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1990/91

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TECHNICAL CONFERENCES

| | | |
|------|------------------|----------------|
| 1947 | Landskrona | Sweden |
| 1949 | Milan | Italy |
| 1951 | Paris | France |
| 1953 | Cambridge | United Kingdom |
| 1955 | Arhus | Denmark |
| 1957 | Madrid | Spain |
| 1959 | Stockholm | Sweden |
| 1961 | Wiesbaden | Germany |
| 1963 | Helsinki | Finland |
| 1965 | Edinburg | United Kingdom |
| 1967 | Stresa | Italy |
| 1968 | Brussels | Belgium |
| 1970 | Sandefjord | Norway |
| 1972 | Seville | Spain |
| 1974 | Prague | Czechoslovakia |
| 1976 | The Hague | Netherlands |
| 1978 | Orlando | United States |
| 1980 | Vienna | Austria |
| 1982 | Kallithea | Greece |
| 1984 | Paris | France |
| 1986 | Port El Kantaoui | Tunisia |
| 1988 | Edmonton | Canada |
| 1990 | Venice | Italy |

FIRST GENERAL SESSION

"AMMONIUM NITRATE & NPK FERTILIZERS"

Chairman: J. Jolly, Freeport McMoRan Resource Partners, USA

Rapporteurs: M. Handley, IMC Fertilizer Inc., USA
D.W. Leyshon, Jacobs Engineering Group Inc., USA

TA/90/1 Fertilizer Supply and Demand Trends in the 1990's.
K.G. Soh and K.F. Isherwood, IFA

Q - P. Orphanides, Duetag, France

In your table N°4 you estimate the N plant availability = $\frac{\text{supply}}{\text{capacity}}$ to be only 69%.

This seems to be very low! Most of developed countries N plants have 80%-85% availability and a lot of N plants in developing countries have availabilities higher than 75%. This brings the balance, are the figures much higher than those indicated by you!

Ans: The figures are derived from the feed-back of the survey amongst our members, for the non-IFA members, the figures are based on projections.

Additional notes:

| | | |
|---|------------|------------------|
| <u>1990:</u> Name-plate capacities | | <u>116 399</u> |
| - Minus allowance for operation of plants at sub-optimal rates* | - 17 744 | |
| - Minus non-fertilizer use | - 11 470 | |
| - Minus processing and distribution losses** | - 7 784 | |
| <u>Total</u> | - 36 998 = | <u>79 401</u> |
| | = | available supply |

* Estimated on a country basis, based on performance

** Estimated on a regional basis, varying from 7 to 11% according to the region.

Q - A. Horkko, Kemira Oy, Finland

Could you tell me how do you find that the fertilizer consumption growth of 4% per annum in developing countries have the possibility to build up own fertilizer capacity quickly enough 4 m.t. N, 2 m.t. P2O5, 1 m.t. K2O in 5 years? Or does this mean expanding export from the developed countries?

Ans: It is usually a combination of both. As developing countries expand their capacities, importation will fill the shortfall. The latter is critically dependent on availability of foreign exchange as is clearly evident in the case of India and China.

TA/90/2

Spherodizer Granulation of NPK Fertilizers by Nitric Attack: Process and Environmental Aspects.

G. Ciaccasassi and G. Gelli, Enichem Agricoltura S.p.A., Italy

Q - G. Kongshaug, Norsk Hydro, Norway**1. What is the % water solubility P₂O₅ in your product (eg: NPK 15-15-15)?****Ans:** The P₂O₅ water solubility is as follows:

| | | | |
|----------|------------|-----------------|-------|
| 15-15-15 | : 70 + 85% | 12-6-18 S + MgO | : 60% |
| 25-10 | : 25% | 12-12-12 | : 20% |
| 20-10-10 | : 25% | 16-35 | : 70% |

2. How do you achieve a recycle ratio of 1 to 1.2 when the slurry moisture is almost 20%?**Ans:** We set recycle ratio so that we may have the best flow rate in function of the granulometry.**3. Are you not operating product screening in your granulation process? (recycling operation not indicated in your flow sheet).****Ans:** We have screening in the granulation process.**4. Are any of your products produced as B-grades (giving self-sustaining decomposition)?****Ans:** As B-grade we produce 12-12-12 (limited amounts).**5. Have you experienced any burning accidents in your granulation process since you are operating with as high as 300° C of the inlet air temperature?****Ans:** We have not decomposition in spherodizer since we use 2% of H₃PO₄ in formulation.**Q - B.K. Jain, FAI, India****1. Product moisture varies from 1.2-1.8. Is there any limit of this? In India, only 1% is allowed. How is it measured?****Ans:** The final humidity of the product is 1.5%. There is no legal limit on moisture in Italy. It is only a matter of product quality. For each formula, there is a critical moisture level of the slurry below which it is impossible to granulate. Final moisture varies consequently. Moisture is measured with this system: evaporation in the vacuum.**B.K. Jain's comment:** In India, the legal limit is 1% moisture on NPK & DAP.**Rapporteur:** The latter figure appears to be inconsistent with most international producers' specification which is 1.5% moisture in DAP usually, but dependent on the method of analysis which was not discussed here.**2. In paragraph 3: In Effluent Scrubbing Unit, the scrubbing performance in respect of nitrogen oxides at 85% and fluorine at 94% appears to be low. Although the quantities of pollutant emission are below the legal limits, is any action being taken to improve on the scrubbing performance?****Ans:** The projected limits are in accordance to public law; the limits are satisfactory at the present time.

Q - M. Bencharrada, SLAPE, Tunisia

Can you indicate the difference in efficiency between units operating with and without effluent recovery?

Ans: With scrubbing system, we have recovered:

4.0 % Nitrogen
0.2 % P₂O₅
0.2 % K₂O

Q - L.K. Rasmussen, Kemira, Belgium

1. What kind of pump are you using for the pumping of the slurry to the spherodizer and have you installed any safety equipment related to this pumping?

Ans: Centrifugal pumps with packing seal, 30% Cr material, with variable turns, hydraulic variator controlled by slurry FTC. There is a block for slurry high temperature.

2. Can you confirm that your recycle rate is 1-1.2?

Ans: We confirm recycle ratio is 1 - 1.2. Only for 12-12-12, it is 1.5.

3. Your flow sheet shows addition of ammonium sulphate to the MAP mixing reactor. Is this to adjust the P₂O₅ water solubility?

Ans: Ammonium sulphate is added to increase P₂O₅ water solubility.

4. What is normally the P₂O₅ water solubility in your products?

Ans: The P₂O₅ water solubility is as follows:

| | | | | | |
|------------|---|----------|----------------|---|-----|
| 15-15-15 | : | 70 + 85% | 12-6-18 S + Mg | : | 60% |
| 25-10 | : | 25% | 12-12-12 | : | 20% |
| 20-10-10 | : | 25% | 16-35 | : | 70% |
| 20-10-10 S | : | 70% | | | |

5. How often do you clean the spherodizer and how?

Ans: Normally we wash spherodizer once a month with industrial water.

Q - J. Moeller Birkebaek, Kemira Danmark A/S, Denmark

Hourly capacity. What are the limiting factors to the capacity of the spherodizer for 15-15-15 and 20-10-10?

Ans: Formula 15-15-15: for above 800 t/h charge, holding time into spherodizer decreases so that granulometry reduces.

Formula 20-10-10: the limiting factor is low fusion temperature of the salt.

Q - I. Bubalo, INA-Petrokemija Kutina, Yugoslavia

Did you change the distance between spray nozzle and falling curtain or it is constant?

Ans: The distance between spray nozzles and falling curtain is fixed.

Q - J.H. Straatman, Kemira Pernis BV, Netherlands

Please explain scrubber liquid. Are you using H₂SO₄ and H₃PO₄ in a certain ratio? How is the ratio H₂SO₄/H₃PO₄ determined? Or are you using H₂SO₄ or H₃PO₄ separately?

Ans: It uses separately H₃PO₄ and H₂SO₄: phosphoric acid in first section, sulphuric acid in second section.

Q - M.S. Srinivasan, EID-Parry (India) Ltd, India

What is the frequency of spherodizer blockage?

Ans: It is a blockage for high temperature in the furnace. It is never operated. We stop spherodizer once each month for washing.

Q - S. Swanström, Kemira Oy, Finland

1. In the reactors Nos 7 and 8, there is an "internal mixing device". Can you please give some details, or description?

Ans: Agitation system consists in an agitator operating into a conveyor pipe made in 316 L AISI coaxial to reactor. The agitator has a three-blade propeller conformed to push slurry to above. Agitator, also, is made in 316 AISI. Electric power is about 40 kw.

2. What is the maximum feed of ammonia, in t/h, for one neutralising reactor?

Ans: Maximum feed of ammonia is 2000 kg/h.

Q - J. Benes, Uhde GmbH, Germany

1. Can you specify the relative water and citrate solubilities of the products mentioned in paragraph 2.1.1.?

Ans:

| | Water Solubility | Citrate solubility |
|------------------|------------------|--------------------|
| 15-15-15 | 70 + 85% | 99% |
| 25-10 | 25% | 99% |
| 20-10-10S | 70% | 99% |
| 12-6-18S + 2 MgO | 60 % | 99 % |

2. What is done to prevent reversion to citrate-insoluble phosphate during neutralization of 20-10-10-5 and 12-6-18 + 2 MgO?

Ans: If we add K₂SO₄ in acid phase, we have not reversion to citrate insoluble P₂O₅.

3. Can you specify typical conditions for the effluent from the spherodizer scrubber system, i.e.: specific flow, m³/ton, temperature, °C, pH, composition, % N, P₂O₅, F, SO₄?

Ans: We have two liquid currents from gas treatment system:

Current 1

Ammonium phosphate sol. : 50%
 Temperature : 45° C
 Density : 1.3
 N/P2O5 (weight) : < 0.12
 Chlorine : < 100 ppm
 Fluorine : 750 ppm
 pH : 2

Current 2

Ammonium sulphate sol. : 37%
 Temperature : 45° C
 Density : 1.18
 Chlorine : 2500 ppm
 Fluorine : 1500 ppm
 pH : 4.5

Q - A. Van Brempt, Kemira, Belgium

In some formulae you add ammonium sulphate salt (crystals).

1. What is the impact on granulation (e.g. on dust load in the sphero air)?

Ans: We have not negative impact on granulation for the reason that in the reaction section ammonium sulphate reacts completely.

