

IFA Technical Conference

The Hague, The Netherlands
6-8 October 1992

WELCOME ADDRESS
by
C.E. Childers
Chairman, Technical Committee

Ladies and gentlemen. On behalf of the Technical Committee, I wish to welcome you to 24th biennial Technical Conference the history of which dates back to the inaugural conference at Landskrona, Sweden in 1947. We are pleased to be in this wonderful city again. The previous occasion we enjoyed the warm hospitality of Den Haag was in 1976. Only Paris can claim the distinction of hosting the Technical Conference twice.

We are meeting here under very difficult circumstances for the fertilizer industry. Fertilizer consumption has declined for three consecutive years: even the most ardent optimist will not predict that this trend will reverse soon. It is not that we are not in need of fertilizers or that the world population has stopped growing. A large part of the world is in dire need for fertilizers. The fact that in the countries where there are agricultural surpluses, and that good productivity is maintained by adequate doses of fertilizer inputs is a good testimony of our role to feed the masses.

Today, we are faced with multi-faceted problems. Many of the more prosperous developed countries are reducing their agricultural surpluses. Even among the developing countries, there are steps being taken to reduce or abolish agricultural subsidy programs altogether. Further, the industry in the developed countries is faced with many difficult environmental regulations which quite often arise from the misconception of the significance and magnitude of the problems. As a result, research has to be carried out just to verify the magnitude of the problem.

The gloom in the fertilizer industry will have a long term effect on the technological development of the fertilizer industry. As the industry restructures and cost cutting measures are adopted to stay afloat, the research and development department becomes the most vulnerable component of the company. Manpower is diverted to production based on existing technology. Innovation, the cutting edge of any industry, is being blunted. In Europe, several of the large fertilizer manufacturers have cut production by one to two million tons and several thousand jobs disappear in the process. It will appear that the burden of meeting environmental regulations in production will no longer rest solely on the shoulders of the individual companies but increasingly by consultant companies with strong innovations and as well as research institutions.

This conference attracts a good response for the offer of papers. We are glad to receive several papers for the first time from research institutes who are not members of IFA. This is healthy trend and should be further encouraged. We like to see more offers of papers on emission abatement technology, construction materials, removal of micro-impurities, and ways to improve operational and maintenance efficiency.

We are also striving to make the Conference more attractive. One way to encourage more offers of papers is to waive to registration fee of the principal authors. We are holding a technical exhibition for the first time.

We are fortunate to have Dr. Haldor Topsoe who has so kindly consented to spare us some time from his busy schedule to give us the keynote address to precede the start of the general working session. Dr. Topsoe needs no introduction. He started his technology company before World War II.

It is my pleasure, on behalf of all of you to thank Klaas Horstra, President of VKP - the Dutch Fertilizer Producers Association, and his members, for their kind invitation to a reception and dinner tomorrow. We would also like to thank BASF, Hydro Agri Sluiskil and Rhône-Poulenc for hosting the technical visits and lunches. These companies possess the state-of-the-art technology and I am sure you all will benefit immensely from the visits.

I have been told that there is a record-breaking 47 countries being represented here. Reflecting on the current situation of the industry, we have just over 200 participants compared with 350 participants from 45 countries when the previous record was set in Paris in 1984. The fact that we have the record number of countries represented here indicate clearly that the industry clearly attaches importance to technological innovations.

Finally, I wish all of you good deliberations for the next three days.

WELCOME ADDRESS
by
K. Horstra
President, VKP, Netherlands

Mr. President, Ladies and Gentlemen,

The Netherlands, often referred to as Holland, is a country where fertilizer production has taken place for more than a century.

In 1875, the first superphosphate was produced in a factory not too far from here which used to make dye out of madder roots.

The production of dye on the basis of this agricultural cashcrop became economically impossible due to chemically produced dyes.

The chemical fertilizer superphosphate used for the improvement of agricultural production took its place in this factory. Production of superphosphate had for that matter already started some 40 years earlier in England. In Holland, other factories followed soon in 1882, 1891, 1895, 1907, 1910, 1914 and the last superphosphate factory was built in 1920.

The production was at the beginning aimed at the local agriculture, but the geographical position near big ports and waterways made export very soon more important than the home market and the inland sites disappeared one after the other. Of the original sites, only two superphosphate is still produced; these 2 are Amsterdam and Sas Van Gent.

Nitrogen production started later with some 50-100 t N/d ammonia units; in 1928, near Ijmuiden, a unit was built based on gas from near by steel works. The same year work started on a unit at Geleen based on coke oven gas from coal mines and a third unit was built in 1930 in Sluiskil, based on gas from a nearby coke-oven complex, that obtained its coal from overseas.

In a very short period, Holland then changed from a country importing all its nitrogen fertilizers, mainly from Germany into a self-supporting and even an exporting country.

After the 2nd World War, the industry rose from its ashes; new technologies and new products were introduced. Superphosphate was more and more replaced by TSP and compounds and the N-fertilizers, ammonium sulfate and calcium nitrate were mostly replaced by CAN and urea. Especially after the discovery of large amounts of natural gas, the ammonia industry expanded dramatically; on the existing sites 1000-1700 t/d units were built and a fourth site at Roosenburg was added and at Pernis an ammonia unit completed the phosphate and compound production units.

Nitrogen fertilizer production then reached more than 4 times the local consumption. Exports of nitrogen phosphate and compound fertilizers to countries around the globe took impressive dimensions. Geographical position and low energy prices were one reason, but evidently these developments were only possible with the help of well skilled enthusiastic technologists, chemists, agronomists, and so on. All these industry experts took great advantage from the exchange of knowledge and ideas in committees of industry organizations which later came together to create IFA.

What goes up too, often comes down later. This old knowledge is also valid for our fertilizer industry. Fertilizer consumption has reached its peak in Western Europe.

Firstly, the success of modern high input agriculture has led to problems with overproduction of agricultural products on one hand and to environmental constraints on the other.

Furthermore, production of phosphate fertilizers and intermediates on the mines in amongst others Northern Africa has changed the outlook of the European Phosphate Industry. And thirdly, production of NH_3 with very cheap energy compared to the nowadays high taxed European gas has created a difficult competition on export markets.

It is therefore evident that the industry has once again to live up to a challenge, which is "how to adapt to this changed outlook for the Dutch and West European Industry".

One of the necessities will certainly be to find the best technological solutions with which we will be able to produce and process intermediates into custom made products needed by the local agricultural at the lowest possible costs.

It is therefore that I welcome the 1992 IFA Technical Conference to The Hague. And on behalf of the Dutch Fertilizer Producers, I also wish you a fruitful conference to the advantage of all IFA members.

I also do hope, of course, that you will all have a pleasant stay in our country.

KEYNOTE ADDRESS

by

Dr. Haldor Topsoe

Chairman, Haldor Topsoe, Denmark

Forty-seven countries and barely the two sexes, and so I have to start by saying, Mr. Chairman, dear lady and gentlemen.

As one or two of you may know, we focus on working in catalysis. I could not think anything more proper in this audience than to look at catalysis - the field of physics and chemistry where we make things happen so fast that we can economically produce fertilizers to make things grow. There is a unique relationship, symbiosis or synergy between the field of catalysis and the fertilizer industry.

Indeed, no other field of chemistry has had such importance for the progress in catalysis as the fertilizer industry starting with sulphuric acid going on to nitric acid and ammonia. As we all know now, our industry is a father of the process industries and of the tremendous development we have seen in catalysis in industry.

I hope you will excuse me for submitting general views and not just talking about scientific or technical matters related to our industry.

Let me first say that you cannot look at the world surrounding us without being strongly impressed by the tremendous events unfolding. By a situation where we now can see that besides the many problems of detail our industry faces, we have some over-riding problems that must be solved. All of them have a very great impact on this industry. I am thinking of the problems of population, of development of resources, of the environment, and of capital transfer between rich and poor.

I would like first to say a few words about population as Mr. Childers already did.

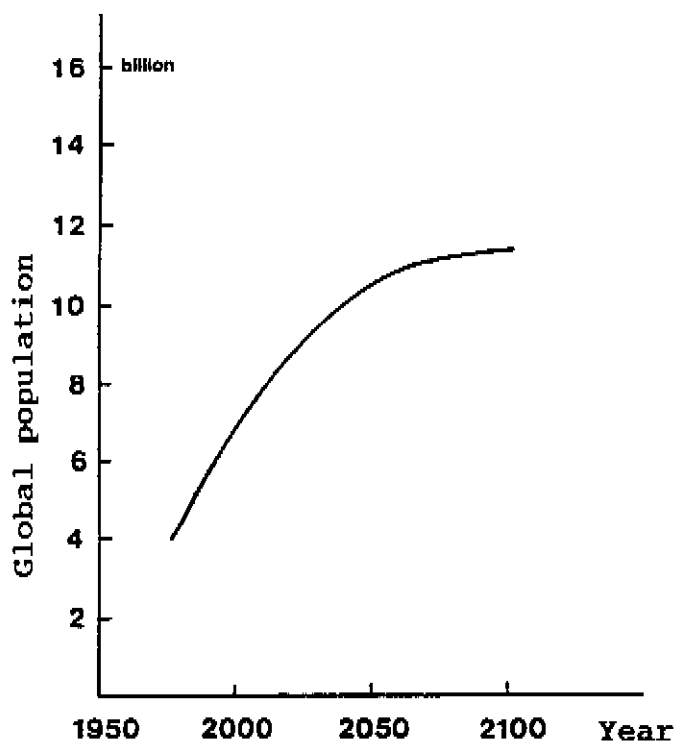
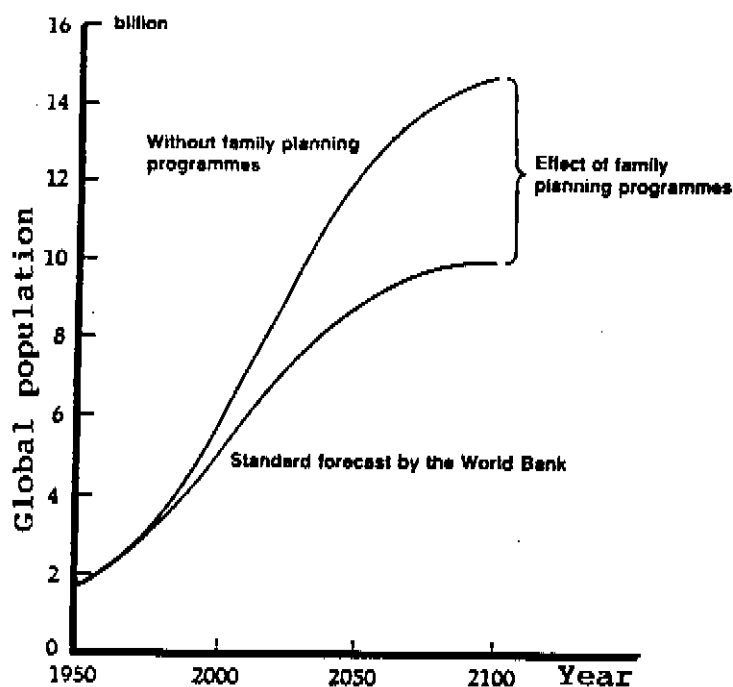


Figure I - Global Population

Figure I shows an analysis of the population growth based on the forecast developed under the auspices of the World Bank. You will see that it will even out towards the second half of the next century at around a dozen billion people, more than twice that we are today. This is, of course, a tremendous growth causing many difficulties but it could have been much worse.



**Figure II - Population Size in the Developing Countries:
Alternative Forecast, 1965-2100**

The two forecasts in Figure II relate only to the population of the developing countries and you will see that the upper curve shows already by year 2050, 13 to 14 billion people in the developing countries, but the lower curve is the forecasts generally believed in today. The top curve is the forecast without taking into account the effect of family planning efforts, the lower does. Some five to ten years ago, it was most uncertain whether the family planning efforts had any real effects. I have myself been involved a little bit in that being on the board of an organization called the Population Council. It has given me very great satisfaction to be allowed to contribute a little, little bit to this area. It is beyond any doubt that recent statistics confidently show us that family planning has an effect and that we can now believe that the population growth will be "less unacceptable". It is difficult to find a better term. It is thus possible to believe that in the developing countries, towards year 2050 we will come close to 10 billion and then the growth may flatten out. Of course, this is of the greatest importance for the problems in our industry. Let us quickly see what is now forecast as growth rates.

