

IFA Technical Conference

**Johannesburg, South Africa
30 September-4 October 1996**

WELCOME ADDRESS BY J.B. SKEEN President of The Fertilizer Society of South Africa

Ladies and gentlemen, it gives me great pleasure on behalf of IFA and the Fertilizer Society of South Africa to welcome you to the 1996 IFA Technical Conference.

This is the second IFA Conference held in South Africa in less than a year. Last November, we were honoured to host the Enlarged Council Meeting in Cape Town. This week, less than 10 months later, we are again privileged to host IFA's Technical Conference.

Representatives from no less than 101 companies and organizations from 41 countries have registered for this conference. For many it will be their first visit to our country, and given the high profile of South Africa in the world media in recent times, you have no doubt formed mental images of what you might expect to find. You may have heard or read that the political transformation in South Africa was nothing short of a miracle. It has also been said that South Africa has arguably experienced the biggest peaceful political revolution during the 20th Century.

Your stay will probably be too short to form a clear picture of a very complex society in transition: a society in which virtually everyone had to and still has to redefine their role in society. In fact, many South Africans are still unsure as to where the transition will lead in terms of individual and group expectations in the social and economic spheres. However, South Africans of different cultural and social backgrounds have the capacity and collective will to make the peaceful transition a lasting one. I trust you will experience some of this « spirit of South Africa » and take back with you lasting pleasant memories of our country.

Of course, without the dedication of Luc Maene, Secretary General of IFA and his staff, meetings like this would not be possible. To him, and his colleagues responsible for organizing the conference, I also want to extend a hearty welcome.

I wish you all a good conference, and a pleasant stay in Sandton, Johannesburg.

WELCOME ADDRESS BY C.R. GIBSON Chairman of the Technical Committee

Ladies and Gentlemen,

On behalf of the Technical Committee, I wish to welcome you to our 1996 Technical Conference in Johannesburg. As we approach the fiftieth anniversary of our Technical Conferences, I note with pride that this is the oldest continuous series of international conferences on fertilizer manufacturing technology. I have been told that we started ahead of the Fertiliser Society of the UK by one year and of the Fertilizer Industry Round Table by two years. In the process we have brought this conference to 24 different cities in 19 countries.

When the first Technical Conference was held in 1947 in Landskrona, Sweden, there were only 2.5 billion people living on Earth, less than half of the population today. At that time, more than 55% of the people were involved in farming. They used 2.6 million tons of nitrogen, 4.8 million tons of P_2O_5 and 3 million tons of K_2O , less than one-tenth of the current consumption. Since then, nitrogen consumption has increased by 30 times while both phosphate and potash consumption have increased by about seven times. We are proud to be associated with the production of nutrient products that feed the plants which feed the world.

Now that we are about to start the second half century of these beneficial conferences, we face many new challenges. As regards manufacturing, nitrogen production technology has reached a point of decreasing improvement in energy conversion efficiency. However, there are opportunities of incorporating new technologies and construction materials that would result in a more reliable and cleaner production with lesser turnaround. This is a considerable challenge. On phosphates, we face dwindling reserves of high grade ores and must be satisfied with medium or low grades in the years ahead. Consumers are putting greater emphasis on the quality of the product. Ways must be found to deal with the gypsum problem. We should not forget that phosphate reserves are finite. Potash is the only product that has an envious position as it does not face the production problems and environmental constraints associated with the other nutrients.

But there are other challenges ahead. In an era where every industry faces public scrutiny and increasing regulations, and we are certainly no exception, the industry should respond positively. I am pleased to note that our member companies are living up to these challenges and are keen to share their experiences with others. If we are to continue as a leading forum for fertilizer technology and related issues, I would urge you to contribute new and important findings to future IFA Technical Conferences. Help us to make future events even better than we have now. After all, we do have the most widely represented audience - from over forty countries - and participants are most receptive to innovations.

I wish to thank all those who have offered papers for this conference. I am grateful to the Committee who has guided me, and to the Editorial Board for their many hours of diligence. In this connection, I wish to single out Mr. K.C. Knudsen, our Vice Chairman for many years, for his outstanding contribution. My special thanks on behalf of all the participants go to Dr. Skeen of the Fertilizer Society of South Africa and his members for the kind invitation to come to this country and for the preparations made, to ensure that this conference is truly memorable. My thanks also go to Foskor and Mr. Nesor for the arrangements in connection with the field visit to Phalaborwa and to Sasol.

Before closing, let me recognize the excellent cooperation received from the Director of the Fertilizer Society of South Africa, Mr. Venter and its Conference Manager, Ms Vosloo in the organization of this event.

Thank you.

KEYNOTE ADDRESS

M. MacDonald, General Manager
Industrial Development Corporation of South Africa Limited and
Chairman of Foskor Limited

« A brief South African overview and some interesting developments in the local fertilizer industry »

I would like to welcome all the visitors to South Africa and say to you what an honour it is for us to be able to host the 1996 Technical Conference of the International Fertilizer Association.

As chairman of Foskor, our domestic supplier of phosphate rock, it is a privilege to be invited to give the keynote address today, and I would like to thank you for the opportunity.

As general manager of the Industrial Development Corporation of South Africa, I am also pleased to say that my organisation is currently promoting a number of interesting fertilizer related projects which I would like to tell you about.

But let me first give you, very briefly, a bit of background about this country on the southern tip of Africa:

- South Africa is the 24th largest country in the world;
- It is mainly arid with only 8% arable land, mostly marginal.
- Despite this, it is the largest food producer in Africa and a major exporter of maize, sugar, forest products, fruit and wool. It ranks as one of the world's seven net exporters of food.

AGRICULTURAL STRENGTH

	Production p.a.	World Rank
Fruit	1 mil t	3
Wood Pulp	1.8 mil t	5
Maize	7 mil t	6
Wool	60 mil kg	8
Sugar	2 mil t	15

- The country is a treasure house of natural resources producing a significant share of the world's minerals putting us in the same league as the USA, Canada and the CIS.

MINERAL TREASURE HOUSE

	World Production %	World Rank
Platinum	54	1
Gold	27	1
Vanadium	46	1
Chrome ore	34	1
Titanium	21	2
Manganese ore	9	4
Diamonds	12	4
Coal	5	6
Nickel	4	6
Iron ore	3	8
Phosphate rock	2	8
Copper	2	13

- We are also major beneficiators of minerals:

BENEFICIATION

	Production p.a.	World Rank
Ferro-alloys	1,4 mil t	3
Stainless Steel	1 mil t	5
Aluminium	640 kt	8
Steel	8,7 mil t	20

- It is the world's 16th and Africa's largest producer of electricity and has a first world economic infrastructure of railroads, harbours, roads, telecommunications and air services.
- The population of 45 million is the world's 24th largest with a GDP per capita of US\$3 200. Although this is high by African standards, it is way below the OECD average of US\$23 000, representative of a wide disparity of income levels within the country.
- The economy measured by GDP is the 29th largest in the world.
- South Africa is the hub of Southern Africa (comprising Angola, Botswana, Lesotho, Malawi, Mozambique, Namibia, Swaziland, Zaïre, Zambia and Zimbabwe), with which its infrastructure is closely linked.
- With a population of 146 million, Southern Africa is one of the world's largest unexploited markets. South Africa is accordingly the focal point of world interest in the sub-continent.
- In addition, Southern Africa contains one of the largest unexploited agricultural production capacity blocks in the world.
- The country's peaceful transformation to democracy recently has received world acclaim and is regarded as a model. President Mandela is generally acknowledged as one of the world's true statesmen.
- As regards the fertilizer industry, we are self-sufficient in nitrogen and phosphate, but import all our potash and the majority of our sulphur.
 - The installed fertilizer capacity is about 2,85 million tons with a local market consumption of plus minus 2 million tons per annum product (740 000 tons nutrients).
 - Consumption of nutrients were 371 000 tons N, 243 000 tons P₂O₅ and 134 000 tons K₂O during 1995.
 - Fertilizer exports reached 600 000 tons last year and phosphoric acid exports (including processed phosphates) were estimated at plus minus 460 000 tons P₂O₅, whilst direct phosphate rock exports reached plus minus 1 million tons.

On the economic policy front, the Government has committed itself to a market economy, combined with fiscal and monetary discipline. Steps have already been taken to reduce import tariffs drastically, and open up the economy to international competition. Foreign investment is encouraged and capital has started flowing into the country since the April 1994 elections.

The Government's greatest challenge is to redress the imbalance in the distribution of wealth and social services inherited from the apartheid era through accelerated economic growth.

My organisation, the Industrial Development Corporation, or IDC as it is called, is a state controlled development bank actively involved in promoting economic growth. We do this by providing risk capital for new developments, expansions and modernisations, and also by identifying opportunities for exploitation. Our objective is to encourage the private sector to invest and we often lead the way. We have total assets of US\$3 billion, have been profitable since inception in 1940 and are totally self-financing. We pay tax and also a third of our profits to the state in dividends. We are rather unique as far as development agencies are concerned.

One of the IDC's focus areas is national resource beneficiation, and over the years we have been very successful in this area. During the past few years we have been a key player in establishing a number of important projects:

- One of the world's largest aluminium smelters at Richards Bay with a capacity of 460 000 tons per annum currently being commissioned at a cost of US\$2 billion;
- A US\$220 million expansion of the world's largest dissolving pulp plant at Umkomaas on the Natal south coast;
- A heavy minerals mine and a 200 000 tons per annum smelter on the west coast employing a new DC are furnace slag smelting technique and costing US\$250 million;

- The world's largest single site stainless steel plant at Middelburg with a 600 000 tons per annum capacity, costing US\$800 million;
- A US\$110 million acrylic fibre plant in Durban;
- A 10 000 ton synthetic lysine plant based on fermentation of sugar - a new technological break-through - at a cost of US\$65 million, also in Durban;
- A US\$620 million expansion of the country's only aluminium rolling mill at Pietermaritzburg, currently under construction;
- A US\$1,5 billion steel mini-mill at Saldanha Bay with a capacity of 1,25 million tons per annum hot rolled coil, also under construction. It will be the first plant to use iron ore rather than scrap as input using the new corex direct reduction technology developed in this country.

These projects, will have a major impact on the country's export earnings and will contribute significantly to the national economic growth targets.

Moving the focus from the past to the future, however, what is interesting is that many of the new projects in the IDC's pipeline have an impact on the fertiliser industry and are mostly connected to Foskor.

As many of you probably know, Foskor produces 3 million tons per annum of high grade phosphate rock from ore with an average head grade of 7% P₂O₅, mined from a volcanic pipe at Phalaborwa in the north-east of our country, making it one of the unique phosphate deposits in the world. Some twenty one million tons per annum of tailings are discarded in the process. It is an equal partner with OTP of Togo in a 460 000 tons per annum phosphoric acid plant at Richards Bay. Four interesting projects are currently being evaluated with a view to adding value to Foskor's output:

- After many years of research a laboratory scale process was developed to economically extract the valuable minerals from Foskor's waste stream, namely alumina, magnesia and potash. Following on the encouraging findings of a feasibility study for a scale-up, it was decided in 1995 to build a commercial scale demonstration plant at a cost of US\$25 million, financed by the IDC. This was considered an essential step for producing commercial samples using the new technology. The critical magnesium sulphate separation process has been successfully proved and we now hope to prove the entire process during 1997. The objective is to establish a US\$1 billion world-scale plant producing 330 000 tons per annum of high-grade alumina, 300 000 tons per annum of magnesia, and 230 000 tons per annum of potassium sulphate. The project is a very exciting technology development promising enormous benefits for Foskor and South Africa. It would also make the country essentially self-sufficient in potash.
- Adding value to its phosphate rock has long been an objective for Foskor and since the dropping of sanctions against South Africa this has become a real possibility. Foskor together with local and foreign partners are well advanced on a feasibility study for the establishment of a 1 million tons per annum world-scale NPK plant for the international market, to be located either at Richards Bay or at Maputo. We hope to make a final decision on the project during 1997.
- The purified phosphoric acid market offers an interesting opportunity for added value, and Foskor is currently investigating the establishment of a joint-venture adjacent to the Richards Bay phosphoric acid plant. If this materialises, it will provide a valuable diversification, particularly during the periodical cyclical down-swing in the phos-acid market.
- The fourth project involves the expansion of Foskor's rock output by 1 million tons per annum to meet increased export demand as well as the requirements of a possible expansion of Fedmis' phosphoric acid plant adjacent to Foskor at Phalaborwa. This offers Foskor better scale of economy and the opportunity to diversify its customer base further.

A project which is not directly related to Foskor, is an envisaged 240 000 tons per annum world-scale zinc smelter being investigated by the IDC and Gencor, a major international metals group. The plant, to be located at the coast, will produce 380 000 tons per annum of sulphuric acid as a by-product which offers interesting potential for further phosphoric acid production or substitution of sulphur imports for present phos-acid production.

The fertiliser industry in South Africa is likely to see a lot of investment activity during the next few years which will present some demanding technical challenges. This will add to the viability of our industry and also add a further measure to the economic growth so desperately needed in this country.

I hope these few remarks will help give a better flavour to your South African experience and I trust that the conference and your stay in our country will prove to be thoroughly rewarding.

Thank you.

PLENARY SESSION: "GENERAL I"

Chairman: C.R. Gibson, Firstmiss Fertilizer Inc., USA

Rapporteurs: J. Jolly, Freeport McMoRan Resource Partners, USA
P. Smith, Société Chimique Prayon-Rupel SA, Belgium

PAPER 1 Future development of fertilization: The right NPK ratio, physical properties for even distribution, site specific farming/computer-directed fertilization
T. Kankaanpää, Kemira Agro Oy, Finland and M. Nielsen, Kemira Danmark A/S, Denmark

PAPER 2 Implications of precision farming for agriculture and the fertilizer industry
R. Conolly and C.R. Thorpe, Kynoch Fertilizer Ltd, South Africa

Q - K.G. Soh, IFA

How many comparative trials have you conducted between conventional and precision farming methods?

What is the average saving in fertilizers input compared with normal/ conventional recommendation? I saw the figure of about half a ton increase in yield per hectare. Is this result consistent with what you have obtained in your trials?

What is average increase in yield when compared with historical yields?

Ans: M. Nielsen, Kemira Danmark A/S

At our National Research Institute in Denmark, the last three years data indicate higher yields for wheat and barley. We also saved about 5 to 10% on fertilizer. We, therefore, have had higher yields, approaching 200 to 500 kg; and savings on fertilizer, especially for nitrogen, were around 5 to 10%.

Ans: C.R. Thorpe, Kynoch Fertilizer Ltd

Our experience has also been of higher yields, and about same order-of-magnitude. However, we didn't always have savings on fertilizer. As a matter of fact, sometimes we required additional fertilizer which was in line with the higher yields. In terms of fertilizer per ton of yield, we, however, do experience lower fertilizer usage.

Ans: M. Nielsen, Kemira Danmark A/S

Please allow me to make some additional comments. In Denmark, as well as in some of the other European countries, there will be a limitation on the maximum amount of fertilizer that we will be allowed to use. On a good land, if the yield is below the optimum level, we can then really utilize the satellite system. This is because we can take away fertilizer from an unproductive land and use it on a productive land. This way, we can have much better utilization of the maximum fertilizer usage limit. This also has a beneficial impact on the environment. Our studies show a reduction of about 10 to 15 kg in nitrogen leaching using site specific precision farming.

Q - P. Deigner, BASF AG, Germany

In Europe, we have a lot of small-size farms. To use the precision farming system, what would be the lower limit on land size? Also, could you please give us a rough estimate for the break-even size?

Ans: C.R. Thorpe

That's hard to say because it will depend to some extent on how intensive the crop is. For wheat, perhaps in the order of 5 or 10 hectares. However, for a much more intensive crop, such as fruit crops, one could apply this system to smaller size farms. So I guess it would depend on the intensity of the crop and the value of the crop. It may reach a point, say below half a hectare range, where it would probably be not sensible at all.

Ans: M. Nielsen

At the moment, the breakeven size would be about 150 hectare on the calculations that we have done. Based on fertilizer savings alone, we think that one can pay for this system for a 150-hectare farm. A yield measuring system costs approximately 120,000 Danish Crowns (USD 20,000). To this, you will have to add cost of other systems. So the total cost could be around 150-200,000 Danish Crowns for a currently available system. But in the future, when you are going to make a fertilizer map and you are going to use it the way we have recommended, you will have to think not in terms of a field but in terms of points in the field. You will be looking at your field in a different way. Each field will have a low point, high point, sandy soil, loamy soil, etc. Once you have defined all the characteristics of your field, using this system will be no more difficult than using your personal computer. Everything we need to know to utilize a GPS System is presently known. GPS systems are now being commonly used for ships and automobiles. I think it will be 5 years before this idea takes off because such a system will need to be mounted on each different tractor, in each different fertilizer spreader, in each combine harvester, and then adjusted to each other. In Europe, I think we already have over 300 combined harvesters with yield measuring systems.

Q - R.J. Tricklebank, Kemira Agro UK, United Kingdom

Data shows that up to 50% of farmers don't even calibrate their spreaders. How do you reconcile the use of this new GPS technology with the gains that can be achieved with more basic calibration improvements?

Ans: M. Nielsen

I think that was a good question because with a 20% coefficient of spreading, which some calculations have shown, it would be better to adjust your spreader and to buy a good fertilizer than to proceed with site-specific farming. Because utilizing precision farming requires a well calibrated spreader, a good fertilizer and complete knowledge of what you are doing, it is almost essential to know the basics of farming before one can proceed to use advanced fertilization techniques.

Ans: C.R. Thorpe

Perhaps if I can add to that: we believe that as agricultural subsidies diminish, the better farmers will slowly displace the poorer farmers. This is already happening. The farmers who don't farm efficiently and who are poor farmers are being bought out by better farmers. This process is always taking place. So I believe it is important to develop systems that can help the better farmers improve their skills and get even better.

Q - L.K. Rasmussen, Kemira Agro Oy, Denmark

I have a question to both gentlemen. How are you going to satisfy in practice the need for the N, P and K level in the different spots in the field?

Ans: M. Nielsen

For algorithms we have made in Europe, it's quite normal to have two applications for cereals. Using an NPK fertilizer with the same ratio as we used in the cereal removed the year before, we prepare a yield map for phosphorus and potassium. So we go out directly after nutrients with low leaching properties and use exactly the amount of P and K used the year before. We also use a small amount of nitrogen as first application. In second application, we go after nitrogen. For this second nitrogen application, we can not utilize the yield map. It's very difficult to use a yield map because if you have high organic matter, then you can achieve high yield with limited amount of nitrogen. So the yield map can be very confusing for nitrogen. Then we have other algorithms. In the first application, i.e. P and K, we adjust only for very sandy soils where we have leaching. We will adjust for potassium and use a little higher potassium on very sandy soils.

Ans: C.R. Thorpe

It's a good question because it is not easy to vary the N to P ratio as you are traveling across the field. Our equipment has been adapted so that when we are applying liquid fertilizers, we can vary between a trace element solution on the one hand and a liquid fertilizer on the other. That's easy

because you can put a small tank on the applicator for the trace elements. We have been looking at the possibility of having two large tanks on an applicator, one with an N-rich material, one with a P-rich liquid and varying the ratio in that way, but we have not done that yet.

PAPER 3 Physical properties of fertilizers and spreading: standardization, regulations and manufacture
F. Samec, Grande Paroisse S.A., France

Remarks: J.R. Polo, IFDC, USA

We congratulate Dr. Samec for the very important work his organization is conducting in trying to come up with standard ways to determine the physical properties of fertilizers. The information on physical properties included in the 1979 Fertilizer Manual was not intended to give procedural details for carrying tests, but only to convey information on what physical properties are and how they affect the performance of fertilizers. A manual dedicated to physical properties has since been prepared by IFDC, which we will make available to Dr. Samec to assist him in his work. This manual is also available to others who may request to IFDC. The Fertilizer Manual is being re-edited, and the new version will be available in 1997.

F. Samec

If I may add to this one, the fertilizer manual of the IFDC, if it is available, my advice is try to get hold of it. It's a book that has been out of print for the last 15 years, and those who have it consider it a treasure.

J.R. Polo

With regard to the fertilizer manual, we also recognize that it is a rather old document and we are working on the new one which will hopefully be out in the first quarter of 1997.

Remarks: P.W. Yelverton, The Fertilizer Institute, USA

Regarding regulations of physical properties I would like to make one comment. About 10 years ago, as you know most of the North America manufacturers adopted a system of voluntary guidelines. I think that it is an appropriate alternative to expanding regulations of physical properties and I believe it has been very successful within North America.

Q - P.W. Yelverton, The Fertilizer Institute, USA

Are there significant differences at this point between your work and the Canadian work which have been adopted in the U.S. primarily with Jean Chaval who I know you're familiar with?

Ans: Thank you for the question. I didn't want to bore you with details and I have just mentioned all the work done in the U.S. and Canada. I believe you are talking about size guide number and the uniformity index and this is one of the most successful ways of having a bulk blend which does not segregate. Provided you follow the rules in size guide number and the uniformity index, I think that would be sufficient. Unfortunately, this is only for the fertilizer manufacturers. The problem is with the regulatory matters. You can not enforce controls before you produce the material. You can only enforce control on the finished material and therefore the size guide number in the case of regulatory affairs would be of no use. The fact is that you have to have something which can control the final product and this is why today, the Commission seems to be going in the direction of granulometry and says that if you want a bulk blend, it has to have a D50 above something and if you don't want it to segregate, it has to have a granular metric spread below something. Because both of these, when you have a sample of the finished product which is put in the market, you take the sample, you seal it and you can check. You can't do that with the size guide number. I have talked this over with Mr. Jean Chaval on the phone at least 3 to 4 times. I am still expecting him to come to Europe because he has a trip planned, and I want to have a meeting. But this won't happen until the end of the year. So I think we can all progress on the work that has been done in the U.S. and Canada and on the work we are now doing in Europe.

Q - A. Van Brempt, Kemira SA/NV, Belgium

- *Is the dynamic angle of repose also a good indication of the flowability of the fertilizer? If yes, one could consider a graph, indicating the expected flow rate as a function of this angle of repose, more reliable and easier to define.*
- *Also, I believe fertilizer producers should accept some minimum requirements on sieve analysis in order to guarantee a good spreading.*

Ans: Diplomatically speaking, this is a very good question. Now the answer to this question, in my opinion, is very difficult because we have done no work on the correlation between the angle of repose and the flowability. If you ask for it, we may try but I'm not sure what will be the outcome. In my opinion, the angle of repose is a way of measuring something which can be useful in storage, in calculation of amount of storage you need, and partly how the materials flows. Is there a relationship between the angle of repose and the speed of flow through an opening? Frankly I'm not sure. Unfortunately, variations in the angle of repose are quite small and perhaps Mrs. Kankaanpää would concur with me that despite the rather very low spread we have found in the flowability measurements, the flowability measurements are still more reliable than the measurements of the angle of repose. Because angle of repose would be between 26 and 35 or something like that, it is not very precise.

Remarks: A. Van Brempt

Our concern is not to add additional measures to classify fertilizers. Fertilizers producers may not be too happy with more regulations concerning the seed analyses spectrum, but I believe that we have to follow the demand of the market to assure that our fertilizers can be spread out as it should be and there the seed analysis spectrum is very important. I am expressing my personal beliefs. Thank you.

Q - M. Nielsen, Kemira Danmark A/S, Denmark.

We all want correct application of fertilizers in the field. In Europe, there is a trend toward mandatory fertilizer planning. In Denmark, farmers are fined for discharging 15 kg of nitrogen in excess of the regulated levels. The fine can be as much as \$45 per kg of nitrogen. This further reinforces the need to correctly spread fertilizers in the field. So, in your opinion, what is the most relevant activity to raise the quality of spreading at the field level?

Ans: Another diplomatic good question! The most important properties, in my opinion, would be the granulometric spread and the D50. These would be the most important properties. Humidity is another important variable because I do not believe that the rate of flow for the same fertilizer will remain systematically constant when ambient relative humidity is changing from 60 to 90%. With a critical fertilizer humidity of 80%, there is something bound to happen if you spread a fertilizer which readily picks up water. If you spread it in a dry atmosphere or if you spread it in a dry climate, you shouldn't have problems. However, fertilizer which easily picks up moisture and it is spread at atmospheric conditions of 90-95% humidity, it is bound to cause problems. The flow will not be the same. I know this does not completely answer your question, but this is how I feel on the subject.

Q - N. Louizos, Drapetsona Fertilizers, Greece

I would like to have a comment by Dr. Samec about compacted fertilizers, their physical properties, and any specific classification they belong. Also, are there any specific regulations regarding their physical properties and their safe handling?

Ans: As far as I know, and I'm taking that limitation, there is absolutely no differentiation between compacted fertilizers and other fertilizers. Thus, in my opinion, compacted fertilizers are not very much different from compound fertilizers. In a compacted fertilizer, just as in a compound fertilizer, you have in each grain approximately the same NPK ratio. So whether you call it a compound or compacted fertilizer it doesn't matter. You can, of course, use different compacted materials to make bulk blends. So I would say, and as far as I know, there is no special regulation going to come up from the Commission to regulate the compacted fertilizers. However, the market may or may not pick up the fact that compacted fertilizers, whatever they are, do not behave the same way as prilled fertilizers. That's all I can say. I hope that answers your question.

Q - M. Larouci, Institut Mondial du Phosphate, Morocco

We know that ground phosphate can be usefully applied in acidic soils. In this regard, are there any standards with respect to products such as ground phosphate rock?

Ans: I would say that rock phosphates used directly as fertilizers and other single nutrient fertilizers are already in the statute books of the CEN (Comité Européen de Normalisation). In the last meeting of the CEN Technical Committee 260, it was agreed to put out standard for denomination and specification of single nutrient materials which are not regulated for the moment by the European community in order to have a common reference point. A standard is now being prepared and currently it is in the CEN inquiry stage. What does it mean? It means that if any member state has comments on the draft standard, this is the time to make the comments.

Remarks: C.R. Gibson, Firstmiss Fertilizer Inc., USA

I would like to take Chairman's privilege to make one observation. It seems to me that bulk spreading by centrifugal force would become obsolete. Due to the precision required to place fertilizer in the future, both the rates and placement will have to change continuously as you move across the field. This means that you will have to have a much more precision type of applicator than the spreader.

PAPER 4 NPK quality problems and improvement measures
Y. Al-Yami and V. Jayaraman, SAFCO/IBN AL-BAYTAR, Saudi Arabia

No response received to the questions forwarded.

PLENARY SESSION: "GENERAL II"

Chairman: J.B. Skeen, Kynoch Fertilizer Ltd, South Africa
Rapporteurs: M. Newton, Kynoch Fertilizer Ltd, South Africa
 R. Rabie, Omnia Fertilizer Ltd, South Africa

PAPER 5 Best Available Techniques in European legislation and the position of the European Fertilizer Industry: A follow-up
 R. Bauer, EFMA and T.K. Jenssen, Hydro Agri Europe, Belgium

Q - P. Mertens, Kemira Agro Pernis BV, Netherlands

How can we ensure that the member states actually enforce the limits mentioned in the document?

Ans: It is a requirement of the E.U. that all member states implement the directives. Actual experience has not always been positive. A case in point is the Seveso directive where the enforcement in some countries has been less enthusiastic than in others. But it is better to have an E.U. directive that is intended to be implemented than not to have one at all.

Q - Is EFMA or IFA going to take a proactive approach in obtaining standardised methods for analyzing emissions?

Ans: B. Christensen, Kemira Agro Oy, Denmark

In the BAT booklets you will find references to the analytical methods used. Stack analyses are particularly difficult and there is room for further standardisation.

Q - J.D. Crerar, United Kingdom

What is the scope & content of the BAT document?

Ans: EFMA have so far discussed ammonia and nitric acid. I do not have all the details on the E.U. document, and will supply a written answer. There is, in the directive, a paragraph stating that the Commission must establish an exchange forum where all these BAT systems will be put down in writing or in computer form. The process got so complicated that the commission has asked not to take existing plants into account. This has caused quite an uproar, and it is expected that they will change their minds. The process will take a long time and it is not certain that there is enough money available to make it work.

PAPER 6 Improved training of process workers for the South African fertilizer industry
 G.C. du Plessis, Indian Ocean Fertilizer (Pty) Ltd and J.W. Lotz, Department of Education of South Africa, South Africa

Q - Anonymous

Is the time spent in training considered as normal working time and paid as such, or is it on a free basis?

Ans: The workers already employed will continue in their normal jobs and will study in their own time, mainly through "distance education". There are many cases where in service training occurs. Workers are taken from their jobs for a day or two or a week and trained and developed. This training is in house and not necessarily accredited to an external training board or system.

