e. Biuret formation depends upon liquid composition and exposure time at certain temperatures. Because biuret formation in granulation is considered to be negligible, we could partly calculate and determine the biuret contents taking into account for instance the dust recycle from the shaping facility.
- f. As far as I know Agrico does not operate a Stamicarbon prilling tower and therefore is referring to difficulties experienced with another prilling technology. I can only discuss and comment our prilling technology.

Q - Mr. T.S. HARIHARAN, Fertil, Abu Dhabi

Locations where high ambient temperatures exist, which is suitable in: prilling or granulation?

A - Both for prilling and granulation a high ambient air temperature makes it difficult to reach a sufficiently low final product temperature unless the final product is cooled in a separate cooler with conditioned air. I have not compared the shaping technologies for high ambient air temperatures, though the paper offers the possibility to calculate air requirements for high ambient air temperatures etc... Offhand I do not anticipate that I have to change my conclusions to the contrary.

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

While you have emphasized lower cost it would be of special interest to estimate the increased value of high quality product. This may be more difficult in developing countries where there is no free market. Farmers have only the choice of using the poor quality "Standard agricultural grade" or nothing. In hot, humid climates there has been much difficulty with losses, and with fine damp, caked material that is either unusable, or difficult to spread evenly. Have the authors tried to estimate the value of the various products?

A - The paper compares product characteristics of Stamicarbon prills with granules from certain granulation technologies. I am very much aware that prills made by some other technologies are inferior to ours. We have been consulted recently by different producers on this problem, and possibly we can at least partly correct their problems. I have tried to fight the bad reputation of prills, I have tried to supply information on our prilled

product, and I am certain that this product certainly cannot be considered as "poor quality" standard agricultural grade urea.

Q - Mr. E. HOLTE, Norsk Hydro, Norway

On page 8 the author states that only TVA Fallen Curtain has produced Urea Supergranules (USG).

Norsk Hydro has since 1979-80 supplied most of the USG utilized in the research and testing done both by IFDC, IRRI and FAO. The USG is produced in the NH-High Temperature pan granulation process. The recycle ratio in this process is about 3:1, not 7:1.

A - I am aware that Norsk Hydro has produced supergranules in their pan granulator; however, I made it clear in the beginning which processes I would compare and Norsk Hydro was not part of that list. Yes, I apologize for not having mentioned your ability to make supergranules.

TA/84/11 Compacted fertilizers by A. Seixas, J.D. Ribeiro Marçal & J. Correia, SAPEC, Portugal (Rapporteur N.D. Ward, Norsk Hydro Fertilizers Ltd, United Kingdom)

Q - Mr. M. BARLOY, Office Togolais des Phosphates, Togo

a. Concerning the addition of SSP to the formulation:

- What are the water and free P<sub>2</sub>O<sub>5</sub> contents of the SSP?
- What is the curing time requested before SSP is used?
- What is the maximum amount which can be added in the formulation?

b. What is the maximum permissible moisture content of the compacted materials and where is the limit: compacter clogging or quality of the finished product?

c. Is natural moisture evaporation significant during manufacture and can it be enhanced?

d. Can you indicate what would be, at present US \$ rate, the relative investment cost of a 20t/hr compaction unit as compared to a conventional granulation unit of the same capacity using the same raw materials?

A - a. When it is produced, our SSP contains 10% H<sub>2</sub>O and 3-4% free acidity, expressed as P<sub>2</sub>O<sub>5</sub>.

An increase in the curing time of SSP is favourable to compaction. Minimum time: 10 days.

To obtain a satisfactory output in t/hr, 10-20% SSP are not exceeded.

b. Since we use a mixture of raw materials, it is important to know the humidity of the mixture. The quality of the finished product limits the humidity.

c. Since the process involves a high recycle rate, the



temperature of the product increases and the humidity decreases.

d. The facilities described (10 t/hr) cost \$ 1 million in 1980.

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

How do compacted NPK fertilizers compare with granulated products in terms of uniformity in composition, particle to particle?

A - In view of the successive operations of compaction, grinding and screening, and the high recycle rate, the particles produced in a conventional granulation plant and in a compaction plant are similar in their composition.

Q - Mr. Y. COTONEA, CdF Chimie, France and Mr. P. ORPHANIDES, PFI, Greece

Can you indicate the raw materials used in the production of 20-20-20?

A - To manufacture 20-20-20 we use potassium chloride (0-0-60), ammonium phosphate (20-50-0) and urea (46-0-0).

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

a. How much dust becomes detached from finished granules during handling, packaging and transport?

b. 1-1.5% moisture seems high for urea containing fertilizers. Please give experience of storage life and characteristics.

A - a. The finished product contains less than 1% of products below 1 mm. We carried our abrasion tests on granular and compacted products and the results were similar (0.5-1%).

b. The humidity of the finished product varies according to the composition of the mixture. When we add urea to the mixture before compaction, the humidity of the finished product is below 1%.

Q - Mr. M. HANDLEY, IM & C, USA

What is your experience with roll life on NPK's?

A - The rolls we use have a life time of about 8000 hrs, if we carry out intermediate fillings.

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands

I presume it is necessary to coat urea-containing granules to prevent caking in storage. Can it be done effectively bearing in mind that the granules are far from spherical?

A - The finished product is coated in a drum with anticaking and antidust agents.

Q - Mr. J. LE PAGE, ICS, Senegal

- a. Did you try to compact powder MAP either alone or mixed? of which origin?
- b. How does it behave?
- c. What is the maximum permissible moisture content?

A - a. We did not compact MAP alone, but mixed.

- b. We used Tunisian MAP. It has a good behaviour, although we had dedusting problems.
- c. The moisture content is about 2%.

Q - Mr. N. LOUIZOS, SAHPEC, Greece

- a. What is max. urea percentage used and corresponding production capacity?
- b. What are limitations regarding size distribution of granules i.e. is it better to use the same size, or different size of granules?
- c. How often is it necessary to repair or change the compacting cylinders and could this be done in your own installation?

A - a. The amount of urea used depends on the nature of the mixture. So far we did not exceed 30%.

- b. In the mixture to be compacted we can use materials of different particle size.
- c. The finished product contains 90% of product between 2 and 4 mm. The rolls of the compacter have a life time of 8000 hrs, if there is an intermediate filling.

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

Could the author give more details of the "special-difficulties" experienced with SSP?

A - Since SSP has important plasticity properties, it raises difficulties. We have tried to compact SSP alone and we incorporate it in some fertilizers we produce.

Q - Mr. R. SCHOEMAKER, UKF, Netherlands

Have you got any experiences on the compaction of urea-ammonium sulphate mixtures, such as the 41-0-0-5, as was mentioned by Mr. Bizzotto in his presentation?

Is it necessary to crush urea before the compaction stage?

A - We do not grind urea before compaction.

So far we have not tried to compact mixtures of urea and ammonium sulphate to produce 41-0-0-5.

3RD WORKING SESSION

TA/84/12 Simultaneous production of merchant grade calcium sulphate and high strength phosphoric acid by A. DAVISTER & F. THIRION, Prayon-Rupel, Belgium (Rapporteur E. Holte, Norsk Hydro, Norway)

Q - Mr. J.C. ABRAHAMSE, UKF, Netherlands

Can you give information about cadmium distribution over the acid and calcium sulphate?

A - During the tests we concentrated on making high grade phosphoric acid and calcium sulphate and, although we monitored the impurities in the products, we do not so far have data on the distribution of cadmium.

Q - Mr. K.L. PARKS, Agrico Chemical Company, USA

In table 2 in the paper giving values for the test with Kola/Bu Craa blend you give an increase in the Fe<sub>2</sub>O<sub>3</sub>/P<sub>2</sub>O<sub>5</sub> ratio from the rock of 0.0087 to 0.0108 in the acid. Does this suggest severe corrosion?

A - We utilize stainless steel in our test equipment and the corrosion analysis carried out on the test equipment indicates no corrosion whatsoever.

Q - Mr. R. SMITH, Duetag, France

Could you advise further the uses for the calcium sulphate produced by this process?