2. What is the average granulometry of the ammonium sulphate you add?

Ans: Average granulometry of ammonium sulphate is as follows:

15% up 1 mm
 55% 1 to 0.5 mm
 20% 0.5 to 0.2 mm
 10% less than 0.2 mm

Q - H. Hero, Kemira Oy, Finland**1. Product quality: Are those values mean, target or minimum delivery values?**

Ans: Product quality targets are as follows:

Caking index : less than 500 g/cm²
 Granulometry : 85% min up 2 mm
 : 98% min up 1 mm
 Moisture : less than 2 %
 P2O5 water soluble : in accordance with client's request

2. Do you guarantee the free flowing properties? Which are the critical grades, have you any difficulties with?

Ans: Critical grades for free flowing properties are: 20-10-10 and 25-5-5.

Q - B. Christensen, Kemira Danmark, Denmark**1. Why is ammonium phosphate added as a raw material and not formed by reaction between ammonia and phosphoric acid?**

Ans: We cannot use exclusively phosphoric acid as water soluble P2O5 for following reasons:

Limited storage capacity
 Limited ammonia evaporator capacity

2. Is it necessary to grind ammonium sulphate in order to obtain good granulation in the spherodizer?

Ans: It is not necessary to grind ammonium sulphate for the reason we use ammonium sulphate from caprolactam with small granulometry:

- 15% up 1 mm
- 55% up + 0.5 mm
- 20% 0.5 to 0.2 mm
- 10% less than 0.2 mm

3. Why is acidic magnesium used instead of kieserite as raw material?

Ans: Economic reasons.

Q - E. Busnach, Negev Phosphates, Israel

1. What is the residual P₂O₅ in the final product, and how is it controlled?

Ans: Residual P₂O₅ in the final products is as follows:

- 5% maximum in 12-12-12
- 1% in all other formulations.

2. How much does rock type effect the solubility of the P₂O₅ in final NPK?

Ans: No effects if ratio HNO₃/phosphate rock is correct. The value is different for each kind of raw material.

Q - E. Yanik, Toros Fertilizer & Chemical Co., Turkey

Nitrophosphate products are getting powdered if you store it approximately 1 month. The condition is at approx. 10-15° C. That is why for the eastern countries it seems impossible to use it, it is getting powdered and caked. Do you have any improvement?

Ans: It is necessary to use coating power with low CaCO₃ content and opportune granulometry.

Q - M. Dekmil, SIAPE, Tunisia

1. The flow out of the granulator seems high. Why?

Ans: We confirm that optimized spherodizer gas flow rate is as it is in our presentation.

2. What type of densimeter do you use? What is the reliability of this machine?

Ans: Two pointing out membranes are on the tank wall, one above the other, at a fixed distance. The instrument surveys pressure difference between the points. Differential pressure is in dependance from hydrostatic weight, that is from liquid density. The controller effects a valve to introduce diluting water. A LIC sets the out flow rate from the tank.

3. Electric consumption of the unit of washing seems very high.

Ans: Electric consumption of gas treatment plant is as follows: 900 kwh (18 kwh/ton of product).

Q - G. Thorvardarson, Kemira Danmark, Denmark

1. Do you have any special reasons for using MAP (solid) instead of phosacid plus ammonia in the reactors? (In fact, the desired precipitation of DCP could also be obtained under such conditions).

Ans: We use MAP or phosphoric acid or both depending on the market price.

2. Special reasons for adding Mg either as an acid solution or as dolomite?

Ans: We use Mg in a acid solution only if there is a request for water soluble MgO.

TA/90/3

Production of Calcium-Ammonium-Nitrate: The BASF Process
 Part A: J. L. Engelmann, BASF Antwerp, Belgium
 Part B: R.E. Nitzschmann, BASF, Germany F.R.

TA/90/3A

Q - A. Van Brempt, Kemira, Belgium

1. The process vapours of the neutralizer are cleaned in a packing tower, demister and washed with an AN solution. Can you give the typical load of nitrogen in the vapours before and after cleaning (quantity and form)?

Ans: The process vapours before washing are about 3 to 4 grams of nitrogen per liter calculated as condensate. After washing, the vapours contain 100 milligrams nitrogen per liter.

2. MgO salt is mentioned as product stabilizer. If you add this MgO salt, what is the impact on the CHR and water absorption rate of your final product?

Ans: We do not use magnesium salts. We use some special mixture of different inorganic additives which have been described in the answer to Mr. Jain's question (refer to page 10).

Q - J. Moeller Birkebaek, Kemira Danmark A/S, Denmark

Safety of production

1. What measures have been taken to ensure that no contamination of the raw materials to the mixing vessels occurs?

Ans: Separation of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and the conversion to CaCO_3 and AN is a sufficient cleaning step to avoid contamination.

2. Can you accept the use of defoamer agent in the digestion reaction?

Ans: Yes, we can accept the use of defoamers in the digestion of rock phosphate.

Q - P. Orphanides, Duetag, France

Is 60% HNO_3 minimum concentration used to obtain 93% AN solution? What is the concentration of AN solution for 63% HNO_3 ?

Ans: We normally don't use 63% nitric acid for production of AN-solution.

Q - G. Kongshaug, Norsk Hydro, Norway

Is the BASF Odda process limited to production of 10 t CAN per ton P_2O_5 since the AN-content in the wet CaCO_3 is 5%?

Ans: I don't understand this question fully, but the ODDA process makes a very clear amount of calcium nitrate.

In addition, we have, depending on the NPK formula, a quantity of ammonium nitrate available from the ODDA process.

Dr. Engelmann also indicates there is synthesized ammonium nitrate available. So the relation between ammonium nitrate and NPK is very open.

In another way, we have lime contaminated with ammonium nitrate solution of 60% concentration which, however, brings only a very small quantity of ammonium nitrate into the CAN plant. We avoid washing and drying of the lime.

I hope I have understood the question. There are no limitations under these circumstances.

Further, the Odda process is very flexible. Regarding lime, if you intend to use all the lime coming from the Odda plant, you can produce approximately 10 t of CAN/t P₂O₅. If this quantity is not desired, we use an equipment to wash and dry the lime for other purposes.

My question is that you produce a certain amount of calcium carbonate which has to go to CAN. That means for every ton of P₂O₅, you must produce 10 tons of CAN. That is the limitation I refer to.

Ans: No, we have other processes and plants that can use the calcium carbonate as well.

The Odda process brings out approximately 1 t AN-N/t P₂O₅. This is less than 4 t CAN/t P₂O₅. So we always use an additional AN-synthesis. We refer to page 3A-3 of the paper of Dr. Engelmann.

TA/90/3B

Q - A. Van Brempt, Kemira, Belgium

You mentioned that the mixture of CAN solution is sprayed in a granulator drum. We have two questions:

a) Are you spraying on a falling curtain and is the granulation drum a classical spherodizer?

b) You are spraying airless, can you give some idea about the droplet size?

You mentioned that no heating of process air is required (neither for start-up). It is clear that you can run a granulation CAN plant with a thermal balance in equilibrium, but in your case, you cannot accept a turndown, and I believe that the start-up of a cold plant could be rather long resulting in some off-specific production?

Ans: I mentioned that we spray directly on the recycled material. We don't use a curtain. We use a very special spray system that is very difficult to describe.

The start-up time is approximately 20 minutes. Neither at start up time or at the end of a production period, we produce any product outside specifications.

Q - L.K. Rasmussen, Kemira, Belgium

1. Can you give any details about your metering system for moist lime?

Ans: We use a special design chain conveyor and we use the absorption of beta rays for measuring the content of limestone in ammonium nitrate.

2. You have not installed any cyclones on the gas streams from the granulation drum and the dryer. Can you give a figure for the dust load in normal operation to the scrubbing unit?

Ans: Yes, between the granulation drum and the packed column are cyclones for pre-cleaning.

The dust load after the cyclones is following the normal possibility for cyclones, about 50 to 100, sometimes 100 mg/m³ of gas.

3. Have you installed any special safety system for your slurry pumps?

Ans: All details concerning this question are given in the paper of B. Purucker, BASF Germany "Safety Requirements in Fertilizer Plants", 1988, Tech. Conf. Edmonton, Canada, Preprints: TA/88/15.

Q - H. Hero, Kemira Oy, Finland

1. What kind of granulators have you used? Is it possible to granulate AN in the same equipment?

Ans:

Part I. Refer answer to Mr. Van Brempt's question earlier.

Part II. Up to now, we don't produce AN in such an equipment.

2. How can you prevent stress corrosion in the drum?

Have you installed any special safety system for your slurry pumps?

Ans: Normal stainless steel is sufficient for the granulator because we have no chlorine present, and I see no problems in this case.

Q - V. Olevski, Agrochim, USSR

1. What is the size of particles CaCO₃ do you use for mixing with AN? Is it directly after conversion of Ca(NO₃)₂·4H₂O?

Ans: 60 ± 20 μm.

2. Did you compare drum-granulation and prilling? What is better?

Ans: BASF decided after careful studies to change from prilling towers to granulation.

Q - M. Iznaguen, OCP and M. Smani, CERPHOS, Morocco

Does your process require a special quality of rock?

Ans: No, if you have a quality with 67 BPL min. No other requirements.

Q - B.K. Jain, FAI, India

1. Paragraph 2: Could you give some details of the lime-mixing vessel? Is it steam jacketed-blunger type or single agitator type?

Ans: The mixing vessel is a standard mixing vessel of some cubic meters volume. It has a steam jacket and has a slow turning agitator.

2. It is mentioned that inorganic additives for improving the product quality are added. Could you please elaborate the actual function and what particular quality characteristic of the product is improved by this additive since the final product is again coated with anti-caking agent?

Ans: Yes, regarding inorganic additives, these are not added mainly to improve anti-caking quality, but the important point is to slow down, a little, the hygroscopicity. It is important to get a tailor-made grain hardness, let me say, 60 newtons plus or minus 5 to 8 points. The main point is to keep the granules stable during temperature cycles around 32° C.

Q - H. Hero, Kemira Oy, Finland

We know that CAN is normally not stabilized against thermal cycles. From a safety point of view, is it an advantage?

1. Do you stabilize your product? If so, how?

Ans: Yes, we stabilize it as we have described by using anti-caking agents and by stabilizing mainly against thermal cycles at 32° C to avoid high volume change in the ammonium nitrate.

Yes, we think it is an advantage because we can avoid the dangerous increase of volume. Please see details in the reply to Mr. B.K. Jain.

TA/90/4 **Manufacture of Ammonium Nitrate : The Use of a Pipe Reactor and a Total Air Recycle system for Prilling Tower Pollution Control.**
J.L. Lopez-Nino and J.A. Zurbano, FESA and L.M. Marzo, ESPINDESA, Spain

Q - B.K. Jain, FAI, India

Apart from pollution abatement, does the recovery of ammonium nitrate dust from prilling tower by total recycling of the air compensates the extra operational costs (extra power consumption stated as 16 kwh/MT of ammonium nitrate).

Ans: Yes, one of the side-effects is to improve the efficiency of nitrogen to near 100%, depending on the pollution existing in any given plant. Improvement can be 0.5% to 1.5% in nitrogen efficiency.