Q - B. Christensen, Kemira Agro Oy, Denmark

After training it would be expected that more responsibility would be given to the lower level operators. Can you tell us what increased responsibility is given to lower level workers after completion of the training programme?

Ans: The people will only take full responsibility for the work they are already doing, instead of relying on support from supervisors or technical experts as they do now. They will have the opportunity to develop to higher levels and to take on more responsibilities.

Ans: *B. Bredenkamp, Sasol Fertilizers, South Africa*

South African fertilizer manufacturers accept that in the past operators had insufficient opportunities. They are now working to rectify the situation in an accelerated mode.

PAPER 7 **Organo-mineral fertilizers: A new concept towards sustainable fertilizer use**
N. Louizos, Drapetsona Fertilizers and K. Rettos, Synel S.A., Greece

Q - *T. Kankaanpää, Kemira Agro Oy, Finland*

If you do the composting in the fertiliser plant, how do you control the smell produced by the composting? Have they had complaints from the public?

Ans: The composting, which takes 20 to 30 days, is done at a distant site. The product is then brought to the fertilizer factory by road transport. There are some odours produced during composting but because the site is far from the people there have been no complaints.

Q - *How do you ensure that there are no health hazards? Are the temperatures during fermentation high enough everywhere in the manure to ensure that Salmonella, Newcastle disease and other bacteria are all killed?*

Ans: During fermentation the temperature rises from 45°C to 70°C. At this temperature most of the pathogens are killed. All the tests done have shown that pathogens are absent. These tests are repeated again and again. There is plenty of time during the process to do frequent checks to ensure that there are no pathogens present. In the fertilizer plant the temperature is raised to 100°C to 110°C in the granulator and in the dryer.

Q - *Even assuming that the chicken manure has a zero or negative cost, the fermentation process must be relatively capital intensive. Can you give us an idea of the cost of the organic nutrients as delivered to your granulator?*

Ans: I am not prepared to answer in detail. If it replaces filler, the organics are cost effective. They do contain some NPK, but the value is not so much in the NPK as in the organic matter.

Q - *You are indicating a maximum of 20% poultry manure in mineral fertilizer. On what basis is this 20% calculated? Allowing for nitrogen losses during processing, you will find it difficult to reach 1% organic nitrogen in the final product. There is a proposed EU standard for organo mineral fertilizers which, if I am well informed, specifies a minimum of 1% organic nitrogen.*

Ans: The proportion of organic raw material is 100 to 200 Kg per ton of fertilizer. This raw material contains 3% to 5% of nitrogen. Even if you have a 15% loss during processing, this is only 15% of 3%.

Q - *L. Scheers, BASF AG, Belgium*

Have you done tests on thermal stability? This could be a dangerous product, if it decomposes.

Ans: No. We have not done tests on thermal decomposition, but have not had any problems. We do not use ammonium nitrate, only ammonium sulphate.

Q - *F.V. Samec, Grande Paroisse SA, France*

More a comment than a question: The European community, through DG III has undertaken to establish regulations regarding organic fertilizers in Europe. CEN Technical Committee 260 has been mandated to

provide a report proposing an outline or draft for regulations for organo-mineral fertilizers. The safety aspects of these fertilizers are already set out in several European Directives. The commission would like to issue the directives for organic and organo-mineral fertilizers at the same time to ensure a level playing field.

Q - M. Nielsen, Kemira Danmark AS, Denmark

On what crops have the fertilizers been used, and have you details of the responses.

Ans: The initial quantities of fertilizers were distributed by the local farmer's co-operative. It was applied to plant species that would not be harmed even if there was pathogenic bacteria present.

Q - M. Larouci, IMPHOS, Morocco

Animal manure is known to contain high concentrations of heavy metals. Have you studied this aspect?

Ans: We have analysed the original material and the final fertilizer for heavy metals. We found traces, well below the accepted limits. We do not think that this is a problem.

Q - T. Koivumäki, Kemira Agro Oy, Finland

You argue that "continuous fertilisation year after year enriches the soil and improves its physical and mechanical characteristics". If you use 10% to 20% organic material in fertiliser and the application rate is 500 to 1000 kg/ha, the organic material addition to the soil is 5 to 20 g/m². Do you have any proven evidence that this small amount of organic material has any effect on the soil, either in the short or the long term?

Ans: No extensive large scale tests have been done. Small scale experiments have been carried out. The benefits of organic material are generally known.

Q - C.R. Thorpe, Kynoch Fertilizer (Pty) Ltd, South Africa

Are there any reasons for the fermentation step in the treatment of the chicken manure, apart from destroying pathogens?

Ans: The main reason is to destroy pathogens. It also produces a homogeneous product.

Q - M.S. Kachere, Zimbabwe Phosphate Industries Limited, Zimbabwe

The nutrient concentration of the final product is comparatively low. How would this affect its use in a developing country where the soils are low in nutrients? In Africa we have very high temperatures. Would this not cause decomposition of the humus?

Ans: This would require investigation. A general answer is not possible. Experiments would be necessary.

PAPER 8 Ecological and economical aspects of the BASF nitrophosphate process
T. Meyer and R.E. Nitzschmann, BASF AG, Germany

Q - A. Kossir, CERPHOS, Morocco

1. What is the concentration of phosphoric acid obtained in your case compared to the sulphuric acid route?

Ans: The BASF process does not produce pure phosphoric acid as an intermediate. The process results in a mixture of phosphoric acid, rock phosphate and nitric acid similar to the mixture used in the "Mixed acid" process. The water content of this nitro-phosphoric acid is significantly reduced by the separation of calcium nitrate tetrahydrate, so that a further evaporation of water is not necessary.

Q - 2. *Is the separating out of calcium sufficient at 0°C? In some publications, they indicate -5°C.*

Ans: The crystallization temperature is determined by the CaO:P₂O₅ ratio, i.e. the nutrient concentration and P₂O₅ water solubility in the product, which should be achieved. Also the nitric acid concentration and the rock phosphate grade effect the crystallization temperature. For high analysis products, cooling temperatures down to -8°C may be necessary. Normally, temperatures from 0°C to +5°C are sufficient. Example: normally NPK 15+15+15 requires a temperature of +3°C.

Q - 3. *When you produce Ca(NO₃)₂ you inevitably coprecipitate the impurities which are present in the CAN. This is detrimental to your process compared with the sulphuric route because in this case, we can always purify the acid before using it.*

Ans: This question cannot be answered generally. Normally, the impurities from the rock phosphate are no problem for the Odda-process. Worldwide available rock phosphate grades can be used. Concerning for example, cadmium, it is economically not reasonable to remove it in the Odda process. There are already developments in progress to remove cadmium directly from the rock phosphate.

Q - 4. *What is the energy consumption of the process?*

Ans: Typical consumption figures for example for the production of 1000 Mt/day NP 22:22 are (without nitric acid production, with export of lime and AN): Electric power 193 KWh/t product, steam 370 kg/t product, fuel oils 218 MJT: product. It should be mentioned that generally the cooling energy for crystallization can be provided by the evaporation of the amount of liquid ammonia, which is needed in the neutralization of the NP-acid and for the synthesis of ammonium carbonate.

Q - 5. *Does the product necessitate a fine grinding of the phosphate?*

Ans: No. If the particle size of rock phosphate does not exceed 4 mm. Worldwide usual specifications are sufficient.

Q - J.D. Crerar, United Kingdom

Statement: *Conclusions from economic evaluations are applicable only to the basis selected. BASF selects a basis to favour the nitrophosphate route. It lacks flexibility with regard to product analysis, and I think urea is more economic where agronomically acceptable as well as being more convenient to transport than ammonium nitrate.*

Q - *What is the minimum N to P₂O₅ ratio achievable in the BASF nitrophosphate process, compared with N free TSP and 0.2 to 0.4 to 1 for ammonium phosphates via the phosphoric acid route?*

Ans: We naturally chose a basis for our economics that makes our process look good, but in any case you can do the calculation for yourself. You can get the investment price and can do the calculations for your own situation. Prices for sulphur, phosphate rock and lime will vary from place to place, but in our opinion, the end result will be the same. The minimum N to P₂O₅ ratio is about 0.7.

Q - *What is the price of nitrogen in the form of CAN in the Far East, compared with urea, currently about US\$ 480 per ton of N?*

Ans: Of course, if you transport CAN from Europe to the Far East it will be expensive compared with urea made in the Far East. But with low gas prices in the Far East and with the conversion efficiencies we quote, you should achieve comparable prices with CAN.

Q - *What is the NO_x level in your gaseous discharges to atmosphere?*

Ans: We regularly achieve 400 Mg/Nm³ and have done better than this in some plants.

Q - A. Van Brempt, Kemira SA/NV, Belgium

Can you always valorise your calcium carbonate? In some situations you could have an excess of calcium carbonate. How do you handle this?

Ans: We have never had a problem with excess calcium carbonate. Most people with a nitrophosphate plant have an additional ammonium nitrate plant to increase the production of CAN. The lime could be used for soil pH correction or can be given away to cement manufacturers. We do not dump it anywhere in the world.

Q - Can you give an answer to the rapidly growing demand from the market for more "Tailor Made" fertilizers with low phosphorous content?

Ans: This concerns the flexibility of the process. At one extreme, we have a plant in India which produces a single product, N:P: 20-20-0 all the year round, and a plant in Antwerp producing 60 formulas and over 100 product changes per year.

Q - S. Corton, Fertiberia, Spain

You use an ion exchange unit. If nitric acid and ammonia are used as regenerants to obtain an ammonium nitrate solution, what concentration do you get?

Ans: We no longer use this process. As far as I remember the concentration was approximately 20% to 30%.

Q - Do you have experience of these units? Do you know of licensors?

Ans: No. Our only reference is our own plant.

Q - Are there any adverse safety aspects of using nitric acid for regeneration?

Ans: You need to use special ion exchange resins, but the problems are solvable.

Q - Have you experience of reverse osmosis for this process?

Ans: I do not personally have this experience. I suggest you contact Mr. Scheers of BASF Antwerp. He has experience of reverse osmosis plants.

Q - P. Suppanen, Kemira Agro Oy, Finland

Do you have to have an ammonia plant to supply the CO₂, or could you use another source, such as a power station? Are there other methods of converting Ca(NO₃)₂, not using CO₂.

Ans: You can use carbon dioxide of a lower concentration if necessary. I do not know of any other conversion method, which does not use CO₂.

Q - J.R. Polo, IFDC, USA

What is the water solubility of the phosphate in the end product?

Ans: On the degree of cooling, up to 80% in water, and as high as 99% in citric acid.

Q - T. Koivumäki, Kemira Agro Oy, Finland

It is difficult to compare two different process from an ecological point of view. One fairly objective way to make the comparison is to do a Life Cycle Analysis for the same production capacity. Have you made this kind of study, and what has been the result?

Ans: No we have not done that yet.

Q - B. Christensen, Kemira Agro Oy, Denmark

In the nitrophosphate process, how do you overcome the need for sulphur in the NPK products without using expensive raw materials such as ammonium sulphate and potassium sulphate. In the mixed acid process it would be obvious to use zero value gypsum.

Ans: If you need sulphur as a nutrient you must, of course add potassium sulphate or sulphuric acid. We believe that this is better than using gypsum.

Comment from B. Christensen: In Denmark, gypsum is regarded as water soluble.

Q - *Due to sulphur deficiencies in NW Europe we see a clear trend away from traditional CAN to specialised NS products based on ammonium nitrate and gypsum. How does the nitrophosphate process fit into this new generation of fertiliser products?*

Ans: BASF believe that this need can be met by the use of sulphuric acid, not gypsum.

Q - *If a comparison of capital and operating cost were made with the mixed acid process with rock digestion where about 50% of the P_2O_5 in 20-20-0 comes from the rock, the nitrophosphate process would look less favourable. The nitrophosphate process produces NPK and CAN while the mixed acid process produces NPK+S and AN+S. Please comment.*

Ans: It is not essential to combine the two processes. You do not have to produce CAN. You can produce ammonium nitrate as a product or you can put it into the NPK mixtures. It is correct that the mixed acid route can look cheaper, but whatever variation of this route you use, you have the problems of producing phosphoric acid and gypsum.

Q - P. Mertens, Kemira Agro Pernis, Netherlands

In the nitrophosphate process all the contaminants in the phosphate rock report to the product. In the wet phosphoric acid process, there are stringent controls on the gypsum. This makes it necessary to select the phosphate rock carefully. As long as there are no similar regulations governing NPKs, the nitrophosphate process is not so constrained. As a consequence soil contamination may occur. How do you view this aspect?

Ans: The components you refer to occur in low ppm levels and they are diluted in the final product. In areas where there are very stringent regulations we may have to select special grades of rock.

PAPER 9 Effect of $NH_4NO_3 \cdot K_2SO_4$ particle size on granulation efficiency in production of NPKs using the pipe reactor process
Wang Jin Ming, Sino-Arab Chemical Fertilizers Co., Ltd, China

No question.

PAPER 10 Tailor made liquid NPK fertilizers - a unique production, distribution and application method
M. Hochberg and Y. Nitzani, Deshen Gat, Israel

No question.

PAPER 11 Phosphate fertilizers of Russia
V.V. Babkin, JSC AMMOPHOS and A.A. Brodsky, AOOT NUIF, Russia

No question.

PARALLEL SESSION: "NITROGEN I"

Chairman: K.I. Al-Obaidat, GPIC, Bahrain
Rapporteurs: A.S. Al-Shahri, SABIC, Saudi Arabia
 A. Cadet, Fertil, United Arab Emirates

PAPER 12 Kellogg advanced ammonia process (KAAP): review of commercial experience
 J.R. Le Blanc and S.A. Knez, The M.W. Kellogg Technology Company, United States

Q - S. Stalin, SPIC, India

1. *It has been mentioned that the catalyst has been tried under different space velocities and different hydrogen to nitrogen ratios. It may be clarified whether these factors were deliberately manipulated to monitor the performance of the catalyst or whether the performance was checked whenever there was any change in the plant with respect to these factors.*

Ans: The wide range of space velocities and H/N ratios that the KAAP catalyst has operated over at Pacific Ammonia was as a result of the normal operation of the ammonia plant and under planned conditions. Initially when KAAP was installed in 1992, the ammonia plant operated at about 544 MTPD. Following the addition of a new front end based on the Kellogg Reforming Exchanger System (KRES) in 1994, the KAAP synthesis loop has operated as high as 860 MTPD. The H/N ratio at the Pacific Ammonia plant, because of use of a nitrogen wash unit, is easily adjusted. Normal operation of the synthesis loop is about 2.0 H/N ratio.

When KAAP was put into operation at Pacific Ammonia, we closely monitored its performance. During the 1st 6 months of operation, Kellogg conducted a number of planned tests to demonstrate the operation of KAAP over a wide range of conditions. Of course, in the life of the ammonia plant, sometimes these changes were planned.

2. *How is the mechanical strength of KAAP catalyst? What is the trend in increase in pressure drop?*

Ans: The mechanical strength of the KAAP catalyst is good. As I mentioned, we took samples of the KAAP catalyst after more than 3 years in service and found the physical properties after more than 3 years in service and found the physical properties to be within the original specification. The KAAP catalyst can operate at higher pressure with no deleterious effects.

3. *Grassroot plant is designed for 90 bar. But a retrofit can be anywhere between 140-210 bar. Can a catalyst designed for low press (90 bar) operate on high press (140 bar) in case of a retrofit? Is it difficult to control the reaction?*

Ans: For grassroot applications, the KAAP ammonia synthesis loop operates at around 90 bar. In retrofits, such as the Pacific Ammonia - KAAP operates at the prevailing pressure, around 140 bar. The KAAP catalyst has no limitations on higher pressure operation.

Q - J. Ohri, IFFCO, India

1. *What is the cost of KAAP catalyst and increase in cost during last 4 years?*

Ans: The KAAP catalyst being precious metal based does cost more than the iron catalyst on a per unit volume comparison. But when we compare it on a performance basis, because the KAAP catalyst is up to 20 times more active than the conventional catalyst, it actually costs less.

2. *Do you use same drive for Syngas compressor and ammonia compressor and how is the performance?*

Ans: The KAAP ammonia plant is designed with a tandem drive synthesis gas and refrigeration compressor. The low pressure operation allows Kellogg to employ this cost saving feature. Tandem drive systems have been excellent performers in other process industry applications.

3. What is energy consumption for 1350 MT/day ammonia?

Ans: As I mentioned, with the KAAP technology, we can achieve very competitive energy consumption in the range of 6.5 to 6.9 Gcal (MT). This in addition to our 10% capital cost savings. Of course, site specifics will dictate the energy level achievable.

4. Do you use molecular sieve in syn loop?

Ans: For the KAAP ammonia plant, Kellogg still uses molecular sieve drying of the make-up gas. The molecular sieves are located upstream of the single case synthesis gas compressor.

Q - S. Nand, FAI, India

For retrofitting a high pressure loop with KAAP, there may not be much energy savings. Is the objective only to increase the production capacity by incorporating KAAP reactors?

Ans: The KAAP technology is applied to retrofits for both capacity increase and energy savings. Of course, the economics of the project became more attractive at the high capacity increase achievable. For a current KAAP retrofit project, capacity will be increased by about 25%, while for another KAAP retrofit project, a 10% capacity increase is being specified. Site specifics dictate the overall approach for the KAAP retrofit project.

Q - L.J. Connock, British Sulphur Publishing, United Kingdom

What has the onstream factor of the Pacific Plant been since installation of KAAP? Have there been any unscheduled shutdowns and what caused them?

Ans: We do not maintain onstream information on the Pacific Ammonia Plant, since this is a retrofit application of KAAP and KRES but both KAAP and KRES have been 100% available since their respective start up.

Q - V. Stepanov, SC "Concern Stirol", Ukraine

1. Energy consumption per ton of ammonia?

Ans: With KAAP technology, not only do we achieve a 10% capital cost savings over conventional technology, but we can also design the ammonia plant for low energy, depending on site specifics, in the range of 6.5 to 6.9 G.Cal/MT.

2. Level of conversion in the syn. converter?

Ans: We achieve about 21~22% ammonia at the outlet of the KAAP reactor.

Q - H.R.L. Tobing, PT Petrokimia Gresik, Indonesia

What is the increase in capacity (without KRES), after KAAP was implemented?

In case the syn. reactor is contaminated by CO, up to 4000 ppm, what is the minimum time required for re-activation and what precaution shall be taken?

In KRES, do we have to use air enriched by O₂? What is the alternative in case the oxygen generation plant is not available or has limited capacity?

Ans: Name plate capacity was 544 MTPD, when KAAP was installed in 1992, the plant continued to operate at about 544 MTPD, since it is based on purge gas from the methanol plant. When the new KRES based front end was installed in 1994, the Pacific Ammonia plant increased production to over 800 MTPD.

After the CO contamination, both the KAAP and magnetite catalysts recover full activity shortly after fresh synthesis gas was reintroduced.

The same precautions to avoid poisoning that are used for the conventional iron catalyst are also employed/recommended for the KAAP catalyst.

In a grassroot application, the Kellogg Reforming Exchange System (KRES) is designed to operate with air enriched to about 30% mol oxygen; the enriched air supplies the required heat balance around the KRES system and provides the nitrogen required for ammonia synthesis. Air separation plants are very reliable, and are typically available 100%.

Q - S. Stalin, SPIC, India

1. *Table II indicated that KAAP synthesis catalyst is active at H/N ratio of 2.3. Normally, synthesis loops are designed for H/N ratio of 2.8 to 3.0. If KAAP retrofit is used, it has to operate under the normal ratio only and not at 2.3. Does it affect the activity of the catalyst?*

Ans: One of the fundamental differences between the KAAP catalyst and the conventional iron catalyst is the KAAP prefers to operate at a lower than stoichiometric hydrogen-to-nitrogen (H/N), that is, it has higher activity, relatively, at less than 3:1 H/N

For grassroot applications, Kellogg designs the ammonia synthesis loop to operate at about 2:0~2:5 H/N ratios.

For retrofit applications, Kellogg would evaluate the baseline operation of the overall ammonia plant to assess the optimal H/N ratio. This has typically ranged from 2.0~3.0. Please note, magnetite actually prefers to operate at about 2.7~2.8 H/N. Lowering the synloop H/N ratio, typically only requires a minor adjustment to the front end of the ammonia plant, i.e., operation with slight excess air, which could benefit the reforming operation at higher loads.

Again, site specifics would dictate at which H/N ratio Kellogg would design KAAP for the retrofit application. In any case, the KAAP catalyst exhibits considerably higher activity than the conventional iron catalyst regardless of H/N ratios.

2. *As the new KAAP catalyst has been in service for quite some time, will you please inform about the trend of DP across the converter?*

Ans: The KAAP catalyst has now been in service at Pacific Ammonia for more than 4 years. The pressure drop trend is stable, with the radial flow KAAP reactor operation at a relatively low pressure drop.

3. *If KAAP converter is retrofitted to an existing converter, the operating pressure of the loop is about 140 bar (Refer Table II). However, if this is employed in a grassroot plant, the operating pressure is about 90 bar only. It is presumed that the catalyst is developed having a 90 bar option in mind. If this catalyst is used in a retrofitted converter operating at 140 bar and above, will it result in temperature excursions because of higher operating pressure than designed?*

Ans: In a retrofit application of the KAAP technology, a KAAP reactor is added downstream and in series with the existing ammonia converter. Because the KAAP reactor attains high ammonia concentrations (20+%), even at the higher operating pressure, equilibrium limits the temperature rise across the KAAP beds.

PAPER 13 Production of ammonium sulphate from utility boiler waste gas
G.N. Brown, GE Environmental Systems, United States
 Paper presented by Mark Dutchess

Q - A. Van Brempt, Kemira SA/NV, Belgium

1. *Will it be possible to obtain a premium (and how much) for your granular quality (1-3.5 mm) compared to the standard quality (0.1-0.8 mm) and is the premium sufficient to cover compacting costs?*

Ans: In our economic models prepared to date for developed nations, the premium of \$30-\$50 per short ton for granular over standard grade easily pays for the capital price of the compaction system. In fact, the payback period is usually in 1-2 years.

2. *Is there a difference in the market value (pricing) between your granular quality and the real granulated quality available in the North American markets?*

Ans: We have not seen a price difference between the compacted product and the "grown" large crystalline product. They are treated as equal products.

3. *You apply a coating as we have seen from your flow sheet. Can you communicate what kind of coating agent you are using?*

Ans: In the compaction process, we do not apply a coating as a coating. We do dissolve the surface of the final product and then dry it quickly to improve the product hardness. Down stream of the compaction process, we have experimented with coating the particle surface with mineral oil and wax. Both have been used equally effectively.

4. *Slow release treatment for the granular quality have been mentioned, through "addition" of polymers.*
 a. *We presume that the treatment consists of a polymer coating. Is this correct?*
 b. *Is this CRF already available for the market, and on what price compared to the untreated ammonium sulfate?*
 c. *Could you give some indication on the controlled release performance of this product?*

Ans: Currently, the system does not apply a slow release agent of any kind. We have the ability of adding it, however, at various process points, either as a coating or mixed into the compacted crystal. This has not yet been done on a commercial scale. The price that such a product would demand is relative to its application. The most interest has been expressed by those fertilizer companies servicing the golf course industry. We are not prepared to discuss potential product characteristics without first proving their ability to perform the function desired without detrimental impact on system operation.

Q - S. Stalin, SPIC, India

1. *The boiler flue gas contains CO₂ also. Does it not interfere in the reaction to form (NH₄)₂CO₃?*

Ans: The North Dakota installation has approximately 30-40% by volume CO₂ and no effect of any has really be identified.

2. *Can this system be integrated using sulphuric acid plant? (to the stack gases from H₂SO₄ plant)?*

Ans: We are currently evaluating this potential for a couple of customers. Preliminarily, it appears very feasible and economic. The concern remains dealing with the sulphuric acid mist created from the presence of SO₃ in the inlet gas.

3. *The S content in fuel is said to be 5-7%. Does it mean that heavy furnace oil fired boiler flue gas can be the input?*

Ans: This process is ideal for scrubbing 5-7% sulphur oils. What we have observed is that with liquid fuels, the presence of a higher concentration of sulphuric acid mist at the scrubber inlet can result in a plume opacity problem.

4. *What is the effect of heavy metals such as V (vanadium) present in the fuel oil?*

Ans: The effect of vanadium in the fuel oil will be to catalyze the SO_2 to SO_3 conversion in the boiler. Production of large amounts of SO_3 in a wet FGD system produces sulphuric acid mist which can cause scrubber off gas opacity of 40-60% or higher. This is a result regardless of the scrubbing agent.

5. *What is the opacity of stack?*

Ans: The process developed by GEESI minimizes the formation of the ammonium sulfate particulate by minimizing the quantity of free ammonia in the gas phase. The experience at the North Dakota Installation is that the opacity can be controlled by control of pH in the scrubbing vessel. In recent tests, the opacity of the scrubber exhaust gas remained unchanged with no ammonia flow and pH levels of <3.0 through the standard operating pH levels of 5.0 to 6.0. Above 6.0, the opacity increased dramatically by formation of an ammonia and ammonium salts plume.

6. *The flue gas will contain soot during soot-blowing operations of boiler. Can this system continue to work during this period also?*

Ans: At the North Dakota installation, which is also a liquid fuels fired boiler (5-8% S by design), soot blowing takes place frequently. The soot particles are caught for the most part in the scrubber (~70-90% removal). Depending on the amount of soot relative to sulphur, the soot may be acceptable to leave with the final product. At the North Dakota Installation, we have installed a filter press which removes the soot from a slip stream and minimizes the quantity in the final product.

7. *Whenever ammonia is used for removal of sulphur oxides from gases, the stack has an appearance of thick white plume because of finely divided particles of ammonium sulphate/ammonium sulphite. What is your experience?*

Ans: This species has not been found in any commercial or pilot work we have done to date.

8. *Can this process be deployed to treat flue gas from boilers burning fuel oil with 5-7% of sulphur?*

Ans: Heavy furnace oil may be ideal. See answer to question N° 3.

9. *What is the effect of CO_2 present in the flue gas under process chemistry?*

Ans: At the North Dakota installation, the opacity of stack is not accurately measured because of the water vapour associated with a wet stack. In addition, the presence of sulphuric acid mist results in a measured opacity of 40-60% regardless of the ammonia flow and the operating pH of the scrubber within the normal operation band. See answer to question N° 5.

10. *Whenever fuel oil fired boilers are sootblown, the soot generated also comes along with the flue gas. Will the soot interfere with the process?*

Ans: Yes, see answer to previous question N° 6.

11. *Can this process be integrated with sulphuric acid plant which emits SO_2 and SO_3 to atmosphere?*

Ans: See answer to question N° 2.

Q - B. Christensen, Kemira Agro Oy, Denmark

Have you considered to simplify the wet oxidation stage by using catalytic oxidation before the scrubber?

Ans: During the development stage of the process, the kinetics of the oxidation with and without catalyst were evaluated closely. The net effect was that the process was not enhanced substantially enough at high dissolved salts concentrations to warrant any use of a catalyst.

Q - J.D. Crerar, United Kingdom

1. *Will there be a heavy wet plume from the fired absorber?*

Ans: Without reheat, there will be a vapour plume all year round from the saturated flue gas existing the absorber.

2. *Should some of the "free" energy be used to reheat the final exhaust?*

Ans: Some energy from the flue gas can be used to reheat in the traditional way with the use of a gas reheater. But most U.S. installations do not use GGH's because of cost and maintenance of these systems.

Q - S. Nand, FAI, India

What is the lowest level of sulphur in coal when ammonium sulphate process can be viable?

Ans: The economics of this system are somewhat dependent on location relative to ammonia supply and by product/fertilizer outlets. Relative to other forms of SO₂ control, the ammonia system is more economic down to ~0.5 wt% S. This is a conservative analysis. For other conditions, the relative economics are difficult to assess, however, the cost per ton of ammonium sulphate produced is more attractive at higher sulphur concentrations than it is at lower sulphur concentrations. Since each system must be evaluated individually for relevant economic factors, the sulphur content of the fuel is only one variable which should be considered in the decision process.

PAPER 14 Reliability improvement in coal-based ammonia plants

M. Newton, Kynoch Fertilizer Ltd, A. Marneweck and G. Tennant, PGBI-Industrial Plant Enhancement, South Africa

No question.

PAPER 15 Reliability of operating an atmospheric ammonia plant and its improvements

Li Zhijian, Liang Xihui and Cui Huahui, China National Chemical Planning Institute, China

No question.

PAPER 16 Long-term experience with catalytic reduction of NOx

J. Vlasak and J. Scharf, Lovochemie a.s., Czech Republic

Q - J. Vlasak, Lovochemie a.s., Czech Republic

1. *Catalyst used?*

Ans: TU6-03-234-72 from Russia.