A - For upgrading of Ca sulphate, we have already the Central Prayon process. Virtually for all the production of Ca sulphate from that particular process in our own plants, and at the customers' location in Sweden, Japan and Africa, upgrading is done for the production of products which are currently obtained from natural gypsum in the production of  $\beta$ -hemihydrate. If we look into the results, we would find little differences in the quality of  $\beta$ -III-hemihydrate, whether it be phosphogypsum or natural gypsum. We can also use it as cement retarder or as Ca supplier raw material in the manufacture of cement clinker as is done in some countries, like for instance South Africa.

We have developed the PH3 Process because we wanted to have concentrated acid, but we also wanted to retain this possibility of upgrading Ca sulphate. Because of the mechanical properties of the  $\alpha$ -hemihydrate, we have found new outlets. Introducing a controlled proportion of II-anhydrite is even more interesting, because II-anhydrite does not need to be produced by high temperature thermal treatment, as is done in plaster works. It is present in the crystals and requires no drying energy. That way we could, of course, replace certain production of plaster by traditional means and also cement in terms of strength and in terms of material design.

Fillers and whatever you might have in mind will be another market.

Q - Mr. K.L. PARKS, Agrico Chemical Company, USA

What results would you expect with Florida 68 BPL rock?

Does this process reduce the radioactivity of the gypsum, i.e. the radium content?

A - We have just completed a test of six weeks on Florida rock. I do not have any electronic microscope presentations and I do not have the results with me, but I can tell you that with the Florida grade 70/72 BPL, we had 46% P2O5 acid obtained with yields of about 99%. The operating factor was just as good as with the Morocco grade and the shape of the crystals was more or less similar, so I can tell you that the grade for the Florida product was easier to achieve than with the Morocco grade, which was pretty good in any case.

During the tests we monitored a certain number of ingredients. Included in these is the monitoring of radium, but the results of these tests are just now under processing.

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

The operating conditions given for the 2nd reaction stage are 18-23% P2O5 and for the 3rd stage 24% P2O5. Thus there can be little recovery of P2O5 at the dihydrate separation. How difficult is this operation? Is it by-passed on the pilot plant?

A - What I can say is that the control of the quality of the final Ca sulphate with  $\alpha$ -hemihydrate or the mixture of  $\alpha$ -hemihydrate and anhydrite II requires essentially that we have a full rehydration in the second phase and this is a must of the process. Separation after the second stage is optional and has nothing to do with the recovery of the characteristics of the products.

Q - Mr. P. MORAILLON, CdF Chimie AZF, France

How can you explain the balance of impurities in the phosphoric acid and Ca sulphate that correspond to it? We altogether find only part of the metal impurities in the phosphate, for instance 82% of Al2O3 in the Kola/Casa grade or 50% Na2O in the Kola/Bu Craa grade according to the figures given in table II.

A - When you have 80% Al2O3 balance when doing experimental work in a pilot plant, it is not that bad. In return, it is pretty obvious that 50% when you do a Na balance is something which is not right. It must be a mistake somewhere. By all accounts, Na in the acid will ultimately be of a slightly higher concentration by the time full equilibrium in the system has been reached.

We have to realize that in a pilot plant, with a 20 kg P2O5/day capacity, there is from a human point of view a maximum you can do. We also have to produce series of samples and samples which in fact require additional purging. At the time of sampling on liquid products at a laboratory level, we can work on the product as

such, but, for reasons of overload, we do it away from the clear liquid. Consequently, we might have precipitation. The sodium might not be present in the liquid phase. That is why you have to do it on the solids rather than on the liquid.

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

In the Ca sulphate handling section III  $\alpha$ -anhydrite seems to be an intermediate between  $\alpha$ -hemihydrate and II-anhydrite, but putting yourself in intermediate conditions, you are going to get a mixture of  $\alpha$ -hemihydrate and II-anhydrite. How can you account for the absence of the  $\alpha$ -hemihydrate III, if put this way?

A - The crystallization phase in the chemistry operations is sophisticated. A lot of literature has been published on this and I think that things are changing pretty fast. So everybody has his own ideas on the issue, and I would say that III-anhydrite is often present. But in doing the analysis and the X-ray diffraction for determination, we cannot make the distinction between  $\alpha$ -hemihydrate and III-anhydrite as the spectrums are superimposed on each other. When working out the product, you will have a lot of III-anhydrite present which will activate the setting times.

Q - Mr. P. BAEKEN, Coppée SA, Belgium

On page 2 on the paper, you say that one can resort to silica, which could be added. Could you say more about this? That particular thing is already patented and covered. Please elaborate.

A - Adding silica or the role played by various foreign matters (impurities) present at various levels in the phosphate rocks used, like organics, silica, alumina and many others have been brought forward many years ago. In the beginning of the 60's, recommendations were drafted on the use of these products.

As a matter of fact, later on (some 20 years back) a very interesting paper by Mr. Gilbert, USA, gave a lengthy description of which role is played by a certain number of those impurities or foreign matters and what effect they can have on the crystallization behaviour of Ca sulphate and also on the transition from one form to another. As a matter of fact, as we go along, we get to know how to make the most of the phenomenon and to use it to maximum advantage.

Q - Mr. M. MIYAMOTO, Nissan Chemical Industries, Japan

On figure 3, there is no outlet of filtrate from the dihydrate section. Where does it go?

A - Separation of dihydrate is an operation which is still optional as shown on our flowsheet. But when we incorporate it, that does not necessarily give any outlet because we have the possibility to recycle.

Answers to questions in writing

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

The P2O5 recovery of 99-99.5% probably excludes mechanical losses from the filters. With three separation stages this must be significant. Can the authors quantify the overall recovery for an industrial plant?

A - Generally, our design includes one closed circuit for process and cooling waters. In this case, the overall recovery is 99 to 99.5% for an industrial plant. The possible losses will be accidental.

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

Since phosphoric acid contains less impurities than a dihydrate free acid, the final hemihydrate will be more impure. Please comment.

The phosphates indicated in table II contain little Na. Have you tried phosphates with a higher Na-content? In that case, does the hemihydrate contain more fluorine?

A - No, the final hemihydrate is purer than with a dihydrate process, because impurities are dissolved at this concentration. The impurities are in the product acid.

Yes, we have treated the Casa phosphate rock 68-70 BPL. There is a relation between Na and F content in solids; but values are less than in a dihydrate process.

Q - Mr. M. MIYAMOTO, Nissan Chemical Industries, Japan

How do you positively proportioned  $\alpha$ -hemihydrate and II-anhydrite in dihydrate hemihydrate conversion stage, by temperature or by H2SO4 concentration therein?

Is it possible to make II-anhydrite free of hemihydrate?

What is the allocation of 98% H2SO4 into the three stages?

A - The concentration of II-anhydrite in hemihydrate and dihydrate conversion stage is controlled by adjusting temperature, P2O5 and H2SO4 concentration in the liquid.

In the wet phosphoric acid processes II-anhydrite is made from hemihydrate and dihydrate conversion. There are some free II-anhydrite crystals in the produced solids.

The distribution of sulfuric acid introduced in the three phases of the process is about:

- hemihydrate attack section	50%	25%
- dihydrate conversion section	25%	25%
- hemihydrate conversion section	25%	-
- II anhydrite conversion section	-	50%

Q - Mr. M. BARLOY, Office Togolais des Phosphates, Togo

Does the first hemihydrate reactor include several compartments?

Does the process described have an economic advantage if the calcium sulfate is not processed? In particular, can you eliminate the third stage?

A - The multicompartimented single tank coupled with the new low level flash cooler organized to ensure separate control on the three flows (through the tank, through the flash cooler and inside each compartment) as well as to minimize the power rating by the use of helicoidal pumps and carefully shaped agitators has now reached full maturity and strength. It is of course one of the main topics of the hemihydrate reaction together with the digestion tank(s) in which the slurry is adjusted and stabilized before filtration.

If the gypsum is not processed, the third stage is of no interest except in case of dry handling to disposal, which is more easily achieved when the process delivers a self drying mixture of  $\alpha$ -hemihydrate and II-anhydrite. But of course who can do the most can do the least and as a fall out of the PH3 process we have developed a full range of hemihydrate processes named PH for Prayon hemihydrate. These are either straight hemihydrate in one stage (PH1) or in two stages (PH12), or hemidihydrate in two stages (PH2).

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

Results are given only for the rock mixtures including Kola-apatite. How does the plant perform on a sedimentary rock alone?