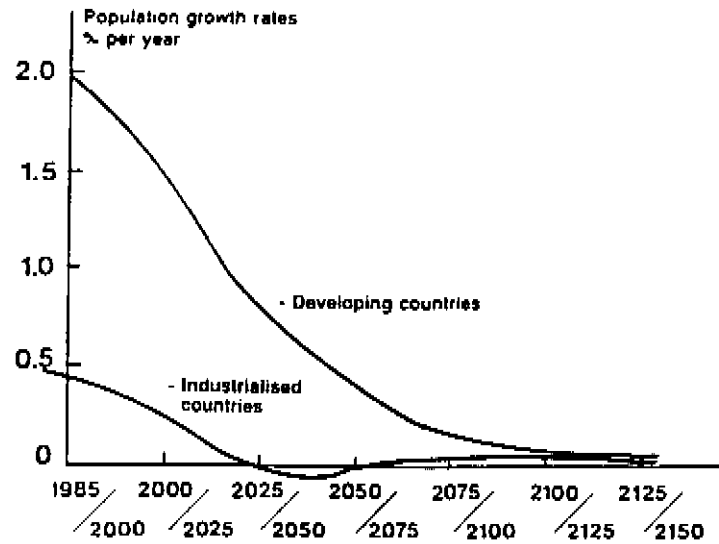


Figure III - Population growth in Industrialized Countries and Developing Countries (according to Bulatao Bos Stephens and Wu, 1989-90 Edition, Published for the World Bank, The John Hopkins University Press "World Population Projections").

Figure III gives the growth rates as percent per year in all the developing countries in the top curve going down from 2% which it is at present and probably coming down below 1% in about twenty years' time. You will see that in about 3 generations, the two curves for the industrialized countries and for the developing countries will more or less be the same. It is, however, not enough to look upon such overall curves, we have to ask ourselves what are the problems caused by population growth year by year and therefore let us look at the next figure.

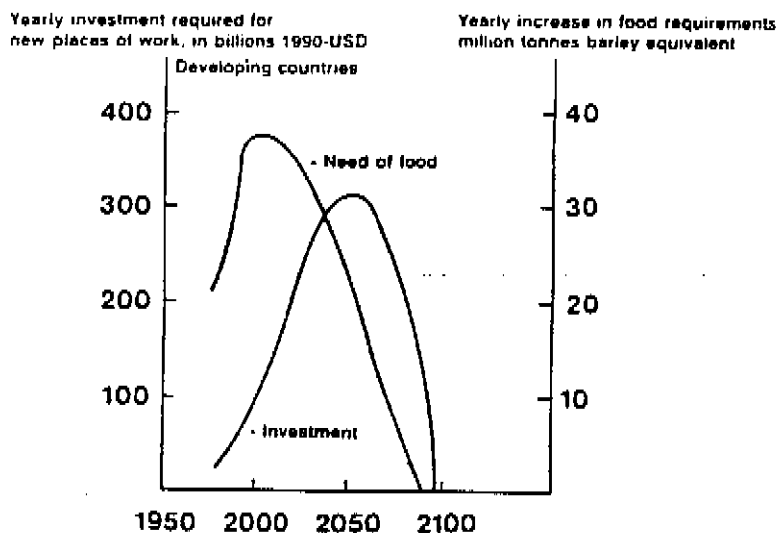


Figure IV

Figure IV shows, again for the developing countries, to the left: the yearly investments required to create new places of work in billion 1990-dollars, and to the right we see the yearly increase in requirements for feeding the population without change in diet. Around 1990 the investments required are in the order of 52 billion dollars a year, not a very high sum, but you will see a drastic increase. This is going to place a greater demand on all of us in this world than any political leader has had the courage to tell us. These curves as far as the coming generation is concerned cannot be disputed.

Now, what does this mean for our industry. It, of course, does mean what Mr. Childers already said that there is a tremendous demand for fertilizers out there. A necessity to meet the rapid growth in demand for food. So, I submit to you that one must focus on the yearly increases instead of focussing on some prognosis for year 2000 or 2025.

What does it say about the fertilizer quantities needed to create this growth in food production? It simply means that during the next two generations, we have to triple the output of fertilizers. There is no technique, no scientific development anywhere that would indicate to us that we can meet these requirements without an increase of that order of magnitude. There is nothing in new bio-technology, genetic manipulation and what have you that indicates that we can meet these requirements without such an increased output in fertilizers. Some people say: yes but there are so many other things we can do in agriculture, we can increase the total area under cultivation, we can may be cut down - excuse me for saying it - the rainforest and grow food there and we can change the diet and thus reduce requirements for primary agricultural production - but all this will not change the picture.

Then, why do we have the present trouble in our industry? Why do we, everyone of us, experience over-capacity? Why do we have difficulties getting reasonable prices for the products? Of course, most of you know the answer. But it is unbelievable that we are unable to supply and distribute towards what is needed to give a decent diet to all the five and a quarter billion people living here today.

If you are an optimist, you may think that about 3 billion people have a reasonable diet, but to give a decent diet to everybody, you would need a fertilizer input much larger than the present. Why do we have this trouble? Because of economic dislocations. Because where the need is greatest, the monies are smallest. We all know that.

But then, there is another peculiar thing that has greatly disturbed all of us. It is a fact that the fertilizer industry has developed to be a cyclic industry. Yes, when you ask industrial economists, they tell you certain industries are cyclic, certain are not. Heavy chemical industry is cyclic and TV is not. Why is that? People, particularly economists, talk as if it was an inherent quality, an inherent property of industries like ours. But it is not that we do not know how these cycles happen? They happen whenever there is a small imbalance between supply and demand, for instance, a small under-supply, then, all of us - and the financial people behind us - decide to build new capacity in great secrecy. Also the contractors who are asked to build these new plants, are, of course, working under secrecy. And then, after some three to four to five years, new capacity comes on stream and everybody observes that everybody has made the same secret decision. Then, in a couple of years this new over-capacity destroys prices and the industry decides not to build anything for some years etc.

Therefore, the cycle length is the time it takes for the financial people to observe that there is a demand plus the time it takes to build new capacity plus the time it takes to realize one has created over-capacity. If you add these reaction times, it is quite understandable that such an industry as the fertilizer industry has to live with cycles between 8 and 12 years for quite some time to come. Unless the industry should be intelligent enough to find ways of avoiding this unpleasant situation.

What could you hope would happen to the industry in the near future? When would the industry take off and start developing in order to meet the requirements I just mentioned. My guess is that it would be some years before we will see a solid improvement. I am optimistic if you look further ahead but quite pessimistic for the immediate future, and particularly so because of the considerable difficulties in providing finances. It is more and more difficult to provide finances for the developing world, the poor world and the newly poor world in the East.

Well, the industry is an energy-heavy industry, particularly the nitrogen fertilizer industry. We are pestered with all sorts of forecasts about scarce energy resources. Figure V shows known reserves and the number of years of use at present level which these reserves can cover.

Reserves and Future Span of Consumption ¹⁾					
	Oil	Natural gas	Steam coal ³⁾	Lignite	Uranium ⁴⁾
Reserves, billion tonnes	-150	-110	-620	-300	3,2
Oil equivalent, billion tonnes	-150	-110	-425	-95	
Future span of consumption, years	-50	-50	-175		-75

Fig. V - Known Reserves ¹⁾ in 1992 for Energy Raw Materials

(Sources: Shell, BP, NESO/IAFA, OECD/IFA, Häfele 'Energie Systeme im Übergang' (Poller 1990).

- 1) Only reserves which can be economically extracted using existing technology are included
- 2) Years until depletion of reserves at 1991 level of consumption
- 3) 'Hard Coal'
- 4) Certain and relatively certain reserves, excl. the eastern countries (P.E. Grönwall, Kul- Kernekraftredogørelsen (Chapter 3) Danish Ministry of Energy, 1984, mio. tonnes

You will see that if you take oil, natural gas, coal, brown coal lignite and uranium, you have reserves corresponding to between 50 and close to 200 years the largest reserves of course being coal reserves. On that basis, some people say that we have to be very careful and already now plan for what happens two or three generations later. But it is a fact that we keep discovering reserves even to a larger extent than what corresponds to running use. It is also a fact that many reserves - like very heavy oil, shale oil, tar sand and remote natural gas resources and coal resources - are known to be there but so far not taken into account because they are too remote or too costly for present use. If we take these resources into account, we have energy reserves for more than 500 years.

In the public debate, we cannot at present speak about nuclear energy but we may very well have to go nuclear. Then maybe those forcing us to go nuclear will be the green propagandists who have been so violently anti-nuclear. If the concern about CO₂ force us to go nuclear, we have to improve the nuclear technologies involved in order to make them acceptable to the public. We have to design nuclear plants at least in the same careful manner used in modern fertilizer industry to deal with operational and hazard problems. This has not been the case. If, for instance, ammonia plants were built, the way some operating nuclear power stations have been built, you would be quite shocked.

Now, if we take into account the energy reserves for nuclear power production, and assume that breeder reactors will be introduced, we have known energy reserves for a thousand years. That seems, at least to me, to be such a long period that we do not have to worry too much about energy resources.

Let us look at how the use of energy can be forecasted.

Period	Index global energy consumption 1990 = 100 (Fig. III.14)	Yearly "increase" mio. tonnes oil equivalent 1 MWh equivalent to 1.800 t oil	Yearly "increase" MWh	Total increase is covered by nuclear power, Exchange by conventional plants. Investment per year, billion 1990 USD for		
				Power plants Total	Workshops	Total
1990-2000	100 → 99	-10	(5.600)	0 (25)	0 (0)	(25)
2000-2010	99 → 108	90	28.000	140	40	180
2010-2020	108 → 117	96	31.000	155	7,5	162,5
2020-2030	117 → 134	106	59.000	295	70	365
2030-2050	134 → 179	140	77.000	385	45	430

Figure VI - Nuclear Power Alternative Indicates Yearly Increase in Energy Consumption, in tonnes oil equivalent in MW-electric

We will use 1990 as hundred. You will see that until 2000, we will have a near constant use of energy and that is caused particularly by developments in Eastern Europe and the former Soviet Union and by quite efficient introduction of energy saving technology. But then, whatever people forecasting environmental problems are saying, you cannot keep consumption down and in about two generations, you will have a consumption which is 80% higher than the consumption at present. If you look upon this, you will see that we will necessarily experience a considerable increase in the release of CO₂. Increase in nuclear energy will have an impact only after twenty-thirty years.

So, the political statement sometimes issued as a must to all of us, that we must cut down in energy so that by the year 2000 we have reduced emission of CO₂ by 20%, is unrealistic and unacceptable for social and other reasons.

Now, we all know that the energy picture, which we depend on in our industry, has caused a great geographical migration of heavy industries. Let me describe what is at the bottom of all this.