2. *Temperature?*

Ans: 230–240°C.

3. *Experience between selective and non selective catalytic reduction?*

Ans: Control of SCR is simpler and more comfortable.

4. *Quantity from absorption is 2000~2500 p.p.m.v. (NH₃/Nox. About 6 kg NH₃/MT NA).*

Ans: Outlet from absorption is 2000~2500 p.p.m.v. ----> CO NS.CCA 6 kg NH₃/MT NA.

Q - M.R. Barouni, Groupe Chimique Tunisien, Tunisia

1. *After a long experience with the two processes of selective and non selective catalytic reduction, can we make an economic comparison?*

Ans: No economic study has been achieved.

2. *Following figures 7, 8, 9 10, NOx contents have been relatively high until 1991/92. Do you have register real case of professional diseases provoked by NOx?*

Ans: No.

PAPER 17 Chimco catalysts

G. Christov, Chimco, Bulgaria

Q - S. Stalin, SPIC, India

Copper promoted HT shift catalyst CK-O3: Chimco guarantees complete absence of sulphur and the catalyst does not require desulphurization period. How are they eliminating sulphur during the catalyst manufacture? (All other manufacturers indicate presence of sulphur in the Cu promoted HT shift catalysts)?

Ans: The catalyst CK-O3 is the last modification of catalyst for conversion of carbon dioxide with water vapour, which manufacturing started four years ago.

The catalyst is completely sulphur free and does not require a period of desulphurization. The preservation of the primarily high activity for a long time, at the copper availability, requires a complete absence of sulphur. The high specific surface and the catalyst activity is a consequence of the possibility of valency change of the main component - iron, in the presence of copper.

For a big regret, I could not give more details regarding the technology of this catalyst.

PAPER 18 Revamp of urea plant to improve efficiency

A. Karsono, E. Anwar and M. Djohan Safri, PT Pupuk Sriwidjaja (Pusri), Indonesia

Q - S. Stalin, SPIC, India

1. *What is the modification done in hot water compartment of high pressure absorber cooler to accommodate increased heat duty of the plant after revamp?*

Ans: Hot water compartment of high pressure absorber cooler itself was not modified, but the original hot water passed into this compartment was changed with cooling water in order to accommodate increased heat duty of the plant after revamp.

2. *With increase in plant load by 50%, did you face problems in HP absorber? Was there any CO₂ slip? Did you change the packings?*

Ans: No problems faced in HP absorber, no CO₂ slip and no change of packing made.

3. *What were the modifications carried out to perforated plate of HP decomposer?*

Ans: No modifications carried out.

4. *Did you modify HP absorber cooler to handle additional heat duty?*

Ans: No modification carried out.

5. *Did you not consider acoustic granulators and additional air or chilled air for enhancement of capacity of prill tower?*

Ans: No.

Q - I.J. Ohri, IFFCO, India

1. *What is the material of construction of tubes in NEW ACES process of urea stripper? Inline since when? Any corrosion/loss of thickness in urea stripper tubes?*

Ans: No corrosion in stripper tubes experienced.

2. *Do you have hydrogen recovery arrangement from CO₂? And what is oxygen content in CO₂?*

Ans: No hydrogen recovery arrangement from CO₂ installed. O₂ content in CO₂ about 500 ppm.

Q - F. Graneli, Snamprogetti, Italy

1. *How much is the payback time?*

Ans: 5.5 years.

2. *33 million US\$ seem very high for 500 T/D more capacity, even though there was a benefit of the energy, but not for NH₃ consumption?*

Ans: This much investment does not appear too high.

Q - A. Karsono, PT Pupuk Sriwidjaja, Indonesia

1. *Number of shutdowns before and after the revamp?*

Ans: It is the same, because the main shutdowns are due to external problems.

2. *The problem due to external sources, is it the same for all the plants?*

Ans: Yes, all the plants have gas shortage.

3. *From where the equipment is coming?*

Ans: Spain or Korea.

4. *Prill size before and after the revamp?*

Ans: The same.

PARALLEL SESSION: "PHOSPHATE AND POTASH"

Chairman: D. Vorster, Foskor Ltd, South Africa
Rapporteurs: A. Bruhn, Rohphosphat H. Huckfeldt, Germany
 G. van der Linde, Foskor Ltd, South Africa

PAPER 19 Technology of production of NaCl and KCl using the method of crystallization
 V. Novoselov, Yu.N. Savvatin and G.L. Slobodkina, JSC « Sylvinit », Russia

No question.

PAPER 20 Experimental production of KCl and MgO utilizing carnallite as raw material
 E. Cekinski, E.F.M. Camargo and M. Danese, Institute for Technological Research,
 Brazil

Q - L.K. Rasmussen, Kemira Agro Oy, Denmark

Have you considered the use of $\text{Ca}(\text{OH})_2$ for $\text{Mg}(\text{OH})_2$ precipitation? That would give you a mother liquid with CaCl_2 which you could send to the sea. What will you do with the huge quantities of NH_4Cl ?

Ans: No, we did not make any tests with calcium. I don't think NH_4Cl will be a problem.

Q - Have you considered the production of MgSO_4 (Kieserite)? Many companies would like to see an alternative supplier of this material.

Ans: We have no experience with this material.

PAPER 21 Improved reactor control and increased filtration capacity by upgrading filtration equipment in a phosphoric acid plant
 G.C. du Plessis, Indian Ocean Fertilizer, South Africa and S. Kurowski, Process Filtration and Liquid Equipment S.A. (Profile), Belgium

Q - S. Stalin, SPIC, India

What are the modifications done of filter pans? More details like slope on each side and collection manifold area may kindly be provided. Photos are not very clear and hence clear prints may please be provided.

Ans: Modifications done on filter have been the replacement of existing 2 slopes pans by 3 slopes pans (see Figures 1 and 2 in the 1996 Technical Conference paper, page 173). In general, the surface increase is due to the fact that bigger cells with different shape and other minor changes could have been designed on these 2 filters. More details cannot be provided.

Q - J.D. Crerar, United Kingdom

What effect does the crystal habit modifier have on :

a) The shape and
 b) the size
 of the gypsum crystals?

Ans: The crystal habit modifier actually makes the crystals shorter. The needle shaped crystals then become shorter and also wider. The size of the crystals is also much more uniform. It depends on the amount of crystal habit modifier that you use and the control over that. Uncontrolled use can give you the opposite effect.

Q - Who is the designer of the new larger area pans?

Ans: The company, Profile, manufactured all the equipment discussed here.

Q - *The pictures of the cloth fixing method are not clear in the preprint. Can the presenters give some description?*

Ans: Unfortunately we have no picture of the cloth fixing method, but we have improved on this point. We now have a cloth fixation system that is done by clamps. No screws or bolts are used any more. We put a certain rubber with a certain shape around the cell and the clamp just falls into this rubber into a groove and the cloth is pinched between the clamp and the rubber. According to information, a cloth change takes about 10 minutes per cell.

Q - *What future plans does IOF have to further de-bottleneck the plant to more fully exploit the new filtration capacity?*

Ans: Our plant is limited by the sulphuric acid side and not by the phosphoric acid side, so we are focusing on increasing the capacity on the production of sulphuric acid. If we want to increase the capacity of the plant further, and we are evaluating that, we can increase the capacity of the reactor and reactor cooling and evaporation on the concentration side without having to increase too much on the filtration area.

Q - *P. Mertens, Kemira Agro Pernis BV, Netherlands*

Was the filter performance deviating from design from start-up, or did it worsen because of the declining mechanical condition?

Ans: Mister Chairman, the answer on that question I don't fully have. I was not involved with the plant from the original start-up. I know the plant achieved its production capacity but I don't know whether the other performance results that we indicated here were actually measured and compared. I know the plant from about 1987, the filter was then in a better condition than when we replaced the equipment. It was performing as I have described from 1987.

Q - *The filtration performance, I assume, is closely linked to the vacuum being obtained. To what extent did the performance improve because of the improved pan design and what was the contribution of the overhaul e.g. the improved vacuum?*

Ans: Filtration capacity was certainly influenced by vacuum. Often when we had a severe leak on the pan filtration was poor. Even when we had a good vacuum you had the problem that we had no control of the recycle to the reactor. We tried all sorts of actions to gain control of the recycle but it was just not possible.

Q - *Has the cause of corrosion been examined?*

Ans: Yes, the corrosion is caused firstly, by the filtrates in the filter and the filter acid. We also have high values of fluoride in the wash water and that can also contribute to the corrosion.

PAPER 22 Valorization of Taiba phosphate slimes by hydrocyclones D. Fam, Industries Chimiques du Sénégal, Senegal

Q - *T. Mehari, Compagnie des Phosphates de Gafsa, Tunisia*

What is the quality comparison between the merchant grade rock and the phosphate recovered from slimes?

Ans: In the merchant grade rock we have:

35,5% P ₂ O ₅	1,1% Al ₂ O ₃	0,8% Fe ₂ O ₃	6% SiO ₂
-------------------------------------	-------------------------------------	-------------------------------------	---------------------

while in the recovered slimes we find:

29% P ₂ O ₅	3,9% Al ₂ O ₃	2,4% Fe ₂ O ₃	18% SiO ₂
-----------------------------------	-------------------------------------	-------------------------------------	----------------------

Besides a strong increase of silica the ratio R_2O/P_2O_5 is notable which rises from about 0.05 in the merchant grade rock to 0.2 in the slimes.

Q - S. Saidi, Groupe Chimique Tunisien, Tunisia

1. The recovered slimes are rich in iron, aluminium and silica. What would be the chemical and industrial yield of an acid plant operating with and without the addition of slimes?
2. Could we have an indication of the analysis of impurities in the concentrated acid produced?

Ans: 1. Actually, we observe a slight reduction in chemical yield with the increase of the slime proportion added. For example, the following results have been obtained:

Phosphate rock without slimes	chemical yield:	94.67%
Phosphate + 10% slimes	chemical yield:	94.63%
Phosphate + 15% slimes	chemical yield:	94.42%
Phosphate + 20% slimes	chemical yield:	92.84%

This slight reduction in yield, however, is combined with an increased filter capacity.

Above the level of 20% of added slimes, we start to observe a more important reduction of the yield.

Ans: 2. The acid coming from attack-filtration is desaturated and thereafter decanted before being concentrated to 52-53% P_2O_5 . The level of dissolved impurities is measured by the following formula:

$$I = \frac{D - D_0}{D \times D_0} \times 167\%$$

D being the density of the acid produced

D_0 being the density of pure acid of the same concentration as the acid produced

Without addition of slimes, 6-7% of impurities are obtained and with slimes, this is 8-9%.

Comment from J. Gregorio Filho, Fosfertil SA, Brazil

Fosfertil is operating an installation for concentration of slimes at their Catalao GO phosphate rock mine. In this process, impurities are removed and the product is a good fine concentrate all < 325 mesh. Normally, the concentration plant discards the slimes, but now they are sold to SSP producers who can use them without the need for grinding.

Q - H. Hakim, Groupe Office Chérifien des Phosphates, Morocco

1. What is the influence of the utilization of slimes on the water balance of the reaction-filtration units?
2. What treatment do you apply to the fines of < 10 μ which represent the tails of the slimes recovery unit?
3. What is the date of commissioning of these units for slimes recovery?

Ans: 1. The phosphoric acid reactor is fed a phosphate slurry containing 60% solids. The merchant grade phosphate having a water content of 20%, the slurry is prepared by adding water. The enriched slimes have a solid content of about 50%. Therefore, with the utilization of slimes water for slurring is saved and the water balance of the reaction-filtration units remains the same with or without slimes.

2. The fines coming from the hydrocyclones first are treated in a settling tank "clariflux" for water reclaiming. The underflow of these settling tanks at a concentration of 150-200 g/l is pumped to a pond about 2 km from the plant. It was tried unsuccessfully to recover water from these ponds as evaporation is very high due to the large surface area of the ponds.

3. The first slimes unit treating 100 T/h of slimes expressed as dry solids were commissioned in 1987. A second unit of same capacity is under construction and is expected to start up during 1997.

Q - A. Kossir, CERPHOS, Morocco

When adding the slimes you are experiencing an improvement in crystallization. What could be the reason for this improvement?

Ans: In fact, an improvement in crystallization is observed. When using the merchant grade phosphate only, crystals of sea-urchin shape are obtained while with the addition of slimes parallel-epipedic crystals of larger dimensions are observed. This change of crystal form is accompanied by an increase in filterability of the slurry. This improvement is certainly due to the increase of aluminium in the blend.

Q - M. Larouci, IMPHOS, Morocco

1. *What is the origin of the slimes?*
2. *Why has attrition not been tried for a better separation of P_2O_5 in the hydrocyclones and possibly a flotation could also result in better recovery of P_2O_5 and a reduction of ferrites?*

Ans: 1. The mineral coming from the mine, after removal of flint and reducing the mineral to less than 25 mm, is treated in a preparation unit before flotation. This treatment is applied to reduce the mineral to between 40 and 800 μ . The slimes consist of the particles of less than 40 μ and contain on average 23% P_2O_5 - 10-12% ferrites - 25% SiO_2 . These contents depend on the particle size as is indicated by the granulo-chemical analysis.

2. Flotation trials actually have been run on the slimes residues but the results have been clearly less attractive than upgrading by hydrocyclones.

PAPER 23 Encouraging method of cadmium removal from phosphoric acid

A. Kossir and M. Ghayor, CERPHOS, Morocco

Q - J.D. Crerar, United Kingdom

1. *How much is the loss of P_2O_5 in the anhydrite / cadmium stream?*
2. *What is the fate of the calcium sulphate after the Cd is removed? Is it recycled to the phosphoric acid process?*

Ans: 1. The P_2O_5 content in the anhydrite is about 1 to 1.5%, most of which is recovered during recrystallization of the anhydrite into gypsum.

2. After recovery of cadmium from the anhydrite you obtain a much cleaner calcium sulphate than the phosphogypsum, from which the anhydrite was produced. This calcium sulphate can either follow the same fate as the phosphogypsum, or it can be recycled in order to adjust the solids content at the entry to the concentrator.

Q - G.C. du Plessis, Indian Ocean Fertilizer, South Africa

What is the temperature and the SO_4 content required in the concentration step for co-precipitation of Cd with $CaSO_4$?

Ans: The sulphate content is about 40 g/l in 30% P_2O_5 acid. The temperature should be about 80 to 90°C.

Q - J.L. Bovens, Kemira SA/NV, Belgium

When will the process be ready for industrial use and how much will the investment and operating costs amount to?

Ans: At the moment we are waiting for finance for construction of a semi-industrial pilot plant. The investment cost for an industrial plant can only be defined after realisation of the semi-industrial pilot plant.

Q - D. Fam, Industries Chimiques du Sénégal, Senegal

The classical concentration of phosphoric acid without cadmium removal yields an acid of 52-54% P_2O_5 while with cadmium removal a concentration of 56-57% P_2O_5 is obtained with a yield of cadmium removal in the order of 90-95%. Is there a correlation between acid concentration of 53-57% P_2O_5 and the yield of cadmium removal?

Ans: Cadmium removal is achieved by co-crystallization in the crystal lattice of the anhydrite. The crystallization of anhydrite based on gypsum requires a certain content of P_2O_5 and sulphates.

Q - A. Bourgot, Prayon-Rupel Technologies S.A., Belgium

What are the principal differences between the cadmium removal process developed by CERPHOS and the process presented by Mr. P. Becker during the IFA Technical Conference, held in The Hague, Netherlands in 1992?

Ans: There are various differences between the CERPHOS process and the Becker process:

- The Becker process intervenes outside the phosphoric acid production process as it uses concentrated acid, while the CERPHOS process intervenes during the acid production process (concentration stage);
- The Becker process utilizes phosphate as calcium source and precipitates a combination based on calcium, phosphate, sulphates...
- The Becker process makes use of a succession of reactors with agitators operated at a temperature of 120°C, which results in a very corrosive and abrasive environment.

PAPER 24 Beneficiation of Eshidiya low grade rock

A. Ghosheh, M. Al-Muthakar, A. Salameh and L. Dabbas, Jordan Phosphate Mines Co. Ltd, Jordan

Q - A. El-Houari, Groupe Office Chérifien des Phosphates, Morocco

When do you expect to start the mining of the south area of Eshidiya?

Ans: We have finalized the feasibility study, phase two, for which we go for finance in the beginning of next year (1997). It will be over two hundred million U.S. dollars. We aim to commence operation within two to three years from now.

Q - *Could you give us an idea about the percentage of cost increase of commercial rock due to the new investment made by JPMC?*

Ans: You need about a 100 U.S. dollars new investment for each ton of concentrate produced. This can give you an indication of what the cost can be.

Q - A. Van Brempt, Kemira SA/NV, Belgium

One would expect different Al_2O_3 values in the different ores A1 to A3, but all your Al_2O_3 values are the same. Can you explain this please?

Ans: You mean that there is no big difference in the Al_2O_3 content of the final concentrate? Because of the intensive agitation and attritioning we get rid of all materials relating to clay materials. This is a necessary step to successful flotation. This step essentially removes all clay materials relating to alumina. We thus end with very clean phosphate particles in the final concentrate. We can blend run of mine material, which is rich in alumina, with final concentrate to give the customer a blend with the required amount of iron and alumina.

Q - P.A. Smith, Société Chimique Prayon-Rupel SA, Belgium

You showed in one of the tables a P₂O₅ recovery of 91.7% on the flotation sections. Could you give a range of recoveries for each section and the overall recovery (Run of mine to concentrate)?

Ans: I cannot give you an exact figure in this regard. Actually it is a variable ore and I cannot give exact figures for each stage, but we know exactly what the flotation recovery is, because it is very important that we have a highly floatable ore.

Q - P. Suppanen, Kemira Agro Oy, Finland

My question is also about recovery. You have two systems: one with and one without flotation. What is the difference in recovery between these two systems?

Ans: Actually recovery is very important in terms of economy. The only way to recover A3 is by flotation. We choose the process not according to recovery but according to the need of the customer.

Q - You have coarse and fine materials. Do you have different flotation recipes for the two materials?

Ans: That is a very good question. The coarse flotation circuit applies to 0.5 to 1 millimetre with conventional flotation cells. This means that we also use the same cells even for the fine flotation. The flotation recovery is not the same for the two systems. The flotability of the coarse material is different, the reagent consumption is different, so this is a difficult area to work in. We could not go to column flotation because we believe there is not strong experience in this area. We can float unsized material in conventional flotation cells but the split between coarse and fine give a little bit more recovery.

Q - Y. Zahidi, Groupe Office Chérifien des Phosphates, Morocco

Can you give us a cost comparison between conventional conveyors and pipe conveyors which you are using now?

Ans: You have to add at least 50% more to your investment cost.

PAPER 25 Development of a method to beneficiate and purify sedimentary phosphate rocks with a carbonate matrix

B. Guedri and K. Lahmadi, Compagnie des Phosphates de Gafsa, Tunisia

No question.

PARALLEL SESSION: "NITROGEN II"

Chairman: P. Deigner, BASF AG, Germany

Rapporteurs: L. Scheers, BASF AG, Belgium

T. Meyer, BASF AG, Germany

PAPER 26 Regional shifts in nitrogen fertilizer production
J.R. Polo and G.T. Harris, IFDC, United States

Q - T. Koivumäki, Kemira Agro Oy, Finland

- *Can you see, that the similar development has happened in compound fertilizers?*
- *Can you see, if there has been any major structural changes between single nutrient and compound fertilizers?*

Ans: We have not yet followed up on NPK. We have distributed the request for information to many companies and will evaluate the data.

There are differences between USA and Europe. In the US, you have the phosphate in the south, Florida, natural gas in the center for ammonia and potash in the west. That is the reason for the production of single nutrient fertilizers and rather using bulk blends instead of NPK compound fertilizers. The future is difficult to predict. But regarding trends to global positioning systems and precision agriculture, I would expect an application of nutrients on the field according to the field nutrient map. This requires concentrated single nutrient fertilizer mixed on the field. Therefore, I would expect a worldwide tendency to single nutrient fertilizers. Exceptions are small or poor countries.

Q - I.J. Ohri, IFFCO, India

- *Comment on energy consumption of ammonia plants: new plants with a capacity of 1350 MTPD NH₃ operate with an energy level of 7.5-7.7 Gcal/MT ammonia in India. Naphtha based plants consume around 10 Gcal/MT ammonia. However, new plants under implementation in India will consume 7.1-7.2 Gca/MT ammonia based on gas.*

Ans: Yes, I agree, I wanted to say that a point is reached where it is very difficult and uneconomical to achieve a further major decrease in energy consumption.

Q - Li Zhijian, China National Chemical Planning Institute, China

- *China has the goal to become self-sufficient in N-fertilizers. If the goal is achieved, what is the effect on world N-fertilizer-market?*
- *Could you please analyze the future of N-market in the world?*

Ans: My field is not the market side. But as far as I know, China is more in the market of P than N, where China already has a relatively high level of self-sufficiency. Of course, as China consumes about one fourth of the world fertilizers, it will have influence on the prices. We already saw such effects in the past.

PAPER 27 Fertilizer ammonium nitrates: present concerns regarding regulations
F. Samec, Grande Paroisse S.A., France

Q - H.T. Kiiski, Kemira Danmark AS, Denmark

- *Thank you for the nice summary on the dangerous properties of AN. There is also another concern related to mixtures of AN and AS. They are not fully covered by regulations. In some cases, the process with crystalline AS causes low density, porous products, which may not pass the detonation test. Are these new types of fertilizers safe enough in relation to misuse?*

- *Are there any actions toward changes in regulations?*
- *Could it be better to change regulations and/or classification to be based on testing rather than on composition?*
- *What is the acceptable level of AS in straight AN-fertilizers and how much can be used as stabilizer in CAN?*

Ans: New regulations will never prevent a wilful misuse of AN-fertilizers. Should a discussion start on AN/AS-fertilizers, which I do not hope, I would recommend to look the German regulations and experience regarding transport and storage of such mixtures. They are based on investigations and experience derived from the Oppau explosion and mixtures admitted in aftermath have proven to be safe, nothing attributable to such mixtures having happened since. There seems to be no necessity in discussing new regulations or classifications and, as far as I know, there is no action right now. I would not recommend a classification based solely on testing methods since chosen methods may not fit existing products or worse, inventions or new product compositions. As an example, ammonia was classified as "very toxic" for the aquatic environment based on test carried out at pH between 6 and 8.5 and despite existing results, reclassification only as toxic was not accepted. This in turn means that if the same test were run at pH 8.5 on AS, AS would be classified as "very toxic"! As for the last question regarding acceptable levels of AS, I have to look it up in my papers. There is no simple answer and another example can be given: according to EU regulations, AN can be mixed with secondary nutrients such as sulphur, calcium, magnesium or sodium but I would very strong advice against mixing AN with powdered sulphur since you get a highly explosive mixture.

Q - R. Rabie, Omnia Fertilizer Ltd, South Africa

- *Can AN be stabilized in terms of cigar burning or explosion by addition of a chemical (i.e. MAP)?*
- *What do you consider a safe pH for NPK fertilizer containing Cl and AN?*
- *Is not it true that 4% organic matter is circa stoichiometric correct for complete reaction, but AN with less oil (0.5-1%) is more sensitive?*
- *Then 4% max oil absorption regulations make sense?*

Ans: Straight AN is not subject to cigar-burn; it melts before sustainable ignition is reached and does not have the necessary chloride component. Stabilization against "explosion" has been advocated many times for many addition products and even patents have been taken as shown by the developments of the Oklahoma case. I do not believe that there is an efficient additive capable of preventing a purposely engineered explosion. Regarding the pH question for chloride- and AN- containing NPKs, I would say that low pH (<4) would accelerate the decomposition rate of a slow cigar-burner but, on the other hand, I have no example of a fast burning composition being made non-burning by changing the pH to more than 6. Furthermore, the trough test results are different whether the same trough is used the "UN way" (horizontal) or the "DIN way" (vertical). As for the 4% limit of oil retention imposed by the EU regulations, this is well below the stoichiometric amount needed for ANFO explosives and the absorption test is carried out on sensitized material since it has been subjected to thermal cycling. As far as I know, the force of explosion plotted against the amount of oil shows a maximum which corresponds approximately with the stoichiometric amount of oil calculated for a complete "combustion" of AN but this does not indicate anything regarding sensitivity of a 4% mixture versus a 1% mixture. Having discussed this with specialists, there is no reason to believe that a 0.5-1% oil mixture is more sensitive than a 4% one. However, past literature indicates that there may be a maximum for sensitization of pure ammonium nitrate with 1-2% organic matter, see Cook & Talbot (1951), Haid & Koenen (1952) and Brinkley (1958).

Q - P.J. Parekh, Tata Chemicals Limited, India

At low temperature, AN is quite stable. The presented data are all at high temperatures. Is not that an important point that is not covered in regulations?

Ans: Yes, it is an important point since even at "low" temperature, AN losses ammonia and becomes more acidic, therefore more sensitive. There are regulations that prohibit sending AN to storage when above a certain temperature, and are also manufacturers' recommendations.

PAPER 28 Towards more uniform methylene-urea, slow-release fertilizer
J. Luhtala and T. Koivumäki, Kemira Agro Oy, Finland

Q - LI Zhijian, China National Chemical Planning Institute, China

MU can be produced with granulation processes. Is it possible to produce MU with prilling tower process, are there experiences?

Ans: No, we have no experience with prilling MU containing fertilizers.

Q - R. Nitzschmann, BASF AG, Germany

- *How do you handle the formaldehyde in the offgases?*
- *What is the final formaldehyde content in the offgas?*
- *How exactly can you control the chain length of the oligomers?*

Ans: We have not measured the offgases from the dryer. But in the offgases from the reactor, where you expect the highest formaldehyde emission, the formaldehyde was below detection limit. Formaldehyde level in the control room was higher due to tobacco consumption. Methanol is emitted via stack gases, but is calculated below governmental limits. The chain length is controlled by U/F molar ratio, pH and temperature.

Q - V. Stepanov, SC "Concern Strol", Ukraine

Were there problems observed related to the connection of oligomeric chains together, resulting in formation of polymers?

Ans: No, not under these process conditions. Oligomers keep a chain length under 10 urea units.

Q - F. Samec, Grande Paroisse SA, France

- *Can you specify the costs of MU-nitrogen?*
- *Did you compare MU-fertilizers with sulphur coated urea?*

Ans: The costs of MU-nitrogen are about 5-10 times the costs of ordinary nitrogen units. But slow release N-fertilizers are used for special applications, not as a general N-source. We do not have comparable data from sulphur coated urea.

PAPER 29 Modifications done on synthesis gas compressor drive turbine
A.B. Tipnis, V.B. Waghulde, T.M. Bhagwat and H.N. Gakhar, Rashtriya Chemicals and Fertilizers Limited, India

Q - P.J. Parekh, Tata Chemicals Ltd., India

- *What is Siemens' experience on their previous supply to other plants?*
- *When the rotor length was reduced, how was it connected to the compressor and L.P. turbine?*

Ans: As per our knowledge, many of the users have faced similar problems with dash 3 machines. M/S Siemens' have therefore discontinued dash 3 design and their modified dash 2 versions only are now available.

By increasing both side coupling spacer length, the connection has been carried out.

PAPER 30 Co-production of ammonia, methanol and/or power for export
S.E. Nielsen and Ib Dybkjaer, Haldor Topsoe A/S, Denmark

Q - S. Stalin, SPIC, India

- *Is there a mix ratio which determines how much of CH₃OH can be produced? Is it possible to produce NH₃ and CH₃OH in any ratio?*
- *When CH₃OH is integrated, air flow has to be adjusted to get the proper CO:CO₂ ratio for the CH₃OH converter. Does it not lead to high methane slip exit by reformer and less of steam generation in reformed gas boiler? It means low energy efficiency in NH₃ plant.*
- *How do you really estimate the costs of production for CH₃OH and NH₃, because the production is thoroughly integrated?*

Ans: You can play with the figures. There is, in principle, no limit to the ratio between CH₃OH and NH₃ production. Typically 20-40% of the feed is taken for the CH₃OH production.

Yes, the air flow is adjusted to fulfil the requirements of the ammonia synthesis loop. The influence on the energy efficiency by this adjustment will be negligible, since the effect on the methane slip and steam generation will more or less balance out.

We calculate the cost for one ton of product. We do not distinguish between NH₃ and CH₃OH.

Q - A. Alexandron, Haifa Chemicals Ltd, Israel

- *What is the criteria to build a grass route co-production of NH₃/CH₃OH regarding relative market prices, investment, etc.?*
- *In the revamps you showed the NH₃/CH₃OH ratio was 3:1, in the grass route plant it was 6:1?*

Ans: Earlier one criteria has been the very high market price for CH₃OH. But also the revamps were optimized to as high a CH₃OH production as possible. Local conditions dictate these things. If you have requirements of CH₃OH in relatively small amounts, it is a cheap way to produce CH₃OH in a co-production plant. The co-production plant was invented as a add-on unit for revamps and has comparable investment costs per ton of CH₃OH as world scale plants only producing CH₃OH.