A - During the lecture, we have shown results obtained with commercial Kouribga rocks and indicated figures obtained during a run with commercial Florida rock. Both behaved perfectly well. If our tests were started with Kola apatite containing rock mixtures, this is simply to adapt it to our usual plant feedstock.

Q - Mr. T.J. THOONEN, UKF, Netherlands

Can you give figures in p-Curies/g over the different steps (HH-DH-HH)?

A - Without special treatment the radioactivity is the same over the different stages.

Q - Mr. N. TURKI; SIAPE, Tunisia

Do you add chemicals to prevent formation of dihydrates in the first crystallization?

A - No, but it is necessary to adjust the conditions for hemihydrate crystallization.

TA/84/13 Energy consumption in ammonia production. Influence of external conditions and key process parameters by Ib. Dybkjaer, Haldor Topsoe, Denmark (Rapporteur N.D. Ward, Norsk Hydro Fertilizers Ltd, United Kingdom)

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

It is mentioned that production of sufficient CO<sub>2</sub> for conversion of all the NH<sub>3</sub> to urea will require some additional energy consumption.

I suppose the endeavour should be to produce maximum CO<sub>2</sub> and utilize this in the production of urea even, if need be, by importation of additional NH<sub>3</sub> to consume all the CO<sub>2</sub>. The incremental urea production would perhaps offset the marginal increase in energy consumption when it is apportioned to the total urea production. Your comments please.

A - The problem of production of CO<sub>2</sub> and the match between the CO<sub>2</sub> and the NH<sub>3</sub> in the urea plant can be tackled in different ways. The problem is only relevant when you have a rather clean natural gas. If you run on naphtha or heavy natural gas, the balance between H<sub>2</sub> and C in the feedstock is such that sufficient CO<sub>2</sub> is produced automatically.

If you have dry NG or pure methane, as we have assumed, then it is necessary in some way to increase the production of CO<sub>2</sub>, if it is not possible in the local conditions in which the plant is built to coproduce NH<sub>3</sub> and to export that out of the plant.

It can be done, as you can see from some theoretical equations included in the paper, in two different ways: one is to add an excess of air to the secondary reformer. In this way you will upset the balance between the H<sub>2</sub> and N<sub>2</sub> in the synthesis gas and you will have to spend energy and investment to take the excess N<sub>2</sub> out of the synthesis gas. But you will be able to achieve the balance between NH<sub>3</sub> and CO<sub>2</sub>. It cannot be done in this way without additional energy consumption.

It can also be done, and that is what we have assumed and realized in plants built in various parts of the world, by producing an excess of synthesis gas. This is done simply by having a somewhat larger front end which produces sufficient CO<sub>2</sub> for the conversion of the ammonia. The excess synthesis gas is then taken out before the compression, i.e. after the CO<sub>2</sub> removal, and used as feed in the reformer. The extra energy consumption, by doing it this way, is marginal. In such a case, it does not pay to install a purge gas recovery unit, which is installed to reduce the production of synthesis gas.

Q - Mr. M. SIVONEN, Kemira Oy, Finland

Is the steam/carbon ratio of 2.5 already used continuously in any conventional ammonia plant?

A - No, this steam/carbon ratio is not yet used in any conventional ammonia plant. Plants are under construction where operation at this ratio will be possible. But similar operation conditions are



used in conventional steam reforming plants for other productions, such as H<sub>2</sub>, where the conditions in the primary reformers are very similar to what they are in an ammonia reformer plant. Even lower steam to carbon ratios are used in steam reformers producing oxo-synthesis gas or gas for other purposes.

Q - Mr. D. BANQUY, Foster Wheeler, France

Did you evaluate the effect of the synthesis pressure on the overall investment cost?

A - Yes, we did. It is of course not a simple thing to do. We have studied the effect of the synthesis pressure. Since we found that we could not by analyzing the energy consumption alone find any optimum pressure, we selected it on the basis of other criteria, like:

- Size and cost of equipment
- Reasonable layout of the compressor section
- Reasonable integration of the heat recovery system.

I would like to emphasize that we see no problem whatsoever in supplying a synthesis loop at 200 atm or at 80 atm, as we have the technology for it and we are quite willing to do that. For our own complete process scheme, we have found that a pressure of approx. 140 atm is advantageous when we consider the investment cost and the overall integration of the energy system.

TA/84/14 What ICI's ammonia process does for low tonnage ammonia plants by J.G. Livingstone, ICI PLC, United Kingdom (Rapporteur N.D. Ward, Norsk Hydro Fertilizers Ltd, United Kingdom)

Q - Mr. O. HANSEN, Haldor Topsoe, Denmark

In the paper you refer repeatedly to "conventional processes" and claim e.g. a 20% reduction in overall capital against such processes. Please give the main features of the "conventional process" used as a reference.

A - I think the point here is that the conventional process used for comparison was in fact as stated on the slide. I showed the last plant that ICI designed. What we tried to do in this exercise was what ICI, with ICI's standards and ICI's approaches, would pay for this sort of process as opposed to what it paid for its last process. So the basis for a conventional process was the last ICI designed/built. The N° 4 ammonia plant in Billingham was the basis.

Q - Mr. J. SARLABOUS, CdF Chimie AZF, France

At what time will the start-up of the CIL unit with the ICI AMV process be anticipated? Why such a delay?

A - The CIL plant at Courtright in Ontario is in fact the first with this new process to be operated. Unfortunately, that plant was designed to cater for the American fertilizer market, but, as that market suffered badly in 1982/83, the Canadians decided to slow down the project because it was no point in making ammonia they could not sell. The plant is presently in the precommissioning stage. The demineralization units are being precommissioned. The plant will first produce ammonia in July of 1985. Everything seems now to be on program, the American economy is hopefully going in the right direction and the plant will produce ammonia.

Q - Dr. S.K. MUKHERJEE, FAI, India

What has been the longest continuous run in a commercial plant on the low pressure (68-75 atm) ammonia synthesis catalyst?

A - The catalyst is perhaps one thing I did not clarify in the presentation. One constraint put on the designers on this process with ICI was that all the new steps in the process had to be proven. We do not like prototypes in ICI. We have suffered too many times from prototypes. All must be proven in one of our plants somewhere in the world, before it goes into the new process.

The low pressure synthesis catalyst, that is mentioned here as the major step forward in the new process, has now been operating just over 4-1/2 years in the N° 1 ammonia plant in Billingham. I am happy to report that after 4-1/2 years, the activity of the catalyst is still well above initial activity of our conventional catalyst at start of life. So it is quite a dramatic step forward in the performance of ammonia synthesis catalyst.

Q - Mr. T.S. HARIHARAN, FERTIL, Abu Dhabi

Refer to page 14-6, first §. How does H<sub>2</sub>/N<sub>2</sub> ratio of 2.2 to 2.5:1 in the synthesis loop improve operability as a certain ratio still needs to be controlled to avoid operational upsets and compressor performance problems? Could you please elaborate.

A - The simulation of the low pressure synthesis loop has shown that sensitivity to inerts in the H<sub>2</sub>:N ratio of 2.2.-2.5:1 is much, much less than it is with a conventional plant. What we have simulated and shown on the slides is that a 50% increase in the methane slipping into the synthesis loop will almost certainly shut down a modern plant, but will hardly affect this process.

The reason for this is that the actual conversion in this process is less than in conventional processes. It is about 77%. What improves the overall situation is the fact that the H<sub>2</sub> recovered across the circulator without recompression goes back into the process with 90 odd percent. The plant now is much less sensitive to effects of the upstream catalysts. In a modern process something like 80% of the methane that goes into the synthesis loop is contributed from the make gas catalysts and 20% from the reforming section. What we have done with the AMV process is to completely reverse that, like 80% of the methane going into the synthesis loop is from the reforming section and only 20% from

the make gas catalysts.

The sensitivity at low pressure to CH<sub>4</sub> and N<sub>2</sub> concentration, as can be demonstrated by real time simulation, is much less than in conventional processes.

If you then look at the point that has been made in the extra compression cost for trying to improve the conversion, the overall economics is very much in favour of going for high CH<sub>4</sub> slippage, high N<sub>2</sub> and low pressure.