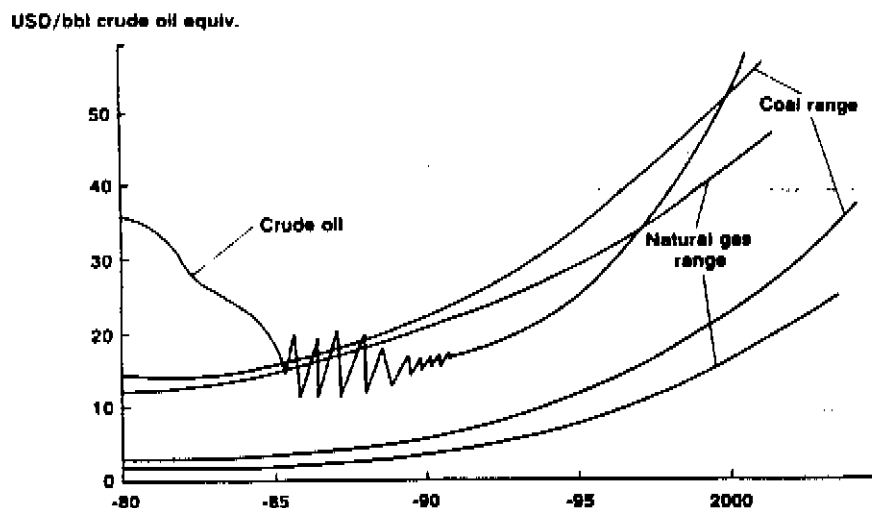


Figure VII - Ranges of Variation for Coal, Oil and Natural Gas Prices
[Source: official quotations for coal and oil, private communications for natural gas]

Figure VII shows how energy prices have varied and a guess for how they may develop in future. It gives the energy raw materials prices per barrel of oil equivalent expressed in dollars - I suppose we still have to express ourselves in dollars although it is a peculiar thing to let such an important part of our society as the energy industry and everything derived from energy depend on the gyrations of the dollar. Maybe some day one will find an alternative and relate all such very important matters to a basket of currencies. I do not believe so.

Anyhow, crude oil prices do not vary much from place to place but for coal and for natural gas, two curves show the great range of variations - with location. The range for natural gas is between a dollar per barrel crude oil equivalent and about 12 dollars, a ratio of some 15 to 1. Also a very great range for coal exists. Of course, it is impossible for our industry not to go where you find low cost raw materials and let us ask ourselves exactly what it means in some figures. Let us take the Middle East. You all know that one can have natural gas at 50 cents per million BTU - you may not even always have to pay for the gas! And we all know that in our part of the world, we have to live with somewhere between 3 and 5 1/2 dollar per million BTU. This difference for a modern ammonia plant means in the order of 80 to 100 dollars per ton. It is not possible to bridge this gap, so the heavy chemical industry including ammonia, methanol and so on, migrates to the Middle East and other places with cheap natural gas.

Now, what does it mean? It means that our industry has a big problem of location for future expansion. We could recently hear from Mr. Nojaidi, the President of SABIC Trading, at a meeting recently in California, that he and all friends in the Arabian Gulf area were looking forward to glorious years in their area, but chemical industry elsewhere would have a difficult decade. Obviously, this is part of a general problem between industrialized countries and many of the other regions.

What do we see as future distribution of tasks between different regions? Do we, for instance, in Western Europe, here in Holland, believe that we can manage a situation where heavy energy dependent industry is built elsewhere, in the Middle East, in the Far East and that we then continue developing and dealing with more advanced industrial activities. Do we believe that this will create a situation which will give political balance between the regions, employment to people in the industrialized countries and a balance in macro-economy, not the least in currency. Do we believe that? I do not. I cannot see what would keep developing regions with cheap energy from going all the way and producing even the most advanced industrial products. I can see that it will take a lot of time, maybe twenty or thirty years but I cannot see that we can base an equilibrium between the energy rich countries and our part of the world on such a distribution of tasks.

Well, of course, the situation in the nitrogen industry and the P_2O_5 -industry is quite different, as known to all of you so I shall not deal with this. But I do believe that what happens to the nitrogen industry is going to be quite a determining factor for what happens to the whole fertilizer industry.

Let me say a few things about technical trends. We have seen over the last two decades tremendous development in our industry giving us much better plants, larger plants, more energy efficient plants. I believe that for the coming years, we will still see a small increase in energy efficiency. Maybe we will come in with new plants using down 6 1/4 to 6 1/2 gcal/tonne. But I think that the more important developments will be in reducing investments and in increasing reliability and I know that you are going to deal with all this during the coming days!

Notwithstanding the progress made, there will still be important opportunities for development of new technology. It is of great interest for all industries, and certainly for ours, that we improve the process of coming from research and development to industrial operation. This process has for many years been somewhat neglected but it is - although in a different way - just as important a process as the ones our chemical engineers deal with. It is really amazing that in the present situation where everybody recognizes the importance of new technology, one does not pay more attention to research. To my colleagues and me it is surprising that we still seem to be one of the very few organizations which emphasizes this integration between science and industry.

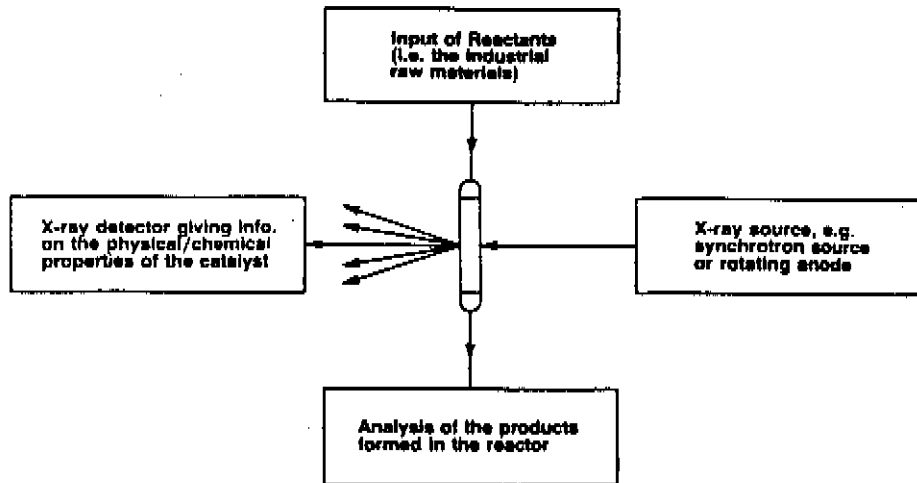


Figure VIII

Let me show you just an example of how quite fundamental research can be carried out in such a manner that laboratory research results are directly applicable to industry. Figure VIII shows what we term *in situ* work where you place a catalyst inside a small reactor and flow feed gas through the reactor while it is exposed to radiation that will give information about physical and chemical properties of the catalyst and particularly its surface. Radiation can for instance be X-ray or γ -ray capable of penetrating the reactor wall. In our work, we use for example Mössbauer spectroscopy, synchrotron, and X-ray radiation.

You will understand that it is possible in such experiments to vary feed composition and reaction parameters to vary feed composition and reaction parameters over a very wide range and thus reproduce conditions in any part of an industrial reactor. It is possible to determine kinetics selectivity, and in many cases also life of catalyst. You can then correlate these "industrial" parameters with the physical and chemical data determining catalyst properties. Such *in-situ* experiments can be refined and enable one to introduce research results into industry a good deal faster than by using more classical research procedures.

I would like to end this speech by saying a few words about how we observe the world around us. Changes have been very dramatic. The circumstances under which the cold war and the terror balance left us were most surprising. It is frustrating to note how political balance and equilibrium went away at the same time. We have to hope that development in the East will come under reasonable control and be carried through in a sustainable tempo so that some years in the future, a new equilibrium between the industrialized Europe and the East will be established. I am surprised to see how little emphasis political leaders in the West are placing on the importance of this development and particularly on finding ways of helping the CIS republics to develop and integrate into a greater Europe. This integration would present a great long-range opportunity for all of us. One task of importance for all of us here is to find ways of integrating our western advanced technology into CIS' vast raw material base.

I am surprised to see that here in Western Europe - and the EEC - we concentrate our matters of rather minor importance and forget these very big problems and opportunities.

May I end by saying that no Dane could be the country of Maastricht without submitting a word or two about the Common Market. We voted no and why did we? We did for many reasons: there was no clarity in the Maastricht document which some people really term a very poor and unclear paper, full of contradiction and unreadable sentences. Some people are surprised how one, e.g. in a highly intellectual country like France, could put such a poor document to public vote. Many of us felt that we lacked knowledge about the meaning of the term "subsidiarity" and of what we gained as a *quid pro quo* for giving up a good deal of sovereignty.

Nevertheless many of - I also - thought we should have voted yes because of the overriding importance one should give to the visions of creating a Europe free of conflicts and risks.

No doubt the Danish no gave great service to the debate in Western Europe and forced people to look again at Maastricht. Hopefully, in the coming weeks and months, one will develop information and papers showing clearly to our populations what is really the content of Maastricht, and not the least where each country will still have the right to decide and where decisions must be made in Strasbourg or Brussels.

Certainly, one has during these months been reminded of Napoleon's statement that a constitution should be short and unclear. I think it will be very bad if we must accept one which is long and unclear.

FIRST GENERAL SESSION

'AMMONIA, UREA TECHNOLOGY'

Chairman: C.E. Childers, PCS, Canada

Rapporteurs: L. Pritchard, Kemira Agro UK Ltd, United Kingdom
R.J. Tricklebank, Kemira Agro UK Ltd, United Kingdom

PAPER 1 Product quality improvement on urea prills
Younis Ahmed Ibrahim, Abu-Qir Fertilizers & Chemical Ind. Co.,
Egypt

Q - A.K. KUKKADY, SAFCO, Saudi Arabia

a. Formaldehyde is a suspected carcinogen. Has any study been done to determine any adverse health effects it may have on personnel handling urea which is conditioned using formaldehyde?

b. Secondly, are there any commercial bulk flow heat exchangers in use other than at Cominco, Canada?

Ans: a. Formaldehyde is not directly used, but supplied in the form of hexamine (HMT) which is directly injected to the evaporation section, to react with urea at evaporation temperature to give formaldehyde and ammonia, i.e. no direct personnel handling with formaldehyde.

Reaction of formaldehyde with urea is a polymerization reaction we think has no health effect. It is commonly used as conditioning. We do not carry out studies about the health effect.

b. We have no information about the commercial bulk flow H.E other than at Cominco, Canada.

Q - D.C. THOMPSON, ICI Fertilizers, United Kingdom

In the impact test for urea prills, how is the percentage of unbroken prills assessed? Is the method visual? Could it be assessed by a sieving method? Could the method be adapted into an International or European Standard?

Ans: The percentage of unbroken prills is determined by analytical method (UKF.1032-E), determination of shock resistance is visual and assessed by an analytical sieving method (UKF 10 11- E). Roundness is determined according (UKF 1081-E).

Q - C. NAJAB, Trinidad and Tobago Urea Company Limited, Trinidad

Could you please indicate at what temperature the urea is stored and under what conditions? Secondly, do you have problems of caking when shipping your product to cold climates, what precautions have been taken and how successful have they been?

Ans: Urea prills are stored at temperature 60°C - 75°C in a bulk store-max 7 metre height heaps, not ventilated. We haven't any problems of caking either in storage or shipping.

Q - B.K. JAIN, Fertilizer Association of India

Certain advantages have been claimed in using bulk flow heat exchangers as compared to fluidized bed coolers. What cost savings were estimated (per tonne) for bulk flow heat exchangers over fluid bed coolers, taking into consideration the respective costs of installation and operating costs?

Ans: The cost estimated by Cominco, Canada, are:

The capital cost \$ 900,000 and \$ 1.5 million for bulk flow HE and Fluidized bed cooler respectively, and the working cost of power and maintenance \$ 53,000 and \$ 170,000 per year bulk flow HE and FBC respectively, for a plant 1350 t/d.

PAPER 2 Reliability of ammonia plants

M. Altieri, B. Ersini and A. Stauble, Enichem Agricoltura SpA, Italy

Q - P. ORPHANIDES, Orphanides Consultants, Greece

a. The first question is about the catalyst changing programme to reach this 5 yr turnaround period as well as the maintenance system you have applied for the primary reformer tubes.

b. Do you change them every 10 yrs or what is the philosophy you have developed?

c. The third question is about your circulator. I understand this is rotating equipment, do you have a spare or is it a unique unit, and how do you cope with this 12,000 hrs MTBF?