Depending on the prices of energy and nitrogen, talking only of operating costs in most cases, the recycled air system will pay for itself, apart from the pollution aspects.

Q - M. Dekhil, SIAPE, Tunisia

1. What is the investment expected to adapt your cleaning system for the prilling gas to a unit of 1000 t/d Kaltbach-Thuring process?

Ans: The investment shall be in the order of US\$ 1.5 million.

2. Can we economically justify such a modification?

Ans: Further to our answer of Mr. Jain's question, we might add that there are some savings, difficult to evaluate, in subjects such as maintenance in the AN and adjacent plants which sometimes can be important due to the corrosive effects of the AN upon ladders, platforms, instruments, electrical equipment and the like. Generally speaking, the economical justification of such a modification depends on the dust emission of the prill tower.

Q - B.K. Jain, FAI, India

What is your view with regard to applying the system of total recycling of the air to urea prilling towers, particularly in some of the old plants where urea dust emission may be as high as 500 mg/Nm³.

Ans: Our view is optimistic. In fact, when we applied for a patent, we have covered also the cleaning of urea towers and we are working on it. Basically, what happens in a urea prilling tower is the same: the air becomes polluted and hot. Therefore, the same principles can be applied.

Q - A. Van Brempt, Kemira, Belgium

Pipe Reactor

1. What is the typical capacity of such a unit? (Range "Min and Max")?

Ans: Up to 1000 TPD or in that order of magnitude.

2. How much temperature delta T exists between the operating temperature and the solidification point of the 95% solution (bottom concentration)?

Ans: We are working at about 132° C. The solidification temperature - I do not remember the number for 95% solution. (Someone in the meeting gave the solidification temperature as 120° C). So we are working about 10° C to 15° C above the solidification temperature.

Q - G. Brusasco, Agrimont, Italy

1. What is the construction material for R-01, C-01 and E-04?

Ans: The pipe reactor is Uranus B-6. The towers are 304L or 316L, where the operating temperatures range from 30° C to 80° C.

2. What is the maximum capacity that can be reached by a single apparatus?

Ans: We have tested up to 500 TPD in one of our plants. The pipe is a modification of a DAP pipe reactor which we are providing currently for Iraq at 700 TPD. We see no problem in going to 1000 TPD. We are, in fact, designing such plants with one single pipe.

3. Can you tell, please, the NH₃ and NH₄NO₃ content in the exit condensate and the solution pH inside C-01?

Ans: We have analyzed the steam exiting the pipe reactor and received the following figures, in the separation chamber:

0.3% free NH₃
 1.0% ammonium nitrate
 0.02% nitrous acid
 pH was of the order of 9.0

In other cases, we have worked with much lower pH's, which means we are getting efficiencies of 99% or not far from that.

Q - G. Kongshaug, Norsk Hydro, Norway

1. What is the inlet air temperature to the prilling tower?

Ans: The temperature of the air leaving the prill tower is about 46° C. Air entering the prill tower is about 35° C. What is important is to keep the product below 80° C which depends on the height of the tower.

2. What is the water content and the average particle size of the product?

Ans: The specification for porous ammonium nitrate, which is the normal product, is less than 0.2% moisture. In fact, the products are below 0.1%, last week's analyses were 0.07%-0.08% water.

3. Do you add any stabilizing agents to the product (dense and porous)?

Ans: We do, but in fact, what we presented were the aspects of the process which are new contributions. The original process was provided by Kaltenbach who are present here, so these questions can be directed to them because they are in the middle of our two technologies.

SECOND GENERAL WORKING SESSION**"UREA AND AMMONIA"**

Chairman: P. Martinet, Soferti/Grande Paroisse, France

Rapporteurs: J. Basset, Soferti/Grande Paroisse, France
L.J. Carpentier, IFA

TA/90/5 Energy Reduction in Urea and Ammonia Production at the Agrimont Ferrara Plant.
R. Botti, C. Burlando, G. Gramatica, Agrimont S.p.A., Italy

Q - B.K. Jain, FAI, India

On page 6, it is mentioned that the use of new materials for higher corrosion resistant piping and with improved mechanical characteristics, brought about a reduction of the piping thickness. Please give details of this new material of construction. The passivation technique used on the vessel surfaces to inhibit corrosion may please be explained.

Ans: The IDR section piping construction material is the ferritic-austenitic stainless steel 2205 called SAF2205 produced by SANDVIK. This material besides to have an high corrosion resistance, tested both by lab tests and by practical experimentation on the semi-industrial urea plant of S. Giuseppe di Cairo, is characterized by mechanical properties higher in comparison to the stainless steel AISI 316 urea grade previously used in the Montedison urea plants.

This has allowed, at the same project conditions, to reduce the piping thickness with consequent savings not only of the material of the piping but also of the supports and of the structure.

The passivation technique is based on the combined action of the passive air introduced into the IDR loop either alone by means an appropriate compressor or together with CO₂ and of a liquid passivation agent consisting of a hydrogen peroxide solution, fed by means an appropriate pump at some key points of the IDR section.

TA/90/6 Steam and Power Balances in Ammonia Plants and Ammonia-Urea Complexes
 I. Dybkjaer, Haldor Topsoe A/S, Denmark

Q - V. Olevski, GIAP, USSR

What is your opinion about the possibility of a deep integration of ammonia and urea plants by absorbing NH₃ with CO₂ - water solution from the synthesis loop under the urea-synthesis pressure (the suggestion of Snamprogetti company)?

Ans: There have been many suggestions for such processes where the ammonia production is in some way integrated so that you recover the ammonia either by washing it out with water and using this ammonia water to remove CO₂ or even more integrated concepts where you have the exit gas from the synthesis gas converter going to the urea reactor. To my knowledge, these schemes have never been successful in industry although they have been around for more than 20 years.

In my opinion, it is an indication that you can integrate too much. You can make a plant too complicated and too integrated so that it becomes too difficult to operate. There are also certain evident disadvantages with this integration and the main one is that in all situations there is a significant risk of carryover of water into the synthesis loop where you could poison the ammonia synthesis catalyst. I have not seen a recent analysis of any of these schemes in combination with modern low energy ammonia processes but I have the suspicion that the advantage in efficiency would be marginal to-day because optimization has taken place inside the ammonia plant and inside the urea plant to a level where you would not gain very much by this extra installation.

Q - U.P. Jhaveri, Gujarat Narmada Valley Company Ltd, India

Does extra cost of combined cycle installation justified by savings of energy consumption?

Ans: There is of course no general answer to that question. The payback time of the installation of a combined cycle of cogeneration unit must be evaluated in each individual case on the basis of the local cost of energy, etc. What I think can be said to-day, without any doubt, is the reliability of these cogeneration units, or of other ways of using gas turbines, is not inferior to the reliability of steam turbine installation. The onstream factor obtained with gas turbines to-day is very good. In your individual case, you have to evaluate whether it is justified to install such units.

Q - P. Orphanides, Duetag, France

1. In many modern granular urea production units of 2000 MTPD or more, there is an export surplus of about 40 TPH low pressure steam. How this steam production is fitting in our energy scheme?

Ans: Where we put the low pressure steam would depend on a lot of factors inside the ammonia plant. Firstly, there may be a significant consumption of LP steam inside the ammonia plant if you have a Banfield or similar CO₂ removal and also to compensate stripping, if you do that with LP steam boiler feed water deaeration it is inside the battery limits; if you noted in the balance shown for the Kaltim plant, the boiler pump was on a back pressure turbine to provide LP steam for these purposes. If you have a more energy efficient CO₂ removal system and if you do stripping of process condensates with MP steam, with the process steam, the consumption of LP steam may be significantly reduced inside the ammonia plant battery limits. If you are forced in such a case to accept an amount of LP steam from outside, then the only solution I have readily available is to inject it in one of the steam turbines and to get power out of it in that way but you might redesign the ammonia plant to choose another process option in order to make use of some free energy

which may be available to you. I understand that not all urea plants have to export so much LP steam. It could also be a choice of urea process.

2. How it fits in your Figure 5 scheme a HP steam boiler, with MP steam export from the ammonia unit?

If we have a production of HP steam in the auxiliary installations, then we would probably have too much HP steam used in the synthesis gas compressor turbine alone, then the condensing part would be too small in practical. That would mean that the two compressors would be on HP steam. In some other solutions, we have seen in recent cases, it is suggested to have the ammonia compressor on a back pressure turbine between HP and MP level. So, there are many possibilities and the actual choice would depend on individual cases.

3. You use a MP steam drive for the CO2 compressor and my question is have you made a comparison with a HP steam drive for the CO2 compressor?

I think it also dealt with the question whether the CO2 compressor on HP or on MP. The fact is that you could use both and it is a matter of detailed evaluation of individual cases.

4. Why use steam for water cooling water pumps where you produce electricity within the boiler and not use steam to produce more electricity in the turbo alternator and then drive them with electric motors?

The same goes for the boiler feed water pumps. The actual situation is that every often you have two drives available for the cooling water pump. You may either use, motor or steam turbine. There is no fundamental change in the arguments whether you take this 15 t steam and convert it to energy before you use compressor drive or not.

Q - E. Groensmit, Kemira bv, Netherlands

You have optimized energy systems by using the heat of the chemical reaction to produce steam and on top of that extra steam in outside boilers or gas turbines to get an optimum picture for the whole sites. Have you also considered situations where you would minimize steam production, in other words, much more heat integration within the process steams and you would eliminate the steam as a factor in this type of process completely?

R - In my paper I tried to describe that in an ammonia plant which depends on the process concept, there is a certain minimum steam production. In a conventional plant, with a conventional type of steam reformer, there will be enough heat available in the plant to produce slightly below 3 tons of HP steam per ton of ammonia. This amount of steam would roughly be able to produce the amount of power required inside the ammonia plant battery limits meaning large drivers shown in the figure for the Indonesian plant. So, there is a nice fit between steam production and steam consumption for these drivers. This is the situation you would try to reach in your own ammonia plant, using that process concept.

It is possible to consider other process schemes. In some other schemes, there will be a minimum steam production which is larger than what is required for drivers. In such cases, the ammonia plant becomes steam exporting. You cannot get rid of this extra steam if there is no consumer for it. In a situation where you cannot export steam, you would choose a concept not exporting steam.

You can also reduce the amount of heat available for steam production but in that case, you will be, at least, in the situation with heat exchange reforming which has been discussed so much, selectively take away from the steam generation high level heat, the heat available at high temperature, the most valuable one, and what is left would just be able to produce MP steam. That represents a loss of efficiency; you can produce less power from MP steam.

In my opinion, heat exchange reforming is only relevant in cases where this extra energy you need for the drives can be supplied either from steam from some other process units or in the form of very cheap electric power.