A/84/15 A new concept for ammonia production by P. Orphanides & Ch. Polychronides, PFI, Greece (Rapporteur J.E. Reynolds, Grace W.R. & Co. USA)

Q - Mr. H. ALLYOT, TECHNIP, France

1. What are the references of the recovery boiler the reformer gases of the Haldor Topsoe/Nuove Pignone concept?
2. Why did you choose a different technology for the boiler in the synthesis loop?

A - 1. The reference list can be obtained from the IFA Secretariat.

2. At the time of selection, BORSIG had the best references for a boiler for the gas pressure, temperature and composition prevailing in the synloop. However, we believe that Topsoe/Pignone could also be a valid solution.

Q - Mr. T.S. HARIHARAN, Fertil, Abu Dhabi

1. Who is responsible for engineering, construction and commissioning?
2. What is the overall completion period of the project?
3. After introducing feed gas to the plant, within how many days you expect ammonia production?
4. What is the expected on stream capacity of realization for first year operation?
5. Could you please give reference of any other plant using PSA process for hydrogen purification?
6. What type of adsorbent is used for PSA H<sub>2</sub> purification, and during operation what is the frequency of regeneration cycle change?
7. Waste heat boiler for reformed gas - what precautions have been taken to avoid tube fouling problem?
8. What type of training is foreseen for operating and maintenance staff for Foxboro's Spect: IV system?

- A - 1. KTI of Holland. Project is on "turn-key" basis.
2. 33 months.
  3. Approximately 24 hours.
  4. 330 days per year.
  5. The reference list can be obtained from the IFA Secretariat.
  6. For adsorbent type please ask Union Carbide Corporation. Full cycle-time of each bed is 2 minutes.
  7. He has supplied a very sophisticated water treatment based on VGB 64 (for high pressure boilers) though steam pressure is 45 bar.
  8. Two-weeks operators training course for 2 process and 3 instrument engineers and two weeks maintenance course (for 3 instrument engineers) at Foxboro educational centre, Soest, Holland.

TA/84/16 Energy saving in urea and ammonium nitrate plants by G. Brusasco, R. Monaldi, G. Pagani & M. Santini, Fertimont SpA, Italy (Rapporteur J.E. Reynolds, Grace W.R. & Co, USA)

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

The ammonium nitrate granulation process shows that the drier is heated indirectly using steam. We use direct heating with natural gas combustion. Would Fertimont do the same with a new plant today?

A - Porto Marghera plant has been designed using steam at 8 bar for heating the drying air at a maximum temperature of 160°C; at that time the decision was connected with safety reasons. With a new plant, we consider important the energy saving connected with the use of the steam coming out from the AN reactor as indicated in the paper; nevertheless we consider feasible the use of natural gas with the proper safety devices.

Q - Mr. P. CHINAL, CdF Chimie AZF, France

In figure 5 of your paper, it seems that the scrubbing of the mist out of the reactor is done with a nitrate solution at the same concentration as that of the reactor. Is it the case?

In this scrubber, do you introduce make up nitric acid? If so, how is it controlled?

What is the operating pH of the reactor and of the scrubber?

A - The washing of the vapours from the reactor operates in two steps inside the column C101:

- in the first step AN solution at 15-25% and pH = 1 is recirculated;
- in the second step AN solution at 2-3% is recirculated.

Nitric acid is added with a pH control in the reactor and in the lower part of the column. The pH value in the reactor is 3.

Q - Mr. Z.H. PAN, CNCCC, China

1. If H<sub>2</sub>S and H<sub>2</sub> contents in CO<sub>2</sub> are about 10 mg/Nm<sup>3</sup> and 0.8% (vol.) respectively, is your special passivation technique still applicable and does the explosive atmosphere exist in your new process? If it does, where? And how to avoid explosion?
2. What is the minimum load at which the new IDR section can still be operated?
3. What are the factors which influence the performance of the 2nd stripper?

A - 1. H<sub>2</sub>S must be reduced through an appropriate adsorbant to 1 ppm maximum.  
The content of 0.8% vol. H<sub>2</sub> in the CO<sub>2</sub> may give the same problems due to the presence of O<sub>2</sub>. In our case we use two types of passivation agents:

- the first one is a liquid oxidizing agent in order to protect the strippers' surfaces. No problem for this substance;
- the second one is air for passivating all the remaining parts of the plant.

In order to avoid any operation near or in the explosion field due the contemporary presence of O<sub>2</sub>, H<sub>2</sub> and NH<sub>3</sub>, (that can occur during the inert gas washing) we operate in the H.P. scrubber always in the presence of an ammonia excess; it means out of the explosion field. In the M.P. scrubber we add nitrogen before the final washing of inert gas in order to reduce the H<sub>2</sub> and O<sub>2</sub> concentration below the inferior flammability limits.

2. The minimum operation load of the IDR plant is about 40-50% of the design figures.
3. The performances of the second stripper of the IDR process are affected mainly by:
  - temperature of inlet solution that must not exceed 210°C. We prefer to operate in the range 200-205°C;
  - NH<sub>3</sub>/CO<sub>2</sub> ratio of the inlet solution. We operate quite well when this ratio is equal or higher than 3.8-4 (by weight).

Q - Mr. F.A. KARS, Stamicarbon, Netherlands

1. On page 2, §4 in the english version of the paper is stated:

"the innovative aspect of the process is the removal of excess ammonia in the urea solution mainly achieved through using carbon dioxide in the stripping operation". I always believed that we were the true inventors of the CO<sub>2</sub>-stripping process in which CO<sub>2</sub> at synthesis pressure is used to remove excess ammonia but also non-converted carbamate from the urea solution. Can some clarification be given?

2. How do you compare your process with Mitsui Toatsu's ACES process? And is your process and/or your patent situation, if you have any, not in conflict with M.T. patents or others?
  3. Is it correct that you have no commercial experience with the scheme as given in figure 2, such as:
    - the ejector added to the carbamate condenser
    - the high pressure scrubber that replaces the separator of scheme 1
    - the heat exchange in the L.P. distiller
    - the heat exchange in the vacuum system?
  4. . Is your anticipated steam figure based on full heat removal with cooling water in the ammonia recovery system and
    - . Is it the difference between import and export steam, if not, what is your anticipated export steam figure?
  5. Is it correct that your anticipated steam figure is based on a plant with an electrical driven compressor, and do you agree that the energy picture is completely different for a plant with cogeneration in which the compressor is steam driven?
  6. Modern granulation technologies have lowered investments and utility costs in comparison to earlier technologies by reducing recycle ratios and air requirement by evaporation of water. Can you explain to us why it is advantageous to move in the opposite direction again?
  7. Is your multiple curtain granulator, without water spray nozzles, more or less identical to TVA's falling curtain granulator?
- A - 1. The main innovative aspect of our process is the stripping of ammonia by CO<sub>2</sub> in a urea solution very rich in free ammonia, without significant change of the carbamate content in the solution. In this way, it is possible to recycle big quantities of ammonia to the reactor directly in the H.P. loop and this permits to obtain very high conversion in the reactor and, in consequence, low energy consumption.

This is completely different from the STAMICARBON process, in which mainly carbamate is stripped by CO<sub>2</sub>. In the last version of IDR process, our philosophy is to produce carbamate in the stripper evaporating ammonia in order to cut the M.P. steam consumption. It is clear that our process is completely different from STAMICARBON CO<sub>2</sub> stripping process both from a conceptual point of view and for the final results.

2. Our process has been patented on May, 1977, that is before the ACES MITSUI-TOYO Patent. In any case, our process is different from ACES process. In consequence, our patent situation is good.

3. We have commercial experience on:

- the ejector added to carbamate condenser. This ejector has been operating for 3 year in the S. Giuseppe di Cairo plant with good results.

- high pressure scrubbers are in operation at 80 bar in our Ferrara factory and in several other plants.

No problem to operate at higher pressure (for instance 200 bar).

- The double effect technique between M.P. distiller and L.P. distiller has been operating in commercial scale since 1967 in our S. Giuseppe di Cairo factory, in the conventional urea plant.

- The double effect technique between M.P. distiller and 1st vacuum distiller is in commercial operation from June 1982 in the IDR plant of S. Giuseppe di Cairo factory.

4. Our steam figures are overall M.P. steam duty from our side. No imported LP steam is needed and no exported LP steam is foreseen.

5. The steam figures are process figures and don't take into account the possible driving with steam turbine of the CO2 compressor. For this subject, please see our article "IDR technology cuts to a minimum the energy consumption in urea plants" - NITROGEN 145 Sept-Oct 1983.