Ans: a. The policy for catalyst changes to achieve a five year turnaround is as follows:

- to select the catalysts giving the best results in lab tests. Generally the best catalysts are more expensive;

- to operate the plant in a very smooth way thus avoiding even minor damages. The careful installation is also important for longer runs.

b. We perform on the primary reformer tubes ultrasonic examination every shutdown to verify the growth of cracks due to creep phenomena, and we have zero point at the installation of the reformer tubes in 1978 and we relate to these initial values all the ultrasonic examinations we perform during that time. We established four classes of suitability of the reformer tubes and we change at every shutdown tubes that are considered in 4th class and we will assume that with tubes we consider belonging to third class may give operation for 4/5 years and so we changed last shutdown 5 tubes that were considered in 4th class and in 1985 shutdown we changed 8 tubes.

c. For the circulator, we have only one circulator with a spare part and when we have to change the circulator we have a system that lowers down the charge of the plant to 40% and in eight hours we can change the circulator with the spare part we have.

Q - I. GHOSH, Shriram Fertilisers and Chemicals, India

Remark: Speaker has mentioned in his paper that reciprocating plants in the world are found to be inactive for 60.2 days per year with an average 16.5 shutdown per year. I would like to bring to the notice of my fellow delegates that our own factory at Kota, commissioned in 1969, having Reciprocating Compressors, are operating at a very high onstream day efficiency and the average of the last 3 years onstream factor (%) was 96.8% and downtime days/yr average was 13.47 and number of shutdown/yrs average was 2.67 with a capacity utilization of more than 110%. In a non turnaround year SFC kept the plant running for 362 days and 352 days when the plant had to be stopped for 10 days for statutory inspection of waste heat boilers.

In India, we are to take a shutdown once in two years for statutory inspection of waste heat boiler with the plant remaining out of operation for a minimum of 10 days. Will you please tell us the frequency of the inspection of waste heat boilers by statutory authorities in your country.

Ans: I spoke a year ago with an Indian committee who were studying laws to apply to waste boilers in your country and we pointed out all the problems you find out on such equipment and how we solved them. In Italy, requirements, law requirements are of a visit of a vessel every 5 years for our boiler vessels, so it is possible to verify with inspectors every five years this equipment. Another consideration to do on the relief valve that an Italian law requires to verify every three years, but if an adequate system of the plant is able to guarantee no excessive pressures and temperatures and this is documented by recordings, we can go up to 4 and half years and in certain cases up to 5 years but we had to request this situation to our inspectors at the plant facilities.

Q - G. MUTHUKUMARASAMY, SPIC, India

a. What is the energy consumption for CO₂ removal after switching over to glycine solution?

b. Is there any change in corrosion rate after switching over to glycine solution?

c. The turnaround in Enichem Porto Marghera ammonia plant is reported to be once in five years. Is the naphtha reformer catalyst able to operate for five years? How do you meet the statutory inspection requirement of boilers, vessels, pipelines, etc.?

Ans: *a.* After switching over to glycine solution the electric energy consumption remained constant. Regarding the steam consumption, you have to expect an increase. In our plant, in effect, we had a 5% decrease due to some improvements in the CO₂ removal section. In particular, we have installed an adjoined regeneration column working at atmospheric pressure.

b. After the switching over to glycine the stress corrosion cracking, which is frequently controlled with electrical potential measurements, is decreased.

c. The feed in our ammonia plant is natural gas, consequently we don't have experience with virgin naphtha. Regarding the statutory inspections of the pressure vessels we have provided the authorities with scientific data showing that the monitoring system and the construction materials enable safe operation. In such a way we obtained special permits.

PAPER 8 Urea technology optimization
V.M. Olevsky and M.E. Ivanov, GIAP, A.G. Bogatov and
T.M. Bogdanova, GNKP, Russia

Q - P. ORPHANIDES, Orphanides Consultants, Greece

a. You indicated in your paper that the free fall height required to obtain the calibrated prills out of your acoustic device in a moderate climate conditions of Europe should be about 70 meters. Can you indicate the required free fall height for even hotter climates like those existing in the Arabian Gulf or India where air temperatures of about 35-38°C are usual?

b. Please give references of your total enclosed system, you indicated here to capture emissions in Russia or whenever this is already applied for full industrial applications.

Ans: *a.* First about the height of the prilling tower for hot countries. We have a calculation program for computer and we can easily calculate it in a very short time for any humidity, temperature, air flow, air velocity, melt velocity, any changeable conditions that you like. I cannot give answer without calculations but I can calculate this very quickly if you let me have the data. The answer is very simple, it is somewhat higher if you still want to have the middle size of prills about 3 mm. Certainly if you can go a bit lower if we say 2-3 mm but you will not be near 3 but a bit more than two. But you will have this interval but you will have most 2 mm for instance, but then I think it is enough with 50 m height. But if we prefer to have nearer to 3, I need to calculate and then we can guarantee this size.

b. Your second question made references about the closed loop. We made investigations, calculations and pilot plant experiments and we have been going to build such an installation in one of our plants in our country but events of the last two years made it impossible because the plants say that at the moment they cannot spend money because this won't earn them money. It will only improve ecology and they say they will wait one or two years, so at the moment we didn't build it in industrial scale but we are ready to realize it anywhere if we find a partner. We are absolutely sure of the results.

Q - B.K. JAIN, Fertilizer Association of India

It has been stated that reconstruction of plant with close cooling air cycle may add up to 5% of total investment. What would be the additional cost for a new plant with built in such facility? Would it not be preferable to go for urea granulation rather than prilling which may be more cost effective with respect to pollution abatement and improved product quality on a comparative basis?

Ans: When you build a new plant and use this closed circuit it is also about 5 not more than 7% increase in expenditure of investment, this being for improved ecology. Different possibilities exist: building closed circuit, reconstruction or to build a new, for instance, fluidized bed or maybe pan granulator or drum granulation. I can answer very simply, the investment for these alternative methods of granulation certainly are lower investments, if you remember every ton of urea by granulation are 5-7 dollars more expensive than you produce by prilling and by other mentioned methods of granulation. So you while gain in investment but you lose very much in the price of fertilizer. You must decide what quality of urea you need. If you use it directly into the soil in the region of the production, then you must use prilling, because it is good enough and it is much cheaper. If you want to transport it round the world, then you must have a more mechanically strong granule, and then certainly you must use alternative methods of granulation.

Q - *S. AL-MUTAIRAN, Al-Jubail Fertilizer Co., Saudi Arabia*

a. Does this system work for a natural draught prilling tower?

b. How do you overcome the caking in the recycling cooling system? Urea is a high caking substance. Will dust plug the pipes?

Ans: a. Yes, the natural draught prilling towers may be reconstructed this way.

b. No special additional problems with caking. You mean, for instance, the humidity of the circulating air after it has left the tower. If your installation is in a hot and humid country, you have air humidity of 95% and we have in some regions of our country, the same humidity and no special difficulties exist, because in the tower the temperature is much higher, so the conditions are approximately similar and not much different from that in hot and humid countries.

Q - *A.K. KUKKADY, SAFCO, Saudi Arabia*

Can you please provide the addresses for:

a. urea plant locations where vibro prilling is in service?

b. urea prilling plants (or pilot plants) using a closed air system where performance data can be obtained?

Ans: a. Vibro prilling systems for urea are in service at a number of urea plants, including the 450 000 t capacity per year in Perm (Russia) and Gorlovka (Ukraine), as well in Dorogobuzh and Novgorod (Russia) in ammonia nitrate production.

b. All necessary investigations concerning the fertilizer prilling with closed air system in pilot plant scale are already accomplished. We are ready to design an industrial installation, fitted to your specific conditions.

PAPER 4 Reliability - a must in ammonia production
Ib Dybkjaer, Haldor Topsoe A/S, Denmark

Q - *B. GROTZ, Brown & Root Braun, USA*

a. In calculating the net energy consumption values of 8.85 and 7.02 on Table 3, what is the quantity of export steam (per tonne of ammonia) and what is the pressure and temperature of the exported steam?

b. Also in each case what is the total natural gas feed to the two plants (in Gcal/MT).

Ans: Fauji plant, no figures available here. For the Kaltim plant the natural gas consumption during test run was 7.64 Gcal per tonne. The steam balance of the plant is rather complicated, there is an export of steam as high pressure steam 105 kg/cm²; of medium pressure steam about 40 atmospheres; of 22 atmospheres steam as process steam for the urea plant and also as low pressure steam. All the figures are available in a paper which we have outside at our small exhibition - a paper written by the operators of the Kaltim plant and you can check the exact figures there.

The latest ammonia plant we have started in Bangladesh, Jamuna Fertilizer Co. has a feed plus fuel consumption during test run of, as far as I remember, 7.35 Gcal per tonne and the net energy consumption after export of medium pressure steam and import of low pressure steam is 6.96 Gcal. In all cases both for the Kaltim plant and for the Jamuna plant we calculate the steam credit or debit as the full calorific value of the steam according to steam tables with zero degrees centigrade as the basis.

Q - *B.K. JAIN, Fertilizer Association of India*

a. How would you optimise plant costs considering flexibility versus reliability? While too liberal a design would ensure high capacity utilization rates but consequential higher capital cost (even with marginal lower operating costs at higher loads) may result in higher production costs. While operating costs can be assessed based on guarantee performance figures, estimation of realistic capital cost of plant is rather difficult, and to that extent quantitative evaluation of different technologies becomes difficult.

b. Normally, what over-design allowance is provided for reforming and synthesis sections of an ammonia plant?

Ans: a. It's not very easy to give a short answer to these questions. Let me first comment on the first point how you would optimise plant costs considering flexibility versus reliability. First of all I would like to say that ammonia plants are big industrial installations and they are certainly big enough to justify individual design in each case. So in each case there will be discussions between the engineering design company and the client to determine to what extent extra capacity shall be built in, to what extent future capacity increase shall be foreseen and so on. Each company may have its own philosophy concerning design capacity and design margins and that is something which is discussed and determined in the design phase. Whether liberal design meaning probably an over-design of certain equipment would lead to higher capacity utilization rates, I think that is a point which is could be debated, I am not completely sure that it is true. I think that most equipment will operate best if it operates close to the point that it is designed for. Then there is a problem which is certainly a real problem for people who want to select technology for ammonia plants or for other plants, how do you evaluate the various technologies. With respect to ammonia plants, there are today, I think, four main suppliers of technology and these four suppliers, I can name them, they are Topsoe, Brown & Root Braun, Kellogg and Uhde. These four suppliers they are well experienced, they have all constructed a number of ammonia plants, and there is no doubt that they can supply technology with all that implies. So the competition is of course on energy efficiency, which as it is stated is easily evaluated and investment cost which is much more difficult to evaluate, and then on a lot of less tangible factors, where do you get the credit, who is providing financing, and a lot of immaterial things, which may in many case be more important than the technical points, unfortunately, I think. I don't think I can give any good advice how you compare capital cost for different technologies, except by going all the way and asking for fixed costs bids entirely on consistent basis from the different suppliers. This is a very big workload for the suppliers to produce such complete bids, and it's certainly also a very significant workload for the receiver of that information to evaluate it. So it's not something you embark on without having a very solid reason to do it, but I think it's the only real way you can compare technologies.

b. The last question was what over-design allowance is provided for reforming and synthesis sections of an ammonia plant. Also to that question I don't think there is a clear answer. If you look at the reformer for instance, then the design of the reformers is such that if the reformer operates at its design point then the average life time of the reformer tubes will be 100,000 hours that is the normal practice.