TA/90/7Peat as a Raw Material for Ammonia
J. Koljonen, Kemira Oy, Finland

Q - L.J. Carpentler, IFA

Concerning the environment, in your paper, you say that peat is not a renewable resource but exploited peatland could be planted with trees thus supplying a new source of energy. However, a very beneficial aspect of peat, namely the high water holding capacity which means that peatland plays a very useful role as water reservoir, would be lost. Have you considered this aspect and can you comment?

A. In actual fact in Finland peatland covers 10 million ha of which 500,000 could be economically utilized. It means that the area is large enough and not too far from cities or places where peat could be used. At present, about 100,000 ha are in reserve for production and 20,000 ha are exploited at the moment. In my paper, I mentioned that 21,000 ha would be needed for operating the 80,000 t/yr ammonia plant. They are already under continuous production and that area could supply peat for about 15 years. It is not too much in relation to the total existing area. There has been discussion with local authorities because of the risk of spoiling small streams. We must be careful with water and ensure a good drainage. On the other hand, in the area where peat is exploited, there is a big unemployment problem and there is some pressure for that utilization of peat.

TA/90/8

Urea Granulation Plant at Ferrara

G. Brusasco, A. Cavallaccio, M. Ognibeni, M. Santini, Agrimont S.p.A., Italy

Q - H. Hero, Kemira Oy, Finland

1. With your granules, have you any improvement concerning evaporating of urea compared to prills?

Ans: It is not a problem with our system which is mechanical.

2. Normally, the highest acceptance biuret is 1.0%. Now you have 1.1%, does it affect to marketing? Have you any way to decrease it?

Ans: It is possible to reduce the percentage of biuret. However, it is necessary to operate at a temperature during transfer of urea melt slightly above crystallization temperature. Biuret concentration increases with temperature, residence time and melt concentration. Our production is arranged according to the quality of the finished product; so it is difficult to change the biuret concentration.

Legislation in Italy allows in production the concentration of biuret to the level of 1.2%. Just now, we have concentration of 1.1%, we have quite a margin and we maintain it.

Q - P. Orphanides, Duetag, France

Can you please summarize the advantages of your process in comparison with other urea granulation plant in terms of:

- a) Investment cost
- b) Operating cost
- c) Plant-process flexibility
- d) Product quality
- e) Environmental aspects
- f) Revamping easiness of existing drum granulation plants

Ans: Concerning the investment cost, we have reported 11 billion Lira for the cost of revamping at the Ferrara plant but we have to consider that we changed some equipment, we recovered and adapted others. The calculation we did arrived at the conclusion that for a new plant of the same capacity, the cost would be 15 billion Lira, all included. It is difficult to compare with other systems, because it is always difficult to reach realistic figures under the same conditions. The plant is very simple, the granulator is very small (the drum is 3 m in diameter and 9 m in length for a 1700 t/d capacity). Considering the easy operation, the investment cost is at its lowest level.

Concerning the operation cost, I am sure that the cost of steam for the concentration of the recovered 45% solution in the abatement system can be higher in comparison with other systems, but the quantity of this solution is small, and it may not be meaningful.

For the energy consumed in the cooler, under hot weather conditions, we have a disadvantage because we take away more heat in the fluidized bed cooler than others and, in this case, since the difference in temperature is reduced, we probably spend more. If this plant was established in a factory which contains other plants such as ammonium nitrate and nitric acid and there is a need to evaporate ammonia, if it was possible to take advantage of the frigories produced by the evaporation of ammonia, we could reduce the size of the cooler, which may improve the situation in this case.

The comparison of product quality is difficult. We can produce granules with a better crushing strength if we use more formurea. I have seen in the literature that there are

higher figures on this point. The process can produce granules with a higher crushing strength. I think it is possible to reduce the formurea content but we have to check it again in a big plant.

Environmental aspects are better than in a prilling system. In comparison with competitors, I think it depends on the selection of the manufacturer of the abatement system and the efficiency which the process people calculate. We have designed the system to comply with the values requested by local authorities.

Concerning the revamping easiness in granulation plants, there are not so many granulation plants for urea and the process is designed for urea. It can be used for ammonium nitrate but with some difficulty.

Q - E. Lopez Tallada, Fesa-Enfersa, Spain

How is the consumption in Kwh/mt between prill and granulate?

Ans: Concerning the energy consumption, we have to distinguish between the case of Ferrara and the general case. In the case of Ferrara, where we have a prilling plant and we have added a granulation plant, we have increased the energy consumption. The increase is fairly big (we have quoted 22 Kwh/t in our paper) but if an entirely new plant was constructed, this energy consumption could be reduced. For Ferrara urea plant, the consumption is as follows:

- 28 Kwh/mt (prilled urea)
- 50 Kwh/mt (granular urea)

Q - B.K. Jain, FAI, India

1. Your paper mentions addition of formaldehyde to concentrated urea solution to improve the physical characteristic of the product granules. Since use of formaldehyde is not recommended (being carcinogenic), could you suggest some other additive like lignosulphonates to get proper quality product granules?

Ans: We tried to use some other additives to improve the physical characteristics of our granular urea but, as anticaking agent, nothing is better than formaldehyde.

However, we don't use formaldehyde directly as that. A stabilized solution based on urea/formaldehyde polymers is added to the concentrated urea. This way, it is possible to avoid possible problems give by free formaldehyde.

Lignosulphonates are very good as hardening agents but they induce a brown colour to urea.

2. Under effluent treatment, it is mentioned that a small quantity of sulphuric acid is added to fix ammonia which may be present in air sucked from granulator. What is done to this dilute ammonium sulphate liquor?

Ans: We added a small amount of sulphuric-acid directly into the scrubber. $(\text{NH}_4)_2\text{SO}_4$ obtained is recycled with the urea solution to the concentration section. The final product, granular urea, contains about 2000/2500 ppm of SO_4 .

3. In Table 2, the particle size of granular urea is given as more than 93.5% between 2-5 mm. What would you suggest as the optimum particle size for granular urea, considering the production, application and use efficiency aspects?

Ans: We think that the optimum particle size for granular urea is in the range 2-5 mm because it can be used in blended fertilizers also. That is in addition the mean particle size of the different N.P.K. grades; this way it is possible, for the farmer, to use the same distribution system for both products.

Q - P. Niehues, Uhde GmbH, Germany

What is the dust formation expressed as kg dust per tonne final product considering prilled product as seed product?

Ans: When we use prills as seed product, the dust formation is about 30 kg/ton of final product in the Porto Marghera plant and about 35 kg/ton in the Ferrara plant.

Q - O. Lerner, Haifa Chemicals, Israel

As far as formaldehyde content in the final product urea is concerned, what was the content in the old coating-drum (NSM) technology as compared to granulation technology used presently, and to what extent this improved or changed the mechanical strength of the product and dusting sensitivity of it?

Ans: The old coating drum technology (N.S.M.) used 0.06-0.08% of free formaldehyde as anticaking agent, distributing it on the surface of the prills.

In the granulation technology, we use 0.25-0.35% of free formaldehyde dissolving it into the melted urea. Only in this case is possible to get hard granules.

If we treat only the surface of granular urea, we loose about 0.3-0.4 kg of hardness.

TA/90/9

The ICI Leading Concept in Ammonia (LCA) Process

D. Kitchen and A. Pinto, ICI Catalysts & Technology Licensing, United Kingdom

Q - P. Orphanides, Duetag, France

In case of an integrated NH₃-Urea plant, is it not more efficient to have the CO₂ removal section upstream the PSA unit (smaller PSA unit, higher H₂ recovery efficiency, elimination of desaturator, better use of low enthalpy syngas condensation heat)?

Ans: For any system where you are looking essentially for a 100% recovery of CO₂ then, economically, it will be more efficient to put a conventional CO₂ recovery system after the shift with pressure swing separation following it.

Is your picture concerning NO_x released in the atmosphere a correct one when you have to import a lot of electrical energy to run your LCA process and to produce these KWH, somewhere, there is burning fuel and it creates NO_x.

Ans: The mass balance depicted on that slide is the mass balance taken over the ammonia plant. If you are taking in electricity, you want the overall balance for the society as a whole, then clearly you have to include what happens at the power station.

Q - H. Suroto, P.T. Pupuk Kaltim, Indonesia

1. What the maximum duty of GHR, due to the maximum flexibility design?

Who is the manufacturer of this fixed tubes catalyst reactor?

Ans: The two GHR at Severnside, one for each plant, are of the design there that would cope with something of say 400-600 t/d ammonia. There is no theoretical or technical reason why those GHRs could not be built a single units for much larger flowsheets, 1000-1500 t/d. In licensing this technology, though, we have a practical difficulty which is that a prospective customer would wish to see an operating unit and the two operating units that we could show you at the moment are clearly not 1000 or 1500 t/d. And if this technology follows the way that other technologies have developed over the years, it is likely that people perhaps increase capacity by 20 or 25% in one throw but not double or treble it. It is unlikely that someone will go out and be sufficiently brave to order a 1500 t/d single stream GHR even though technically, there is no reason why it cannot be done. If we were looking at this technology for a large plant, 1000-1500 t/d, technically, we could offer two solutions:

1. Is a large single GHR which has the disadvantage that we would not show you a demonstration unit.
2. To use a parallel system for two or even three GHR units. That would have the advantage of getting a direct copy of the units which are already demonstrated. It would have the disadvantage of inevitably being more expensive than a single stream unit.

The second part of the question has to do with the manufacturer. The GHR is a proprietary piece of equipment and the knowhow rights to that belong to ICI. It is made by licensed engineering contractors. At the moment, Mannesmann in Germany are approved and can do it. There are some other contractors which are now going through that development process and are going to make one certainly very soon but I am not prepared to give its name. One of them is Chiyoda Corporation, in Japan, which has just signed. There is a third contractor in England which built the Severnside site and we are negotiating with them for the future. So there will be about half a dozen contractors which can build this.

2. The catalyst: Please explain the special trace element or active ingredient for low steam to carbon ratio in the reformers, isothermal shift converter and low pressure NH₃ converter.

Ans: There is no special catalyst in the primary reformer to cope with these conditions. It is a conventional primary reforming catalyst already on the range. The catalyst is the smallest size that we actually make and it is purely a physical concentration of packing this into the annular tube.

As far as the other catalysts are concerned, there is no catalyst in the LCA flowsheet specially designed for the problem of low steam/carbon ratio. The only catalyst that we have specially designed for that, we made two high temperature shift catalysts 71-3 and 71-4 that would enable conventional plants to run at lower steam/carbon ratios. You don't have those in that flowsheet because we have taken out the high temperature shift catalyst. We are on a single stage shift. It is copper, zinc, aluminium, Catalyst 83-1 on our range.