6. Our consumption figures are on the same level as other granulation processes. Presently our recycle ratio is about 2 without affecting the product quality. According to our opinion, advantages of our process are simplicity of operation and low investment cost.

7. Fertimont has experience in the curtain granulating drum since fifties. In comparing our drum with the one of TVA we have these differences:

- melt is sprayed on two or more parallel curtains in order to reduce the drum volume, without affecting the quality of the product. In consequence, we have a very high length/diameter ratio in the drum with relevant low investment cost;

- the crystallization heat is removed mainly by cold recycle.

Q - Mr. L.K. RASMUSSEN, Superfos A/S, Denmark

What is the amount of  $(\text{NH}_4)_2\text{SO}_4$  per ton in your 26% AN and why do you add AS?

A - The maximum amount of  $(\text{NH}_4)_2\text{SO}_4$  is 4 kg/t 26% AN. We add ammonium sulphate in order to prevent the reaction between limestone and nitric acid to form  $\text{Ca}(\text{NO}_3)_2$ ; this reaction occurs mainly in the scrubbing step with the limestone contained in the entrained powders.

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

The storage temperature for 26% N ammonium nitrate of 70-75°C seems very high. It is surprising you have no caking in storage. Can you comment further particularly:

1. Storage time before bagging
2. Temperature at bagging?

A - The storage time before bagging varies between 1 and 5 months depending on the shipment programs.

The bagging temperature is less than 45°C in winter; summertime it can be sometimes more than 45°C.

Q - Mr. C. HOEK, UKF, Netherlands

What is the bulk density of ammonium rate and urea granules?

A - - AN 26% bulk density is 0.8 t/m<sup>3</sup>; real and apparent density are respectively 1.86 and 1.49.

- granular urea bulk density is 0.76 t/m<sup>3</sup>; real and apparent density are respectively 1.32 and 1.26.

Q - Mr. P. ORPHANIDES, PFI, Greece

What is the recycle ratio in your granulation AN 26% process?

A - The recycle ratio in the AN plant is in the range 2-3.

Q - Mr. G. CALIS, UKF, Netherlands

Do you expect any disadvantageous effects on product quality of ammonium nitrate, due to the III to IV transition, occurring during transportation and handling?

A - No. We have tested the same resistance to thermal shock in the cooled products.

Q - Mr. J. MOELLER BIKEBAEK, Superfos A/S, Denmark

What is the maximum acceptable particle size of limestone fed to the AN-granulator?

A - The limestone size is 60% passing through a 400 mesh screen; rather than the size, we consider the outside specific surface, that is more than 5000 cm<sup>2</sup>/g.

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

What is the concentration of ammonium nitrate in the granulation



plant scrubber?

How is granulation controlled:

- by addition of steam?
- by control of on-size material as recycle?

A - The concentration of ammonium nitrate in the granulation plant scrubber is 50% by wt. Granulation parameters controlled are the recycle of on-size material and solution concentration in order to keep in the fixed range temperature, humidity and size of the granulated material.

TA/84/17 Recycle purge gas for additional ammonia production from an existing ammonia plant by N.C. Brahma & S.C. Mittal, Shriram Fertilizers & Chemicals, India (Rapporteur J.E. Reynolds, Grace W.R. & Co, USA)

4TH WORKING SESSION

TA/84/18 The BASF-ODDA NPK process by A.H. Durocher, L. Diehl & H.J. Eisen, BASF, Germany (Rapporteurs L.K. Rasmussen, Superfos AS, Denmark & B. Persson, SUPRA AB, Sweden)

Q - Mr. F. MOSTAD, SUPRA AB, Sweden

Why are you changing from the sulphuric acid route to the nitrophosphate route in the Antwerp plant?

A - Some arguments have already been given. Gypsum deposits are one reason, but switching to the new BASF-ODDA technology by revamping an older plant enables fertilizers to be produced with lower variable manufacturing costs. As you can see from the slide, the manufacturing costs are considerably higher using the mixed acid route, until we reached the practical production limit grade (3x16.7) with the Odda process. These are the main economical reasons.

Q - What are the main improvements in this process compared to the old Ludwigshafen plant?

A - Integration of experience is also sometimes a change in experience and that is the reason why we switched from centrifuges to belt-filter separation technology. Due to erosion/corrosion effects, centrifuges entail higher maintenance costs than belt filters, thus increasing production costs.

Low emissions were also attained, but the main changes were in the separation steps - insolubles and CNTH salt.

Q - Mr. G. KONGSHAUG, Norsk Hydro, Norway

You indicate in your paper that 90% water-soluble P2O5 can be achieved by the use of fractional filtrations and several cooling stages.

What is the purpose of fractional filtration, and is this system installed in any commercial plant?

A - By fractional separation we mean a two-step crystallization procedure. There is a limit to crystallization of the NP slurry in one step because a highly viscous solution is obtained which causes problems in the CNTH-separation section. That is the reason why, if we need a NPK grade with 85 to 90% water solubility, we have to recycle the mother liquor, obtained in a first separation step, to cool it to a lower temperature and to re-separate calcium nitrate ballast. We have so far not installed this second step in any of our plants.

Q - What is the design value for water-soluble P2O5 in your new Antwerp plant?

A - We designed the plant for values between 35 and 85%, but there is

no need in general in Europe for water solubilities above 50 to 60%.

Q - Mr. R. MONALDI, Fertimont SpA, Italy

Which type of neutralizers of the NP-acid are preferred and, if the slurry evaporates, what is the lowest pH before such an operation?

A - The type of neutralizer depends on the granulation process used in the plant, but in general we preferred loop neutralizers. In fact, this piece of equipment is a standard feature, but the optimization of the neutralizers varies from plant to plant.

Answering your question about the pH, it is 2-3.

Q - Is the coating operation carried out before, during or after cooling?

A - There are only two possibilities: before and during, but not after.

Q - What are the main problems of the scrubbing tower where the NH<sub>3</sub> losses in the neutralization section are absorbed with nitric acid?

A - Problems are related to the presence of steam and high temperatures (about 95°C): steam has to be separated from the ammonia, which is recovered in standard ammonium nitrate scrubbers.

Q - Is this tower on line with the equipment for the NO<sub>x</sub> gases where the NO<sub>x</sub> gases are absorbed and oxidized in the solution containing calcium carbonate?

A - No, the two units are not on line.

Q - Percent energy consumption in the different units is reported in figure 13. Is it possible to know the absolute value as steam, electric power, fuel gas and liquid ammonia per ton of 15.15.15.?

A - There are a lot of standard answers to this question. Let me say that absolute figures are difficult to give because energy costs change from plant to plant and that is why an answer to this question is not simple.

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

What is the minimum ratio of N:P<sub>2</sub>O<sub>5</sub> in the final product from the BASF/Odda process?