But there's certainly a possibility to operate the reformer at significantly higher load at the expense perhaps of a shorter average life of the tubes. If you talk about over-design allowance is that for the normal expected life time, or will you allow a reduction of the expected life time of the tubes. In a similar way, of course, the catalysts will have a certain life if they are operated in a certain way, if you press them harder it may mean shorter catalyst life time. I could say that most reformers will without any problems will be able to sustain much higher capacities, than the design capacity. One point which could be a limitation is the firing on the reformer, typically the burners are designed with an over-capacity of 25% and if you want to operate above that then you need to add some additional firing. In a similar way with the synthesis loop, the converter, which is the most critical equipment there, can certainly provide much more than what it is designed for. If you feed more synthesis gas to the loop and take a high purge rate then you can often make 40/50% more than the design capacity of the loop. The bottlenecks in ammonia plants are very often the rotating equipment, the air compressor typically and the synthesis compressor, and that maybe because if you over-design significantly centrifugal compressors, then the performance deteriorates. They have the best efficiency if they operate close to the design point, and if you deliberately over-design and therefore operate at a point away from the optimum then that will cost you money, energy consumption in your normal operation. There is much more to say about these fundamental questions, but I don't think time will allow further deliberations.

Q - G. MUTHUKUMARASAMY, SPIC, India

Both the FCC and the Kaltim plants have the same capacity. However, the energy consumption in the Kaltim plant is less than FCC plant. What are the specific features which lower the energy consumption at Kaltim?

Ans: First of all, the FCC plant was designed in the late 1970's, whereas the Kaltim III plant was designed in the second half of the 1980's.

Optimization of operation parameters for the individual process steps is one of the main reasons for the lower specific energy consumption figure in the Kaltim III plant (the figure for FCC has been, in the same way, decreased from 8.85 to 8.18 Gcal/MT NH_3 in the period from FCC design to Kaltim III design, mainly due to optimized operation conditions).

Improvements in individual equipment (better tube materials in reformer, higher efficiencies on rotating machines, etc.) as well as improved catalysts, have lowered the specific energy consumption figure in modern plants.

Specific improvements made in the Kaltim III plant which bring down the energy consumption figure compared to the FCC plant are:

- Topsoe S-200 Converter (S-100 in FCC).
- Efficient integration of process condensate unit to maximize heat pick-up.
- Purge Gas Recovery Unit.
- Steam Superheater integrated after secondary reformer.
- Use of electric motors for small drives below 1,000 kW.
- Low flue gas temperature.
- Benfield Lo-heat CO_2 removal
- Efficient integration of steam system with urea plant.

PAPER 5 **NO_x absorption rate studies in sodium hydroxide solution**
K. Ofosu-Asiedu, U.S.T. Kumase, Ghana and S.H. Wu, Sino-Arab
Chemical Fertilizer Co. Ltd, China

No question or comment.

SECOND GENERAL SESSION

'ENVIRONMENTAL ISSUES RELATING TO FERTILIZER PRODUCTION AND USAGE'

Chairman: C.H.M. Vinke, Hydro Agri Europe S.A., Belgium

Rapporteurs: C.P. Langeveld, Amsterdam Fertilizers BV, Netherlands
J. Straatman, Kemira Pernis BV, Netherlands

PAPER 6 Physico-chemical properties of the ammonium nitrate - ammonia system and their relation to aerosol and dust emission in ammonium nitrate prilling process
Z. Krawiec, Institute of Nitrogen Industry, Poland

Q - C.H.M. VINKE, Hydro Agri Europe S.A., Belgium

Is the information that you have developed applicable to the calcium ammonium nitrate system?

Ans: I think it will be applicable because the calcium ammonium nitrate in different mixtures also decomposes, and if ammonia exists even in small quantities, it may produce such a phenomenon.

PAPER 7 Advantages of complex fertilizers in logistics, application and environment
J. Platz and K.H. Ullrich, BASF AG, Germany

Q - B.K. JAIN, The Fertiliser Association of India

The effect on cereals production of an NPK complex fertiliser and a bulk blend of the same formula appears to be quite pronounced. Is it due to segregation in blends or are there any other reasons?

Ans: The effect is only due to segregation in blends caused by different physical properties of the straight fertilizers in the blend components.

Q - B.K. JAIN, The Fertiliser Association of India

The types of fertilizers, which are used in the blends are different, and the types of nutrients available will have some effects, as for example calcium ammonium nitrate has a better effect as a source of nitrogen than urea.

Ans: We agree with Mr. Jain that calcium ammonium nitrate has a better effect than urea. This is particularly the case in areas under intensive cultivation of the land where the negative effects of bulk blends are more pronounced.

Q - *J. STRAATMAN, Kemira Pernis BV, Netherlands*

What are the most important factors in segregation? If it is the size of the granules, would this problem be overcome by producing the same particle sizes for the blending materials, or are other factors of importance like of density?

Ans: In Germany, the fertilizer application is the NPK and the diamphos, calcium ammonium nitrate and potash are the components of the blends and are of different granule sizes. There are not available the straight fertilizers of the same physical properties.

PAPER 8 Coproduction of electricity and fertilizer: A key environmental/energy concept for the twenty-first century
H. Lewis Faucett, R.W. Weatherington, D.T. Bradshaw and T.L. Wright, Tennessee Valley Authority, USA

Q - *P. ORPHANIDES, Orphanides Consultants, Greece*

1. In the scheme you indicated, the power generation was named as combined cycle: is it co-generation using steam with the ammonia/urea process?

Ans: Yes, steam turbine and gas turbine on the same shaft.

Q - *2. Type of Wash Heat Boiler considered for synthesis gas down stream gazifier.*

Ans: The details of the design will be in the preliminary design study. We have basic information and will provide you these informations, assuming they are not proprietary.

Q - *3. Types of coal to be tested?*

Ans: Western Kentucky coal, bituminous coals, Illinois N° 6, containing anywhere from 3.5 to 5% sulphur.

Q - *4. What is the ash content of these coal?*

Ans: The details are available in the engineering design stage.

Q - *C.H.M. VINKE, Hydro Agri Europe S.A., Belgium*

You have shown the possible environmental impact of SO₂ and NO_x. Should you not possible to include CO₂ as well?

Ans: It is difficult to track CO₂ although it would be nice to credit for CO₂ to be converted into urea. However, the CO₂ will eventually re-enter in the atmosphere after its cycle in the living plants.

Q - *K.J. ELKINS, ICI Fertilisers, United Kingdom*

What are the comparative economics of the optimised IGCC System versus IGCC/F System e.g. in terms of return on investment/payback times?

Ans: It will be credit on the electricity. The urea is a by-product supplementary to the income of the power cost. In power plant economics, the capital investment becomes part of the rate base and the only concern is the incremental operating costs.

PAPER 9 Existing and forthcoming environmental regulations in Europe
D. Heather, FMA, United Kingdom and F. Samec, Grande Paroisse
S.A., France

Q - P. ORPHANIDES, Orphanides Consultants, Greece

What is the role and the influence potential of the FMA lobby in formulating environmental regulations affecting the fertilizer industry?

Ans: Within the Directorates of the EC, there are officers who will evaluate the suggestions and objections of the industry. Mr. Ripa di Meana of DG XI, for instance, pushed through the Carbon Tax while the "greenhouse effects" are not yet clear. The reality is, while the industry may have suggestions and objections, it can never be certain they would make a case out of those suggestions.

Q - B.K. JAIN, The Fertiliser Association of India

In the EC draft directive on fertiliser packaging and waste, a target of packaging waste per capita will not exceed 150 kg/year. In India, while there is preference for the use of HDPE bags, jute packaging has been used and can be increasingly used for Urea (Paper bags are comparatively uneconomical in the Indian situation). Is there any development towards easily destructible plastics for fertiliser packaging and/or which can be burned for energy recovery?

Ans: There is biodegradable polyethylene packaging available in general and this has been started by Italian and certain French companies. However, I do not know the results. Most polyethylene packagings do not survive 2 years' exposure to sunlight. Moreover, there is some development using starch derivatives which are biodegradable. The EC directive refers to all packaging and this includes fertilizer packaging as well.

Q - I. GHOSH, Shriram Fertilisers and Chemicals, India

Fly ash as solid waste is normally being used for land filling and a time will come when availability of land may pose a limitation. Is any technology developed to usefully utilise fly ash for making bricks or any building materials?

Ans: Fly ash has been used for some construction elements by blending with Portland cement and sand. It has been used as a filler and binder. It is used for road construction together with sandstones, etc. for Europe, it goes to controlled landfills and this is going to be prohibitively expensive.

Q - C.H.M. VINKE, Hydro Agri Europe S.A., Belgium

What do you think, personally, that the industry would experience in the next few years, irrespective whatever the outcome of Maastricht would be?

Ans: Personally, I feel that, given the close shave of the outcome of the French referendum on Maastricht, there will be a slowdown. Only a bit. I do not expect officials to quit the 5th Environmental Programme. There would be more money, more people and efforts to expend on that programme, perhaps at a slower pace.

PAPER 10 Environmental improvements at a UK fertiliser plant
R.J. Tricklebank, Kemira Agro UK Limited, United Kingdom

Q - G.L. MYBURGH, Sasol Fertilizers, South Africa

We would like to receive information on model and make of instruments for measurement of NO_x, NH₃ and dust in off-gases as described in this paper. We either don't have meters or experience problems with the meters that we do have. If you can at the same time supply us telephone/telefax numbers and names of contact persons, it will be very much appreciated.

Ans: The systems you have requested are proprietary systems, commercially available. The NO_x analysis system is chemiluminiscent system, ammonia in NPK stack is measured by infra-red absorption type and the NPK dust monitors are based on flicker measurements. I will be able to provide you with this information personally.

All the companies are based in United Kingdom.

1. NPK Dust Monitors
 Environment Research Technology Limited
 Fax 44 902 29010

2. Ammonia in NPK Stack Monitors
 Procal
 Model Pulsi 210 Infra Red Analyser
 Fax 44 733 235255

3. Nitric Acid NO_x Analyser
 Signal Instruments
 Fax 44 276 691302

Q - G. MUTHUKUMARASAMY, SPIC, India

a. What are the steps taken to reduce noise level especially in compressor house?

b. Noise level is normally high in the area around process air compressor due to high velocity of air.

Ans: a. Our policy is not to limit the noise in the compressor house, mainly out of concern for the safety aspects of full acoustic enclosures for compressors.

b. Outside the compressor house, we have introduced extensive noise abatement. On the air compressor, we completely replaced the intake silencer system, acoustic insulation on the inner pipe work. On the syngas compressor we have acoustic insulation and low noise anti-surge valves, but inside the compressor house itself we recognize it is a high noise area - 100 DBA in our plant and we make it a mandatory ear defender area. The plant outside in general is below 90 DBA, but in the compressor house, we have not attempted this. We contain the noise in the compressor house.

Q - L. ARAUJO, Quimigal Adubos SA, Portugal

In our company, we have difficulties to meet the legal limits concerning NO₃ content in liquid effluents from CAN plants. Can you comment on this point, including your experience?

THIRD GENERAL SESSION

"GRANULATION"

Chairman: W. Rittinger, BASF AG, Germany

Rapporteurs: J. Engelmann, BASF Antwerpen, Belgium
J.G.A. Reuvers, BASF AG, Germany

PAPER 12 Granulation of hygroscopic fertilizer ANP and CAN
R.C. Desai and V.K. Karia, Gujarat Narmada Valley Fertilizers Co.
Ltd., India

Q - A. VAN BREMPT, Kemira SA, Belgium

In order to reduce the reactivity of your ODDA chalk you add a limited amount of ammonium sulphate (max 0,8 % as SO₄).

Questions:

- a) Ca(NO₃)₂ content in your final product with or without the ammonium sulphate?*
b) Can you document the observed brittleness of the product by an increased amount of ammonium sulphate?
(Is there a special ratio AN/AS inside where the brittleness is critical?)

Ans: 1. Our CAN-process is not based on the BASF-technology. We do not normally add ammonium sulphate, we add sulphuric acid in order to maintain a sulphate content of about 0,5-0,8%. Without adding sulphuric acid, the calcium nitrate content increases to about 0,8-1,0%, to be compared with the Indian standards which permit a maximum of 0,5%. When we add sulphuric acid the Ca(NO₃)₂-content remains 0,1-0,2% but always less than 0,5%.