3. What was the present on-stream days performance in Bristol last year?

Ans: Last year, the plants at Bristol operated at from a high of about 450 t/d to a low of 280 t/d that was determined purely by commercial requirements of the market. In terms of on-stream days, the normal criteria when the plants were built were 330-340 d/year.

Q - A. Rodriguez, FESA, Spain

Can you give more details regarding the primary reformer:

- outside tube materials
- inside tube materials
- shell-channel materials

Do you have plugging problems of the outside tubes, due to catalyst dust?

Ans: The answer to the first part of the question is, I am afraid, no. In terms of the metallurgy of the GHR that is clearly one of the key items of the proprietary information that has been developed around this and certainly the identity of the alloys that are used there, we are not prepared to discuss in a public seminar. But we are prepared to say that it is an extremely arduous duty. We have taken out the tube cartridges recently after two years of life for a very close inspection. As a result of this inspection, we would modify the process conditions very slightly and also the metallurgy very slightly. We are not experiencing any serious problem at all.

Concerning the second part of the question, it is not a problem.

Q - B. Grotz, C.F. Braun, Inc., USA

Is your new HTS catalyst suitable for operation at the LCA conditions, and, if so, would you consider revising your design to include it.

Ans: No, we would not consider that. The HTS catalyst is an iron-chromium catalyst which is designed to operate at temperatures around 350° C. The inlet temperature of the unit here is about 250-260° C. We would not be interested by particularly raising that temperature by 100° C. We would not gain anything. The other reason why we don't want to use that particular catalyst is that the use of iron-chromium catalyst is giving increasing environmental health problems which is increasingly undesirable. As far as the modified HTS is concerned to enable it to operate at a low steam/carbon ratio, the market of use for that is purely in conventional technology. We use it for example in RMAV process. The first demonstration plant we used was the AMV plant at Sarnia in Canada. It is now used throughout the world, but in conventional technology.

THIRD GENERAL WORKING SESSION**"ENVIRONMENTAL ASPECTS OF FERTILIZER PRODUCTION"**

Chairman: W. Rittinger, BASF, Germany F.R.

Rapporteurs: F. Hofmann, BASF, Germany F.R.
W. Freitag, Uhde GmbH, Germany F.R.

TA/90/10 Fertilizer Manufacture and EEC Environmental Activities
G. Biberacher, BASF, Germany F.R. ; F.V. Samec, SCGP, France ; K.D. Shah,
ICI, United Kingdom.

Q - A. Benes, Uhde GmbH, Germany F.R.

In the last five years, there is an increasing evidence on the toxic effects of N₂O, nitrous oxide. The fertilizer industry contributes heavily to N₂O-emissions in their production plant and from the utilization of their products. So far, the relation between fertilizers and N₂O has been neglected or obscured by the industry. Can you indicate why?

Ans: N₂O (laughing gas) is actually used as an anaesthetic and analgesic as well as propellant gas for whipped cream preparations. It has been pinned down as one of the culprits (lesser) in the greenhouse effect but not as a toxic. Being a relatively stable chemical, it is not detected by certain NO_x analysis methods of gaseous effluents. I would say that it has neither been neglected nor obscured but rather considered as harmless in comparison to NO or NO_x.

Q - S. Smani, OCP, Morocco

EEC has agreed to allow individual members adopt its own regulations. This is done by Denmark. If a country member requests EEC to adopt their national regulation, what would be the position of the EFMA? What would be the position of EEC towards a common action programme?

Ans: Present EEC position, as far as we know, is that cadmium is priority but not top priority. Should any other EEC country request a regulation, a tendency towards harmonisation will surface and a discussion will follow regarding limits. EFMA is ready for such discussion.

Q - M. Dekmil, SIAPE, Tunisia

The problem of cadmium contained in fertilizers is all made up. As indicated by the author of the paper, the effects of this element are neither recorded nor foreseeable. In your opinion, what is the origin of this issue?

Ans: To my opinion, the origin goes to a misinterpretation of the influence of cadmium as related to the Itai-Itai disease. On the other hand, cadmium has documented influence on lung cancer and renal tubular functions. The latter effects may have a tenuous but still far from documented relation to the cadmium content of soils and thus to fertilization.

Q - S. Ormberg, Norsk Hydro, Norway

Do you have an estimate of the cost increase if one succeeds in removing cadmium by extraction or by calcining of the phosphate rock?

Ans: Taking into account what has been published regarding the "extraction" cost and as stated in my answer to Dr. Engelmann of BASF (refer to page 27), the cost increase per tonne of P₂O₅ produced would be in the 5 to 10% bracket, calcination being on the high side in comparison to extraction from phosphoric acid. Calcination would also be more dependent on energy cost variations.

Q - G. Kongshaug, Norsk Hydro, Norway

Table III is too optimistic! The "degree of lowering in EEC Cd soil input" include only fertilizer input, not acid rain; aerial depositions and sewage sludge.

Ans: I fully agree with your opinion, more so since one can also include cadmium input due to manure spreading, part of European feedstuff being imported, as well as other sources mentioned in table IV. On the other hand, Table III is intended only to compare effects of cadmium "extraction" in different compartments of fertilizer manufacture or related to the amount actually brought in by fertilizers only.

Q - Rus Iskiyatna, PT Petrokimia Gresik, Indonesia

Is it possible to obtain copies of the EFMA studies on "BAT" and "Water Pollution"? I want them.

Do you have a reference that shows which rock contains low level of cadmium?

Some ecology specialists note that there is also mercury in the phosphate rock beside cadmium. What is your comment? Do you have experience for this, especially in Western country?

Ans: There is no EFMA BAT study on water pollution. The BAT project is EEC sponsored work on air pollution based on directive 84-360.

Most references to low cadmium rock can be found in literature. "Phosphorus and Potassium" to name just one.

My comment regarding elements other than cadmium is that phosphate rock usually has nearly all the elements of the periodic table in the ppm or above range. Anybody can pick any element to suit his aims. Major European expertise which has been published concerns liquid-extraction of uranium from phosphoric acid. Some work also seems to have been done on magnesium.

Q - B.K. Jain, FAI, India

EEC funding has been made available for research on Cd removal in the case of Togo. Could you please tell about the present status, whether any progress has been made and the time frame of the R & D Project?

Ans: Sufficient progress seems to have been achieved to justify patenting. I have no idea about the time frame of the R & D project.

Q - M. Bencharrada, SIAPE, Tunisia

What are the limits (actual or expected values) set by the EEC concerning the cadmium contents in the waste waters and in the fertilizers: phosphoric acid 54%, DAP, TSP?

Ans: I have a slide here with some figures from European countries. EEC has not yet set values.

CADMIUM IN FERTILIZERS

Present and Future Legislation

| Country | Status | mg Cd/ kg P ₂ O ₅ | mg Cd/ kg P |
|-------------|---|--|-------------------|
| Norway | Proposals: From 1-1-1991 From 1-1-1996 Annual report to authorities | 44 22 | 100 50 |
| Sweden | Annual report to authorities | | |
| Denmark | Statutory From 1-7-90, max. From 1-7-92, " From 1-7-95, " | 87 66 48 | 200 150 110 |
| Finland | Statutory From medio 1988 | 44 | 100 |
| FRG | Voluntary | 90 | 206 |
| Austria | Statutory From 1986 | 120 | 275 |
| Japan | Statutory | 150 | 344 |
| Switzerland | Proposal From 31-8-96 | 22 | 50 |
| Holland | Guideline in feed phosphate | 15 | 35 |

EEC-limit under discussion in Bundesrat, FRG, has in spring 1990 asked EEC to set a limit on 75 mg Cd/kg P₂O₅.

Q - B. Meyer, BASF, Germany F.R.

1. What quantities of residue coming from the cadmium removal process do you expect?
2. What techniques (available or theoretical) are available to dispose the resulting cadmium containing residue and how much do you estimate the costs?

Ans: I would say from 1 kg residue per kg Cd extracted by an electrochemical process up to over 100 kg depending of the extraction process. These residues are of various compositions with, may be, toxic components. The treatment of such residues will be very difficult and costly.

Q - M. Van Den Heuvel, BASF Antwerpen, Belgium

You said that the rock treatment route would have several advantages and therefore also for the application of P₂O₅ fertilizers in the EEC. Is this also the point of view of the EEC and does EEC also encourage that route?

Ans: The EEC spends money for work to be done on phosphate rock, which - we believe - is not the easiest but the most effective way. They are also looking on phosphoric acid extraction, which is a complicated business.

Q - J. Engelmann, BASF Antwerpen, Belgium

1. Are the EEC officials well informed about:

- a) the investment costs
- b) the production costs per tonne for the different processes for the lowering from cadmium by treatment of the rock phosphate?

Ans: The EEC-officials have a study with some figures about the cost for an extraction unit for H₃PO₄. The production costs for extraction are between 7-8 Ecu/t P₂O₅ lowest and 30 Ecu/t P₂O₅ highest. An Ecu is to multiply with 1.4-1.5 to get dollars. The investment cost is between one and an half million Ecu and 30 million for an 1000 t P₂O₅/day-unit.

2. Has the EFMA Committee asked the EEC to do an epidemiological study about the possible toxicity of cadmium on a basis of maximum 200 mg/kg P?

Ans: We have insisted on this study because we have information, that cadmium is not so dreadful as some people wish to have it.

Q - P. Suppanen, Kemira Oy, Finland

The BAT (Best Available Technology) project of the EEC.

BAT is reacting for two first manufacturing processes: ammonia and nitric acid.

- a) What is the schedule for the rest of listed processes?
- b) Are there other fertilizer related processes for which BAT is expected in near future?

Ans: Ammonia, nitric acid and sulphuric acid have been done. As for the other processes, they will also be done but when and how, sorry, I can't say.

Q - H. Hero, Kemira Oy, Finland

Why are those recognized harmful elements like Mg, Pb, As, Ni, Cr, Sr not included in this work? Although there are even not harmful in soil, they can give very bad label to our industry in the publicity.

Ans: Refer to my comments to Mr. Rus Iskiyatna's question.

TA/90/11 Pollution Control of a Fertilizer Plant Situated in a Populated Area
N. Louizos, The Hellenic Chemical Products & Fertilizers Co., Greece

Q - B.K. Jain, FAI, India

From Tables 3 and 4, the Greek emission standards appear to be much less stringent as compared to EPA standards. Are Greek standards being revised to fall in line with EPA and in that case, meeting the emission standards may be difficult by the existing methods?

Ans: In most EEC-countries, the standards are less stringent than the EPA-standards and more or less the same as Greek standards.

Q - N. Kolmeyer, Hydro Agri Rotterdam, Netherlands

You say to use lime for the waste water treatment. Is this ground limestone or burnt lime?

Ans: Burnt lime.