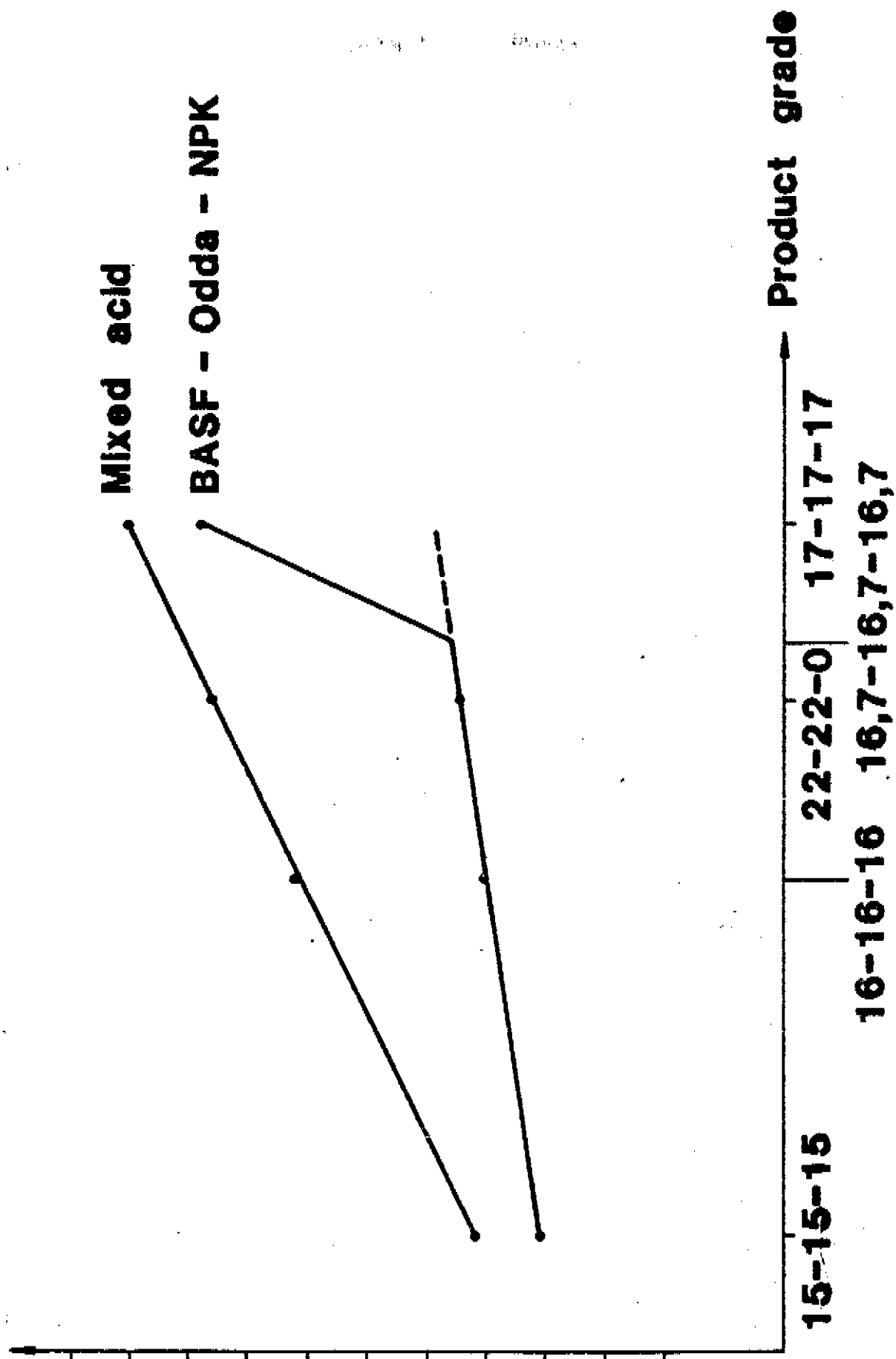
A - Minimum should be at the moment 0.65:1.

Q - What is the maximum water soluble P<sub>2</sub>O<sub>5</sub> proportion?

A - About 85%.

Q - What material is used for the filter cloth for separation of

# Variable manufacturing costs



**BASF**

Product grade vs. variable manufacturing costs

calcium nitrate?

A - We have tested all possible cloths, and have found that polypropylene cloths are the most suitable ones. In fact the cloth construction needs not be very complicated.

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

What percentage increase in operating cost is expected for increasing water soluble P2O5 from 60 to 85%?

A - Between 22-22-0 and 17-17-17. The difference should be 20%.

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands

The conversion of Ca-nitrate to C.A.N. requires CO2. Are you going to do this in Antwerp? And if so, where do you get the CO2 from?

A - It comes primarily from the ethylene oxide plant.

Q - Mr. E. AASUM, Norsk Hydro, Norway

In your paper you describe that phosphate rocks with more than 500 ppm of Cl are serious for corrosion and that the problem can be resolved by appropriate pretreatment. Do you mean a pretreatment of the rock itself or what other solutions could be foreseen?

A - The word pretreatment is indeed not exactly defined. Pretreatments are possible, but very expensive to say the least. The best pretreatment is to blend the phosphates.

Q - How much NOx and fluorine compounds are released to the air in the waste gases without scrubbing with a solution containing calcium carbonate?

A - Regarding the fluorine content, separation with a normal scrubbing system - water scrubbing - gives rates less than 2 ppm of fluorine and for NOx we do not exceed 100 ppm/m3 in any case.

Q - Mr. P. MORAILLON, CdF Chimie AZF, France

To get a low CaO/P2O5 ratio in the NP acid solution, lower than 0.2 in weight, is it necessary to use more nitric acid than the excess of 12-18% required for a good conversion of phosphate rock?

A - What matters here is the acid concentration and not the excess. The excess of 12-18% is present at all times but what matters at levels below 0.3 is that your acid should be thick enough to promote intensive crystallization; this is why we are using an acid that is very close to 60% by weight.

Q - Mr. S.K. MUKHERJEE, FAI, India

How much production have BASF so far provided to market with water-soluble P2O5 content of 70% and above?

A - The production of such NPK grades is mainly for export and BASF is represented in almost all markets.

Q - Mr. M. SIPILA, Kemira Oy, Finland

What particular method is preferred by BASF for removal and washing of insolubles?

A - Sand removal alternatives depend to a great extent on the type of phosphate you are processing. Kola phosphate does not need sand removal; but centrifuge separation is well suited when processing 68 Pebble phosphate.

Q - What is the typical crystal size distribution?

A - CSD is not a big problem and crystallization procedures do not need to be very sophisticated in our process. Anyway, the average crystal diameter should not be lower than 300  $\mu\text{m}$ .

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

On page 13 you mention concentration of NP-slurry by means of single-stage evaporator. Can you tell us:

- the pH of the slurry?

A - Between 2 and 3.

Q - . the material used for the heat exchanger?

A - Stainless steel n° 1.4541.

Q - . the maximum content of Cl allowed in the slurry?

A - 50 ppm.

Q - Mr. S. ORMBERG, Norsk Hydro, Norway

In washing the Ca nitrate with calcium/ammonium nitrate solution you have the risk of getting some  $\text{NH}_4$  ions returned to the dissolution stage. Have you observed any connection between the  $\text{NH}_4$ -concentration and the filtrability of the Ca nitrate?

A - The question is not relevant because our AN-CN washing medium is only a displacement wash and no appreciable  $\text{NH}_4$  ions are returned to the digestion step.

Q - After conversion of calcium nitrate to AN and  $\text{CaCO}_3$ , you concentrate the 61% AN solution to 94-98%. Do you experience scaling in the evaporators?

A - Yes, we do and the evaporators have to be washed from time to time with a special cleaning solution.

Q - Mr. E. HOLTE, Norsk Hydro, Norway

In your 2500 tons per day going on stream in Antwerp in 85 what

concentration of CO<sub>2</sub> will you use for converting the calcium nitrate?

A - Concentrations up to 100%.

Q - Mr. E. AASUM, Norsk Hydro, Norway

Norsk Hydro has in its Glomfjord nitrophosphate plant been operating since 1955 without any equipment for sand removal. Several different phosphates have been used, some with up to 6% acid-insoluble material with size up to 250 micron. The calcium nitrate is filtered off with rotating filters. Would the author comment on this?

A - I would only observe that in this case maintenance costs for pumps, lines, stirrers and the amount of ballast are certainly higher than in our plants using for example phosphate containing up to 6% insolubles. Of course, using Kola phosphate, a plant can be run for 30 years or more without any difficulty.

TA/84/19 Significant energy savings in the ODDA process by M.H.G. Jennekens, Stamicarbon BV & G.H.M. Calis, DSM, Netherlands (Rapporteurs L.K. Rasmussen, Superfos AS, Denmark and B. Persson, Supra AB, Sweden)

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

In the New Stamicarbon batch crystallization process the number of cooling circuits required are more than for continuous process. While integration of the set of crystallizers in various cooling circuits leads to energy savings, the installed costs of equipment may be higher. Some idea of the relative cost of production per tonne P205 may be given for the continuous and the new batch process.

A - The Engineering Department of DSM made an investment estimate of the three variants of the process. It indicates that no significant differences in investment figures, and thus in fixed costs of the product, were noticed.

In my paper I have shown that considerable energy savings may be realized, which of course directly affect the variable costs of the end product. Comparison of the continuous crystallization process with the new batch crystallization process shows that the total price of the end product is 7 US\$ per ton of P205 lower.

Q - Mr. G. KONGSHAUG, Norsk Hydro, Norway

Do you find it correct to compare the new crystallization process with existing conventional processes when this is only based on your own tests and your own understanding of the theory?