2. No, we have not measured this. We have never increased the sulphate concentration beyond 0,8%. To the best of my knowledge, the parameter to be controlled is the sulphate content of the final granules, this should be 0,5-0,8%. Depending up on this value, sulphuric acid is to be added to the ammonium nitrate melt.

Q - G. VENTURINO, Enichem Agricoltura, Italy

In the paper is said that it is necessary to maintain a proper value of the pH of AN melt. I would like to know which way is used to maintain the pH.

Ans: We inject ammonia gas into the ammonium nitrate melt. In the CAN plant, we inject ammonia one equipment before the granulator. Similarly, for ANP the pH is maintained by injection of ammonia gas.

Q - P. SUPPANEN, Kemira Oy, Finland

What are the benefits of BASF granulation compared to spherodizer, technical - economical?

Ans: Question answered by J.G.A. Reuvers, BASF, Germany

In comparing the benefits of a granulator vs. spherodizer, it is not possible to focus only on the drums. Instead, one ought to compare the processes. From our viewpoint, the granulation process offers a number of significant advantages :

1. In the granulation process, the water content of the slurry sprayed is about 6-8% vs. ca. 15-20% in the spherodizer process. This implies that in the granulation process, more water is evaporated more efficiently by using steam (use of heat of condensation). In the spherodizer process, however, the large amount of remaining water (in the slurry) is to be evaporated by means of hot air, i.e. more (expensive) oil or gas is to be used for heating. As such, energy consumption is less in the granulation process.
2. The amount of air required in the granulation process (for forming and drying the granules) is about 1/2 to 1/3 of the amount of air required for the same purpose in the spherodizer process. The economical and environmental advantages are obvious. Also, reducing the amount of air means less equipment for dedusting this air: investment and maintenance costs are reduced.
3. Given the dimensions of the drum, higher capacities per single train are possible in the granulation process. This reflects the higher water content of the slurry, the increased amount of air and the higher recycle ratio in the spherodizer process. Also the granulation process is more flexible with regard to product grades.
4. Increased safety with the granulation process due to the lower air inlet temperatures required for the drying drum.
5. Reduced investment for the granulation process due to the lower recycle ratio. The higher recycle ratio required in the spherodizer process implies larger elevators, screens, mills and the like. As such the specific design of equipment in our granulation loop is reduced.

The sum of these arguments leads us to favor the granulation process. Our continued - operation of this process for over 30 years has seen many developments. The implementation of these improvements resulted in technological, economical and environmental advantages of the process.

Q - B. MOORE, AECI Ltd, South Africa

1. *What raw materials are used to produce the ANP melt?*
2. *Which coating agents are used on ANP + CAN?*
3. *What is the percentage of P + N in final product ANP?*
4. *What are the handling characteristics of the ANP?*
5. *What is the ANP used for?*
 - (1) *direct application? If so - which crops?*
 - (2) *in bulk blends?*
6. *What environmental problems are created when producing ANP?*

Ans: 1. Raw materials used to produce the ANP melt are NP-acid and ammonium nitrate. Both are produced by dissolving rock phosphate in nitric acid, the subsequent separation of calcium nitrate using the Odda-process, and the conversion of calcium nitrate into ammonium nitrate and chalk.

2. The coating agents used for ANP and CAN carry the trade name BILCON 108 and BORAFLO, supplied by M/s Transmetal India Ltd. and the Bombay Oil Industries.

3. The total percentage of N and P_2O_5 in the final product ANP is 46%. Product grade: 23+23+0.

4. The ANP produced in our plant at Bharuch shows excellent handling characteristics.
5. The use of ANP depends upon its water solubility. Since in our case the water solubility is more than 80%, this ANP can be used on all soils and on all types of crops by direct application.
6. The environmental problems center on $N-NO_3$, $N-NH_4$, fluoride and P_2O_5 in the liquid effluents, and suspended particulate matter in the waste air. However, these problems have been avoided by providing proper treatment facilities.

PAPER 13 Granulation and fattening of fertilizers using the Kaltenbach-Thüring fluid drum granulation (FDG) technology
E. Vogel, Kaltenbach-Thüring S.A., France

Q - R. ANTONUS, Hydro Agri Europe, Belgium

Are any granulation additives required, and which ones, in the FDG process for urea and ammonium nitrate?

Ans: We do not need granulation additives or helping agents.

Q - A. VAN BREMPT, Kemira SA, Belgium

Concerning the FDG granulation of ammonium sulphate:

1. Are there special requirements for the particle size of the ammonium sulphate crystals in the melt? That's, is a given maximum particle size required?

2. If yes, how do you achieve this?

Ans: Yes, we need small crystals for the granulation of ammonium sulphate. Usually, we try to stay below 150 microns. We achieve this by controlling the residence time in the crystallizers or in the evaporation unit. It is not very difficult to achieve, but, obviously, if we have large crystals, the quality of the granules suffers, because the surface of the granule is not so smooth. When we do the abrasion tests, we find more dust. The crushing strength is unchanged. It is clear that if you have small particles on the surface of the final granule, we lose these on handling. This is to be avoided. It is less a question of the granulation itself but rather a question of the final quality of the product.

Q - F.S. AL-MUTAIRAN, Al-Jubail Fertilizer Company, Saudi Arabia

1. Does this "Fattening process" granulator act also as fluid bed cooler?

2. Did you check the caking tendency of the prills during storing in the bulk storage? Or do you use anti-caking agents?

3. What is the area this fluid drum granulator and its accessories occupy?

4. If possible, can you please tell us about the budgetary cost of the fabrication and installation of each fluid drum granulator?

Ans: 1. Yes, as shown on the flow sheet, the urea product undergoes, after granulation, cooling. It leaves the drum with a temperature of nearly 100°C, about 90-95°C, so you cannot forward this product to the storage as such. There is a fluid bed cooler, which I had shown.

2. With urea, for us the best caking tests are complaints from clients. We have not registered such complaints, to my knowledge, however. No sophisticated laboratory testing is done. We just measure the resistance to abrasion, which means the ability of the product to generate dust and that is a factor which obviously influences the caking tendency. We did not find reasons to believe that we risk something. On the contrary, we are improving the resistance to abrasion. Secondly, the biuret content is not changed. The hygroscopicity of the product is unchanged. As long as our clients agree to pay a premium of about 10 US\$/T, we consider the product is fine. Our client, Irish Fertilizer Industries, never complained about it.

3. For a plant with a capacity of fattening about 1100 T per day of urea, you need a ground surface of about 11 x 11 m².

4. The fattening plant in Cork for Irish Fertilizer Industries, which was commissioned in about 1988/1989, costed at that time US\$ 4 million turnkey. Further aspects, I will be glad to discuss afterwards.

Q - A.K. KUKKADY, SAFCO, Saudi Arabia

1. *Please explain how emissions from an urea prill tower are reduced? Is air-flow reduced?*

2. *What is the concentration of the urea melt?*

3. *What is the smallest unit practicable?*

Ans: 1. We did not reduce the air-flow through the prilling tower because our client did not want this for various reasons. But the quantity of melt which was sprayed into the tower was about half the amount prilled before. The plant is still producing between 1100-1200 T per day of urea, but half of it is sprayed into the tower and the other half into the drum. It is very simple, when you prill only half the amount of urea, the emissions of solid material, i.e. urea dust, are also halved. This we demonstrated in that plant several times.

2. The concentration of the urea melt is 99,7%.

3. That is difficult to answer. Technically we can design any type and any size of granulation plant. We have a small plant in our pilot unit, with a capacity of 500 kg/h. This plant can be operated around the clock. The smallest plant we built was for Enichem in Italy with a capacity of 400 T per day. I wonder whether somebody would build a smaller unit, nowadays. But there is no limitation in the process itself.

Q - N. LOUIZOS, Hellenic Chemical Products & Fertilizers, Greece

For the compaction plants: can you give energy requirements of the reference plants vs. conventional fertilizer plants? Is it possible that by adding FDG the overall energy requirement of a compaction plant exceeds that of a conventional plant?

Ans: I do not believe, that one can compare a conventional granulation plant with the process described here. If I would be looking for investment, I probably would not build such a granulation plant which includes a compaction unit plus a rounding-off unit.

It was a rescue decision: the product leaving the compaction unit is not any longer accepted by the market. Thus the client had the choice between closing the plant (or accepting a discount on his product) or improving the product quality. To do this, he made several calculations and found that adding such a rounding-off unit could increase the quality and could lead to an acceptable product price and, secondly, could increase the capacity. About 25-30% of fines which were not compactable could be eliminated. These fines were repeatedly recycled and, partially, had to be disposed of.

A comparison between a conventional granulation unit and a system combining compaction and rounding-off turns certainly in favour of a granulation unit. But if you have a compacting plant - and if you have problems with the product as described before - then the presence of such a fattening rounding-off unit is justified.

For a typical fattening unit of a plant which fattens between 500-1000 T per day, the energy consumption is about 5 kilowatt per T of product passing through the drum. You can double this figure if you include the cooling system and the scrubbing system. Thus, about 10 kilowatt per T for the fattening operation is an approximate value.

Q - P. ORPHANIDES, Orphanides Consultants, Greece

When you mention that no additives are used, you restrict yourself to the granulation loop. But the client may add or is adding additives to the melt or to the final product.

Ans: You have put it the right way. We do not need granulating agents or additives in order to granulate. But clients, who store products for very long periods, use additives of their own or from the market. For example, the Irish company IFI does not need to add any additives if they sell the product immediately after bagging. In that case, the melt contains no additives. But, if the product has to be shipped or stored for longer periods, then the client may decide to add some additives. In the case of IFI, a proprietary additive mixture is used. But we do not need such additives in order to granulate. As a matter of fact, we do not like these additives, because they sometimes disturb the granulation.

In other cases, for example the plant in Australia, the client is adding small quantities (1-2%) of a filler in order to colour his product. But, for us, this is a disturbing element of which we have to take care of. It is a kind of local clay which we do not need as a supporting granulation agent but we accept the material and adjust the operating parameters.

Q - R.J. TRICKLEBANK, Kemira Agro UK Ltd, United Kingdom

What is the minimum size increase you can achieve with FDG for ammonium nitrate fattening? I.e. is there a minimum thickness of stable layer you can add to the product?

Ans: It is a simple mathematical relation. We found that we can vary the ratio between liquid and solid when we fatten. When you crystallize ammonium nitrate or other compounds, you have to remove a certain quantity of heat. This implies a certain quantity of air and this amount of air influences the sizing of the drum.

In various simulation tests in our pilot plant - in which we have the possibility to granulate or fatten between 500 to 1000 kg/h of ammonium nitrate or urea - we found that when we are fattening, the optimum ratio between liquid and solid was 1:1. This means that we double the weight of the granules; the diameter of the granules is increased by the cubic root of 2 (about 1.26). The diameter is thus increased by about 30%.

This factor can be changed by varying the ratio of liquid to solid. We made tests in our pilot plant with a ratio liquid/solid of 4/1. The total weight was increased by a factor 5, the diameter was increased by the cubic root of 5 (1.71). We made some tests for Kemira: this procedure reduces the capacity of the plant as the same quantity of air is used to remove the heat of crystallization.

PAPER 14 Operating experience with rule-based control of the CAN/NPK granulation
P. Bo Olsen and K. Sorth, Kemira Denmark A/S, Denmark

Q - O. LERNER, Haifa Chemicals Ltd, Israel

Was a "neutral net" approach considered as for achieving the required "expert system" you were looking for? If yes, why was it overruled?

Comment by Mr. Lerner:

My question was fully answered by Mr. Sorth in his lecture and I will be glad to talk to Mr. Sorth afterwards.

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

1. Why was the screen analysis of product leaving the spherodizer chosen as the primary variable and not the screen analysis of product exiting the screens?

2. Could you give some details about the analytical equipment/instrument chosen for sampling and sieve analysis of product leaving the spherodizer?

Ans: 1. If we had chosen the product leaving the screens as primary control variable, the fuzzy controller would also adjust granulation according to screen performance. Our target was specifically to optimize the spherodizer granulation (quantity and quality) and then leave it to the operators to ensure the best possible screen performance.