Q - N. Hummadi, JPMC, Jordan

Can you please give more details on the odour treatment in your plant?

Ans: We don't treat the offgas on odour so we must select the sort of phosphate rock very carefully. We have to avoid types of rock which gives us odour problems.

Q - A. McGarrigle, Esso Chemical Limited, Canada

1. Do you have special regulations for the sulphuric acid plant on start-ups?
2. What levels of SO₂ do you get then?

Ans: There is no specific legislation for this point. But during start-up, the plants are connected to the ammonia scrubbers, so we are avoiding excessive emissions.

Q - M. Bencharrada, SIAPE, Tunisia

The levels of emissions into the atmosphere in the vicinity of the complex depend on: direction of the winds, height above the complex. To what extent the indicated values are representative of the environment around the complex?

Ans: The figures I have given are yearly average figures and take into account all possible direction of the wind.

Q - Rus Isdiyatna, PT Petrokimia Gresik, Indonesia

1. Do you have a procedure to overcome emergency or disaster condition caused by gas leakage? How do you develop it since you have a complex industry?

Ans: We have no standard procedure for this according to EEC-legislation.

Q - A. Horkko, Kemira Oy, Finland

You say in your paper that theoretically one could expect ammonia emissions from NPK plant. According to my experience, the $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ are harmful even after effective scrubbers. Could you tell the amount of these emissions and also the amount of mist or droplets in your NPK exhaust gases?

Ans: The ammonia emission is very, very low. We are working with low N:P ratios and that helps a lot.

Q - G. Kongshaug, Norsk Hydro, Norway

1. Is the odour from rock acidulation an environmental problem in Piraeus, and do you really include the odour issue in selecting phosphate rock?

Ans: Yes, we have to respect this on the choice of the phosphate, otherwise, we have complaints from the people around us.

2. Do you have any limits for NH_3 emission? (No data for NH_3 to air is given in the paper).

Ans: There are no limits for NH_3 -emissions - at least in this moment - in Greek legislation. But we have done measurements and there is practically no ammonia in the offgases.

Q - J.D. Crerar, Hydro Licensing, United Kingdom

1. Please give more information on use of pyrites cinders for cement production. How much can be used?

Ans: At maximum 5%.

2. Have you considered using phosphogypsum for making building material?

Ans: In Greece, we have very much natural gypsum, so phosphogypsum is not easy to sell.

TA/90/12 Reduction of NO_x Emissions in Low Pressure Nitric Acid Plants
R. Salcedo, J.L. Lopez-Nino and M. Tristan, FESA, Spain

Q - S. Swanström, Kemira Oy, Finland

After the NO_x absorption tower, there is a scrubber separator, before the outgoing gas is sent to the expansion turbine.

1. What is the scrubbing liquid in this scrubber separator?
2. What do you do with the possible surplus of this liquid?

Ans: We use water in the scrubber, and we give this water to the ion-exchanger of the HNO₃-plant.

Q - G. Kongshaug, Norsk Hydro, Norway

The nitric acid tail gas is saturated by water vapour. Does the recycling sulphuric acid absorb this vapour, and require your abatement process' supply of fresh sulphuric acid?

Ans: As I already mentioned, we need about 10 kg/hr H₂SO₄.

Q - J. Strattman, Kemira Pernis, Netherlands

1. According to your flowsheet, the H₂SO₄ is circulating in a closed loop system. How is this controlled since the water input is not necessarily the same as the output from the desorption tower?

Ans: The losses are very low, so we expect to introduce about 10 kg/hr.

2. We tested a similar system in a pilot plant 4 years ago and used concentrated H₂SO₄, because the absorption via nitroxil sulphuric acid is better. Why do you operate at a lower H₂SO₄ concentration of 75%?

Ans: When the concentration of the acid is increased, the efficiency increases too, and so is the temperature. The limit is the corrosion-resistance of the hot parts. We use tantal with a max. allowable temperature of 485-490° C.

TA/90/13 Nitrate in Water : Assessing the effectiveness in terms of water quality and the impact on agriculture of policies proposed for controlling the concentration of nitrate in water.
A.J. Williams and K.A. Ure. ICI Fertilizers, United Kingdom.

Q - O. Oertel, BASF, Germany F.R.

Your paper deals with nitrate leaching in fertilized areas and crops. How do you calculate the high losses coming from application of animal manure?

Ans: Within each MOREC square sources of N include fertilizer additions, deposits from air, and that produced by livestock. Data on livestock is available from the complete annual June (and partial December) census giving numbers and location. This gives total volume of FYM etc and N content can be calculated. No particular differential leaching has been applied to cover say FYM v slurry, or injection v surface spreading. However, this would be handling data at the micro level, and the model is clearly at the macro level.

Q - G. Kongshaug, Norsk Hydro, Norway

How much of the total UK nitrogen input to the North Sea is leachage from farming?

Ans: I don't have this figure, but it is available. About 5-10% of the nitrogen applied to the crops leaches actually into the water. On grassland, it is even less.

Q - L.J. Carpentier, IFA

In your paper, you mention eutrophication as one of the background reasons for a new regulation on the N content of any water. Would this regulation also concern phosphate?

Ans: One can expect that both nutrients will be covered by this regulation, perhaps in a different way.

Remark of the Chairman:

You noted the big influence of mineral fertilization on nitrate leaching. But we all know the much bigger amounts brought in by manures. I hope, when you speak about best farm practice, you mean also the reduction of the number of cows etc... Also the agriculture has to help to reduce the water pollution.

TA/90/14

NO_x Catalytic Reduction by Ammonia in Nitric Acid Plant
P. Gry and M. Prat, Société Chimique de la Grande Paroisse, France.

Q - F. Luck, Rhône-Poulenc, France

1. Nitrous oxide (N₂O) is involved in the greenhouse effect and possibly in ozone depletion in the stratosphere. We ascertained that there is no N₂O formation over the Rhône-Poulenc NO_x reduction catalyst when it treats typical nitric acid plant tail gases.

2. Did you pay attention to formation of nitrous oxide over your catalyst?

Ans: Actually N₂O is not considered as an air-polluting agent. As for the formation of N₂O over our catalyst, I can't answer your question here in this session.

Q - J. Roiron, Rhône-Poulenc, France

Catalysts differ from the NO_x limit value that we can obtain and corresponding residual ammonia. What are the performances of your catalyst? As indicated, RP catalyst can reduce to below 200 ppm NO_x without detectable removal of NH₃.

Ans: Actually, legislation asks for 200 ppm and our catalyst can do it. And about NH₃, I already mentioned that we have only some ppm NH₃ in the off-gas. With another catalyst, we can further reduce some ppm NO_x, but it is not the same type of catalyst.

Q - G. Kongshaug, Norsk Hydro, Norway

1. What is the specific NH₃ consumption? (kg NH₃-N/kg NO_x-N reduced)?

Ans: We use stoichiometric quantities of NH₃, one mol NH₃ for every mol NO_x. The cost per ton depends of the quantity of NO_x, as average cost (NH₃ and catalyst) you can take 0.5-1.0 dollar/t HNO₃.

2. How do you measure and avoid NH₃-loss in the tail gas?

Ans: There exist analytical devices and we find only some ppm NH₃ in the off-gas.

3. Similar abatement processes have been presented during the last 12 years. What is the advantages of your process compared to Rhône-Poulenc, BASF, etc...?

Ans: Many thanks for this question. These processes look all very similar each other, the difference is only the catalyst. The lifetime of this catalyst can be very different, our catalyst works now 6 years without any problems.

TA/90/15 Environmental Improvements and Energy Savings in the SUPRA HDH Plant
B. Persson and J. Heyman, Hydro Supra, Sweden, **B.T. Crozier**, Hydro
 Licensing, United Kingdom.

Q - B.K. Jain, FAI, India

In Section 4, it is mentioned that a mobile pilot plant for cadmium extraction has been in operation. Could you please give some more details of cadmium extraction and whether a full scale plant is likely to be installed.

Ans: At present, we use rocks with a quite low Cd-content, so we are not thinking about the installation of a full scale unit.

Q - N. Hummadi, JPMC, Jordan

1. Could you please explain how did you reduce energy consumption by 80 and 50% in NPK and PK manufacturing respectively?

Ans: We use now higher concentrated phosphoric acid in both cases.

2. The 98%-99% recovery you mentioned, is it the overall P2O5 recovery?

Ans: Yes.

Q - B. Blytre, Jacobs Engineering, USA

What materials are used on the Delcor Filter? What fluid is used for lubrication and how much?

Ans: The wetted parts are Sanicro 28.

Q - N. Kolmeyer, Hydro Agri Rotterdam, Netherlands

Under the chapter fluorine recovery, you state that the fluosillicic acid is aged. What is happening during ageing and what positive influence is aimed for?

Ans: As the acid ages, the silica-crystals grows and can so easily be separated by filtering.

Q - J. Strattman, Kemira Pernis, Netherlands

1. What specification do you have for the mercury level in your H2SO4 and what limit for mercury discharge do you have for your waste water?

Ans: We don't have any waste water containing mercury, the commercial agreement is about 1 ppm.

2. What is your actual P2O5 content in the gypsum as it goes to the island?

Ans: I don't remember this figure, but we have minimized the P2O5 going with the gypsum.

3. Could you please give the amount of cadmium in gypsum before and after the revamp? How do the other heavy metals like zinc, chromium, copper, nickel, lead influence the distribution?

Ans: The influence of the other heavy metals is not big. We used to have 15% of the cadmium in the gypsum and this has increased to 25%.

Q - A. Smani, OCP, Morocco

What is the chlorine content of the de-cadmiumised acid in your pilot plant?

Ans: We can treat many different types of rock with good results.

Q - H. Sirvio, Kemira Oy, Finland

1. Cd-removal

- a) Principle?
- b) Results?
- c) Commercial scale, when?

Ans: I have already answered.

2. Fluorine balance

- a) Fluorine emissions from:

| | |
|----------------------------|--------------|
| * circulation water system | { To |
| * gypsum hill | { atmosphere |
- b) Are these emissions restricted by authorities as well as the emissions from stack?

Ans: We have had problems to measure F-emissions from the gypsum because they were very low.

3. What is the role of active silica in conversion from HH to DH?

Ans: The purpose is to dissolve aluminium fluorides.

Q - P. Smith, Prayon-Rupel, Belgium

1. In your paper, you refer to scaling around the HH filter when using pure Florida phosphate. In which section of the filter was it encountered (the product acid, strong medium or weak section) and how was it cleaned?

Ans: It occurred in the strong-acid section and was be cleaned off by hot water.

2. How is the fluosillicic acid used in the superphosphate plant?

Ans: The H_2SiF_6 acid is produced in the plant. The highly concentrated part is recycled to the mixer of the superphosphate plant.