In the grass roots plant described, the ratio between CH₃OH and NH₃ was adjusted to customers' requirements.

Q - V. Stepanov, SC "Concern Stirol", Ukraine

- *Would you share your opinion to the following matter: which is more effective - a revamp of NH₃ plant with CH₃OH co-production, in which case we lose some market in NH₃, or maybe a construction of a grass route CH₃OH facility?*
- *What is the payback period for the plant in Malaysia?*

Ans: In the jobs I presented, you will see that the CH₃OH production is fairly limited and a construction of a new grass roots CH₃OH plant with this capacity would not be competitive. It would not be economic for this small capacity.

I cannot answer the second part of the question. This is a matter between erector and client. I can only refer to what has been published in the newspapers.

Q - T. Phillips, Fertecon Limited, United Kingdom

- *What is the additional capital cost of CH₃OH compared to a straight 1350 t/day NH₃ plant in the case of Malaysia?*
- *Can you confirm that the Malaysian plant can go up to 1350 t/day NH₃?*

Ans: I am sorry, but I do not have the figures, but there are some additional costs due to the equipment which handles the methanol.

Yes, without CH₃OH production, 1350 t/day NH₃ is possible.

PAPER 31 Stamicarbon's new process urea 2000plus™

K. Jonckers, J. Mennen, J. Meessen and W. Lemmen, Stamicarbon BV, Netherlands

Q - H.R.L. Tobing, PT Petrokimia Gresik, Indonesia

What is the advantage/disadvantage, mainly in investment costs, utility/raw material consumption by using pool condensor type compared with falling film high pressure carbonate condensor?

Ans: With the pool condensor you achieve about 8% reduction of investment in the synthesis unit, that is something of 3% reduction of invest costs for the whole plant. That is not very much. But with the pool reactor, the investment will be 20% lower, regarding the reactor sector, or 10% lower, regarding the plant. This is quite a considerable reduction of investment.

Q - Li Zhijian, China National Chemical Planning Institute, China

- *Should the H₂ removal unit be eliminated?*
- *What is the oxygen content in CO₂?*
- *Do you have any modifications for the high pressure scrubber? Is the chamber at the top of the scrubber needed?*

Ans: No, it should not be eliminated. With this system, we wash out all NH₃ from the inerts before venting them. If we do not do that, we would run into environmental difficulties. And we cannot allow H₂ in the synthesis for safety reasons. We and our mother DSM would not allow that.

The O₂ content is around 0.6 vol % in the CO₂. That is independent of the reactor design.

In the scrubber, there is no more heat exchanger. Previous scrubbers had an additional heat exchange for condensing. We place a heat exchanger close to the scrubber and can avoid the heat exchanger in the scrubber.

The chamber at the top is still there, although we had no explosions, since H₂ is removed from CO₂. The chamber still remains for safety precautions. In case something happens, no high pressure equipment should be damaged.

Q - A. Alexandron, Haifa Chemicals Ltd, Israel

What is the next breakthrough, which you prepare to the next IFA conference, or are you considering implementations of this innovation to another process?

Ans: I think we have been quite innovative for the past 2 years. Right now, I have nothing new. We are thinking about putting the reactor on the ground, reducing even more the height of the plant. But that is future.

Q - F. Granelli, Snamprogetti, Italy

- *How can Stamicarbon be sure that in the pool reactor, specially at the opposite side of the condensor, the vapours containing NH₃ and CO₂ coming from the stripper can be totally condensed and that carbamate can be dehydrated to urea with a good conversion? In fact, the head of liquid in this part of equipment is very low compared with traditional vertical reactors, the residence time goes nearly to negligible time and there are no trays.*
- *From the pictures, it seems that no inspection to the pool reactor is possible?*

Ans: The reactor is equipped with a series of baffles. They are not overflow baffles. They insure a horizontal flow through the reactor. Additionally, there are spargers for each compartment to assure the correct distribution of the gas. So, the retention time is sufficient. We are sure that the design with a reactor temperature of 168°C is right.

The vessel is of course accessible. There is 0,5 m space around the bundle and there is enough room for inspection. There is a problem when down-scaling the reactor below 1000 ST/day, then 0,5 m space can hardly be achieved. So you might install a larger vessel than necessary for that purpose.

Q - F. Granelli, Snamprogetti, Italy

Do you use oxygen or air in the H₂ removal reactor?

Ans: We use air. In one revamped plant, we used enriched air. These are lots of advantages in using O₂, but we regard safety first priority and would not want to use O₂ in our plants.

Q - M. Djohan Safri, PT Pupuk Sriwidjaja, Indonesia

Based on conventional reactor, level in reactor should control by hand through level indicator. For pool reactor, is there any level indicator or not? If any, what type of level indicate will be used?

Ans: Also in this reactor, there is a level control of the last reactor compartment.

Q - V. Stepanov, SC "Concern Stirol", Ukraine

Are there changes in the energy consumption? Compared to previous projects, is the consumption of NH₃ and CO₂ different from state of the art?

Ans: No, there is no difference in energy consumption. NH₃ and CO₂ are used in stoichiometric amounts, which we cannot improve. In revamps though, we were able to reduce the energy consumption from 1200 to 800 kg steam per ton urea.

PAPER 32 Upgradation of ammonia-urea complex at IFFCO-Kalol for sustained high level performance
J. Ohri, IFFCO, India

Q - G.K. Rao, Nagarjuna Fertilizers & Chemicals Ltd., India

You mentioned that you would be changing the solvent in CO₂ removal system. What solvent are you going for?

Ans: We change to MDEA.

Q - S. Stalin, SPIC, India

Even after replacement of mixed feed coil and superheaters, the required design heat recovery has not been obtained. Do you consider any further improvements?

Ans: The basic reason is that the original Kellogg design was intentionally under designed. We tried to improve the design within our possibilities.

Q - M. Djohan Safri, PT Pupuk Sriwidjaja, Indonesia

In 1995-96, your plant can achieve 115% of rated capacity. What kind of modifications did you implement on your urea plant?

Ans: We got in contact with the original designer, Stamicarbon. There were very few major changes: the urea reactor trays were modified, the compressor was replaced, the buckets in the prilling tower were changed, which still is a bottleneck. We are thinking about putting up a small additional granulation plant.

PARALLEL SESSION: "ENVIRONMENT AND SAFETY"

Chairman: K.C. Knudsen, Kemira Danmark A/S, Denmark

Rapporteurs: J.L. Bovens, Kemira SA/NV, Belgium

T. Kankaanpää, Kemira Agro Oy, Finland

PAPER 33 Re-engineering of safety system in a large fertilizer complex

A.N. Aggarwal and R.H. Khamkar, Gujarat Narmada Valley Fertilizers Company, India

Q - S. Nand, FAI, India

1. What is the criteria for classifying accidents into minor and major ones?

Ans: Major accidents: lost time is more than two days. Minor accidents: lost time is less than two days.

2. Is it an internationally accepted way of classifying the accidents or is used only by your company?

Ans: It is accepted by the Indian companies.

Q - S. Stalin, SPIC, India

1. Having revised your systems, do you have plans to conduct any safety audit by an external agency?

Ans: Safety audits have been provided by a team of 7 engineers from a consultant company. Safety audits have been performed in all departments, laboratory, production maintenance, etc. A procedure has been applied to make those audits and check lists have been used in the various departments.

Q - 2. Is there any plans to undergo 5 start safety audit by British Safety Council?

Ans: We have examined that possibility and is considering it seriously.

Q - S. Saidi, Groupe Chimique Tunisien, Tunisia

1. In place of reducing the height of retention wall around NH_3 storage, is it not better to replace them?

Ans: Walls house a height of 60 cm and is designed to contain a small leak, not a big one. There are 2 tanks of 10.000 t and they have been checked for wall thickness, cracks and visual inspection. The ammonia can be transferred from 1 storage to the other to allow for an inspection.

Q - 2. What kind of action have you undertaken to interest workers and supervisors to safety?

Ans: Any safety rules which are transgressed are reported in a bulletin mentioning the names of the offenders. The list is available in work council. People listed several times try to avoid repetition. There is also a competition for best safety behaviour. Rewards are distributed each year directly by top management to show their interest in safety and to stimulate a safe behaviour.

Q - Anonymous

1. Is your emergency plan approved by the government?

Ans: Yes, we have submitted the plan to the government authorities and they have approved it.

Q - 2. How have your ammonia sensors worked?

Ans: We have not yet installed the sensors close to the ammonia tanks; it is only a recommendation to do that, but we are planning to do it.

Q - H. Hakim, Groupe Office Chérifien des Phosphates, Morocco

1. Participative management is an advantage when dealing about safety. Did you use it to have a better adhesion to the safety programme?

Ans: There are daily meetings every day at 11 o'clock but specially every Saturday there is a meeting deliberating about safety issues. This meeting is also attended by management. There is also a team of engineers in HAZOP studies; team work is a good approach for safety. HAZOP studies are presented to people allowing further reactions. There are safety reports and papers on safety aspects that are circulated. Each manager is trained in safety at least once a year. There are also programmes dealing with toxicity of material used in the plants.

Q - 2. Are local authorities involved in the definition of emergency plan?

Ans: We made a documented emergency plan prepared in consultation with our partners and the local authorities have been contacted as well.

Q - Anonymous

Why have Severity Rate (SR) and Frequency Rate (FR) increased during the last two years?

Ans: The workers have got into smaller accidents mainly with their scooters. During shut-downs, slippings have happened. Special instructions for working at high places have been given to improve the situation. There are also special guards during the shut-downs to watch that instructions are followed. Stricter rules to use the safety helmets have been given.

In 1995, there was one fatal accident. The accident happened to a contract-worker, not to our own, this was included into our statistics.

Q - H. Lai Chuck Choo, Mauritius Chemical & Fertilizer Ind. Ltd., Mauritius

Have details on your emergency plan been communicated out of your site and have local authorities been involved in its definition?

Ans: The emergency plan has been prepared in consultation with local authorities and all partners. A leaflet has been prepared to summarize all actions and is available for the population.

Q - P. Mertens, Kemira Agro Pernis BV, Netherlands

Did you execute HAZOP studies on complete production facilities or was it limited to the critical parts of those units? If so, what method was used to make the differentiation?

Ans: At GNFC, we carried out HAZOP study of all the production facilities including steam generation. However, we excluded certain systems such as Drinking Water Network, Plant Air Network, etc., where are not directly connected to the process lines/equipment.

PAPER 34 Safety, health and environment management (SHE) in a large modern nitrogenous fertilizer complex - NFCL's approach

G.M. Koorse and G. Kameswara Rao, Nagarjuna Fertilizers and Chemicals Ltd, India

Q - B. Tricklebank, Kemira Agro UK, United Kingdom

What is your Lost Time Accident Frequency Rate?

Ans: 0.07.

PAPER 35 Latest developments in pollution control of urea plants

F. Granelli, Snamprogetti SpA, Italy

Q - S. Corton, Fertiberia, Spain*Can you give some more details about dedusting system?***Ans:** We have dedusting system in 3 plants in India and 3 plants in Russia. Water is cycled in a scrubber. Air from prilling tower is fed in the bottom of the tower and passes through sprayed water. Losses are the level of 15 mg/Nm³ as mentioned in the article.**PAPER 36** Study of the F/SiO₂ ratio in order to minimize fluorine emissions

K. Lahmadi and A. Ben Attia, Compagnie des Phosphates de Gafsa, Tunisia

No question.

PAPER 37 Concept and ecological programme « Chimco » company*

K. Petkov, Chimco, Bulgaria

No question.

PAPER 38 Corrosion problems and remedial measures in fertilizer plant*

M. Al-Rassi, SAFCO/IBN AL-BAYTAR, Saudi Arabia

No question.

PAPER 39 A novel and economic approach to CAN (LAN) prill tower plume abatement utilizing low temperature prilling

M.J. Burr and M.W. Du Plessis, Kynoch Fertilizer Limited, South Africa

Q - B. Christensen, Kemira Agro Oy, Denmark*How do you produce the liquid magnesium nitrate solution, what are the raw materials and what is the concentration of the magnesium nitrate solution when you add it into the AN-solution?***Ans:** We have a two-stage neutralizer dedusting system which presents about 86% AN-solution and the magnesium nitrate solution is pumped into that tank.

Magnesium nitrate is made by dissolving magnesite (from eastern Transvaal) into nitric acid and filtering the insoluble residues out. The typical strength of the solution is 35-40% magnesium nitrate.

Q - P. Suppanen, Kemira Agro Oy, Finland*What was the cost of development work and pilot plant that you carried out to find the solution you have implemented?***Ans:** There has been no pilot test. Trials of a few days have been made directly on the industrial unit. There were 2 or 3 tests required to reach the present results. We produced a couple thousands tons of products during each run.

ORGANO-MINERAL FERTILIZERS A NEW CONCEPT TOWARDS SUSTAINABLE FERTILIZER USE

N. Louizos, Drapetsona Fertilizers
and K. Rettos, Synel S.A.
Greece

RESUME

Les engrais organiques de diverses origines ne peuvent remplacer les engrais minéraux classiques fabriqués chimiquement car les premiers ont une teneur irrégulière et plus basse en éléments nutritifs, cependant que leur transport et leur manutention sont très difficiles. Malgré cela, les engrais organiques sont employés non à cause de leur teneur en NPK, faible en tout cas, mais surtout pour la matière organique humifiée qu'ils contiennent.

En incorporant de la matière organique à des engrais fabriqués chimiquement, on peut obtenir un produit qui offre encore tous les avantages de ce dernier en terme de teneur (produit à haut dosage) et forme (granules faciles à manipuler) qui en même temps conserve les propriétés valables de la matière organique qui enrichit le sol.

Synel, la coopérative agricole de Grèce, produit un engrais organique obtenu à partir de fumier de volaille après fermentation prolongée. Ce produit constitue l'une des matières premières de la production d'engrais NPK comme le 20-10-0, 11-15-15 (S), etc. en une proportion d'environ 20 %. Ainsi, il est possible de remplacer des matériaux essentiellement inertes autrement nécessaires pour la production des engrais ci-dessus par de la matière organique, qui grâce à une fertilisation continue année après année, enrichit le sol et améliore ses propriétés physiques et mécaniques.

On peut définitivement affirmer que cette combinaison est un pas en avant vers une fertilisation durable et peut donc servir de modèle pour d'autres combinaisons semblables.



1. INTRODUCTION

The idea of sustainability became once again a top subject for discussion by the end of the eighties based on the work of the so-called Bruntland Committee in Europe. In 1992 the European Commission issued the Programme of Policy and Action in relation to the Environment and Sustainable Development: Towards Sustainability. During the same year the United Nations Conference on Environment and Development in Rio de Janeiro established officially sustainability as the principle which should govern human activities, mainly in the developed countries. According to this principle natural resources should be used in such a way so that they continue to serve forthcoming generations.

Historically agriculture has developed based on the recycling of plant nutrients, in the form of crop residues or manure. However with an increased availability of mineral fertilizers from external sources in the 20th century there was a trend towards the breakdown of traditional nutrient cycling practices on-farm, thus less and less organic material was used in arable production.

The ability to maintain sustainable agriculture over decades and centuries will largely depend on our successful management on recycling plant nutrients as well as proper balancing in the use of mineral and organic fertilizers. Our presentation is aiming at this end and could be considered as an example of such a combination or a step towards sustainability.

2. EXISTING SITUATION IN PRODUCTION AND USE OF ORGANIC FERTILIZERS

Production of organic fertilizers is mainly effected in small scale by processing organic materials such as lignite, agricultural residues, animal residues or biologically active materials which subsequently are mixed with inorganic salts. The product resulting from the above mechanical mixing is in the form of powder or pellets but its main characteristic compared to conventional mineral fertilizers is not only that it is in an inconvenient form for handling-distribution (big volumes, difficult application) but also that it has a very low content of main elements i.e. N, P, K, normally less than 10% in total which results in high transportation and storage cost.

On the other hand from the agronomic point of view it is well known that one of the basic factors for the fertility of the soil is its content in organic material which not only improves the physicochemical characteristics of the soil but also leads to the development of biological processes valuable for the development of plants. The use of soils year after year, under intense fertilization practices, normally and exclusively by means of mineral fertilizers results inevitably to the decrease of organic matter in the soil which in turn leads to all negative consequences on the quantity and quality of agricultural products.

To face the above problem Greek farmers as most probably happen in most other parts of the world, use organic fertilizers in a very small percentage of the arable area and only for limited species of vegetable and fruit plantations, while nothing is done for the big majority of soils and plantations.

It is worthwhile to mention that by using exclusively and continuously mineral fertilizers which in certain cases (16-20-0, 20-10-0, 11-15-15) contain inert material (filler) just to balance the grade, instead of enriching the soil at the end of the cycle one ends up with degrading the soil by adding this inert material.

Last but not least from the environment point of view the diminishing or in any case low use of organic fertilizers not only widens the already "open" nutrient cycle of agricultural systems but also creates environmental problems since all these organic materials if not properly used as fertilizers should be massively disposed i.e. dumped in landfills or introduced into rivers, washed to the sea, etc.

3. ORGANO - MINERAL FERTILIZERS

3.1. Outline of the Innovation

To overcome the disadvantages of organic fertilizers regarding handling and distribution mainly as far as the end user (farmer) is concerned and make it possible to re-introduce them to the farming cycle, one can incorporate organic matter (by different ways) into a chemically manufactured fertilizer, thus leading to a new product the organo-mineral fertilizer. This new product has still all the advantages of the mineral fertilizers in terms of content (high analysis product) and shape (granules easy to handle) but at the same time it maintains the valuable properties of the soil enriching humified organic material. In other words the mineral fertilizer is acting on the carrier of the organic matter. On the other hand in cases where inert material is introduced in the fertilizers as filler the organic material is replacing the filler, so a chemically and agronomically inert material is replaced by a soil enriching material which also contributes to the effectiveness of the final product in terms of macro and micro elements.

3.2. Profile of the Partners

The innovation was developed in the framework of the existing collaboration by SYNEL and Drapetsona Fertilizers. Synel is representing the farmers cooperative in Greece which is distributing more than 60% of the country's fertilizers. Synel also owns and operates a small production plant which is producing about 10000 MT/yr of organic fertilizer obtained from poultry manure after prolonged fermentation, according to the OKADA process. Drapetsona Fertilizers is the ex Hellenic Chemical Products & Fertilizers Co., which is the oldest fertilizer producer in Greece producing about 350.000 MT/yr of conventional mineral fertilizers of various grades such as 16-20-0, 20-10-0, 11-15-15, 15-15-15 etc. by the slurry granulation method or compaction.

3.3. Analysis of the organic material

The organic material to be incorporated in the mineral fertilizer was selected among various other materials since it is relatively rich in macro as well as micro nutrients. It is also sterilized (as sterilization is part of the production process of the organic material) and has acceptable physicochemical characteristics, while heavy metal content is zero to negligible. The typical chemical analysis of the organic material is as follows (%):

Molsture	15-25
Organic matter	70-80
N	3-5
P ₂ O ₅	3-5
K ₂ O	3-4
CaO	3-4
MgO	1-1.5
Fe	0.5
Zn	0.05
Mn	0.07
B	0.009
pH	8
Humic/Phulbic acids	44

The results of DTA and DTG analysis for organic material correspond to those of the chemical analysis (39% ash, 61% organic matter) and show that the behaviour of the material is very close to that of humic acids and xylite.

3.4. Production Process

Poultry manure is first of all well mixed and homogenized in specially designed mixer within greenhouse-type buildings where it is undergone aerobic fermentation and drying for about 25 days, according to the OKADA process and technology. The temperature obtained (40-70°C) guarantees the production of a sterilized free from pathogens as well as odorless material which is either palletized and directly distributed as organic fertilizer or used as raw material for the production of organomineral fertilizers.

The production of organomineral fertilizers is taking place either in a compaction plant (Figure 1) or in a conventional slurry granulation plant (Figure 2) where the organic material is introduced in the recirculation stream along with other solid raw materials (Ammonium Sulphate, MAP, Potassium Sulphate, etc.) prior to the granulation step. Several industrial scale runs were made with typical Greek market products such on 16-20-0, 20-10-0, 11-15-15 whereby organic matter was introduced at a percentage of 10-20%. The limiting factor for this percentage was keeping the physicochemical characteristics of the produced fertilizer to the quality standards of conventional fertilizers i.e. to ensure good granulation, keep granule size, hardness, abrasion resistance and caking tendency within the accepted limits. The effect of high temperatures (~100-110°C) developed in the granulator, on the stability of the organic matter was checked and found to be insignificant since in the worst case under laboratory scale measurements the loss of material at such temperature due to volatilization did not exceed 20% which in terms of nitrogen corresponds to a loss of ca 15%.

Alternatively the organic material was also used in the production of compacted fertilizer (18-9-6) in a compaction plant where it was fed together with other solid raw materials. In this case the effect of temperature mentioned above is eliminated.

4. CONCLUSIONS

Organo-mineral fertilizers are produced by incorporating organic matter such as poultry manure properly fermented into conventional chemical fertilizers. By controlling the percentage of organic material so that it does not exceed 20%, the final product is equivalent to conventional chemical fertilizers as far as physicochemical and mechanical properties are concerned. However it is superior to them taking into account the humified material by which it is enriching the soil, an advantage which is magnified in case the organic material is substituting fillers used so far in the production of chemical fertilizers. Beyond this it contains low cost macro and micro nutrients of organic origin, no heavy metals and is practically odourless.

Environmental problems due to the inefficient use and poor distribution of nutrients, as well as growing awareness that raw materials for the production of nutrients are not infinite resources, represent a real challenge and opportunities for the fertilizer industry to work with farmers and policy makers and become once more involved in the recycling of nutrients as the basic principle of sustainability imposes. Potential sector for application of this principle is primarily the sector of animal waste which indeed corresponds to a vast nutrient resource.

Beyond this however there are lots of other cases like food industry wastes (Brewery, dairy, meat, starch, sugar, etc.) as well as urban wastes which following current trends for cleaning municipal waste water are creating an ever increasing quantity of recyclable material.

FIGURE 1
COMPACTED FERTILIZERS GRANULATION PLANT

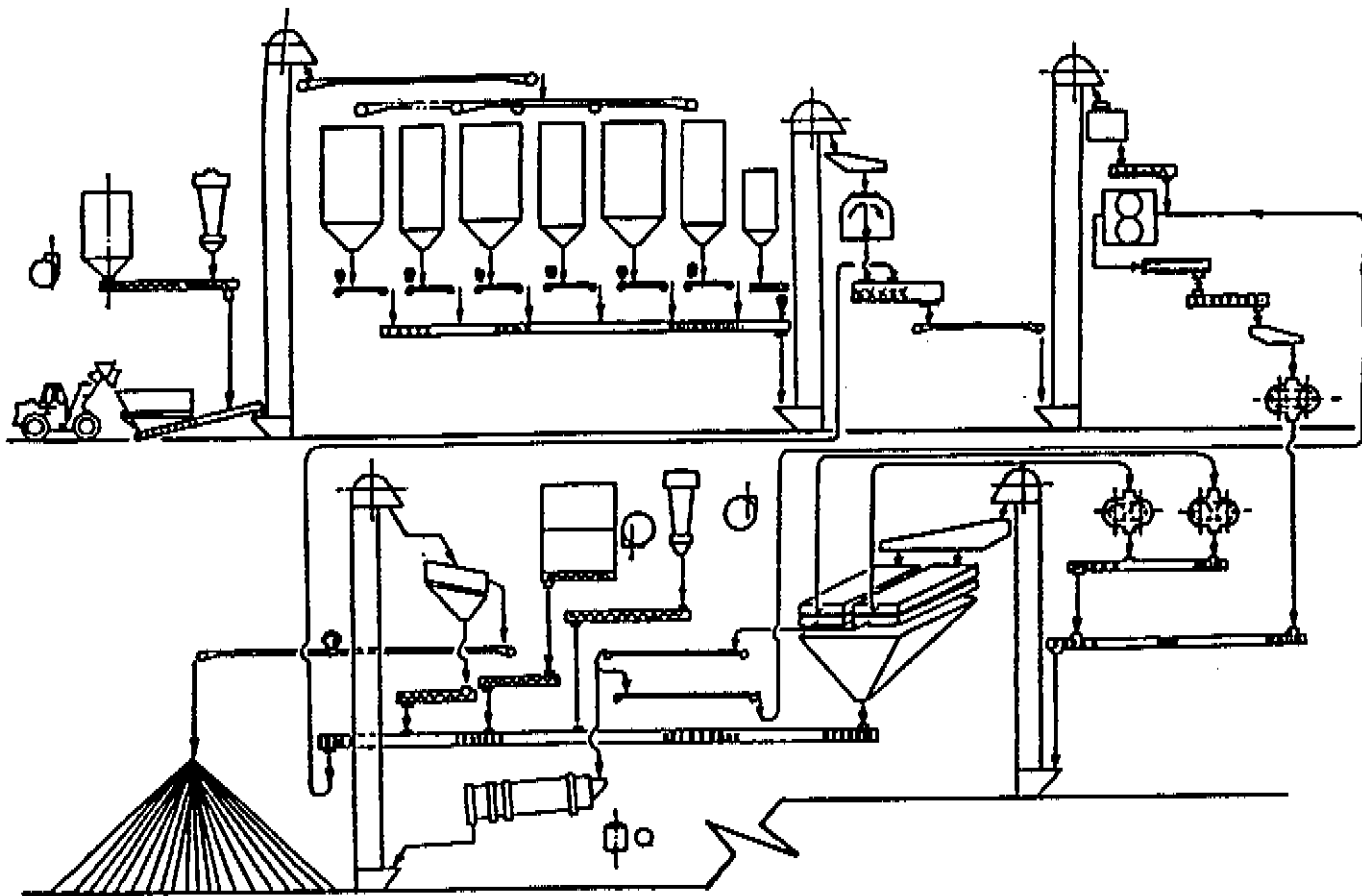
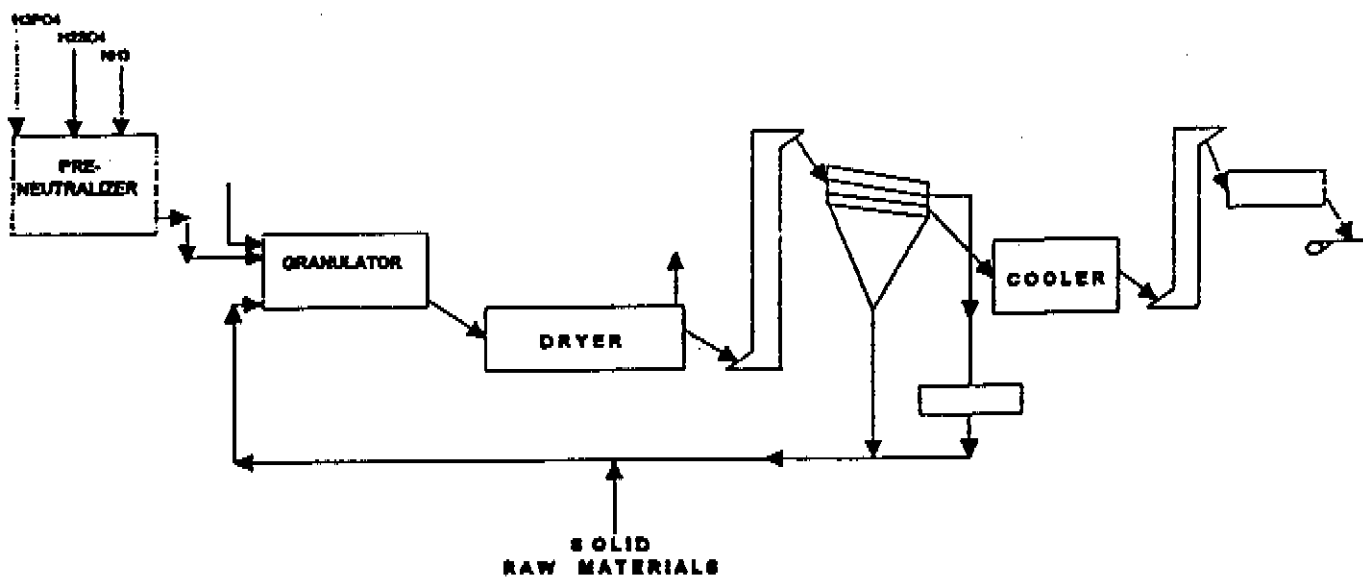


FIGURE 2
CONVENTIONAL GRANULATION PLANT



LATEST DEVELOPMENTS IN POLLUTION CONTROL OF UREA PLANTS

F. Granelli

Snamprogetti SpA, Italy

RESUME

Le problème de pollution par les usines d'engrais, et particulièrement les unités d'urée, s'est aggravé ces dernières années et cette tendance se poursuivra dans l'avenir. Les limites de polluants autorisées ne sont pas uniformes et diffèrent d'un pays à l'autre, et parfois à l'intérieur du pays d'une région à l'autre.