Using the product leaving the screens would furthermore mean 3 times the costs of automatic screen analysis equipment.

2. The sample is taken by a small bucket passing through the free falling stream of fertilizer from an elevator.

This sample falls as a mono layer in front of a video camera..Based on pictures from the camera the particle size distribution is calculated automatically.

PAPER 15 Specialized liquid complex fertilizers in Lithuania
A.M. Sviklas, Azotas, Lithuania

Q - J. POUKARI, Kemira Oy, Finland

1. How pure are your raw materials, H_3PO_4 , MgO, CaO?

2. How much insoluble solids are present in your liquid fertilizers? (or: how big are the holes in your spreading equipments for liquid fertilizers)?

Ans: 1. Phosphoric acid produced thermally was used. $Mg(NO_3)_2$ derived from ammonium nitrate served as the source of MgO.

2. All insolubles were separated off and the resulting liquid was used as fertilizer (the holes are rather small, however, and the apparatus compact).

Q - I. GHOSH, Shriram Fertilisers and Chemicals, India

1. *What is the distribution pattern? By tankers or special container? What is the material of construction? What is the method of application?*

2. *What is the optimum distance to the consumption points in order that transportation charges remain within control?*

Ans: 1. The product was bottled and the bottles were distributed. Part of the production was sold in tanks. No special stainless steel is required. Application is by supplying the roots as well as by spraying on the leaves.

2. We consider the Lithuanian area, but suggest that it is not limited, especially for the bottled products.

PAPER 16 Evaluation of production flexibility and costs by NPK pilot plant experimentation

F. Cocquio and G. Venturino, Enichem Agricoltura SpA, Italy

Q - A. VAN BREMPT, Kemira SA, Belgium

In the granulation of K_2SO_4 , what is the influence of the K_2SO_4 -sieve analysis on the granulation efficiency and on the product quality?

Ans: Generally, the potassium sulphate available in our plants is in the form of very small crystals coming from Sicilian mines. We experienced no difficulties in granulating even coarser K_2SO_4 as available in our Italian market.

Q - L.K. RASMUSSEN, Kemira SA, Belgium

Concerning the precipitation of $CaSO_4$, would you please indicate the pH-range in which the $CaSO_4$ is precipitated as hemihydrate in a stable phase? Does the $CaSO_4 \cdot 0,5 H_2O$ precipitation take place in a pH-range where viscosity problems are frequent? If yes, how do you minimize these problems?

Ans: During the nitric acid attack of rock phosphate, we sometimes added some ammonium sulphate and this ammonium sulphate caused (due to the low reactor temperature) the precipitation of calcium sulphate dihydrate. During the successive ammoniation stages, the temperature increases to the boiling point, and the $CaSO_4 \cdot 2H_2O$ is transformed into the calcium sulphate hemihydrate. During this transformation, many Ca-ions are set free, there with disturbing the equilibrium between the phases and causing the precipitation of calcium phosphate. The solution to this problem was - practically, in industrial plant - to raise the temperature during nitric attack to cause the precipitation of calcium sulphate hemihydrate or to add the ammonium sulphate directly during the ammoniation stages.

Concerning the viscosity problems: there are not clearly related to the calcium sulphate hemihydrate. In any case, by maintaining a stable form in which the calcium sulphate is present and even by maintaining a correct water content in the slurry, a correct viscosity in the slurry can be achieved.

PAPER 17 Fluidized-bed (FB) dehydration on an industrial scale
Yu.Ya. Kaganovich, LenNIIGyprochim, Russia

No question or comment.

PAPER 18 New developments in corrosion resistant materials for fertilizer acids
R.J. Borges, Chas. S. Lewis & Co., Inc., USA

Q - G.L. MYBURGH, Sasol Fertilizers, South Africa

1. *What will be the corrosion and erosion resistance of Lewmet 25 and 125 against 28% P₂O₅ at 135°C?*
2. *Machineability of these two materials?*
3. *Are there DIN or ASTM specifications available for Lewmet 25 and 125?*
4. *Is it known whether flow and pressure control valves are available in these two materials and if so, what is the trade name for the valves?*
5. *Is the material (25 and 125) suitable for scrubbing tower nozzles?*

Ans: 1. Corrosion and erosion resistance of these alloys in 28% P₂O₅ at 135°C: we have laboratory test data at temperatures up to 120°C. These are static tests. We see no change in the corrosion resistance. We have actual plant operating experience with the Lewmet 25 alloy at temperatures up to 100°C. We have some operating experience with the Lewmet 125 alloy, at a plant in Canada, where it also serves as an evaporating surface, with about 42% P₂O₅ at temperatures up to 90°C. To answer the question, I do not believe that the corrosion and erosion resistance will be unchanged by increasing the temperature to 135°C but I have no direct test or plant data that can confirm this. But the material is available in case you want to perform tests.

2. As I mentioned in my lecture, they are both fully austenitic materials. The machineability would be similar to other fully austenitic nickel-based alloys.

3. At present, there are no DIN or ASTM specifications available. These materials have so far been used only with our own equipment. We have never made the effort to get these materials in the ASTM standards.

4. In addition to pumps, my company also manufactures valves: yes, valves are available in these materials. I should add that we got into the valve business because we could not find anybody interested in taking our alloy and making a valve for these applications.

5. Yes, from a corrosion standpoint, there is probably absolutely no problem whatsoever. If you have solids in the circulating stream, abrasion resistance is not going to be tremendous due to the fact that it is a fully austenitic alloy. I should also mention that when you have combined corrosion and abrasion, first you have to beat corrosion and then you can start beating abrasion! It would be a suitable material but I do not know whether it would be the best material.

Q - G. MUTHUKUMARASAMY, SPIC, India

1. It has been reported elsewhere that vessels fabricated of high silicon stainless steel ("Sandwik SK") do not require any lining inside for using as a absorption tower and drying tower in a sulphuric acid plant. Is there any similar development by Lewmet?

2. When Lewmet piping material is used in a phosphoric acid plant, is there any necessity of lining with rubber? If there is no rubber lining, what is the effect of the scaling during operation on the piping? What are the methods used to remove scales during plant shutdown? Do you recommend any chemical cleaning for removal of the scales? What is the expected life of this material?

Ans: 1. At present, the material is not available in plate or sheet form and therefore we can not fabricate vessels or other similar equipment. There is work in development in order to try to generate sheet material, but it is not available now.

2. There is no necessity of lining with rubber. Concerning the effect of scaling on the piping, I cannot answer this question. It really would depend more generally upon your particular operating conditions as to how bad the scaling problems are. From what I have seen, it varies from plant to plant, dependant on fluid velocities through the pipe lines and the like. To remove scales, it is common practise in North America to boil the system out with dilute sulphuric acid. Other chemicals have also been used. It is beyond our scope to recommend any chemical cleaning. We cannot say that boiling dilute sulphuric acid is not a problem, and I do not recall now the other acids that have been used but you do want to avoid chlorine- or fluorine-based acids. Other acids should pose no problems. From a corrosion standpoint, the expected life of this material should probably be a minimum of 10 years, perhaps longer.

When you get into corrosion and abrasion or wear properties, it is really dependant on the types of abrasives that are in the slurry or in the fluid. Gypsum, not being very abrasive, is generally not a problem. If you have significant amounts of SiO_2 or hard silica in your slurries, then shorter lives will result.

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

1. Is the Lewmet 25 alloy available to site repairs by welding?

2. If so, are there currently available welding electrodes?

Ans: 1. Yes, you can repair the material in the field by welding. We have not encountered any problems to date with corrosion in the heat affected zones due to carbide precipitation or anything of that nature. The alloys are generally melted via induction furnace and subsequently treated with AOD or argon/oxygen decarburization, so we are able to get the carbon levels down to very low levels, so you can do repairs by welding.

2. There are welding electrodes available, we have both manual stick-electrodes and wire available for repairwelding, which we use obviously in the repair of our own materials during the casting process.

FOURTH GENERAL SESSION

"PHOSPHATE PROCESSING"

Chairman: N. Hummadi, JPMC, Jordan

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

PAPER 19 Low Grade Rock Phosphates:

- (a) **Alkaline solubilization of Brazilian aluminum phosphate ore**
G.I. Horita, Instituto de Pesquisas Tecnológicas, Brazil

No question and comment.

- (b) **Utilization of low grade rock phosphates in the manufacture of phosphoric acid**
J.L. Thakkar and R.S. Vyas, The Dharamsi Morarji Chemical Company Limited, India

Q - S.A. NIANG, *Compagnie Sénégalaise des Phosphates de Taïba, Senegal*

1. *Could we please have an idea of the cost comparisons for production by these methods for low concentration phosphates and high concentration phosphates?*

2. *Please advise the influence of metallic impurities such as iron, aluminium and magnesium on your process, specifically the filtration problems?*

Ans: 1. (by J.L. Thakkar)

As far as the plant is concerned, it has additional equipment by way of an additional filter because there are two filtration stages, and that the recovery of H_2SiF_6 is done at the concentration stage. A higher capacity evaporation system is required because of the lower strength of the acid. So these are the two additional capital equipment items which would have to be offset against the lower price of the phosphate rock. Presently, the Indian phosphate rocks do not have any commercial value. I know that there are many other flotation tailings which end up as high silica and as well as many mining operations where a lot of silica material is dumped as overburden. So all these will have a much lower value per unit of P_2O_5 . Overall, it depends on a case to case findings as to what constitutes an economically feasible proposition.

As for the process utility, there will be a higher steam consumption.

Ans: 2. (by R.S. Vyas)

The first stage filtration mass is slimy and the rate of filtration is very slow. To improve the rate of filtration, we have tried certain additives. The most suitable of these are certain anionic flocculating agents. When these agents are added at 100-200 ppm, there is coagulation taking place with the slime. Filtration rates improve but this is not very satisfactory.

(c) Utilization of phosphate rock from the Southern extension of the bone valley deposits
B.M. Blythe, S.M. Janikowski and D.W. Leyshon, Jacobs Engineering Group Inc., USA

Q - N. HUMMADI, JPMC, Jordan

Could you please indicate the extent of the South Florida reserves referred to on Page 206?

Ans: In the range of about 100 million tons, but this represents the reserves of only one producer.

Q - L. MEBARKI, EPE-FERPHOS, Djebel Onk, Algeria

Did you have to grind the phosphate of Company "X" prior to the pilot-plant tests, and if so, to what size specification?

Ans: Pebble was typically ground to a maximum of 2% retained on 35 Mesh, and this was fed to the pilot-plant unit.

Q - G. MUTHUKUMARASAMY, SPIC, India

Increased tendency for foaming has been observed when the organic matters in the phosphate exceed 3.5%. Do you recommend any pre-treatment to reduce the organic matter in phosphates?

Ans: Organic carbon does not normally reach 3.5% but there is a trend away from calcination or pre-treatment. The use of defoamer additive of improving effectiveness and mechanical defoamer paddles of proper design with an adequate power input can reduce the problem.

Q - F. DODEEN, JPMC, Jordan

1. In Table 2 of your paper, soluble SiO_2 content in tested samples ranged from 0.5 - 0.58% and F/ SiO_2 ratio is 5.85-6.74. Is this ratio acceptable to convert F to fluosilicic acid or do you need to add active silica?

Ans: Active silica is not normally required.

Q - 2. Filtration rates indicated in Table 3 show 8.8-10.4 tpd $\text{P}_2\text{O}_5/\text{m}^2$. Did you confirm these figures by industrial or semi-industrial tests? If so, what were the results?

Ans: The tests reported in Table 3 are all leaf tests. The results of tests, Column 1 - Plant site, uses slurry being fed to the industrial filter. The plant filter was actually operating at about 9 tpd $\text{P}_2\text{O}_5/\text{m}^2$.