Q - J. Laine and P. Tiberghien, Rhône-Poulenc, France

1. Page 15-15, you mention problems due to scaling on the filter which have reduce the operating factor. You say that you solve this problem by changing the feed system of sulphuric acid into the reactor. Could you give more details on that modification?

Ans: We mix returned acid and sulphuric acid very thoroughly before entering in the reactor. So there is no risk of formation of anhydrite.

2. You mention that you add silica in order to improve the conversion HH to DH in the case of Florida rock. What is the role of this silica (chemical explanation) and is it also necessary with other rocks?

Ans: It is only necessary for the Florida rock.

3. What is the purpose of the drum filter in the fluorine recovery plant?

Ans: It is to remove SiO_2 and so to get a clear solution.

FOURTH GENERAL WORKING SESSION**"REVAMPING, PHOSPHORIC ACID, MISCELLANEOUS"**

Chairman: G. Brusasco, Agrimont, Italy

Rapporteurs: A. Barbera, Agrimont, Italy
P. Becker, Duetag, France

TA/90/16 Energy Management - A Comprehensive View
A.N. Aggarwal and U.P. Jhaveri, Gujarat Narmada Valley Fertilizers Company Limited, India

Q - V. Olevsky, Agrochim, USSR

1. When you speak about reliability, do you mean plant sections, some plants or all the plants?

Ans: As I said at the beginning of my lecture, the "Energy Monitoring Group" looks after all plants of the factory. It includes also facilities, that is cooling water system, instrumentation, steam generators, power stations, everything when possible. Only the nitric acid plant which is still under commissioning has yet to be admitted under the "Energy Monitoring System".

2. Do you have the possibility to increase the capacity of your plants?

Ans: Regarding the possibility to increase the capacity of our plants, limitations are mainly due to oxygen availability, that is from the air separation section, designed with a fixed space-velocity.

No limitation is present in the gasification section.

3. Do you have a programme for de-bottlenecking?

Ans: Finally, about de-bottlenecking, we have no programme if you mean we intend to put up some extra facilities.

Q - L. Araujo, Quimigal, Portugal

It is my opinion that, in your process, there is the risk the catalyst (third conversion reactor) can absorb water. Is your catalyst water resistant?

Ans: We have a water separator before conversion and the catalyst bed has been working for 5 years without any problems. So, we can say it is water resistant.

TA/90/17

Rhône Poulenc DIPLO Process

- Latest Developments with various Phosphate Rocks.

- Its Applications to the Revamping of Industrial Units through the Experience of SICNG (Greece), Rhône Poulenc Rieme (Rieme, Belgium) and ICS (Senegal)

J. Laine, B. Satier, Ph. Tiberghien, Rhône Poulenc Chimie, France
D. Bellis and A. Constantinidis, SICNG, Greece**Q - N. Hummadi, JPMC, Jordan****1. Could you please indicate the reaction volume in the Senegal plant at present and after conversion to DIPLO?****Ans:** The present reaction volume is 900 m³ for a normal production of 720 t/d (based on 22 hrs operation), e.g. a specific volume of 1.15 m³/t P₂O₅/24 h.The conversion to DIPLO involves the construction of a second reactor identical to the existing one. Production will be increased to 1015 t/d (based on 22 hrs), that is a specific volume of 1.6 m³/t P₂O₅/24 h.**2. What is the P₂O₅ content in the rock?****Ans:** The unit will be fed with a mixture of phosphate rock and schlamms (powdered residues) with an average P₂O₅ content of 34.3%.

Phosphate rock and schlamms will be fed as a slurry.

3. What is the phosphoric acid concentration after the filter(s), what is the predicted after DIPLO?**Ans:** The present P₂O₅ concentration of the product acid discharged from the filter is 28.5%. The expected concentration after revamping with DIPLO will be 29.6%.**Q - P. Smith, Prayon-Rupel, Belgium****1. According to the figures 1 & 2 of your paper, the "DIPLO effect" is dependent on the ratio of phosphate fed to the first and second reactors. Surely mass balances round the filter (the quantities of products acid and strong wash) and around the reactors (limitations in acceptable solids contents) require this ratio to be close to 70:30 for all cases.****Ans:** As we always stated, it is possible to vary the P₂O₅ concentration range in the two reactors, including the DIPLO effect, by changing the phosphate distribution between the two tanks.

According to the material balance around the filter and the reactors, there is a variable solid content of the slurry in the first reactor. The solid content at the discharge of the second reactor can be maintained constant by adjusting the mass flow of the filtrate recycled to the two reactors.

The test were carried out in the SICNG plant in Greece with a 50/50 phosphate rock distribution. In these conditions, it was possible to increase substantially the DIPLO effect which resulted in an increase of the P₂O₅ concentration of the product acid to 34%.**2. The use of a high sulphate in the first reactor as stated in your paper surely requires a very fine ground phosphate causing additional energy costs related to this grinding process. What additional energy is required for this grinding especially as wet grinding seems once again to decrease the "DIPLO effect" as shown in Figure 2.**

Ans: The increase of the free sulphuric acid concentration in the first reactor is beneficial to the reaction efficiency as a result of reduced losses of co-crystallized P₂O₅.

The maximum H₂SO₄ content in the filtrate of the slurry of the first reactor depends on the particle size of the phosphate rock fed: the finer the phosphate the greater the phosphate excess and the more important the gain in efficiency.

The additional cost associated with the grinding energy varies according to the type of phosphate rock and its particle size: as a result, each phosphate rock is a special case.

Q - M. Bencharrada, SIAPE, Tunisia

1. Your economic results are based upon hypothesis, could you indicate economic results based on practical experiences?

Ans: The assumptions used in our paper are average values.

The increase of 0.3% in efficiency and 1% in concentration are minimum values.

The costs of raw materials and utilities are indeed estimates.

An accurate economic calculation can only be made for each separate site based on actual costs.

2. What are the variations of these results against the phosphate content?

Ans: We do not believe that the phosphate grade has a direct bearing on the technical economic results.

The gains to be obtained with the DIPLO process are in general the more so important as the rock has low reactivity and filtrability.

Q - J. Basset, Grande Paroisse/Soferti, France

You evaluate the return of investment of the DIPLO/single tank to the maximum of 2 US\$/t of P₂O₅. The saved vapour represents 60% of these two dollars. How do you consider without additional investment the re-use of this saved energy?

Ans: Indeed our technical economic appraisal shows that the most important gain results from the increase of the product acid concentration, which results in steam saving for concentration: this is specially interesting in the plant where steam is used in turbines to produce electricity. It is the assumption we used. It means that the plant should be equipped accordingly.

Q - J.D. Crerar, Hydro Licensing, United Kingdom

1. What is maximum acid P₂O₅ for DIPLO process? Data is given for Yunan rock at 34% P₂O₅.

Ans: The maximum P₂O₅ concentration of acid produced by the Diplo process varies according to the phosphate rock used:

- With Yunan phosphate, we have in fact achieved 33% in the laboratory. For an industrial plant, this maximum concentration would probably be limited to 32%.

- In the SICNG phosphoric acid plant in Greece, we have reached, with Taiba rock, a concentration of 34%, but at the cost of a reduction of about 10% in the production rate and a yield loss estimated at 0.5%.

2. What is the height of the Rieme N° 2 reactor and filter? What is the filter vacuum?

Ans: In the Rieme plant, the height of the threshold of the second drum is 9 m and the height of the filter table is 8 m.

TA/90/18 **Revamping of Ammonia-Urea Complex: Experience at Shriram Fertilizers and Chemicals, Kota, India**
N.C. Brahma and S.C. Mittal, Shriram Fertilizers & Chemicals, India

Chairman's Comment:

The paper is well presented and extremely comprehensive.

TA/90/19 Revamping with Advanced Process Technology and Equipment
A. Bourgot and P.A. Smith, Prayon Development, Belgium

N. Kolmeyer, Hydro Agri Rotterdam, Netherlands

I feel obliged to make a comment. If a company like Prayon, with its impressive reputation, makes a negative comment about certain processes, it must be taken seriously. In my opinion, the statement "the good operation of a straight hemihydrate process is like driving along a mountain road is not correct". We, at Hydro Agri Rotterdam, had for 20 years, plants run by ordinary trained by not impressively educated technicians. For many years, we had 2 plants running in parallel: one on a hemihydrate and another on conventional dihydrate. Of course, we compared all relevant aspects - efficiency, outstream times, maintenance costs, etc. The overall conclusion was that the hemihydrate plant was the more efficient unit. But when the cost of energy became so high and that the dihydrate plant presented us with big losses, we decided to convert it also to hemihydrate. The operators were happy. No phosphoric acid plant processing 42% phosphoric acid is as simple as a single stage hemihydrate plant.

Ans: The comment you made in fact refers to one paragraph of the paper which was purely a simple analogy made solely to make people aware of the possible consequences of mal-operation in the case of a single-stage hemihydrate process.

PRAYON has developed and offers to its clients a single stage hemihydrate process, the PRAYON PH11 Process, and indeed it was this process that was chosen to industrially demonstrate the new PRAYON PH series of High Strength Processes. I believe that, contrary to the beliefs of Mr. Kolmeyer, the operators and supervisors of the HYDRO (Windmill) unit are exceptionally competent and I have always considered them to be "of the best" in the Industry. I am sure that HYDRO should be very proud of their achievements. The single stage hemihydrate process does have a place in certain cases, if not, then PRAYON would not be offering this process to their clients, but one should remember that the potential dangers of a "mountain road" have neither created such a fear that normal motorists taking adequate care are prevented from using them nor that those who are more competent and driving "High Performance" vehicles along the same route are prevented from taking the risks. In any case, if a high strength acid is essential then normally the two-stage hemihydrate-dihydrate process is more forgiving as the second stage acts as a "safety net" for accidental P2O5 losses in the first stage. Of course, the lower strength dihydrate process is less sensitive and more flexible for uninitiated clients.

Q - D.W. Leyshon, Jacobs Engineering Group Inc., USA

1. In the case of the Simplot modification, can the author describe the pumping problems that occurred during start-up that required moving the slurry recirculation pumps from the downleg to the pressure side of the cooler, and where are these pumps located in the current low-level cooler design?

Ans: As outlined in the text and as presented verbally, the SIMPLOT modification was a re-vamp that had very difficult problems relating to the layout. The flash coolers had to be sited on top of the reactor as no overhanging could be accepted. The piping for these two units was extremely difficult to fit in. Initially, the flash cooler recirculation pumps were situated on the outlet of the flash coolers, conditions that are normally considered more favourable, but it was known that with the altitude of the site and the local conditions, the available NPSH was very close to that required for the pump. In fact, the pumps did not cavitate excessively but they ran in conditions that they only gave 90% of the expected flow. It was decided that they should be re-sited on their inlet where their pumping performance reached expectations. As a general comment as to where PRAYON locates the pumps on new projects, the answer is simple; it depends on the local conditions.