Il arrive qu'un pays établisse des limites maxima autorisées à ne pas dépasser pour les polluants, mais les régions, en fonction des situations locales et souvent selon les besoins spécifiques des habitants, imposent des valeurs différentes qui peuvent être bien plus basses que les normes nationales.

La communication décrira les sources possibles de pollution dans un atelier d'urée et la contribution de Snamprogetti à la réduction des valeurs parfois même au-dessous de celles autorisées par les réglementations les plus strictes.



1. INTRODUCTION

We quote the following sentence from the paper we had the opportunity and the pleasure to present at the IFA Conference held in Kuala Lumpur during December 1994: « Antipollution requirements can be expected to be more severe in future and we believe that the solution of the relevant problems will determine the choice of the urea technology ».

This is true today and it will be even more so in the coming years, towards and after the 2000's, for urea plants as well as for any other industrial plants, although a good technology, including all the up-to-date steps for minimizing pollution, is not sufficient: good engineering, plant operation and maintenance play an important role in preventing pollution. Essential is in any case the mentality of the plant owner who has to maximize the benefits minimizing the social costs. The most economical method of running a plant is not necessarily the best one in the long run.

Frequently, in the past, environmental considerations were overlooked, but even today sometimes pollution regulations are not fully respected, also because in some cases they are not clear and often contradictory.

Anyway, today the problem of air and water pollution produced to some extent by every human activity is becoming more and more keenly felt. The problem is of course particularly marked in large-sized industrial plants, specially if located in the vicinity of large towns.

This is the case of the large capacity ammonia and urea plants: at the present time we are designing ammonia and urea plants having capacities of 2500 and 3000 T/D respectively, in one line. But, although with lower capacity, in some factories 3-4 urea lines in parallel are already in operation with a total production around 6000 T/D.

Figure 1 indicatively shows the enormous increase in the urea worldwide production in the last decades, while Figure 2 shows the capacity increase for a single line urea plant in the same period of time.

Such industrial growth in the production of urea was associated with an increase of problems caused by the effluents in gaseous as well as in liquid form.

The polluting elements from urea plants are represented by gaseous ammonia and urea dust in the gaseous effluents and by ammonia and urea in solution in the liquid effluents.

The importance of these problems varies from one country to another depending on many factors and, in the same country, from one area to another depending on the perception of the problem by the nearby population, being in general more acute in those areas that are more densely populated.

So every country has different allowable legal limits for the pollutants, in any case becoming more severe with time. These limits require measures involving considerable amounts of money (particularly for the existing plants), in order to reach the required limits.

Some industrial plants have been shut-down forever due to pollution problems: as far as we know this is not the case of urea plants. Anyway, for some of them, air pollution due to ammonia and urea dust from the prilling tower was considered important enough to compel the plant owner to reduce the plant capacity or to switch the finishing section from prilling to granulation or to produce urea solutions.

2. ENVIRONMENTAL REGULATIONS

In the 1950's and in the 1960's no great attention was paid to the pollution problem by ammonia and urea from urea plants, discharged into the air or into the water.

The consumption of ammonia per ton of urea, that theoretically is around 566 kg, was much higher than 580 kg, which means that at least 14 kg of ammonia (as ammonia or urea) were lost as pollutant to the environment for each ton of urea.

In these years such loss (about 2.5% of the consumption) was tolerated due to the low urea production, but in the 70's and the 80's the enormous increase in the urea production made it unacceptable. Today for a plant having a rather common capacity of 2000 T/D, such loss would be about 28 T/D.

Apart from the pollution consideration this amount of loss means a higher cost of the urea production, having the cost of the ammonia an impact of about 80-90% on the plant battery limit cost of urea. This consistent amount of loss justifies the efforts made to reduce it, not only from the point of view of pollution: easy to say that those antipollution systems leading to the recovery of ammonia would be preferable.

In the 70's the regulations began to set limits that became more restrictive during the 80's. Let us see some allowable legal limits.

2.1. Gaseous effluents

2.1.1 EEC countries

EEC authorities provide the general rules and recommendations. Each country fixes the maximum emission levels which must not be exceeded.

For the urea plant the Italian national rules are as follows:

- urea dust : 100 mg/m³
- ammonia : 200 mg/m³

Inside the EEC countries each region has the possibility to reduce the national limits depending on several parameters, viz.:

- density of population
- density of already existing emissions
- climate (particularly wind)

For example in the Italian Lombardia region (high population density and low wind) the values are as follows:

- urea dust : 20 mg/Nm³
- ammonia : 20 mg/Nm³

The values of three other Italian regions are here given (mg/m³):

		REGION		
		Emilia	Umbria	Puglia
-	urea dust	20	50	80
-	ammonia	35	50	80

while in Germany and Austria they are:

- urea dust : 75 mg/Nm³
- ammonia : 20 mg/Nm³

2.1.2 U.S.A.

In USA the regulations procedure is similar to the European: E.P.A. (Environmental Protection Agency) fixes some maximum emission levels and each State of the Union (as the regions in EEC) can reduce the limits.

Anyway E.P.A. ignores the NH_3 emission to the atmosphere. This is probably due to the fact that ammonia is not so hazardous for human health as other pollutants from fertilizer plants like SO_2 , NO_2 and HF. Moreover ammonia is lighter than air and is therefore dispersed in the atmosphere, for example from an adequate vent stack or from the top of the prilling tower.

Ammonia is contemplated only in the TLV (threshold limit value) list indicated by ACGIH (American Conference of Governmental Industrial Hygienists). TLV's represent the limit concentration in air inside the Battery Limits of the plant, i.e. « the conditions under which it is believed that nearly all workers may be repeatedly exposed day after day without adverse effect ».

The value for ammonia is:

TLV-TWA (time weighed average)	:	18 mg/m^3
TLV-STEL (short time exposure level)	:	24 mg/m^3

Figure 3 indicates the above values and others.

2.2. Liquid effluents

2.2.1 EEC countries

Also in case of liquid effluents EEC authorities give only general recommendations (precise figures for the maximum levels are given only for very toxic emissions). For the most common pollutants each EEC country had rather strict laws since the 70's: regional authorities are just entrusted with the task of getting them complied with.

The national Italian values for nitrogenous pollutants are:

- total ammonia as NH_4^+	:	15 mg/l
- nitrous nitrogen as N	:	0.6 mg/l
- nitric nitrogen as N	:	20 mg/l

EFMA indicates the following values of pollutants in the discharged water:

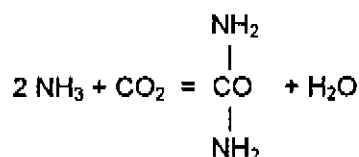
- Urea	1 mg/l
- Ammonia	5 mg/l

2.2.2 U.S.A.

E.P.A. regulations are already rather strict, but any State of the Union can increase or decrease the limits after evaluating all the factors at plant site. Anyhow any increase in emission limits is subject to approval by E.P.A.

3. SOURCES OF POLLUTION FROM UREA PLANTS

All industrial urea production plants are based on direct synthesis between ammonia and carbon dioxide according to the following reaction, represented in Figure 4.



$$\text{i.e. } 566 + 734 = 1000 + 300$$

In fact this reaction leads to a urea solution at about 70% by weight, containing a small quantity of the reactants, NH_3 and CO_2 , and must be followed by a finishing step by which solid urea (prilled or granular) is produced from the solution.

Figure 5 represents the two above steps: the reaction from NH_3 and CO_2 that leads to the urea solution and the solidification of urea from said solution.

During the process of urea production there are continuous losses and discontinuous losses. The points of continuous losses are indicated in Figure 5 at point 1, 2 and 3:

1. Ammonia losses in the « Inerts »
2. Ammonia and urea dust losses from prilling towers or granulation stacks
3. Ammonia and urea losses in the process water.

The discontinuous losses are represented by leakages, drainages and spillages.

In Figure 6 all these losses, continuous and discontinuous, as ammonia or as urea, are summarized. In the following paragraph we examine all of them and we indicate the minimum values that can be reached with the Snamprogetti technology.

4. MINIMUM POLLUTION VALUES ACHIEVABLE WITH SNAMPROGETTI UREA TECHNOLOGY

4.1. Continuous losses

4.1.1 Ammonia losses in the "Inerts"

« Inerts » are the so called components contained in the CO_2 (and partially also in the NH_3), like H_2 , CH_4 , CO , Ar , etc., that enter a urea plant and do not participate to the reaction that forms urea. It is also well known that in all industrial urea plants some passivation oxygen, mainly as air, is introduced in a urea plant just for the protection of the stainless steel surfaces, in contact with the process fluids, against corrosion.

When the « inerts » and the passivation air release the urea plant to the atmosphere they contain some NH_3 , that is a loss and represents a pollutant and at the same time, as all the pollutants, a process inefficiency. The lower the amount of passivation air is, the higher the efficiency of the ammonia recovery may be, without incurring the well-known problem of the explosive mixtures. Those processes are therefore favoured that require (like the Snamprogetti one) a lower amount of passivation air. The ammonia content in the « inerts », plus the passivation air, released to the atmosphere is generally about 1% in volume in this effluent.

As already said in the last years authorities and citizens themselves have become more aware to ammonia pollution problems, especially in case of those plants that are close to crowded areas. In these cases there is the request to strongly reduce the emission of ammonia.

Snamprogetti has already in operation plants in which the ammonia content in the « inerts », leaving the plant to the atmosphere, is reduced to about 10 ppm, totally avoiding the formation of explosive mixtures. Such a result is reached by the addition of an inert gas before the final washing with water to recover ammonia. This inert gas may be nitrogen or, more conveniently, synthesis or natural gas. This inert gas can be added to the « inerts » before their final washing with water in order to recover practically all the ammonia they contain. The resulting ammonia solution is sent back to the urea plant and any problem of explosion is completely avoided. The principle of this deep and safe washing of ammonia is represented in Figure 7.

As well known the composition of the gas mixture leaving a urea plant to the atmosphere can be represented by a triangular diagram, having at the apexes O_2 , N_2 and a flammable mixture made of H_2 , CH_4 , CO , NH_3 . In this triangle the dotted areas represent all the possible explosive mixtures. Supposing that the gas mixture leaving the urea plant is represented by point A in Figure 7.1, after a washing with water in order to recover NH_3 and to reduce pollution, the composition of the mixture, after washing, can be represented in the same Figure 7.1 by point B, i.e. inside the area of the explosive mixture. But if, before washing, an inert gas (like natural gas) is mixed with the mixture represented by previous point A, the new representative point is point C in Figure 7.2.

Washing by water the new mixture, represented by point C, ammonia can be totally recovered and the new composition of the « inerts », plus passivation air, leaving the plant is represented, in Figure 7.3, by any point on the straight line between the apex representing 100% flammable mixture and point D, i.e. for example, according to the quantity of added natural gas, by point E, that is completely out of the composition of explosive mixture.

The gas mixture can be sent to burn in a burner or in a reformer.

In Figures 7.1, 7.2, 7.3 the explosive area has been considered, for simplicity, as constant. In fact it changes with the composition of the flammable mixture.

The flow sheet of said solution is represented by Figure 8 that does not need clarification. The quantity of ammonia in the « inerts » can be limited to 10 ppm.

4.1.2 Ammonia and urea losses from prilling towers and granulation stacks

The air pollution problem due to ammonia and urea dust in the air from a prilling tower is very uneasy to eliminate due to the large volume of air, the extremely low partial pressure of ammonia in the air and to the small size of the particles of urea to be caught.

The effect of the emissions of urea dust on the environment is not very clear: in the vicinity of a plant the effect on vegetation can be even considered as beneficial, but the prevailing winds can concentrate the dust in a small area to cause scorching on vegetation.

Normally from our prilling tower (natural raft) the quantity of urea dust is about 40 mg/Nm³.

In some plants Snamprogetti has installed a dedusting system that can reduce the urea dust emission to 15 mg/Nm³.

If such efficiency does not seem too brilliant, it must be reminded that a large part (80%) of the urea dust has a size lower than 3 µm.

In Figure 9 we have indicated this size and the quantity of air involved. As a comparison we have also indicated the same values for a granulator, Hydro Agri fluidized bed granulator.

It appears clear from this figure that the urea dust pollution from granulation is lower than from a prilling section.

As regards ammonia in the air from the prilling towers it is only in the last decade that authorities, due to the claims of the nearby population, have issued more severe limits. As above said these strict limits made some plant owners to install a granulation section in a urea plant already supplied with a prilling tower.

Apart from a better product achieved, in some cases the granulation section was installed just to reduce pollution problems.

In fact from a granulation section it is easier to catch the urea dust, but it is also possible to reduce the quantity of ammonia sent to the atmosphere.

In order to reach the low values of ammonia, as indicated in Figure 3 (20-30 mg/Nm³), it is necessary a scrubbing with a mildly acidified solution.

Due to the fact that even traces of ammonium nitrate cannot be tolerated in the final urea product, this acid can be used only if the recovered ammonium nitrate is sent to a UAN or DAP/NPK plant.

If sulphuric acid is used for scrubbing, the obtained ammonium sulphate can be recycled to the urea plant if the product is used as fertilizer.

In the 1650 T/D urea granulation plant of Ferrara (Italy) utilizing the double falling curtain drum granulation technology of Snamprogetti, in operation since 1990, a double scrubbing is installed: in the first one urea dusts are scrubbed by means of water, and the obtained solution is recycled to the urea plant, while in a second scrubbing by mildly acidulated water (with sulphuric acid) ammonia is scrubbed and the obtained ammonium sulphate solution is sold separately by the factory.

In the same plant, anyway, for the first 4 years of operation only one scrubbing was installed with acidified water (with sulphuric acid) and the obtained solution of urea and ammonium sulphate was recycled back to the urea plant.

The final urea product contained about 2500 ppm of ammonium sulphate with no detrimental effect on the urea used as fertilizer.

Figure 10, that does not need any clarification, represents the double scrubbing of the granulation plant of Ferrara, while Figure 11 indicates the values of the pollutants in the emission, that are much lower than those reported by Italian as well as by regional laws (Ferrara is in the Emilia region).

As regards the double contemporary scrubbing of ammonia and urea dust on top of the prilling tower there are very few examples in the world, always by scrubbing with a mildly acidified water.

Snamprogetti has tested a different method to reduce the quantity of ammonia in the air from the prilling tower.

The tests have been made, in a urea plant of 360 T/D, by dosing a moderate quantity of sulphuric acid in the molten urea upstream the prilling bucket of the prilling tower. This method, which is simple to implement, has a modest investment and operation cost, ensures easy operability and has reached both the set goal of an appreciable reduction of ammonia emission in the air released to the atmosphere and, unexpectedly, a second result consisting in a remarkable reduction of the free ammonia present in the prilled product, with considerable environmental benefits in the product transportation and storage system.

However, it should be specified that this treatment produces prilled urea with about 2000/3000 ppm of ammonium sulphate.

As already said, this does not restrict in any way the use of urea as a fertilizer, but it can be a limitation for many of the technical uses of the product.

We are still continuing these tests and the results will be the object of the future paper.

4.1.3 Ammonia and urea losses in the process water

As indicated in Figure 4, the theoretical quantity of water produced for every 1000 kg of urea is 300 kg. In fact, due to some injection of steam and water for flushing or washing or for the vacuum ejectors, the amount of the process water can be even as high as 600 kg/T of urea, i.e. for a 2000 T/D urea plant it means 1200 T/D of water.

Unfortunately this process water (also called process condensate or waste water) contains some 4-5% wt of NH_3 and 0.5-1% wt of urea when it leaves the urea plant.

The presence of ammonia is due to the residual ammonia in the 70% wt urea solution sent to the evaporation section, while the presence of urea is attributable to some entrainment of liquid particles from vacuum separators.

Undoubtedly the major problem is toxicity of ammonia to aquatic organisms.

Among the first attempts to reduce ammonia was to strip it out from water and to send it to the atmosphere. Useless to say that the pollution problem is transferred from water to air unless ammonia is fixed as an ammonium salt.

Later on, recovery of the ammonia (as water solution) was introduced.

Today it is possible to strip ammonia to an extremely low value, to recover it, and to couple such stripping with a thermal hydrolysis where also urea can be completely decomposed to NH_3 and CO_2 , which are completely recovered to the urea plant.

The flow sheet of the Snamprogetti process water treatment section is indicated in Figure 12 and Figure 13 represents the values of pollutants in the discharged water:

- Urea : 1 ppm
- NH_3 : 1 ppm

By this treatment three targets are contemporarily achieved:

- ammonia specific consumption reduced
- pollution practically zero
- possibility of reusing process water as B.F.W.

With the values of pollutant contained in the three above examined sources of pollution the specific consumption of ammonia per ton of urea is very near to the theoretical value.

Figure 14 indicates in details such specific consumption of ammonia per ton of urea (565.69) which corresponds to a plant efficiency higher than 99.7%.

4.2. Discontinuous losses

If good antipollution systems can lead at the same time to low specific consumption of ammonia and to low contents of pollutants in the emissions as indicated above, it must be pointed out that these values remain targets to be reached.

In fact they depend not only on the process, but also on engineering proper operation and maintenance of the plant, awareness of hazards of pollution, control by the factory management and authorities, lack of legislation.

Process fluids containing ammonia and urea may be released into the environment due to leakage, spillage, drainage or other accidents which can be even more polluting specially if they are concentrated in pollutants. It is during start-up and shut-down and during other periods of irregular operation that losses from the plant are most likely to occur.

At such time transients the storage of solution in large quantity may present serious problems, which can be alleviated by:

- less frequent plant stoppage
- less frequent plant emptying
- lower inventory of process solution

Obviously good maintenance of flanged joints, pump glands, etc. is important.

But more than that, the very design principles and design details make the most important contribution to a pollution free urea plant.

5. CONCLUSIONS

There are processes available in order to reduce emissions of pollutants from urea plants.

We can supply such process, regarding the continuous losses and we can design the plant in order to reduce emissions during plant irregular operations.

But a great contribution must come also from the plant owner who should have the correct mentality to avoid problems, even in the areas where regulations do not exist or are contradictory.

6. REFERENCES

1. F. Granelli, *Ways to revamp urea units*, Hydrocarbon processing, June 1989
2. F. Granelli, *An improvement in technology has accompanied the enormous growth of urea production in the last four decades*, IFA Conference of Kuala Lumpur, December 1994
3. F. Granelli, A. Cavallaccio, R. Botti, *The Granular urea in the world and the experience of the granulation unit at the Ferrara factory (Italy)*, IFA Regional Conference for Asia and the Pacific, Perth (Australia), December 1995

Figure 1 - Urea world production

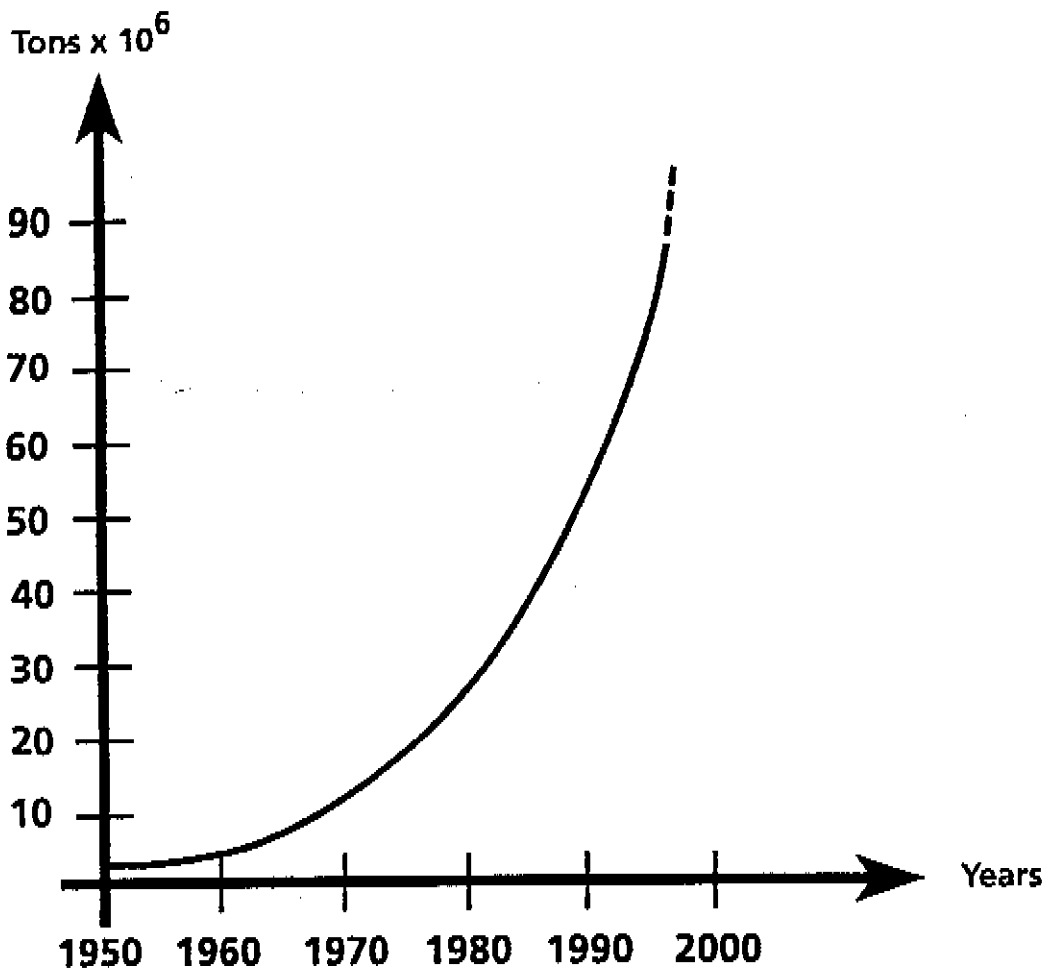


Figure 2 - Single line urea plant capacity

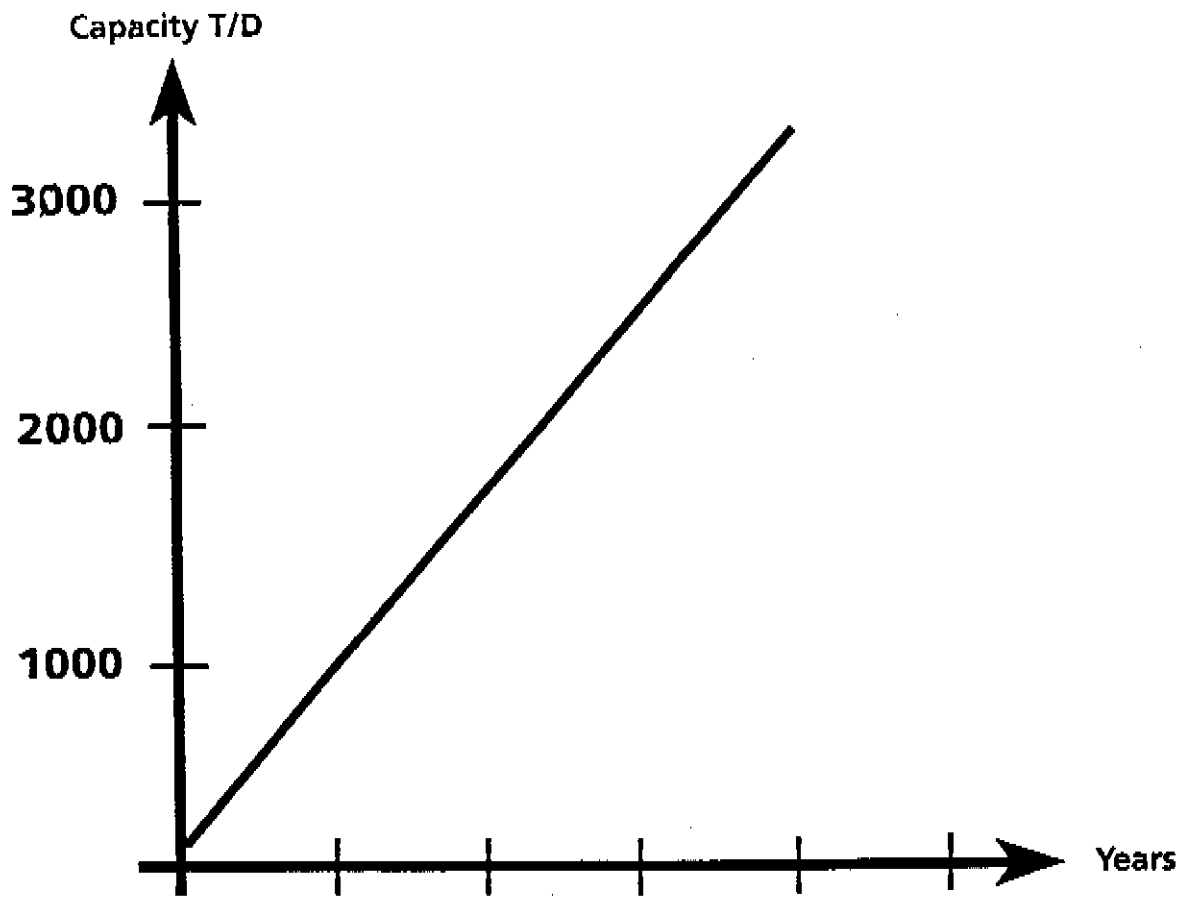


Figure 3 - Urea plant gaseous effluents pollutants limits

		UREA DUST	AMMONIA
ITALIAN NATIONAL LIMITS:	mg/m ³	100	200
ITALIAN REGIONAL LIMITS:			
- LOMBARDIA:	mg/Nm ³	20	20
- EMILIA:	mg/m ³	20	35
- UMBRIA:	mg/m ³	50	50
- PUGLIA:	mg/m ³	80	80
GERMAN VDI 3453 LIMITS:	mg/Nm ³	50	60
AUSTRIAN LOCAL LIMITS:	mg/Nm ³	30	30
EFMA⁽¹⁾:	mg/Nm ³	50	50
WORLD BANK:	mg/Nm ³	50	35
TLV- TWA⁽²⁾ (ACGIH)⁽³⁾:	mg/m ³ ⁽⁵⁾		18
TLV- STEL⁽⁴⁾:	mg/m ³ ⁽⁵⁾		24