There are many differences in opinion as to numbers and assumptions in tables 2 and 3.

A - Well, it is not our understanding of the theory but fundamental crystallization theory which we are referring to. I think that, by the two experiments described in the paper, the validity of this theory is demonstrated clearly. Concerning the differences you note, I would like to invite Norsk Hydro to discuss comparable figures extensively on a later occasion.

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

How did you determine the crystal size distribution?

A - The crystal size distribution has been determined in two ways: The first method is to wash and dry the crystals, followed by sieving. The second method is used for a more accurate size distribution. We applied a microscopy technique in which a photograph of a number of crystals is made. The photograph is scanned with a light beam of very precise dimensions. This is done with 16 different beam sizes and results in a very accurate size distribution analysis of the crystals.

Q - Did you install some kind of automatic equipment for on-line analysis of crystal size distribution in your industrial plants?

A - No, we never did and we don't intend to. However, we do intend to install on-line analysis equipment to determine the CaO/P2O5 molar ratio of the process liquor before the crystallization section and, also, to install a microprocessor-based control system in the crystallization unit. This system calculates the temperature profile for a given dissolution liquor based on this CaO/P2O5 molar ratio. With the process as just described to you, we always have the same coarse and uniform crystals.

Q - Mr. A. DUROCHER, BASF, Germany

A question related to maintenance costs. Can you give some indication of the life of the sieves in your two-stage pusher-type centrifuges?

A - Well, the new process, which is based on batch crystallization, results in very coarse and uniform crystals, which can easily be separated by means of a conventional filter. This is what we are going to apply in our process, and certainly no pusher-type centrifuges. So I think the question is not relevant.

Q - Mr. E. AASUM, Norsk Hydro, Norway

On page 10 the theory of conventional batch crystallization is described where the cooling medium passes countercurrently through the crystallizer in the process.

On the basis of your experiments and of application of theory you conclude for several reasons that crystallization behaviour in technical systems can neither be controlled nor predicted.

In the Norsk Hydro nitrophosphate process the batch crystallization is operated with this countercurrent flow of cooling medium and our experience on crystallization behaviour in technical system does not follow your predictions.



There are now 10 commercial units with a total P2O5-capacity of 3000 MT P2O5 per day operating on this principle, the first one being in operation for 10 years.

A - Well, the most recent paper of Norsk Hydro, which was presented at the Fertilizer Round Table in Baltimore, USA, contains an important typing error. If 85% of the crystals is to be removed, the final crystallization temperature should be -5°C instead of the printed +5°C, which proves that our predictions are right. We have no intention at all of condemning the conventional batch crystallization process, we only wanted to demonstrate that considerable improvements of this process concerning energy savings as well as production flexibility can be realized.

TA/84/20 Computer simulation of fertilizer granulation plants by I.K. Watson, UKF Fertilizers Ltd/Stamicarbon BV, Netherlands (Rapporteurs L.K. Rasmussen, Superfos AS, Denmark and B. Persson, SUPRA AB, Sweden)

Q - Mr. R. MONALDI, Fertimont SpA, Italy

From your paper the computer is used to simulate the plant conditions to optimize the process. Do you intend next time to apply a computerized system on-line to operate the plant?

A - Not yet. The reason for this lies in the last conclusion I gave about the model; that is, the accuracy of simulating the granulator itself is not yet good enough. I think we have only got to go back to one of the earlier papers from Mr. Handley, who was talking about the effects of different phosphoric acids upon granulation to see that to build a reliable model of the granulation step is very difficult indeed. Our approach would therefore be to use the computer model off-line in the manner described in the paper to develop a control philosophy for particular products under particular conditions, and develop this philosophy through to guidelines for operating the plant.

Q - How do you propose to obtain reliable samples of the solid recycle?

A - Earlier I mentioned something about computer models being only as good as the information upon which it is based. As you imply, it is difficult to obtain reliable samples and reliable sieve analysis for recycle and granulate materials. We did obtain large numbers of samples both to determine the sampling variance and also a number of series of samples over short time periods to find out how variable our plant is.

Q - Have you studied an automatic method to obtain sieve analysis on a real-time basis?

A - Several plants in UKF have continuous on-line sieve analysis. One of these works by taking a sample actually of the granulate before screening, in which case the sample is fed to a small screening and weighing system. The problems with this system is that you only need one lump of say 20 millimeters in size, and it

makes obtaining a representative sample very difficult. Another plant tries to get round this problem by doing continuous size analysis of the product after screening, and using the indirect correlation between product sieve analysis and granulate sieve analysis. This is also not an entirely adequate method and a better one is to put a number of weighers on the recycle conveyor and so get a continuous indication of fines, on-size and over-size in the recycle. This is the method that I personally favour, though it is potentially more expensive than the other two.

Q - Is it the same with moisture in the slurry?

A - That has been thought about but we do not do it because in our process the slurry is nearly always under boiling conditions, in which case there is a very good correlation between the water content and the temperature. Knowing the temperature and the mole ratio is therefore sufficient.

Q - How much would a complete on-line automation of an NPK plant cost?

A - John Markham is going to answer that partly when he comes to talk about chemical analysis in the next paper.

Like a lot of other people we are going down the route of putting most of our plant data on to computers. We have been doing this for a number of years via an off-line system and we are now moving to a system of doing it directly on line so that we have got up to date information available on a computer terminal and people can see the trends in process conditions; also statistical techniques can be applied to current data giving suggestions to the operator what action he ought to be taking.

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

On the basis of your model would you say it is essential to include a recycle hopper in the recycle loop?

A - I would not say it is essential, and further there are problems with the practicability of actually doing this. Our own plant for instance was built with a recycle hopper because years ago this was the philosophy of Dorr Oliver. It never worked and has since been removed. In any case, if you have good information on your recycle screen analysis and the required flexibility to control it, e.g. a splitter valve or an adjustable crushing system, then such a recycle hopper is not necessary.

Q - Mr. E. SEUNA, Kemira Oy, Finland

How do your models take different rheological properties of ammonium phosphate slurries into account (e.g. due to different phosphoric acids or varying N/P ratio, etc...)?

A - They don't directly, they do it in an indirect fashion very crudely. If you look at the model, what we have in there is a thing that we call Beta-curves, and we have different Beta-curves for different conditions. I accept that it is extremely

simplistic but that part of the model is being worked out, and hopefully one of these days we will come to the situation where we can say we know enough about viscosity or other properties of AP slurry to put in as a variable in such a computer model.

Q - Do you actually use the model for creating strategies for handling process disturbances?

A - Yes. See my answer to the first question from Mr. Monaldi.

Q - If the recycle loop is burdened up with dust, could you take that into account?

A - Yes. You can use it in any situation where plant is potentially out of control, or is actually out of control (for example, the recycle D50 has gone down to 1.5 mm). To look at potential ways of getting out of that situation you will discover fairly rapidly that some have no chances of success at all, but others show some promise. However, you may come to the conclusion, in practice, that it would be a lot quicker and easier to dump the whole recycle and start again.

Q - How did you collect the data for the models? Was it done manually or by automatic measurements?

A - All the sieve analysis data was collected manually, and let me say once again that to build a reliable model requires the collection of a very large amount of data. This is in effect an advantage of the computer model; it forces you to collect a lot of information which otherwise would probably not be collected. For example, if I take a sample of the granulate, how representative is that? if I take another one in two minutes time, am I going to get the same result or not?

TA/84/21 Computer control of granulation plant NPK autoanalysers by J.H. Markham, ICI PLC, United Kingdom (Rapporteurs L.K. RASMUSSEN, Superfos AS, Denmark and B. PERSSON, SUPRA AB, Sweden)

Q - Mr. A. SINTE MAARTENSDIJK, AMFERT, Netherlands

Although the author refers to the EEC regulations, the P205 determination is done as water-soluble and not as neutral ammonium citrate P205. In our experience the process-control on basis of water-soluble P205, when selling on basis of n.a.c. P205, does (at least when using superphosphates) not give the same results. I would like to hear your opinion on this.

A - I have an easy reply to this as far as we are concerned at ICI. We use Senegal phosphate rock as our basic phosphate supply. This rock produces fertilizers with a relatively high water-soluble P205 content and presents no problems to us in the determination of the phosphate content of our fertilizers.