2. In the Sherritt Gordon modification and others involving the older Mark 2 reactors, is it not necessary to open the slurry underflows and overflows to maintain the freeboard in the reactor? What improvements resulted from the Sherritt mods?

Ans: In general terms, the design and sizing of the overflows and underflows should be evaluated prior to revamping a unit with an increased circulation rate. It varies with each case and also is somewhat affected by the foaming tendency of the phosphate.

Regarding the modifications at the Sherritt Gordon unit in Canada, the results of the modifications giving a higher cooling capacity and a better filtrability allowed the instantaneous capacity to be almost doubled at the same efficiency.

Q - J.D. Crerar, Hydro Licensing, United Kingdom

1. How long was the hemihydrate trial at Engis?

Ans: PRAYON has more than 30 years of experience treating a large number of phosphates in the hemihydrate mode due to its development and industrial operation of the dihydrate-hemihydrate CENTRAL-PRAYON process. Also within PRAYON, we have operated a low-strength hemihydrate-dihydrate process for over 15 years. The industrial demonstration of the PRAYON PH series of processes was effected in March of 1988 by a simulation of the PH11 single-stage hemihydrate process using the equipment of the industrial 400 mtpd P2O5 CENTRAL-PRAYON process. The normal dihydrate-hemihydrate operation offers two important advantages for PRAYON, a high efficiency (98%+) and a pure merchant grade gypsum by-product. As such long term operation in the single-stage hemihydrate process was not possible for commercial reasons (the efficiency and gypsum quality of a single-stage hemihydrate process do not meet PRAYON's requirements). However, during the 14 day period reserved for the test (due to high acid stocks caused by market conditions), the plant even operating with a 2-wash filter (the existing filtration flowsheet) gave efficiencies of the order of 92-94% once reaching equilibrium and the plant availability was marginally higher than the annual average.

2. Details of H2SO4 mixing?

3. What experience with anhydrite formation in hemihydrate system?

Ans: The answer to the second and third questions can be conveniently combined. The formation of anhydrite is mainly affected by three parameters, the acid strength in P2O5, the sulphate content, and the temperature. During PRAYON's development of the PH series of high strength processes, particular attention was paid to maintaining a small but finite content of anhydrite. This technique enabled the best filterability conditions to be selected. Within PRAYON, the process of dilution of the sulphuric acid and the agitation to rapidly incorporate this stream has always been a pre-requisite of any process specification. In fact, the pre-mixing of sulphuric acid with slurry, return acid or water has been practised for many years by most process licensors as a means to prevent high localised concentrations of sulphate ion, in the dihydrate processes to prevent hemihydrate formation and in the hemihydrate processes to prevent anhydrite formation. This basic rule, strangely, has not been followed in certain cases. The proprietary design of Mixing-Tee offered by PRAYON has been developed to be as efficient as possible whilst reducing maintenance, scaling and corrosion problems that can be associated with this type of device.

Q - P. Tiberghien, Rhône-Poulenc, France

You mention two ways of improvement of filtration rate:

1. Agitation of the attack reactor in GELA (ISAF unit).
2. Better homogeneity of the temperature between the attack reactor and the flash cooler (5° C to 3° C in Middle East and Freeport).

Does that mean that these 2 parameters are much more important than the use of multi-compartment attack on the gypsum filtrability?

Is the main advantage of multi-compartment attack reactor its great ability and easiness to switch from one type of process to another? (DH <--> HH <--> HDH)?

Ans: In all PRAYON phosphoric acid processes (dihydrate or hemihydrate attack), for each step of the reaction and crystallisation process, we generally control the constant value of the process parameters by three distinct flows:

- the "pumping" flow in each compartment (maintaining a high level of incorporation of reagents and/or homogeneity in each individual compartment by the selection of the appropriate design of agitator in relation to the objective).
- the control of the temperature of reaction by a circulation flow through low level flash cooling system. (The delta T will be of about 2° C to reduce supersaturation and maintain the best crystallisation conditions for calcium sulphate).
- the control of conditions for each specific process step can be controlled by adjustment of the recirculating slurry flow through all compartments. (This flow can be selected to assure a homogeneous state or to maintain finite gradients in sulphate concentration or temperature as desired).

The optimum conditions for the complete reaction of the phosphate are not necessarily those that give the best crystallisation, this depends on many factors relating to the properties of each individual phosphate, and it is this feature of flexibility of the multi-compartment reactor that enables it to be tuned for each phosphate or even each process route.

The number of compartments selected for any specific project depends among other factors on the total volume of the reactor and the level of flexibility that is desired for each specific case.

The PRAYON reactor technology (process + equipment) was designed in relation with a flexibility such that it could treat a large range of qualities of phosphates and in more recent times, this flexibility has enabled it to be adapted without significant modifications from one process route to another. The main objective in all cases is to produce a high quality of acid and a stable calcium sulphate at the same time, with an optimum recovery.

In summary, the ability to select the point of introduction of the reagents, to adjust the sulphate and temperature gradients by means of the recirculation flow allows the multi-compartment reactor to promote the overall optimal conditions to be selected for each phosphate be it in dihydrate or hemihydrate mode.

TA/90/20**Project and Results for a 500 MTPD Urea Plant Revamp
J.L. Hernandez , A. Rodriguez, Fesa Fertilizantes, S.A., Spain****Q - E. Groensmit, Kemira BV, Netherlands****What is your experience with the acoustic prill heads? Less dust? Reliability?****Ans:** We have reached a more uniform distribution of the grain size, with less dust in the product.

Till now, they have proved to be completely reliable in operation and we don't need to shut down for cleaning or maintenance.

Q - A. Conradsen, Norsk Hydro, Norway**In your paper, you state the increased capacity of 50% with the same prill tower dimensions.****1. To what degree did the installation of acoustic granulators influence this capacity increase?****Ans:** As far as we can see, the use of the acoustic granulators, have not influenced substantially the increased capacity in our plant.**2. Can you give a short description of the acoustic granulator technique?****Ans:** The induced vibrations in these showers promote a more uniform distribution of drops size, together with long, trouble free, operating periods. No obstruction or build-up of them.**3. Is this technology/equipment commercially available?****Ans:** Yes. This equipment was furnished by Toyo Engineering, during the revamping of the urea plant.

TA/90/21 **The New Fertilizer "Calcourea"**
A. Crispoldi, A. Fogli, Enichem Agricoltura, S.p.A., Italy

Q - B.K. Jain, FAI, India

Comment

For nitrophosphate plants, calcourea appears to be a good option, however, in wet land/flooded paddy cultivation, there would be more nitrogen loss through leaching. Also, calcourea will have to be suitably coated because of its low critical humidity.

Question:

Out of the 3 production routes (listed in Enclosure 1), which process gives the best product?

Ans: The best product is given by the "dry process" consisting of the following steps:

- a. Dehydration of calcium nitrate up to 0.1% water.
- b. Dissolution and reaction of the obtained $\text{Ca}(\text{NO}_3)_2$ with melted urea (160°C).
- c. Granulation

Q - F.B. Larsen, Kemira Danmark, Denmark

In Enclosure 2, you have a formula for calcium nitrate showing a small amount of ammonium nitrate:

1. Does it mean that "Calcourea" also has a content of ammonium nitrate? and if it has - how much?

Ans: Yes, it means that "Calcourea" contains some free ammonium nitrate and its percentage is in the range 1-3%.

Q - R. Perander, Kemira Oy, Finland

Where does the calcium nitrate come from and which is the quality (field or horticultural grade)?

Ans: The calcium nitrate is produced by reacting calcium carbonate with nitric acid. The obtained solution is about 80-82% $\text{Ca}(\text{NO}_3)_2$ and 3-6% NH_4NO_3 . Ammonium nitrate is formed in the neutralization step of the excess of nitric acid used to attack CaCO_3 .

Q - G. Kongshaug, Norsk Hydro, Norway

1. Has Enichem decided to commercialize the urea calcium nitrate product?

Ans: It is obvious that Enichem intends to commercialize "Calcourea".

2. Which of the 3 process routes will you go for?

Ans: With reference to which of the 3 routes Enichem would go for, obviously we decided on the "Dry Process" because it provides the best product for quality.

3. Do you generate any ammonium-nitrate during production? (AN content in the product?)

Ans: Finally, about the AN content in the product, it comes only partially from urea hydrolysis which occurs in the reaction between $\text{Ca}(\text{NO}_3)_2$ added to urea melt (160°C). Released NH_3 and CO_2 react with $\text{Ca}(\text{NO}_3)_2$ giving NH_4NO_3 and CaCO_3 .

Q - A. Horkko, Kemira Oy, Finland

Could you tell me a bit more about the physical quality of your Calcourea for example:

- the granule hardness
- caking tendency or what kind of coating it will need

Something else I would like to know is how do you handle calcium nitrate in your process? As we know it is a very sticky and moisture absorbing product. And thirdly, how do you see the economy of your Calcourea? It contains less N than urea and needs an additional process to produce.

Ans: The hardness of granules (size 3 mm) obtained in the pilot plant is in the range 4.5 kg (crushing strength). This result is achieved by adding hardening agents to the process slurry before the granulation step. The coating treatment occurs in two steps by using CaCO_3 powder first and anticaking agent secondly. Given the low content of water in the product, given the efficient anticaking treatment we adopted, and in particular given the fact we bag all the product after the cooling step, no problems of caking have been observed. This was controlled by storing piles of bags for 5 months.

With reference to the problems of product moisture pick-up, this is avoided by performing all the operations after granulation (bagging included) under air controlled conditions.

The relative humidity of the air is maintained below the critical relative humidity of the "Calcourea". About the "Economy of the Product", it is true that it contains less N than urea and needs an additional process to be produced.

Nevertheless, the "Calcourea" once applied to the soil, gives only slight NH_3 emissions in comparison to urea. Furthermore, it is to be taken into account that NO_3 -leaching can be avoided by good farming practices (timing and amount of fertilizers).

Q - H. Hero, Kemira Oy, Finland

During process and storage, how do you maintain that no NH_4NO_3 is formed and form together with the "Calcourea" a wet mass?

Ans: I did not maintain "no NH_4NO_3 is formed in the process". I answered this question previously saying that "Calcourea" contains free AN. Furthermore, I explained that the product is not a wet mass because we work in air controlled conditions.

Q - J. Moeller Birkebaer, Kemira Danmark, Denmark

Have you tested other granulation methods than dry granulation for example, spouted bed or spherodizer?

What will be the maximal crushing strength of the Calcourea granules?

Ans: For the granulation step of the process, we found that the best technique is the "Falling Curtain".

With reference to the crushing strength of the product, I have given the figure previously.