(1) European Fertilizer Manufacturer Association

(3) American Conference Governmental Industrial Hygienists

(2) Threshold Limit Value - Time Weighted Average

(4) Threshold Limit Value - Short Time Exposure Value

(5) At 25° C

Figure 4 - Urea synthesis reaction

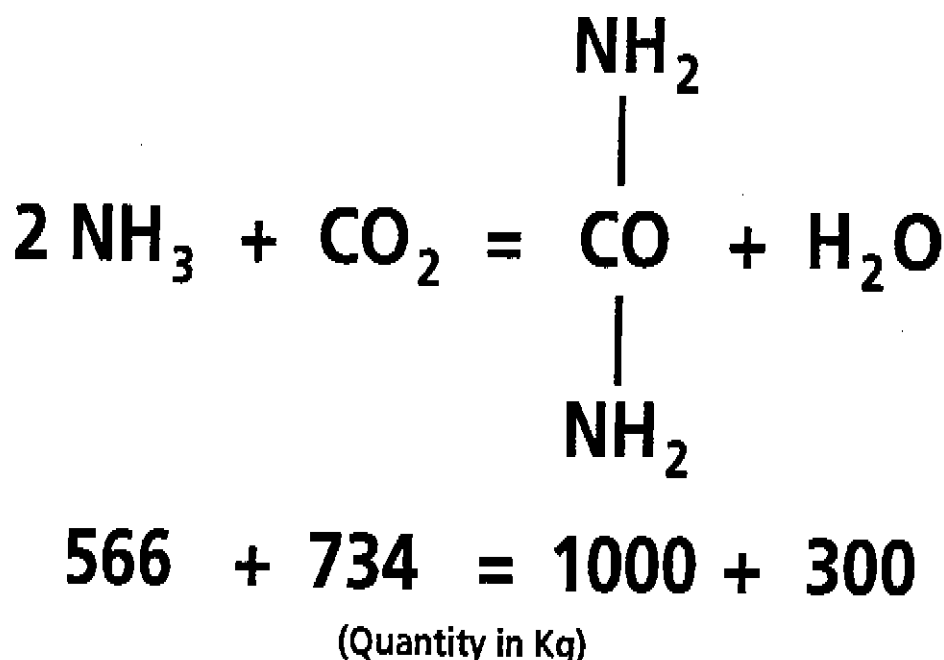


Figure 5 - Block diagram of urea production and sources of pollution

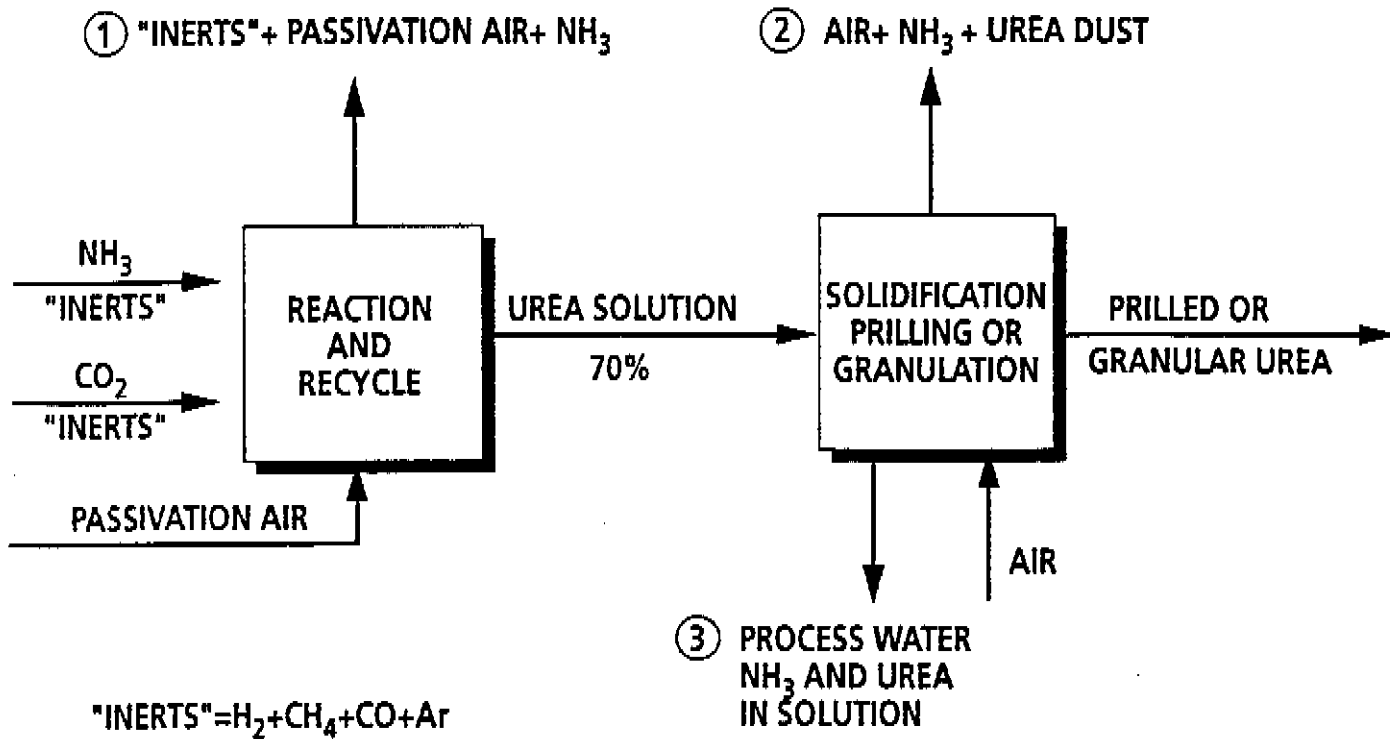


Figure 6 - Losses from urea plants

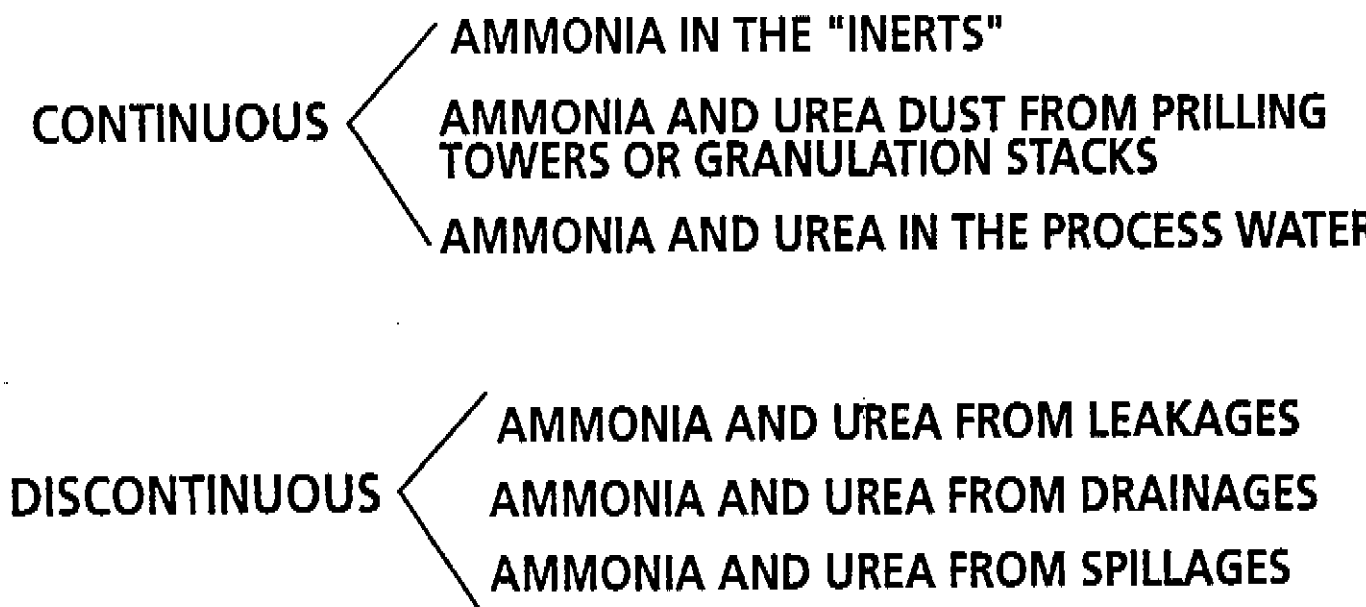


Figure 7 - Total absorption of ammonia from the « inerts ». No explosive mixtures

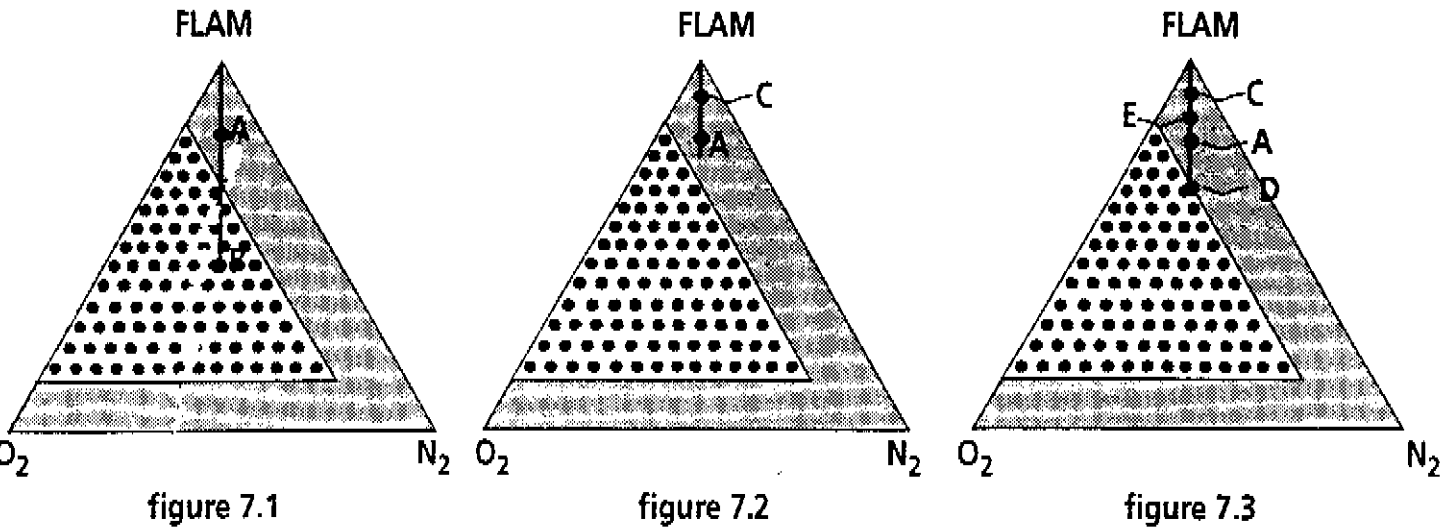


figure 7.1

figure 7.2

figure 7.3

FLAM (flammable)= H₂+CH₄+CO+NH₃ etc

NB. for simplicity the explosive area has been considered constant, while in fact it changes with the composition of the flammable mixture

Figure 8 - Total absorption of ammonia from the « inerts ». No explosive mixtures

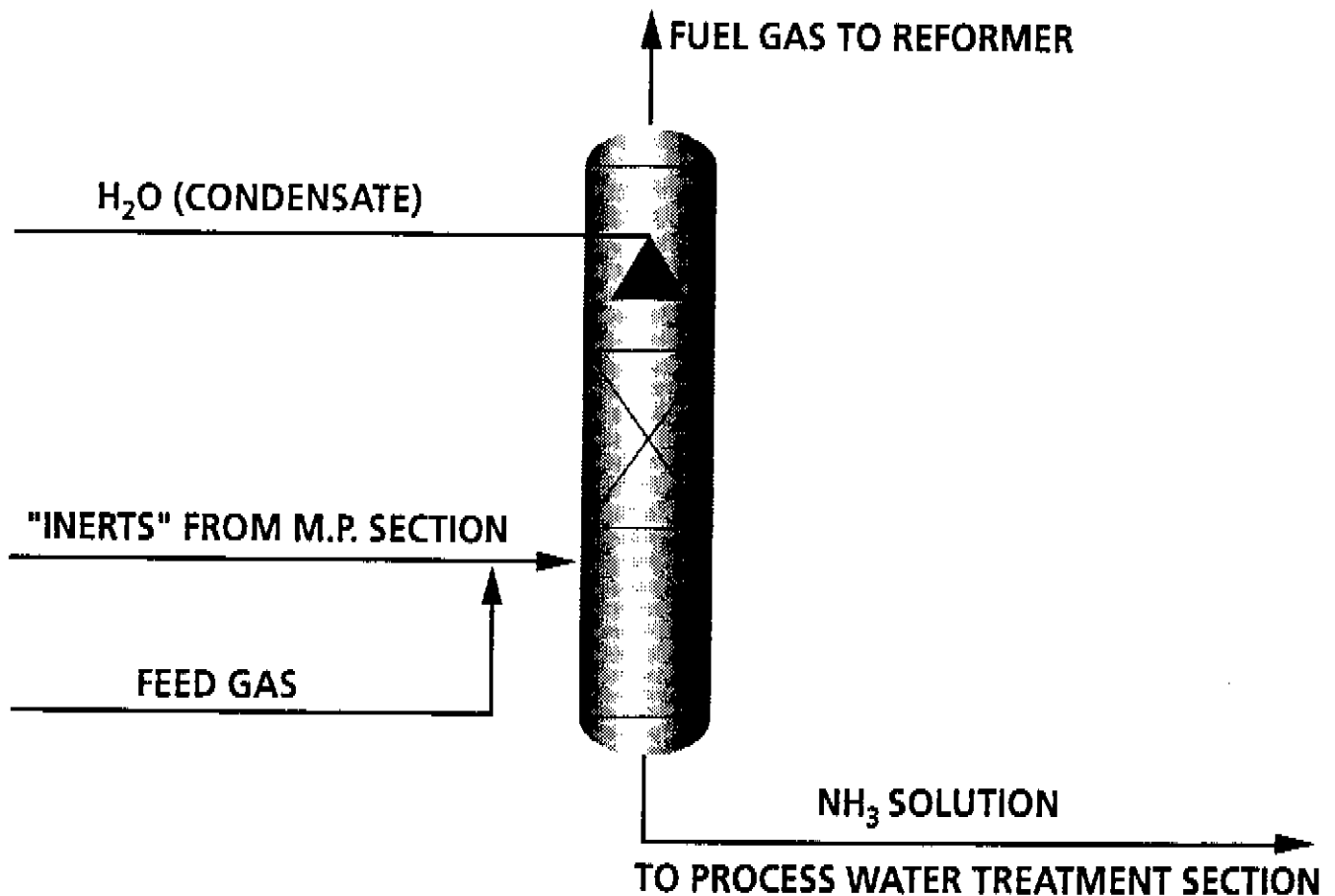


Figure 9 - Urea dust emission characteristics

		PRILLING ⁽¹⁾	GRANULATION ⁽²⁾
AIR QUANTITY	Nm ³ /T of UREA	8,000	5,500
DUST EMISSION	mg/Nm ³	40	25
TOTAL DUST	Kg	0.32	0.14
DUST SIZE MICRON (10⁻³ mm)			
	> 3	18	97
	1 ÷ 3	66	2
	< 1	16	1

(1) Natural draft, without dedusting

(2) From Hydro Agri fluidized bed, not from cooler.

Figure 10 - Separated air washing system for urea dust and ammonia

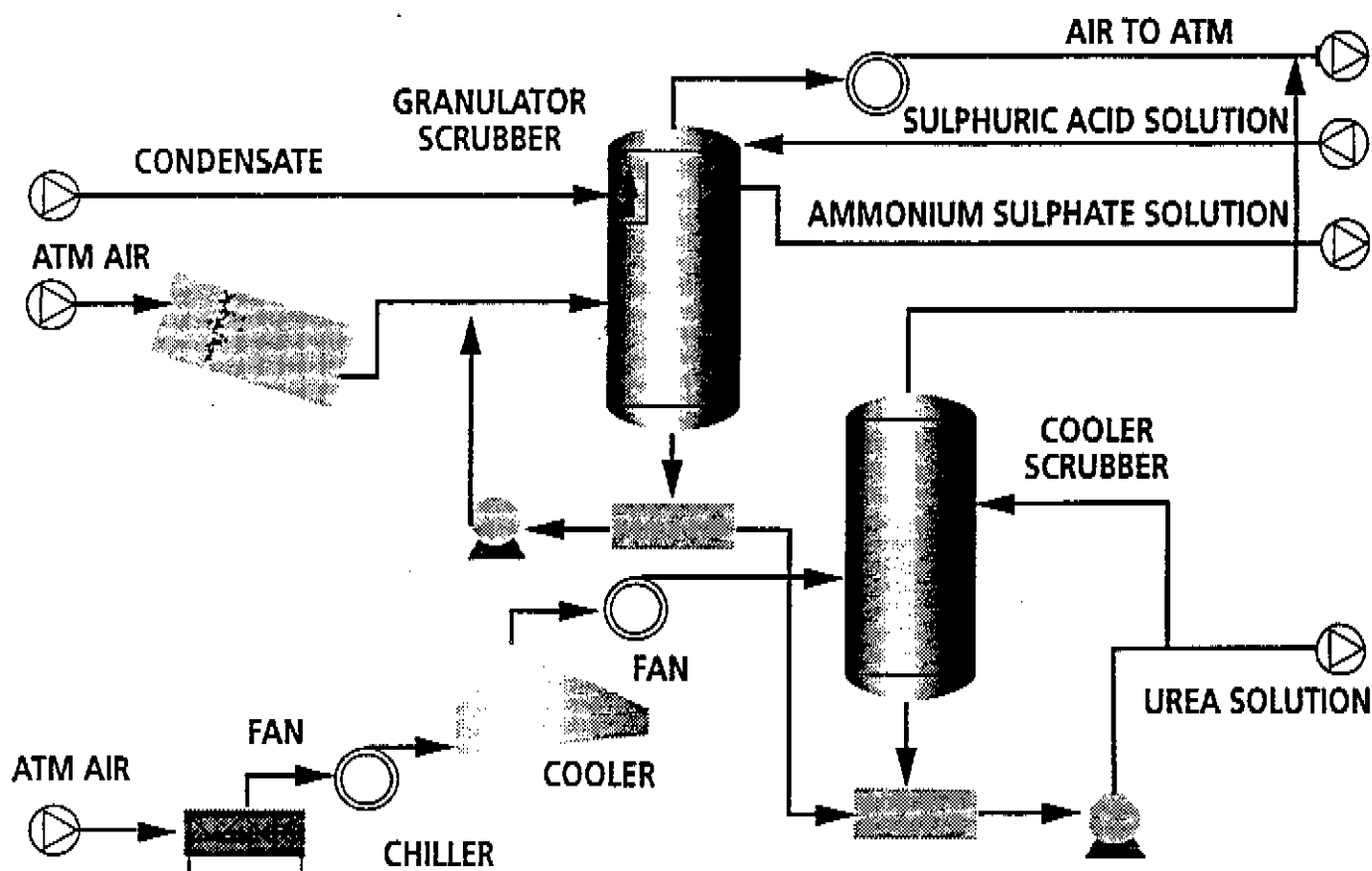


Figure 11 - Values of urea and ammonia in the air to the atmosphere

UREA DUST	15 mg/Nm ³
AMMONIA	20 mg/Nm ³

Figure 12 - Process water treatment section

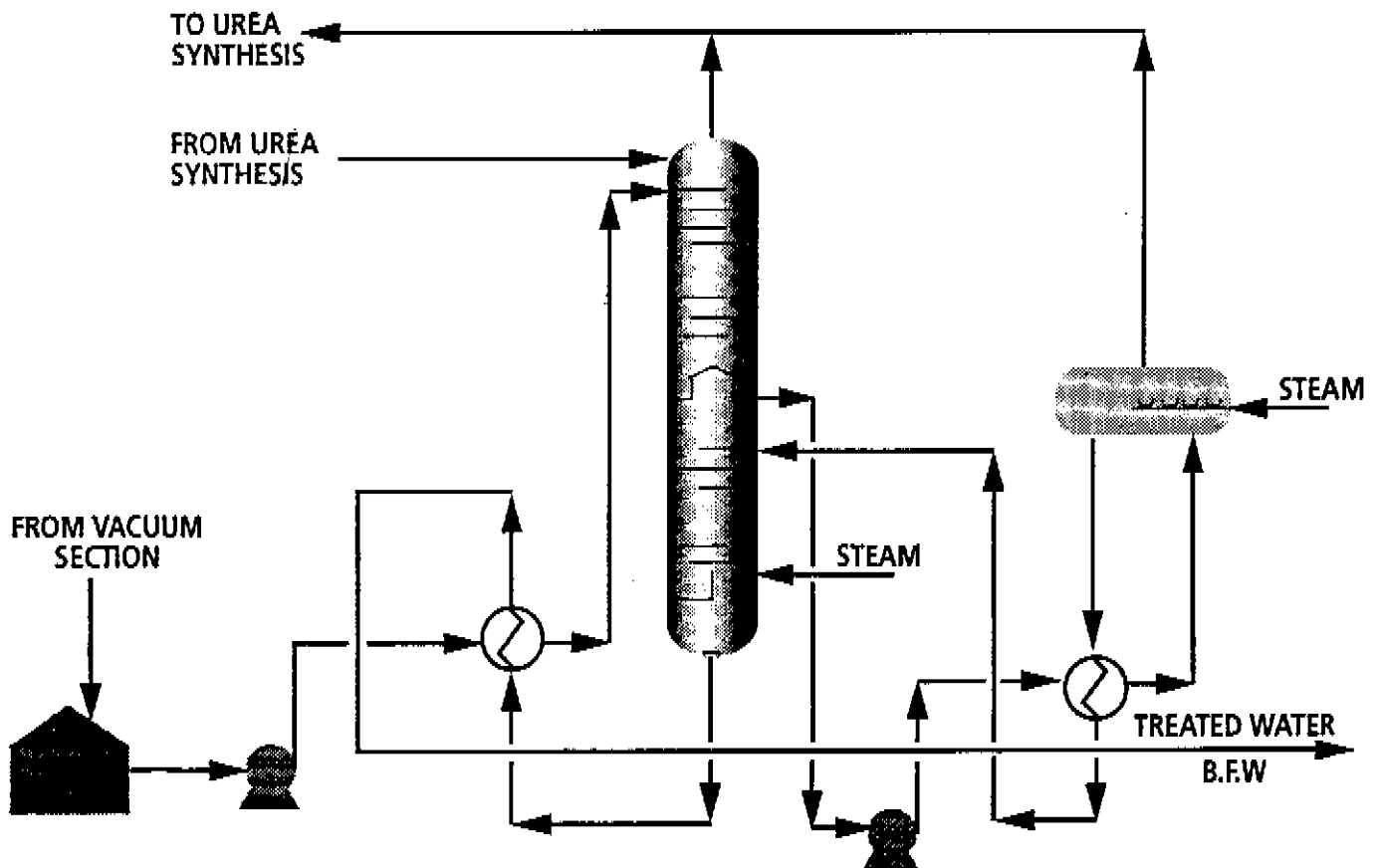


Figure 13 - Process water. Value of pollutants in the discharges process water

UREA: 1 ppm

NH₃: 1 ppm

Figure 14 - Ammonia specific consumption

BASIS:	UREA BIURET MOISTURE	98.8% wt 0.9 % wt 0.3% wt	100% wt	WITH UREA DEDUSTING IN THE PRILLING TOWER
				kg/MT
AS UREA IN THE PRODUCT				559.87
AS BIURET IN THE PRODUCT				4.46
FREE IN THE PRODUCT (150 ppm)				0.15
IN VENTS (10 ppm)				neglig.
IN TREATED WATER FREE AND AS UREA (1 ppm UREA, 1 ppm NH ₃)				neglig.
FREE IN AIR FROM PRILLING TOWER (120 ppm)				1.13
AS UREA DUST IN AIR FROM PRILLING TOWER (15 mg/Nm ³)				0.08
TOTAL				565.69
EFFICIENCY				99.7%

STUDY OF THE F/SiO₂ RATIO IN ORDER TO MINIMIZE FLUORINE EMISSIONS

K. Lahmadi and A. Ben Attia

Compagnie des Phosphates de Gafsa, Tunisia

RESUME

Le travail consiste en une approche sur les formes de silice qui entrent dans la composition des phosphates et leur participation lors de la fabrication d'acide phosphorique par voie humide. En fait, la silice se présente sous deux formes qui ont des comportements différents par rapport à l'acide HF formé.

- *Dans le premier cas une forme de silice dite "Active" qui provient des argiles et qui a la possibilité de réduire l'émission du fluor.*
- *Dans le deuxième cas il s'agit de la silice dite "Non active" ; cette forme ne réagit pas avec le milieu réactionnel, par conséquent elle ne réduit pas l'émission du fluor.*

L'étude de deux phosphates : un phosphate tunisien et un phosphate étranger a permis :

- *d'identifier toutes les formes de silice dans le minerai et dans le phosphogypse.*
- *de prévoir la réactivité de l'acide phosphorique produit par attaque sulfurique en fonction du rapport F/SiO₂ et l'influence de ce rapport sur la filtrabilité du phosphogypse.*
- *de dresser un bilan dans le but de quantifier les formes de silice ayant participé aux réactions dans la bouillie phosphorique.*



1. INTRODUCTION

In phosphoric acid plants, fluorine emissions occur during the reaction of phosphate rock with sulphuric acid as HF and SiF₄ as well as other gases or vapours. Decomposition of phosphate rock follows the reaction:



At present, phosphoric acid producers try to purify polluting gas effluents before discharging them in order to comply with existing regulations.

2. THE ORIGIN OF FLUORINE EMISSION

The following presentation will be limited to secondary reactions of fluorine or siliceous compounds which may occur when phosphate rock is reacted with sulphuric acid. These specific reactions can be summarized as follows:



HF and SiF₄ combine with silica to give H₂SiF₆ which is soluble in the reaction medium.

The distribution of fluorine and silica between the various dihydrate process streams depends mainly on the chemical composition of the phosphate and process water in particular (F, Si, Na, K, Al and Mg).

SiO₂ participating in the reaction is the so-called « Active » silica. It is present in the rock in the form of silicates of various origins. In this case, the theoretical ratio F/Active SiO₂ is close to 1.9. If the ratio exceeds this figure, one can anticipate that the excess fluorine in relation to active silica will be in the form of HF.

The main aim of this paper is to identify all the silicate forms in two phosphate rocks. We will expand on the distribution of active silicates in two phosphate rocks. We will emphasize the distribution of active silicate and its positive relationship with complex fluorine. We will mention fluorine distribution in the various

process streams of the dihydrate process in relation to the mineralogical and chemical composition of the two rocks.

3. MINERAL IDENTIFICATION OF THE ROCKS

Rock 1:

The qualitative characterization of rocks (1) by X ray diffraction (Diagram 1) enabled the following minerals to be identified:

- A carbonate-fluorapatite
- Quartz
- Montmorillonite (a clay containing small amounts of magnesium and sodium)
- Cristobalite
- Zeolite (hydrated aluminium silicate)
- Calcite
- Dolomite (very little).

Chemical analyses of this rock are given in Table 1.

Table 1

P ₂ O ₅	CaO	SiO ₂	MgO	SO ₃	Fe ₂ O ₃	Al ₂ O ₃	Na ₂ O	K ₂ O	F
30.18	48.98	3.41	0.65	3.21	0.26	0.50	1.38	0.07	3.25

Rock 2:

The qualitative characterization of rock (2) by X ray diffraction (Diagram 2) enabled the following minerals to be identified:

- A carbonate-fluorapatite
- Gypsum
- Palygorskite (clay containing magnesium)
- Smectites (a mixture of illite-ferrous montmorillonite)
- Quartz
- Dolomite

Chemical analyses of this rock are given in Table 2.

Table 2

P ₂ O ₅	CaO	SiO ₂	MgO	SO ₃	Fe ₂ O ₃	Al ₂ O ₃	Na ₂ O	K ₂ O	F
30.51	45.94	3.25	0.62	5.14	4.11	0.27	0.080	0.06	2.59

4. DISTRIBUTION OF SILICATES IN THE ROCK

Both rocks have almost the same mineralogical characteristics as a whole. The main difference is the presence of cristobalite and zeolite in the rock (1). Both diagrams show this clearly.

In the rock (1), silica is produced by:

- Clay
- Cristobalite
- Zeolite
- Quartz

In the rock (2), silica is produced by:

- Clay
- Quartz

SiO₂ present in the rock can have two forms:

- A free form made up of quartz and cristobalite
- A combined form with aluminium and, secondarily, with sodium and magnesium in the zeolites and clays.

5. DISTRIBUTION OF SiO₂ IN DIFFERENT SILICATE MINERALS

In the first stage, a quantitative evaluation was made of the silicate minerals present in the composition of the raw materials. Results are given in Tables 3 and 4 for Rocks 1 and 2.

Table 3 - Rock 1

Carbonate-fluorapatite	86 %
Calcite	1 %
Dolomite	1 %
Quartz	3 %
Zeolite	2 %
Clay	3 %
Cristobalite	4 %

Table 4 - Rock 2

Carbonate-fluorapatite	87 %
Gypsum	4 %
Dolomite	2 %
Quartz	3 %
Clay	4 %

The results obtained make possible a fairly accurate calculation as regards the distribution of SiO₂ in the various silicate minerals (Tables 5 and 6).

Table 5 - Rock 1

		SiO ₂ in the rock
Combined silica	SiO ₂ in quartz	1.22
	SiO ₂ in zeolites	0.50
	SiO ₂ in clays	0.60
	SiO ₂ in cristobalite	1.08

Table 6 - Rock 2

		SiO ₂ in the rock
	SiO ₂ in quartz	2.10
Combined silica	SiO ₂ in clays	1.15

At this stage of identification, we can only discuss free silica and combined silica.

In the two identified rocks, the distribution was as follows:

Rock 1:

Combined silica = 1.10%
Free silica = 2.30%

Rock 2:

Combined silica = 1.15%
Free silica = 2.10%

6. IDENTIFICATION OF SILICATES IN PHOSPHOGYPSUM AFTER TREATMENT

As with the rocks, phosphogypsum obtained after filtration was identified by X ray diffraction (Diagrams 3 and 4).

The identified minerals are shown in Tables 7 and 8.

Table 7 - Rock 1 - Phosphogypsum

Gypsum dihydrate
Traces of clay
Quartz
Traces of phosphate

Table 8 - Rock 2 - Phosphogypsum

Gypsum dihydrate
Traces of clay
Quartz
Traces of phosphate

The interesting fact in this process is the complete disappearance of zeolites and cristobalite in the gypsum (1). Quartz is present in both residual products (gypsum), clays are present only as traces.