Q - Could you give a rough estimate of the cost involved in a system

as described and did you make an evaluation of the cost and profits after some years of operation with this equipment?

A - I knew that this would be an inevitable question but I haven't prepared a cost estimate for a modern system to the standard that we have installed at Billingham. From the description I have given, delegates will realize that we have developed this system over a number of years. We have added to and modified the original equipment and this makes it difficult to put a composite cost to the present installation. The equipment used is, in the main, relatively simple and easily obtained. Some of the key items, particularly the sample preparation unit, are produced in our own workshops at Billingham. I have discussed the situation with my colleagues and we believe that Pounds 50,000 might be needed to provide an installation to an ICI standard.

Q - In normal laboratory-analysis the sample is ground before determination of the nutrients, in your equipment it is not; did you investigate whether you got deviations in the analysis due to this simplification?

A - The only problem we have ever experienced in this area has been the determination of total P2O5 content of our fertilizers in our main laboratories. It was found necessary to grind the fertilizer down to a very fine size grading to give an accurate measurement of total P2O5. This problem was never experienced in the water soluble P2O5 determination on the auto-analyzer which provides the information needed for plant analysis control.

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

Sampling of a stream of moving granules is difficult to achieve without segregation. How much work did ICI do to evaluate the performance of the hockey stick sampler with respect to segregation?

A - The hockey stick sampler which I have described is a very simple device and represents an easy solution to the problem of obtaining a representative sample from a moving stream of granules on a conveyor. When we were developing the sampler the major concern was the problem of segregation. Plant operating staff in the audience will be very familiar with this situation where the fine material is predominantly at the base of the granular fertilizer while the large size particles run along at the top. We carried out a series of tests by installing a "GECO" sampler which takes a sweep across the flow of granules at the discharge of the conveyor. The results from the "GECO" were compared with those from the hockey stick sampler and remarkable agreement was found. We are very confident in the representative nature of the sample taken by the hockey stick sampler.

Q - Would you use the same device for sampling for size analysis?

A - We have installed hockey stick samplers on the product conveyors from the two granulation units and they are used to take the routine samples for size grading and quality checks along with the occasional samples taken for check analysis. I believe that this demonstrates our confidence in this device.

Q - Is there segregation in the vibrating feeder to the dissolving stage?

A - There is no segregation because the vibrating feeder is effectively choke-fed. The granules fall down onto the base of the vibrating trough and in effect the granules then pass along the trough in the order that they are presented to the trough itself.

Q - Mr. R. MONALDI, Fertimont SpA, Italy'

Considering that also the NPK plants of Fertimont are controlled by full automated systems developed using the technique of thermometric analysis, I should like to get some information on these points, if possible:

Coefficient of variation % for each determination performed by your system?

A - We all use different terms to describe the accuracy of analysis control. At Billingham we use the term standard deviation. If I use 17 nutrient units as an average nutrient level we would expect to achieve a standard deviation of 0.35 unit. I believe that is approximately 2% in accuracy terms.

Q - What is the accuracy level with respect to the same determinations manually performed?

A - If you are referring to a manual determination in a laboratory then I would not claim that our on-plant analyses give the same accuracy. In the laboratory determination a lot more time is taken to allow the sample and standards to reach the steady state situation which reduces the scope for significant errors. We do achieve, on a continuous basis, a very high degree of correlation with laboratory analysis and I wouldn't want to leave anyone with the wrong impression on this point. Our results from the on-plant analyzers are very close to the results obtained by laboratory determination.

Q - What is the actual time (on yearly basis) required to maintain the system in good performance?

A - We now allow our instrument technician half an hour a day to do a daily standard check and check over the tubes etc... That is two and a half hours a week for this installation.

Q - Mr. S. ORMBERG, Norsk Hydro, Norway

With the auto-analyzer you are determining the total nitrogen content. Is it possible also to get separate values for  $\text{NH}_4\text{-N}$  and nitrate-N?

A - The development of this system has opened up the opportunity to use the automatic equipment on other measurement duties. We have already installed analyzers on the effluent discharge from our ammonium nitrate plants to continuously monitor  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ . It is our eventual aim to use this data to achieve automatic control of certain parts of the plant.

Q - What is the accuracy of the N, P and K analysis and have you managed to reduce the manpower in the laboratory after introducing the auto-analyzer?

A - I have dealt with the question of accuracy. In terms of the manpower used on the installation we used to have one and a half instrument technicians full-time plus additional resources when needed and this has now been reduced to one man working half an hour per day. In addition we are now doing more than 95% of our analysis on the plant and this has reduced the resources needed in the central laboratory. The experience that we have gained by developing the on-plant system has been transferred to the central laboratory situation where many of the systems have now been automated giving further manpower reductions.

Q - Mr. J. FOUKARI, Kemira Oy, Finland

Your NPK-analysis is from year 1975. Have you made development after 1975 and in which parts of the analyzer?

A - We have changed very little in terms of the installation in the last 5 years. I think the 1975 you refer to is mentioned in the paper as being the break-point between phase one of the development which was developing the basic system and phase two of the development which was developing the computerisation, etc... It took a little while to establish the computerisation, the hockey stick sampler, the new house and the new equipment, but for the last five years we have made no significant change to the equipment at all. I know that a number of people in the audience have visited the installation at Billingham and actually seen it operating during that time period.

Q - What is the maximum time for operation without maintenance?

A - It's something we have never tested. When I say that we use half an hour a day of an instrument technician's time I mean Monday to Friday. We do very little over the weekend. We have a shift instrument technician who checks the equipment over during the weekend. I suppose really that relates to two days of continuous operation in that situation, but we have never tested that because we do feel that it is important to go in on a daily basis, doing an absolute determination, checking over the tubes and making sure there are no problems.

Q - Is the weighing procedure precise enough?

A - This is the most accurate weigher of this type that I have come across. For a plant-based piece of equipment it is a very sophisticated piece of precise engineering, which has been developed for this particular activity.

Q - Mr. E. SEUNA, Kemira Oy, Finland

You daily feed a manual standard, which is near the grade being produced. How many different standards of this kind do you need?

A - There are 8 standard solutions used to cover the complete range of NPK formulations produced on the plant.

TA/84/22 Co-audination - A system bringing real time economic performance control to fertilizer production by J. Springell, ICI PLC, United Kingdom (Rapporteurs L.K. Rasmussen, Superfos AS, Denmark and B. Persson, SUPRA AB, Sweden)

Q - Mr. G.H.M. CALIS, UKF, Netherlands

How do you account for product quality in calculating the economic performance of a plant with the Auditor system?

A - Product quality is always an important criterion in the economic assessment of a process. It is more important in some processes than others. One example, important in ammonia production is the hydrogen/nitrogen ratio in the synthesis loop of the process. This ratio has a bearing on the economic performance of the process and we can infer it by using an Auditor to continuously collect a limited amount of process data and to run a theoretical process model which calculates the above ratio and other key operating parameters.

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

The most important element in production costs is raw material cost. Process losses can be accounted for, but there always seems to be losses particularly in solids handling, which cannot be accounted for in this way. Does your system allow for these?

A - Yes, it does help to show where losses occur. As I mentioned earlier, one of the main functions of Auditor is to carry out mass balances and consistency checks. It can be used to calculate the efficiency or losses associated with either individual sections of the plant or the whole process. For example, in NPK production the efficiency of the ammonium nitrate section or that of the whole process can be calculated.

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

Computerised optimisation of individual plants is achievable with some effort where the chemistry and thermodynamics are well documented and understood. This does not apply to granulation to the same degree as ammonia and nitric acid. Does ICI intend to apply Co-audinator techniques to NPK-plants? if so, how and when?

A - In answer to the second part of the question - yes, we are applying Auditor to NPK plants right now. It is obviously easier on large continuous single stream processes to exploit the power of Auditor but we are now extending it to other processes. One of the main functions of the system is to allow coordination of each individual plant into the entire production strategy for the complex as a whole.

Q - ICI Divisional Co-audination seems based on the human shift managers. How good are their decisions for action to deal with sudden problems such as breakdown, lack of transport, full storage etc...?

A - The Co-audination system does not make the problem more difficult

it makes its solution easier by providing instant plant and site information in an easily understandable form. This was illustrated in the slide I showed you of various measures of efficiency on an ammonia plant with the target and actual values displayed. It can only help the user make better decisions.