In our opinion, combined silica is not the only factor responsible for fluorine complexes. Cristobalite, which is a cryptocrystalline silica, participates in the reactive medium for complexing fluorine; it is therefore active. An interesting comparison can be made between the diagram of the rock (1) and a diagram of a siliceous earth generally used as an additive in the reactive medium to increase the amount of active silica. (Diagram 5)

The active silica contents in Rocks (1) and (2) are shown in Tables 9 and 10.

Table 9 - Rock 1

Active silica	2.18
Inactive silica	1.22

Table 10 - Rock 2

Active silica	1.15
Inactive silica	2.10

These measures involved the combination of two identification methods: chemical and mineralogical, but the interesting fact in this case is the identification of the two silicate compounds (zeolites and cristobalite) which exist naturally in the raw material and which will fully play the role of active silica. These two minerals exist frequently in some rocks and their identification should allow the behaviour of the raw material during the reaction to be predicted.

As an indication, we give the chemical characteristics of the phosphogypsum and phosphoric acid obtained after reaction. The results appear in Tables 11 and 12.

Table 11 - Rock 1

	Filtered Phosphoric Acid	Phosphogypsum
P ₂ O ₅	28.60	0.85
CaO	0.20	36.70
SiO ₂	0.35	1.54
MgO	0.55	0.07
Fe ₂ O ₃	0.21	0.06
Al ₂ O ₃	0.34	0.12
K ₂ O	0.03	0.03
F	0.87	0.9
Na ₂ O	0.18	0.65

Table 12 - Rock 2

	Filtered Phosphoric Acid	Phosphogypsum
P ₂ O ₅	25.49	1.53
CaO	0.28	33.24
SiO ₂	0.21	1.65
MgO	0.56	0.03
Fe ₂ O ₃	1.97	2.21
Al ₂ O ₃	0.29	0.11
K ₂ O	0.03	0.01
F	0.62	0.29
Na ₂ O	0.09	0.26

The gypsum factor is roughly equal to 1.45 t/t of reacted rock for rock (1) and 1.36 for rock (2). For the acid produced, the factors are respectively 1.04 and 0.98.

Initially, the amounts of silica were:

- 3.41 for rock (1)
- 3.25 for rock (2)

The silica balance is as follows:

Rock (1) SiO_2 in the gypsum = $1.54 \times 1.45 = 2.23$
 SiO_2 in the acid = 0.35
 Total SiO_2 = 2.58

Rock (2) SiO_2 in the gypsum = $1.65 \times 1.36 = 2.24$
 SiO_2 in the acid = 0.21
 Total SiO_2 = 2.45

The amounts of silica missing in relation to the initial raw material are respectively:

* $3.41 - 2.58 = 0.83$ Rock (1)
 * $3.25 - 2.45 = 0.80$ Rock (2)

Before washing, these siliceous fractions are probably to be found in the gases as SiF_4 . We give below the fluorine and silica balances.

7. SILICA AND FLUORINE BALANCES

Rock 1

	Silica Balance 3.41% = 34.1 kg/t phos.	Fluorine Balance 3.25% = 32.5 kg/t phos.
Acid	$0.35 \times 1.04 = 3.6$ kg/t phos.	$0.87 \times 1.04 = 9.0$ kg/t phos.
Gypsum	$1.54 \times 1.45 = 22.3$ kg/t phos.	$0.9 \times 1.45 = 13.0$ kg/t phos.
Gas	$34.1 - (3.6 + 22.3) = 8.2$ kg/t phos. before washing	$32.5 - (9 + 13) = 10.5$ kg/t phos. before washing

Rock 2

	Silica Balance 3.25% = 32.5 kg/t phos.	Fluorine Balance 2.59% = 25.9 kg/t phos.
Acid	$0.21 \times 0.98 = 2.0$ kg/t phos.	$0.62 \times 0.98 = 6.1$ kg/t phos.
Gypsum	$1.65 \times 1.36 = 22.4$ kg/t phos.	$0.29 \times 1.36 = 3.9$ kg/t phos.
Gas	$32.5 - (2 + 22.4) = 8.1$ kg/t phos.	$25.9 - (6.1 + 3.9) = 15.9$ kg/t phos.

8. REMARKS AND INTERPRETATION

For Rock (1), gases contain 8.2 kg silica capable of complexing 10.37 kg fluorine into SiF_4 gas. The fluorine balance leaves in the gas an amount of 10.5 kg. Considering that the residual amount of fluorine is in the form of HF, this quantity is quite negligible. In any case, washing only concerned SiF_4 gas

For Rock (2), things would not evolve in the same way since the excess fluorine in the gases would be 15.9 kg. The amount of silica present could only complex about 10.1 kg. The amount of fluorine in the form of HF is 5.8 kg. This amount represents 22% of the initial quantity. For this rock, a very intensive washing would be needed to minimize gas emissions.

We give the significant complexation ratios of fluorine to explain the importance of the active silica supply for complexing fluorine in the form of H_2SiF_6 on the one hand and non-corrosive precipitates on the other. Moreover, during the plant maintenance periods, we have identified in the reactor and in the pipes the following products:

- Na_2SiF_6
- Na_3AlF_6

These products can be present in phosphogypsum and in the slimes.

9. COMPLEX RATIOS OF FLUORINE

The significant ratios are the following:

$$\begin{array}{l} * \text{ Active F/SiO}_2 \quad \quad \quad (a) \\ \frac{\text{F}}{\text{Active SiO}_2 + \text{Al}_2\text{O}_3 + \text{MgO}} \quad \quad \quad (b) \end{array}$$

In the first ratio, we only illustrate the value of active silica in comparison with the theoretical value announced initially (1.9).

The second ratio indicates the evaluation of the corrosion of phosphate rock and of the filtrability of the acid produced. In that case, the rock may contain enough active silica, aluminium and magnesium to form non corrosive fluorine complexes.

The following tables give the values of complexation ratios for both rocks:

Rock 1:

$$\begin{array}{l} \text{Active SiO}_2/\text{F} = \frac{3.25}{2.18} = \boxed{1.49} \\ \frac{\text{F}}{\text{Active SiO}_2 + \text{Al}_2\text{O}_3 + \text{MgO}} = \frac{3.25}{2.18 + 0.50 + 0.65} = \boxed{0.97} \end{array}$$

Rock 2:

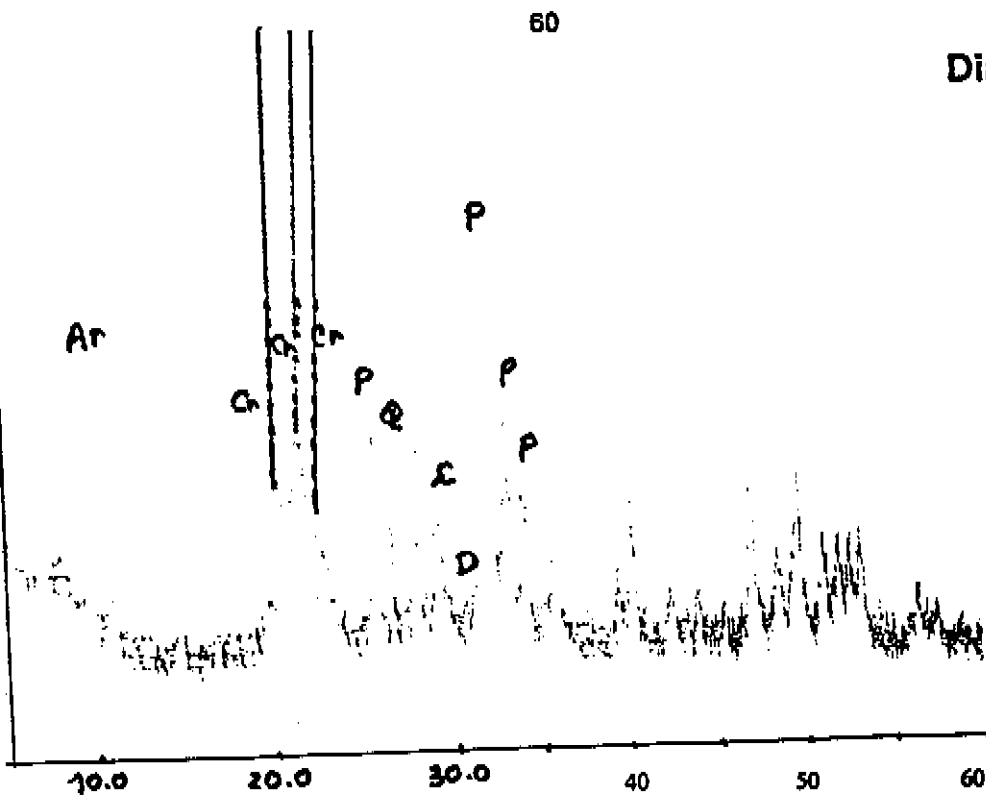
$$\begin{array}{l} \text{Active SiO}_2/\text{F} = \frac{2.59}{1.15} = \boxed{2.25} \\ \frac{\text{F}}{\text{SiO}_2 \text{ active} + \text{Al}_2\text{O}_3 + \text{MgO}} = \frac{2.59}{1.15 + 0.27 + 0.62} = \boxed{1.27} \end{array}$$

The values are significant and demonstrate the corrosive nature of the rock (2).

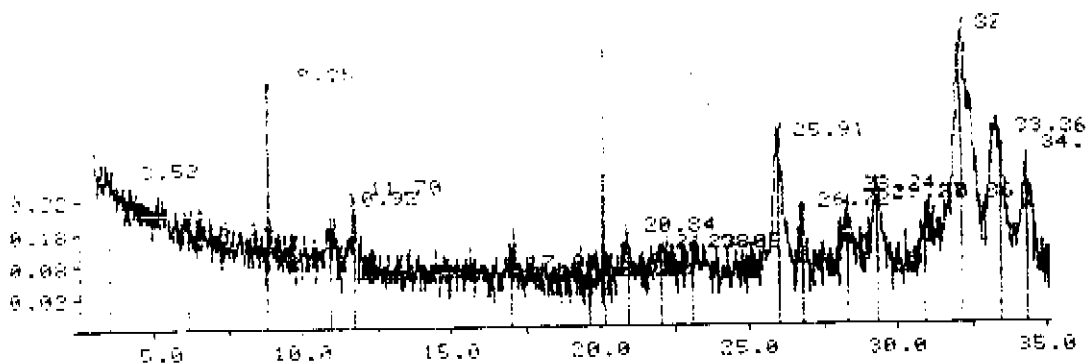
Bibliography

1. Lehr, « Impact of phosphate rock quality on fertilizer market use ». Industrial Minerals (May 1981)
2. Becker, « Phosphates and Phosphoric Acid », 2nd edition (New York, Marcel Dekker Inc. 1989).
3. Schorr, M et al « Use of clays for corrosion control in WPA production », Phosphorus and Potassium (September, October 1993).
4. R. Monaldi, P. Barraco, E. Tomasello, Recyclage du fluor dans les ateliers d'acide phosphorique selon le procédé au dihydrate. (Agrimont SpA, Italie)
5. Groupe Chimique Tunisien, Défluoration des phosphates par les argiles. Internal report. Gabès, 1992.

x10³
1.00
0.81
0.64
0.49
0.16
0.09

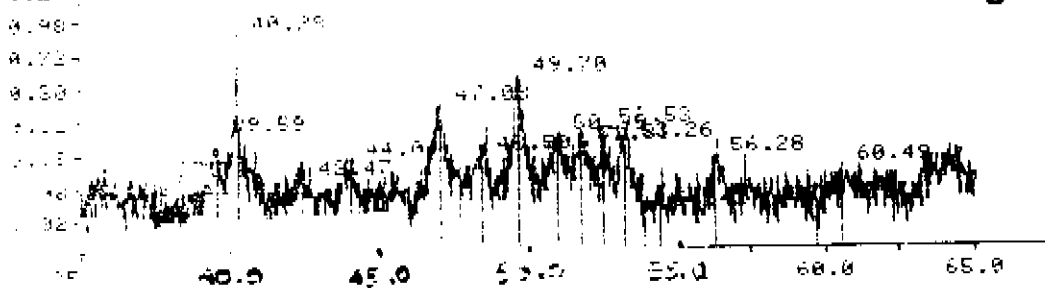


Peak no	Angle (deg)	Fwhm (deg)	Peak (cts)	Backs (cts)	D-spacing (Ang)	I/Imax (%)
1	6.3350	0.10	10.	88.	13.9404	2.32
2	9.8875	0.10	207.	66.	8.9383	47.02
3	11.1725	0.07	37.	36.	7.9130	8.44
4	14.7450	0.15	26.	19.	6.0028	5.90
5	16.9200	0.07	22.	20.	5.2358	5.01
6	19.0350	0.25	10.	22.	4.6585	2.32
7	20.5225	0.40	104.	26.	4.3241	23.59
8	21.6725	0.45	159.	49.	4.0972	36.00
9	22.4425	0.15	31.	81.	3.9563	7.11
10	22.9825	0.30	12.	81.	3.8665	2.62
11	23.7800	0.07	10.	85.	3.7386	2.32
12	25.8600	0.17	213.	27.	3.4424	48.34
13	26.6900	0.12	146.	26.	3.3372	33.20
14	26.1625	0.10	74.	25.	3.1660	16.77
15	29.4900	0.12	81.	35.	3.0264	18.37
16	30.8800	0.15	10.	48.	2.8933	2.32
17	32.0525	0.17	441.	29.	2.7901	100.00
18	32.3450	0.07	306.	29.	2.7655	69.44
19	33.3725	0.17	190.	41.	2.6827	43.18
20	34.2475	0.17	83.	53.	2.6161	18.78
21	35.9525	0.45	41.	37.	2.4959	9.29
22	39.4625	0.20	37.	22.	2.2816	8.44
23	40.3075	0.12	94.	22.	2.2357	21.34
24	42.5000	0.15	31.	22.	2.1253	7.11
25	43.9450	0.25	24.	24.	2.0587	5.44
26	45.4675	0.20	13.	27.	1.9932	2.94
27	47.0875	0.25	102.	20.	1.9284	23.13
28	48.4875	0.30	50.	20.	1.8759	11.43
29	49.6400	0.17	125.	26.	1.8350	28.44
30	51.0700	0.30	55.	28.	1.7869	12.42
31	51.9200	0.20	48.	28.	1.7597	10.80
32	52.5075	0.20	41.	32.	1.7413	9.29
33	53.1525	0.25	45.	36.	1.7217	10.18
34	56.3350	0.30	23.	18.	1.6318	5.22
35	57.4525	0.25	16.	20.	1.6027	3.63
36	58.7225	0.30	10.	25.	1.5709	2.32



2.00
1.60
1.20
0.90
0.72
0.50
0.30
0.10

Diagramme 2



Peak no	Ansle (des)	Tip width (des)	Peak (cts)	Backs (cts)	Π-spac (Ans)	I/I _{max} (%)
1	3.5175	0.25	10.	53.	25.0978	6.77
2	6.1450	0.30	10.	21.	14.3710	6.77
3	8.8450	0.07	14.	12.	9.9893	9.54
4	10.9200	0.30	10.	8.	8.0954	6.77
5	11.7025	0.07	21.	9.	7.5558	13.99
6	17.0075	0.30	10.	6.	5.2090	6.77
7	19.5925	0.12	10.	3.	4.5272	6.77
8	20.0775	0.07	23.	3.	4.4189	15.23
9	20.8450	0.25	12.	3.	4.2579	8.10
10	21.9800	0.50	10.	3.	4.0406	6.77
11	23.0500	0.30	10.	4.	3.8553	6.77
12	25.9100	0.30	64.	4.	3.4359	42.30
13	26.7225	0.20	21.	6.	3.3333	13.99
14	28.2400	0.25	18.	4.	3.1575	12.22
15	29.2700	0.25	28.	6.	3.0487	18.57
16	30.8650	0.20	14.	7.	2.8947	9.05
17	32.0950	0.17	151.	7.	2.7865	100.00
18	33.3600	0.12	59.	12.	2.6837	39.19
19	34.2325	0.25	21.	20.	2.6172	13.99
20	37.1100	0.30	10.	5.	2.4206	6.77
21	37.7000	0.07	10.	4.	2.3841	6.77
22	39.5900	0.12	23.	3.	2.2745	15.23
23	40.2825	0.10	49.	4.	2.2370	32.39
24	42.4700	0.40	10.	9.	2.1267	6.77
25	44.0400	0.35	10.	5.	2.0545	6.77
26	47.0775	0.20	30.	6.	1.9287	19.99
27	48.5000	0.40	11.	8.	1.8754	7.20
28	49.6975	0.12	45.	7.	1.8330	29.67
29	50.9875	0.15	16.	7.	1.7896	10.58
30	51.8075	0.30	10.	12.	1.7632	6.77
31	52.5275	0.15	10.	12.	1.7407	6.77
32	53.2575	0.30	14.	10.	1.7186	9.54
33	54.4225	0.30	10.	10.	1.6845	6.77
34	56.2850	0.15	11.	4.	1.6331	7.20
35	60.4950	0.80	10.	4.	1.5291	6.77

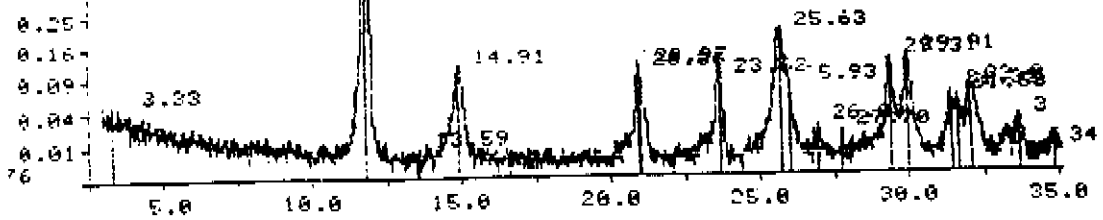
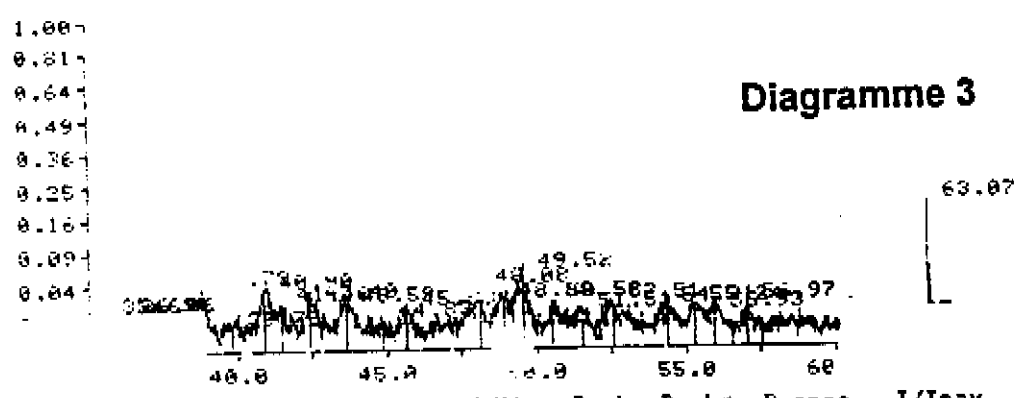
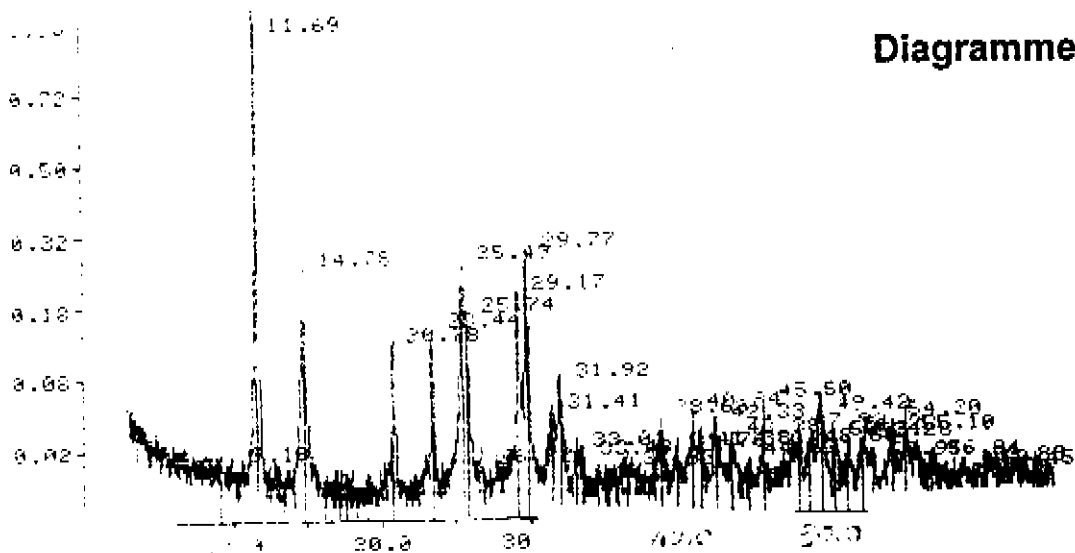


Diagramme 3



Peak no	Angle (des)	Tip width (des)	Peak (cts)	Backs (cts)	D-spac (Ang)	I/Imax (%)
1	3.3275	0.07	10.	37.	26.5305	1.56
2	11.8150	0.22	655.	4.	7.4841	100.00
3	13.5875	0.05	10.	4.	6.5115	1.56
4	14.9150	0.17	110.	3.	5.9348	16.82
5	20.8650	0.10	102.	3.	4.2539	15.57
6	20.9625	0.10	106.	3.	4.2447	16.19
7	23.6175	0.17	121.	3.	3.7640	18.46
8	25.6300	0.20	199.	2.	3.4728	30.34
9	25.9350	0.15	76.	2.	3.4327	11.55
10	26.8775	0.15	12.	2.	3.3144	1.87
11	27.7050	0.07	10.	4.	3.2172	1.56
12	29.3125	0.12	123.	4.	3.0444	18.80
13	29.9100	0.17	128.	5.	2.9849	19.48
14	31.3200	0.07	49.	6.	2.8537	7.48
15	31.5825	0.15	41.	4.	2.8305	6.25
16	32.0050	0.12	74.	4.	2.7941	11.29
17	33.6200	0.20	20.	6.	2.6635	3.09
18	34.7600	0.30	10.	4.	2.5787	1.56
19	35.6350	0.15	10.	4.	2.5174	1.56
20	36.1500	0.20	10.	4.	2.4827	1.56
21	36.2625	0.20	10.	4.	2.4813	1.56
22	38.7900	0.40	24.	3.	2.3196	3.66
23	39.7800	0.40	10.	3.	2.2641	1.56
24	40.9025	0.25	34.	3.	2.2045	5.13
25	41.4575	0.25	14.	3.	2.1763	2.09
26	42.4000	0.15	18.	8.	2.1301	2.82
27	43.5950	0.50	17.	4.	2.0744	2.57
28	44.8350	0.25	10.	4.	2.0199	1.56
29	45.6250	0.40	11.	4.	1.9867	1.66
30	48.0775	0.10	29.	4.	1.8909	4.45
31	48.8800	0.25	21.	5.	1.8618	3.23
32	49.5200	0.12	50.	8.	1.8392	7.69
33	50.5250	0.25	10.	13.	1.8049	1.56
34	51.5100	0.35	10.	5.	1.7727	1.56
35	52.5075	0.12	10.	7.	1.7413	1.56
36	54.3150	0.25	15.	5.	1.6876	2.32
37	55.2450	0.30	13.	5.	1.6614	1.98
38	55.9300	0.25	11.	6.	1.6426	1.66
39	56.9750	0.10	10.	7.	1.6150	1.56
40	60.8700	0.80	10.	3.	1.5206	1.56
41	67.0700	0.07	31.	3.	1.4727	4.79

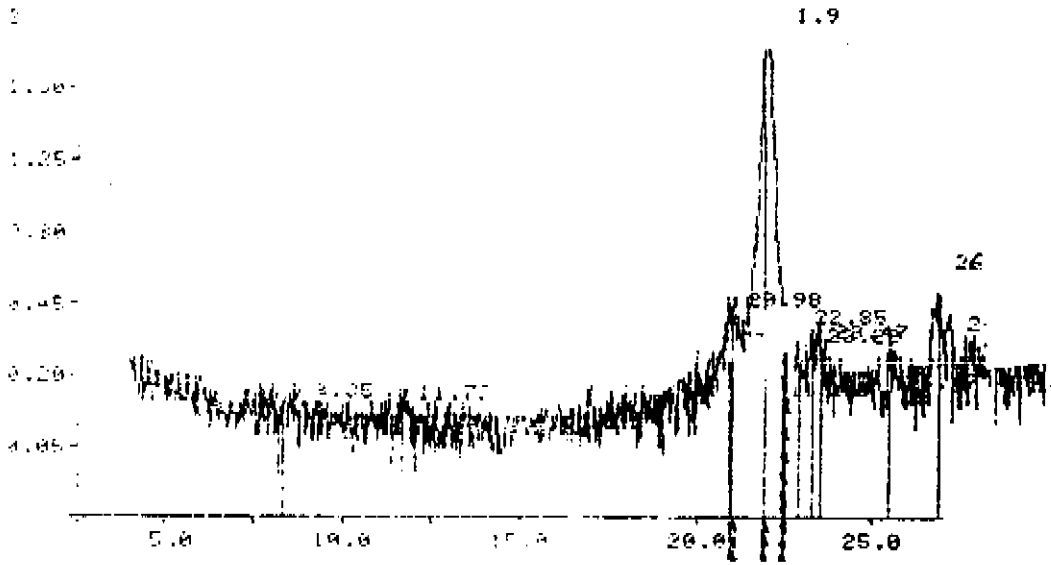
Diagramme 4



Peak no	Angle (deg)	Tip width (deg)	Peak (cts)	Racks (cts)	D-spac (Ang)	I/Imax (%)
1	9.1850	0.40	10.	10.	9.6203	1.02
2	11.6925	0.17	1005.	6.	7.5622	100.00
3	14.7800	0.20	250.	4.	5.9887	24.84
4	20.7850	0.17	114.	3.	4.2701	11.39
5	23.4450	0.20	139.	3.	3.7913	13.86
6	25.4875	0.20	256.	4.	3.4919	25.48
7	25.7425	0.07	166.	5.	3.4579	16.56
8	26.7225	0.20	10.	4.	3.3333	1.02
9	29.1750	0.17	196.	6.	3.0584	19.50
10	29.7700	0.28	262.	6.	2.9986	26.12
11	31.4075	0.20	36.	6.	2.8459	3.58
12	31.9225	0.12	69.	7.	2.8012	6.86
13	33.0100	0.30	10.	12.	2.7113	1.02
14	33.4350	0.15	10.	12.	2.6778	1.02
15	34.5150	0.30	10.	3.	2.5965	1.02
16	36.3200	0.25	10.	3.	2.4715	1.02
17	38.6250	0.25	24.	4.	2.3291	2.39
18	39.7625	0.20	12.	5.	2.2651	1.22
19	40.8375	0.25	27.	7.	2.2079	2.69
20	41.3775	0.25	18.	5.	2.1803	1.76
21	42.3350	0.30	19.	14.	2.1332	1.93
22	43.3750	0.15	16.	5.	2.0844	1.59
23	44.6200	0.07	10.	4.	2.0291	1.02
24	45.5050	0.07	21.	3.	1.9917	2.11
25	47.9250	0.12	21.	4.	1.8966	2.11
26	48.6050	0.30	10.	7.	1.8716	1.02
27	49.4250	0.40	44.	7.	1.8425	4.33
28	50.3100	0.12	16.	6.	1.8121	1.59
29	51.2250	0.25	12.	5.	1.7819	1.22
30	52.2850	0.20	18.	4.	1.7482	1.84
31	52.9150	0.30	10.	4.	1.7289	1.02
32	54.2050	0.40	14.	10.	1.6908	1.44
33	55.0975	0.20	21.	6.	1.6655	2.11
34	56.8400	0.25	10.	9.	1.6185	1.02
35	59.8300	0.12	10.	4.	1.5445	1.02
36	60.4475	0.30	10.	3.	1.5302	1.02

Diagramme 5

Peak no	Angle (deg)	Tip width (deg)	Peak (cts)	Backs (cts)	D-spec (Ang)	I/I _{max} (%)
1	8.3500	1.20	10.	11.	10.5803	4.49
2	11.7150	0.30	10.	7.	7.5477	4.49
3	20.9725	0.35	31.	11.	4.2323	13.75
4	21.9275	0.15	228.	17.	4.0501	100.00
5	22.8475	0.07	10.	21.	3.8891	4.49
6	23.2200	0.12	12.	21.	3.6275	5.07
7	23.4700	0.12	12.	21.	3.7873	5.37
8	25.4650	0.20	10.	18.	3.4949	4.49
9	26.8400	0.07	45.	14.	3.3189	19.69
10	27.1875	0.15	19.	14.	3.2773	8.49
11	28.4325	0.30	10.	18.	3.1365	4.49
12	29.0825	0.20	11.	19.	3.0679	4.78



**PRODUCTION AND CHARACTERISTICS OF
KOLA APATITE CONCENTRATE "SUPER"¹**
V. Golovanov, A. Petrovsky, N. Vasilieva, M. Bykov
JSC "Apatit", Russia

1. Production of Kola apatite concentrate in JSC "Apatit"

The apatite concentrate in JSC "Apatit" is obtained from the apatite-nepheline ores by flotation method.

Before 1993 only "Standard" apatite concentrate was produced. It met the requirements of the specifications GOST - 22275-90, and will be referred to as "Standard".

The following are the requirements of concentrate "Standard":

- P₂O₅, % wt (dry basis) ≥ 39,0
- H₂O, % wt 1.0 ± 0,5
- Residue on sieve No 0.16mm, % wt ≤ 13,5

Sesquioxides (FeO, Fe₂O₃, Al₂O₃) by weight consist of not more than 3,0% guaranteed by the supplier.

The mineral chemical and granulation structures have wide range in composition, depending upon the processed ores and P₂O₅ content in the concentrate (39,0 - 39,4 %)

2. Origin and properties of Kola apatite concentrate "Super I"

The technology for the production of concentrate "Super" was first developed and tested in the plant ANOF-2 of JSC "Apatit" in 1993 from the usual flotated apatite concentrate. Hereinafter, it will be referred as Kola apatite concentrate "Super I".

As expected from the theoretical basis, when a disproportional distribution of apatite by particle size classes in flotated apatite concentrate was present, large particles were enriched, and the small particles, on the contrary, were impoverished of apatite (Table 2.1)

Table 2.1 - The distribution P₂O₅ on size classes of usual apatite concentrate

Size classes, mm	Mass, %	P ₂ O ₅ , %	Extraction P ₂ O ₅ , %
More than 0,200	4,2	39,37	4,3
0,200 - 0,160	8,4	40,12	8,7
0,160 - 0,100	19,1	40,29	19,8
0,100 - 0,071	14,2	40,39	14,7
0,071 - 0,040	12,9	40,45	13,4
0,040 - 0,020	17,9	39,20	18,0
0,020 - 0,010	8,6	37,56	8,3
0,010 - 0,005	6,6	36,47	6,2
Less than 0,005	8,1	31,82	6,6
	100,0	39,90	100,0

On this, existing technology and available equipment, the concentrate "Super" was obtained as a circulating fraction of concentrate's hydrocyclones, which were used in the circuit of flotated concentrate dehydration.

The generalized technological scheme to recover apatite concentrate "Super" is indicated on Figure 2.1.

The main feature of this technology are:

¹ The report has been translated by N. Ryazantseva

- To obtain a given quantity of a concentrate "Super", it is necessary to use about 65% of total usual flotated concentrate output. It means, that during the process time of a concentrate "Super" production, the reorganization of technological scheme almost of the whole factory is required;
- Output of the concentrate "Super" from the usual flotated concentrate is around 20-22%;
- With overflow liquids, there is plenty of lowered quality product from the concentrate's hydrocyclones coming into the production process of usual flotated concentrate. The lowered quality consists of particle size of about 6% more than 0,16 mm, and the P_2O_5 content of about 38,90%.

The results are as follows:

- ⇒ A specific load on thickening equipment grows to 25-40%, and increasing of operations thicking field for usual flotated concentrate is required for avoidance of that.
- ⇒ Kek humidity is increased by 0,3% on average during the filtration of usual flotated concentrate.
- Essential fluctuations of the output and the special features of grain consistence of the concentrate "Super" makes rather difficult the drying process of concentrate and maintenance of its needed humidity (0.8-1.0%) and as the result - the fluidity:
- Losses of apatite are increased during the production process of the concentrate "Super" with changes of technological regimes. The extraction of P_2O_5 in the final product comes down and the economic parameters of plant operation on the whole becomes worse.

The structure characteristics of the concentrate "Super I" are indicated on Tables 2.2-2.4. In comparison with the "Standard" concentrate the first one has more stable mineral, chemical and granulation structure, namely:

- The P_2O_5 of apatite in concentrate Super I increases up to 97-98% while the share of other minerals decrease to 2,0 - 2,7%
- The proportion of grains with sizes more than 0,16 mm increases from 11-13% up to 35% and the particles with size less than 0,071 decreases from 55-58% to 17-20%, including the most dusting class with particle size less than 0.02 mm, which reduce from 23.3% to 5,2%. This accounts for the difference in granulation characteristics
- The difference in the chemical composition consists in increasing of contents P_2O_5 up to 40.0-40.2% and a corresponding reduction the contents of titanium and aluminium oxides .

Table 2.2 - The distribution P_2O_5 by size classes in the concentrate "Super I"

Class size, mm	Mass, %	P_2O_5 , %	Extraction P_2O_5 , %
More than 0,200	14,3	40,05	14,2
0,200 - 0,160	22,4	40,45	22,5
0,160 - 0,100	34,8	40,72	35,3
0,100 - 0,071	11,6	40,61	11,7
0,071 - 0,040	6,6	40,51	6,6
0,040 - 0,020	5,1	39,67	5,0
0,020 - 0,010	2,0	38,59	1,9
0,010 - 0,005	1,3	36,78	1,2
Less than 0,005	1,9	32,93	1,6
	100,0	40,25	100,0

Table 2.3 - Mineral structure of the concentrate "Super I", obtained in plant ANOF-2 conditions

Name of minerals	Mass share of mineral, %
Apatite	97,2 - 98,0
Nepheline	0,8 - 1,4
Aegirine	0,8 - 1,2
Sphene	0,1 - 0,2
Lamprophyllite	0,1 - 0,2
Feldspar and Hydromica	0,1 - 0,2
Titanomagnetite and Ilmenite	Traces

Table 2.4 - Chemical structure of the concentrate "Super I", obtained in plant ANOF-2 conditions

Elements	Mass share, %	Elements	Mass share, %
SiO ₂	1,60 - 2,10	MnO	0,02 - 0,05
TiO ₂	0,15 - 0,25	CaO	50,80 - 51,50
Al ₂ O ₃	0,35 - 0,60	SrO	2,70 - 2,90
Fe ₂ O ₃	0,30 - 0,50	Na ₂ O	0,30 - 0,50
FeO	0,01 - 0,03	K ₂ O	0,10 - 0,20
P ₂ O ₅	40,00 - 40,30	F ₂	3,10 - 3,40
Sum TR2 O3	0,85 - 0,95	H ₂ O	0,10 - 0,20
MgO	0,05 - 0,10		

3. Production of Kola apatite concentrate "Super" using two stage flotation concentration

3.1 Short description of concentrate "Super II" production technology

The defects of concentrate "Super I" production technology, described in section 2.1. necessitated the research to preserve the positive features obtained in concentrate "Super I".

A two-stage cycle of flotated concentrate technology was developed, including a hydrocyclone overflow recleaning after the first step in hydrocyclone of the second stage. Thus, the hydrocyclones of both stages are united and form a final product concentrate "Super II".

Such technology permits increases in output of concentrate "Super" from the concentrate tanks of extraction not only largest, but also intermediate size classes within the specified contents P₂O₅ (see Table 2. 1). This technology was tested in the plant ANOF-2 conditions.

The flow chart for the production of concentrate "Super II" is shown in Figure 3. 1.

Distinctive features of the technology are as follows:

- Initial quantity of flotated concentrate required for the production of concentrate "Super II", was less compared with concentrate "Super I" by 2.6 times;
- The output of the concentrate "Super II" from usual flotated concentrate in comparison with concentrate "Super I" is increased by 22 to 42%;
- The recycled product returned to the production process of normal apatite concentrate indicated in Figure 2.1 is reduced from 418 to 136 t/hour, approximately three times less. In this case, recycled product has lower contents P₂O₅ (38.20%) and class of particles with size more than 0,16-2.0% only.

3.2 Granulation structure of the concentrate "Super II"

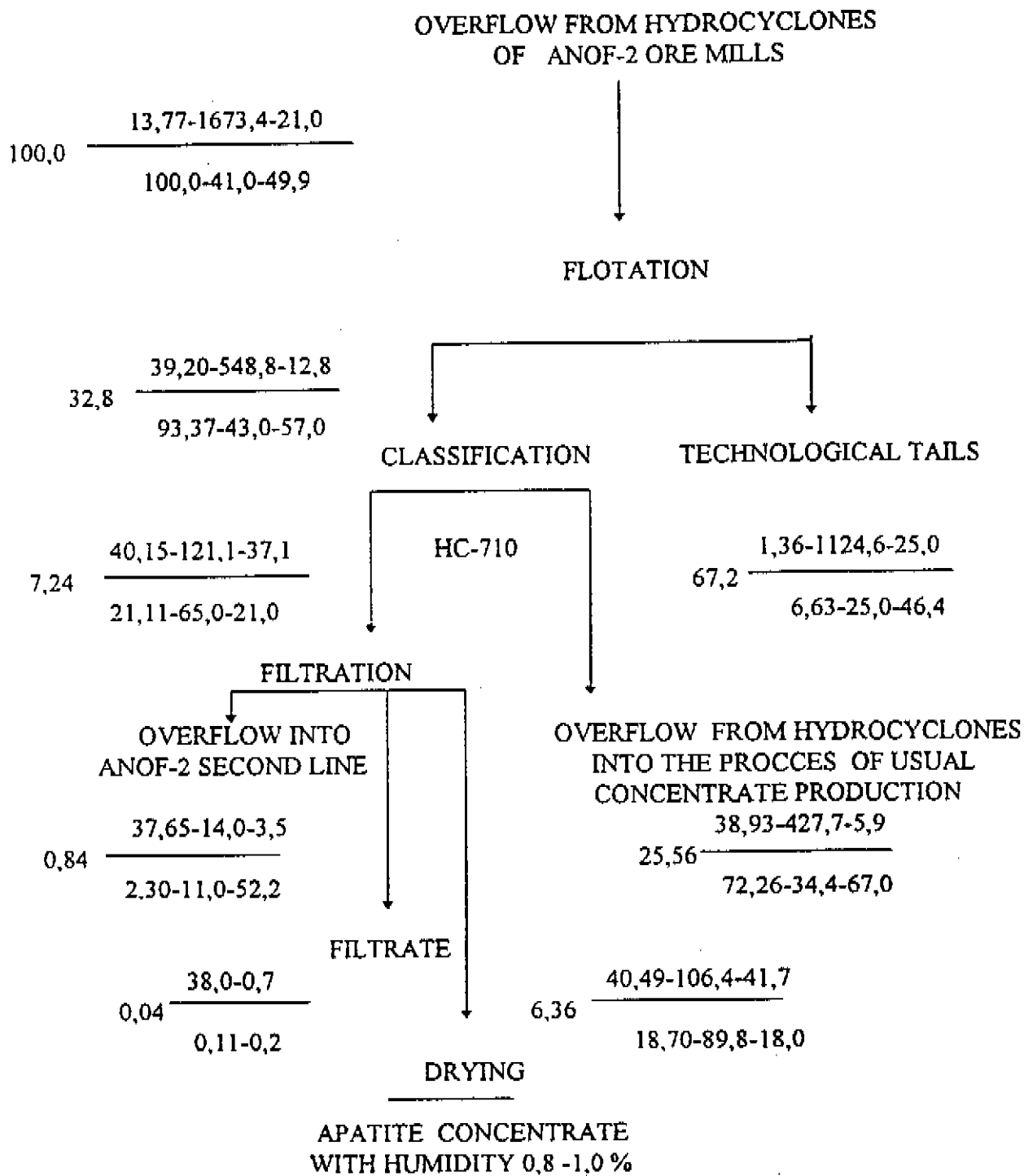
In Table 3.1, we can see the granulation characteristics of all products, obtained from usual flotated concentrate during two stage cycle in the hydrocyclones. The graphs on Figures 3.2 and 3.3 are constructed on the basis of the Table 3.1. It should note, that the particles of the first stage of classification is a concentrate "Super I".

From the indicated data (Figure 3.2), it is clear concentrate "Super II" differs from a concentrate "Super I" mainly by the higher contents of size classes 0.02-0.15 mm and lower contents of classes larger than 0.15 mm. The contents of size classes less than 0.020 mm in both products are approximately the same.

The drawing of size classes extraction from usual flotated concentrate into concentrates "Super I" and "Super II" are indicated on the Figure 3.3.

It is clear from the drawing, that the addition of a second stage of cycle helps to increase the output of the final product by raising all size classes extraction. Thus the largest increase comes with intermediate size classes. It should note also, that the limit size of the median (D50) when changing from one stage into two stage cycle is reduced from 0.11 mm to 0.08 mm.

TECHNOLOGICAL CIRCUIT OF APATITE CONCENTRATE "SUPER I", OBTAINING IN ANOF-2 CONDITIONS



Designations:

	Mass share P ₂ O ₅ , %	Productivity t / Hour	Mass share of class. > of 0,16 mm, %
Output of concentrate	Extraction P ₂ O ₅	Mass share of solid %	Mass share of class <0,071 mm %

Fig. 2.1

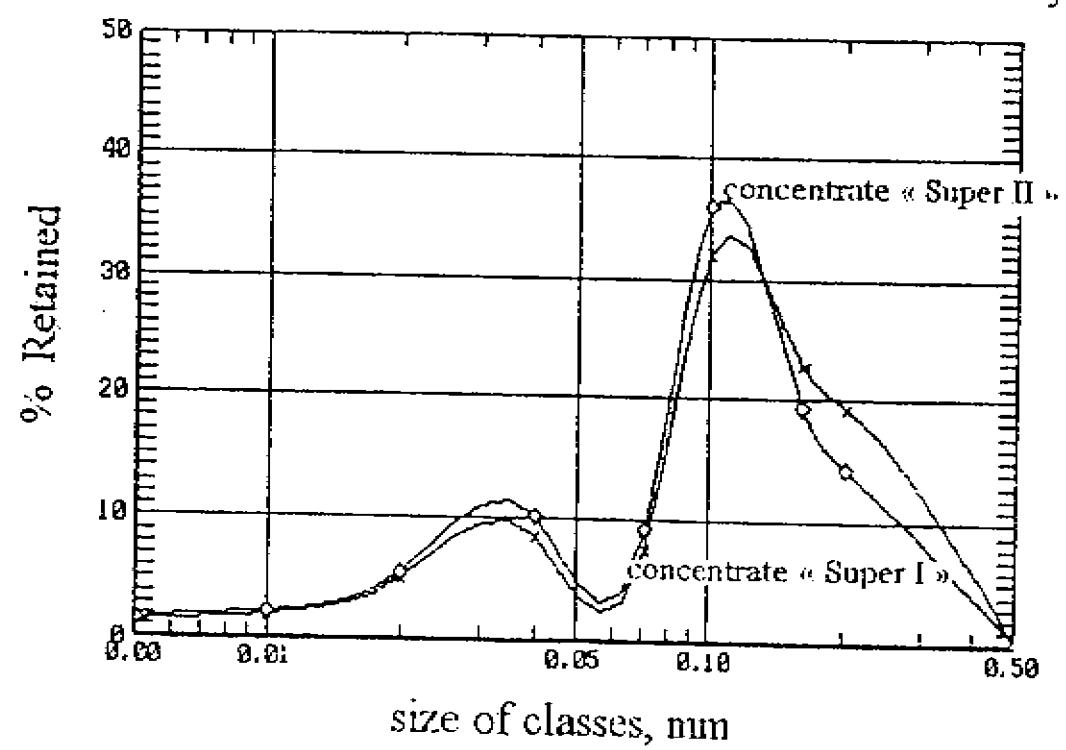


Fig 3.2

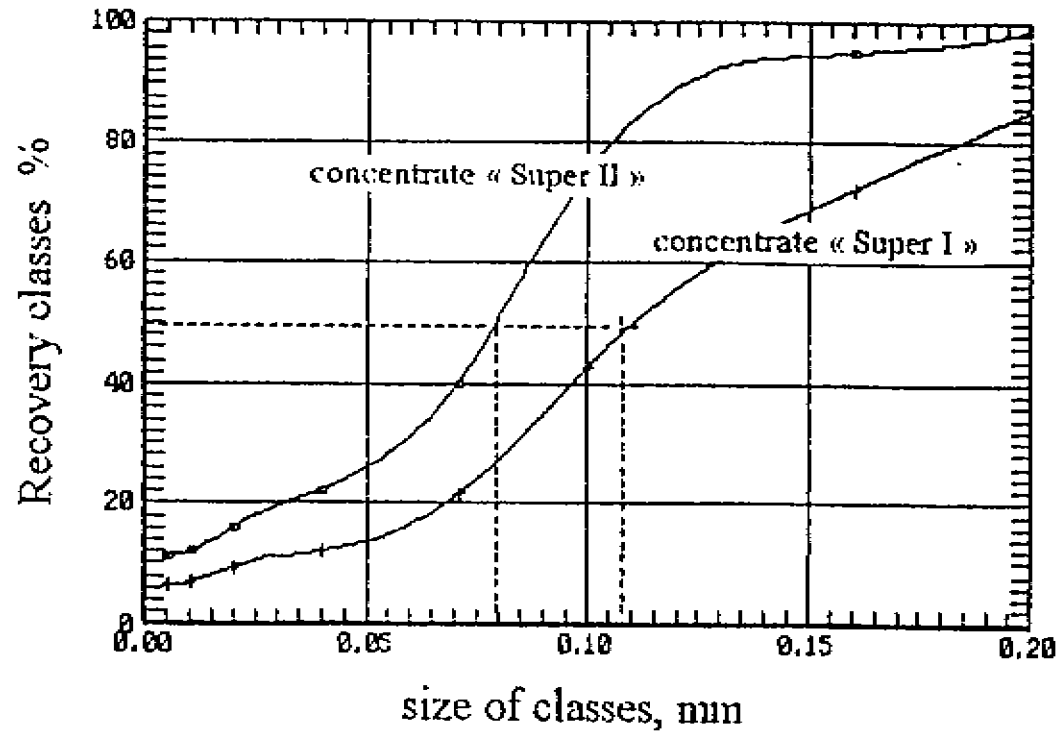


Fig 3.3

Table 3.1. Granulation characteristic of products from two stage usual flotated concentrate classification

Size classes, mm	Mass share of the class, %					
	Flotated concentrate	Overflow of HC one stage	Sands of HC one stage	Overflow of HC two stages	Sands of HC two stages	Apatite concentrate "Super II"
More than 0,200	6,2	1,3	19,2	0,1	5,5	14,2
0,200 - 0,160	8,7	3,3	22,7	0,7	13,0	19,2
0,160 - 0,100	20,6	16,3	31,9	8,9	43,1	36,0
0,100 - 0,071	9,8	10,7	7,5	10,3	12,0	9,1
0,071 - 0,040	19,6	23,8	8,4	26,8	13,0	10,0
0,040 - 0,020	14,7	18,5	4,9	21,9	6,2	5,4
0,020 - 0,010	8,1	10,5	1,9	12,7	2,6	2,2
0,010 - 0,005	5,9	7,6	1,3	9,2	1,9	1,5
Less than 0,005	6,4	8,0	2,2	9,4	2,7	2,4
Output, %	100,0	72,4	27,6	56,9	15,5	43,1

3.3. Mineral structure of concentrate "Super II"

The mineral structure of concentrate "Super II" produced in the ANOF-2 conditions, is indicated in Table 3.2

Table 3.2 - Mineral structure of concentrate "Super II", after two stage cycle on the plant ANOF-2 (26.06.96)

Mineral name	Mineral mass share, %
Apatite	98,3
Nepheline	0,9
Aegirine	0,5
Sphene and Lamprophyllite	0,15
Lepidomelane	traces
Titanomagnetite and Ilmenite	0,1
Feldspar and Hydromica	0,05

It is clear from the comparison of concentrate "Super I" and "Super II", that the mineral structure of both products is practically identical. That is to say, the technology changes in concentrate "Super" production process were not reflected in mineral structure of final product practically.

3.4. Chemical structure of concentrate "Super II"

The complete chemical analysis of concentrate "Super II" structure, as in case with concentrate "Super I" (Table 2.4) was not carried out. However, it practically corresponds with structure of concentrate "Super I" under the contents of such components, as P_2O_5 - 40.45%, Al_2O_3 -0,35% and TiO_2 -0.13%.

4. Dusting of concentrates "Super I" and "Super II"

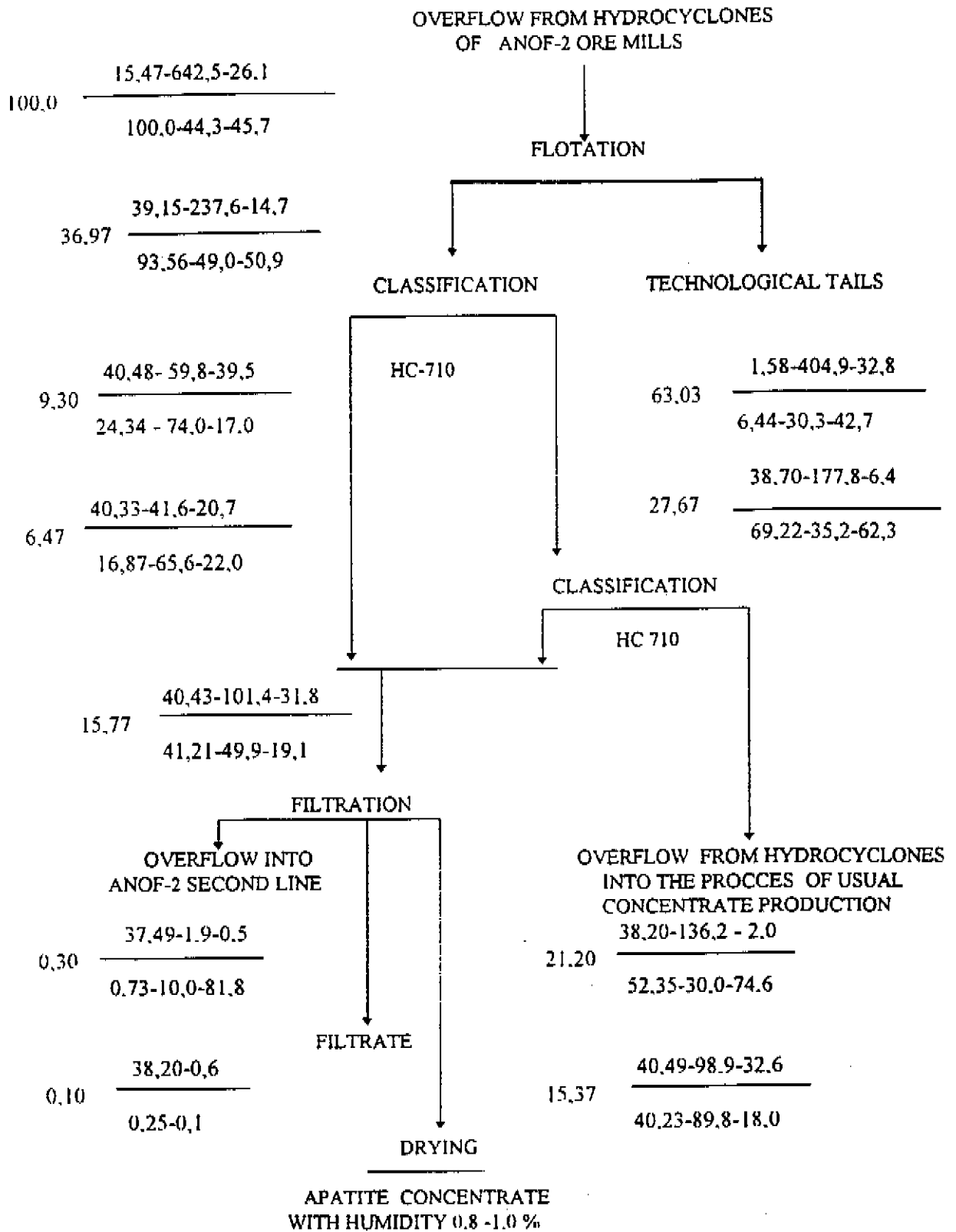
The special tests concerning the dusting of concentrate "Super II" was not conducted. However, the concentrate "Super II" dusting parameters is acceptable our consumers using "Super I".

As described in item 2.2, concentrates "Super I" and "Super II" practically not differ in the contents of fine-dispersion fractions with size less than 0,02 mm which cause large dusting. Therefore, under real conditions concentrate "Super II" in dusting characteristics.

5. Fluidity of concentrates "Super I" and "Super II"

The fluidity of concentrates "Super I" and "Super II" depends of humidity. It was determined under laboratory conditions with a special method, which shows different properties of some loose powdered materials on the basis of the several characteristics. This theory was developed by Karra. Fluidity of a powder estimates on four parameters according with such theory:

TECHNOLOGICAL CIRCUIT OF APATITE CONCENTRATE "SUPER II", OBTAINING IN ANOF-2 CONDITIONS



Designations:

	Mass share P_2O_5 , %	Productivity t / Hour	Mass share of class. > of 0,16 mm, %
Output of concentrate	Extraction P_2O_5	Mass share of solid %	Mass share of class <0.071 mm %

- corner of natural slope
- compactness
- spatula corner
- agglutination

Each of this parameters consists of a 25-mark system. The sum of marks is the common characteristic of loose powder fluidity. Under the Karra table according the marks sum the working characteristics for different stages of fluidity are determined.

On base of above described method the characteristics of apatite concentrates "Super I" and "Super II" fluidity were determined. The results of these tests are in Table 5.1.

From this data it is clear that apatite concentrates "Super I" and "Super II" have the same fluidity with identical humidity of products. The changes of concentrate "Super" obtaining technology does not practically influence to the fluidity of the final product.

In the Table 5.2, the working characteristics of loose products are listed in depending on the fluidity.

Table 5.2 - Mineral structure of concentrate "Super II", after two stage cycle on the plant ANOF-2 (26.06.96)

Characteristic of fluidity	Working characteristic
Excellent	The material does not hang. Auxiliary equipment is not required
Good	The material does not hang. Auxiliary equipment is not required
Satisfied	The material sags at special conditions. Auxiliary equipment is not required usually
Allowable	The material hangs sometime. Auxiliary equipment may be required
Unsatisfied	Auxiliary equipment, vibration is necessary

6. The conclusion

In this report we try to show briefly some special features of apatite concentrate "Super I" and "Super II" production technology on the basis of classification of the flotated concentrate by size classes.

The main properties of these concentrates are described.

It is visible from the indicated data that the concentrate "Super II", which was obtained by two stage method of classification the normal flotated concentrate in hydrocyclones differs in granulation structure with size classes. larger than 0.02 mm from a concentrate "Super I", which was delivered earlier.

The concentrate "Super II" does not practically differ from the concentrate "Super I" on mineral and chemical structures or dusting and fluidity characteristics.

But the industrial tests confirms that the concentrate "Super II" production technology improves essentially the drying process with stabilization of humidity contents in limits of 0.6 - 1.0 %.

Table 5.1 - Research results of Kola apatite concentrate "Super I" and "Super II" fluidity characteristics

Product	H ₂ O %	Corner of natural slope		Compactness (C) %				Spatula corner		Agglutination		Homogeneity coefficient		Marks sums	Fluidity characteristics
		degree	mark	loose weight g/sm ³		C	mark	degree	mark	%	mark	%	mark		
				loose cond.	Compact cond.										
Apatite concentrate "Super I"	1,7	-	0	1,03	1,43	28,0	12	82,5	7	-	-	3,6	23	42	not satisfied
	1,1	-	0	1,17	1,54	24,0	16	72,7	12	-	-	3,6	23	51	not satisfied
	0,6	39,7	18	1,49	1,76	15,3	20	49,0	16	-	-	3,6	23	77	satisfied
	0,0	32,6	21	1,76	1,96	10,2	22	38,2	20	-	-	3,6	23	86	good
Apatite concentrate "Super II"	1,7	-	0	1,04	1,44	27,8	12	80,0	7	-	-	3,8	23	42	not satisfied
	1,5	-	0	1,08	1,50	28,0	12	78,0	7	-	-	3,8	23	42	not satisfied
	1,1	-	0	1,17	1,54	24	16	73,0	12	-	-	3,8	23	51	not satisfied
	1,0	39,5	18	1,41	1,63	13,5	21	55,0	16	-	-	3,8	23	78	satisfied
	0,6	37,8	18	1,64	1,87	12,3	21	46,8	16	-	-	3,8	23	78	satisfied
	0,0	33,0	21	1,73	1,93	10,4	22	38,6	20	-	-	3,8	23	86	good