Q - P.A. SMITH, Société Chimique Prayon-Rupel, Belgium

In your Table 4 the P_2O_5 losses are 4.09% of the P_2O_5 fed. The same test on pilot-plant gave 2.88%. This seems to be a large difference. What are the reasons for such a big difference between your pilot-plant and the industrial results? Is there not also an error between the t dry gypsum/t P_2O_5 of column 1 and column 2?

Ans: The small single-tank pilot-plant with its intense agitation does not fully simulate the industrial plant in question, which is an old PRAYON design having a low recirculation. As such the citrate-soluble (co-crystallized) losses on the pilot-plant are lower and the water soluble losses on the leaf test are normally lower than in the industrial unit.

PAPER 20 A new mineral process for clean commercial phosphoric acid production by simultaneous removal of sludge organics, cadmium and other heavy metals and recovery of rare earths
P. Becker, Duetaq, France and OTP, Togo

Q - F. THIRION, Société Chimique Prayon-Rupel, Belgium

The process as described is relatively sophisticated and complex. Do you not think that treatment or selection of phosphates and/or the selection of phosphoric acid process could affect the cadmium content of the acid? It is well known that two-stage processes have a greater flexibility with regard to the distribution of impurities in the calcium sulphate and the product acid. In a process such as the CENTRAL-PRAYON the level of anhydrite can be adjusted by modifying the operating parameters. Would it not be better to use a process of this type rather than an additional process?

Ans: The details of the CENTRAL-PRAYON process and specifically the possibilities of modifying the split of heavy metals & rare earths between the acid and solid phase are outside my experience but it is obviously an alternative, as is the effect of the source of the phosphate.

Comment: F. THIRION

The problem of the environment is not only a problem of the industrialised countries but also the mineral producing countries specifically those producing phosphoric acid and fertilisers. The production of potentially dangerous concentrates of impurities causes the need to safely store these residues. A phosphoric acid process that produces a self-drying hemihydrate with a relatively low concentration of these impurities where this residue can be used as mine refill is obviously of interest.

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

A slightly frivolous question but quite important to resolve for future orators, is the word "DECADMIATION" a correct word in the English language?

Ans: Personally I do not know grammatically, but I must say that we have to use it as there is no alternative.

Comment: G.J. WITKAMP, Delft University of Technology, Netherlands:

I would like to make the following comment about the first two paragraphs on page 221.

"The Becker text says that the uptake of the PO_4^{3-} ions in the calcium sulphate lattice enables the incorporation of trivalent ions such as Ce^{3+} , La^{3+} , Y^{3+} , etc. However in calcium sulphate dihydrate (gypsum) the uptake of these ions was found to occur in both the absence and presence of phosphate ions, to approximately the same extent. If this non-necessity of PO_4^{3-} ions would also apply in the case of PSY, it would mean that the high distribution coefficients in PSY are due to an altered crystal structure or other changes in physical properties rather than the presence of PO_4^{3-} ions. The fact that in anhydrite and in dihydrate the uptake of the divalent ions is generally lower than that of trivalent ions is also an indication that charge compensation (needed when, for example La^{3+} replaces Ca^{2+}) can take place in other ways than by replacing the SO_4^{2-} by PO_4^{3-} .

The radius of mercury ions, Hg^{2+} , is 114 picometers, i.e. almost equal to that of Ca^{2+} , 112 picometers. The cadmium ionic radius, Cd^{2+} , is 107 picometers. From this we would expect a higher mercury uptake than cadmium".

Q - L. RASMUSSEN, *Kemira SA, Belgium*

To what extent is your process applicable to other phosphates?

Ans: The removal of cadmium and rare earths applies to acids produced from all phosphates, the actual recovery being a function of the concentrations in the acid treated.

PAPER 21 Mass balance of heavy metal ions in hemihydrate phosphoric acid processes
E.T.M.J. Martynowicz, G.J. Witkamp, G.M. van Rosmalen, Delft University of Technology, Netherlands and N.W. Kolmeijer, Hydro Agri Rotterdam, Netherlands

Additional industrial information was presented verbally by N.W. KOLMEIJER during the Fourth General Session prior to the presentation of E.T.M.J. MARTYNOWICZ.

In 1988 Hydro Agri Rotterdam - HAR operated 2 single-stage hemihydrate phosphoric acid plants running on high cadmium phosphate. Capacity was 200,000 tpa P_2O_5 , overall P_2O_5 loss 7%, an overall efficiency of 93%, and the quantity of discharged cadmium with the gypsum-effluent 12 tpa. In that year, the discharge permit was not simply extended for a further three years as before, but the authorities demanded drastic environmental improvements. Main issues at that time were cadmium and phosphate and for both substances basic reduction techniques were considered to be available and hard limits were set.

In a covenant running to the year 2000, a scenario for effluent improvement was agreed upon. The target was that by 1994 there should be an overall reduction of at least 50% in the amount of P_2O_5 discharged and the quantity of discharged cadmium should be 0.6 tpa maximum. This covenant also includes a clause that the cadmium content of the produced fertiliser should not increase; but HAR was permitted time to develop the necessary technology and to convert the existing 2 single-stage hemihydrate plants.

With regard to the behaviour of other heavy metals in phosphoric acid processes, hardly any information was available and therefore a dedicated effort was made to carry out a research & development programme to study this subject. At the same time, HAR had the obligation to investigate the possibilities for re-use of gypsum as an alternative to discharging into the river.

Meanwhile HAR had obtained a new permit running from 1991-1994 with an outlook to the year 2000, and this permit contains stricter conditions than those agreed upon in the covenant of 1988. One of the reasons is that the Dutch authorities wanted to fulfil the agreements reached during the most recent North Sea Conference, the Rhine Action Plan, etc., whilst they also wanted to implement latest available technology.

In the meantime, one of HAR's single stage hemihydrate plants had been converted to a special design of HAIL's 2-filtration stage HDH process. The special features being multiple rehydration or conversion reactors and the possibility of adding silica to the dihydrate section (Figure 1).

However the R&D work showed the importance for the full conversion of hemihydrate into dihydrate gypsum for both the aspect of P_2O_5 efficiency and also the minimisation of the heavy metal content of the discharged gypsum. Full conversion is practically impossible to obtain in a one-reactor system, without silica addition, especially when a high aluminium rock is used.

Aluminium is favourable for obtaining well-filtering hemihydrate crystals of the cluster type, but it also hinders the conversion of hemihydrate to dihydrate in the second stage.

This recycling effect of aluminium from the second stage back to the first stage is shown in Figure 2.

Figures 3 and 4 show practical results obtained in the converted plant as compared with the results in a single-stage hemihydrate plant. Figure 3 shows that apart from lead and mercury which practically fully precipitate with the gypsum, many metals are reduced to levels which are within or close to the detection limit in the gypsum slurry. Figure 4 shows the effect of the high efficiency of the two-stage process on the discharged P_2O_5 content.

Q - *F. THIRION, Société Chimique Prayon-Rupel, Belgium*

1. You have stated that the "formation of anhydrite is to be avoided" if the level of co-crystallization of cadmium and other heavy metals is to be maintained at a minimum. Is it as simple as that, and if so, what techniques do you use industrially to minimise the formation of anhydrite?

Ans: In hemihydrate plants the formation of anhydrite is inevitable. Accidentally, large quantities can be formed. When the reaction conditions are well controlled the level of anhydrite is below the level of accurate detection.

Q - *2. In your paper you have cited many analytical values. Could you please confirm to which basis these are referred, dry solid basis or anhydrite basis (with all crystal water driven off)? PRAYON uses the anhydrite basis as this helps to account for cases where mixture of hemihydrate, dihydrate or anhydrite occur.*

Ans: The analyses are referred simply to the dry solid basis.

Q - *3. The aim of Hydro-Agri, Rotterdam is to minimise the heavy metals and especially cadmium in the calcium sulphate discharged, that is, to maximise their presence in the acid. In the case of many fertiliser producers the presence of these metals in the acid is an inconvenience. Is it not therefore, perhaps, more interesting to use a double stage process like the CENTRAL-PRAYON process when a purer acid is desired?*

Ans: Yes, certainly by changing the conditions to have a greater percentage of anhydrite than normal, a cake with higher cadmium could be produced and an acid with a lower cadmium level.

2-FILTER HDH-PROCESS

38

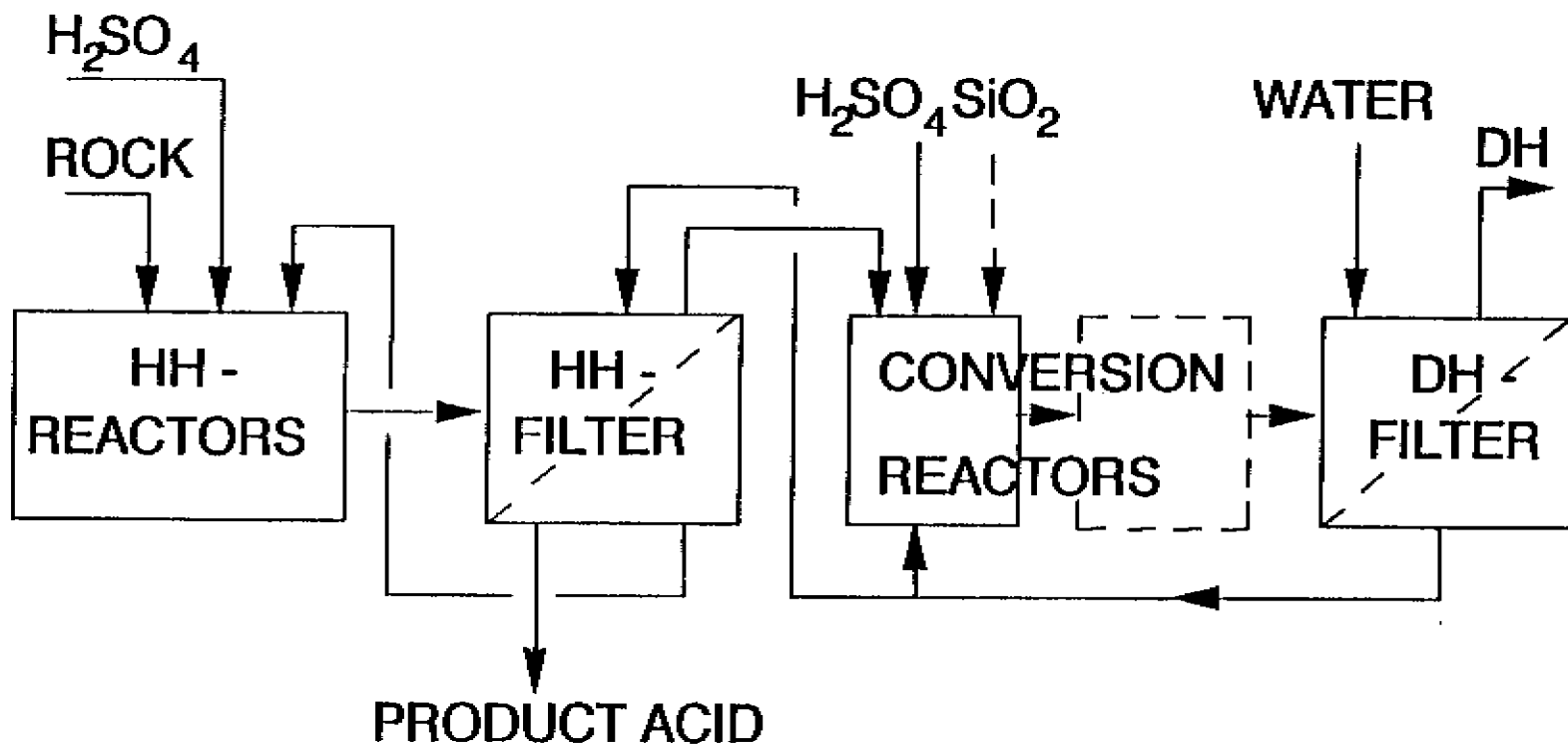


FIG. 1

Al_2O_3 - CONCENTRATIONS IN HDH-PROCESS

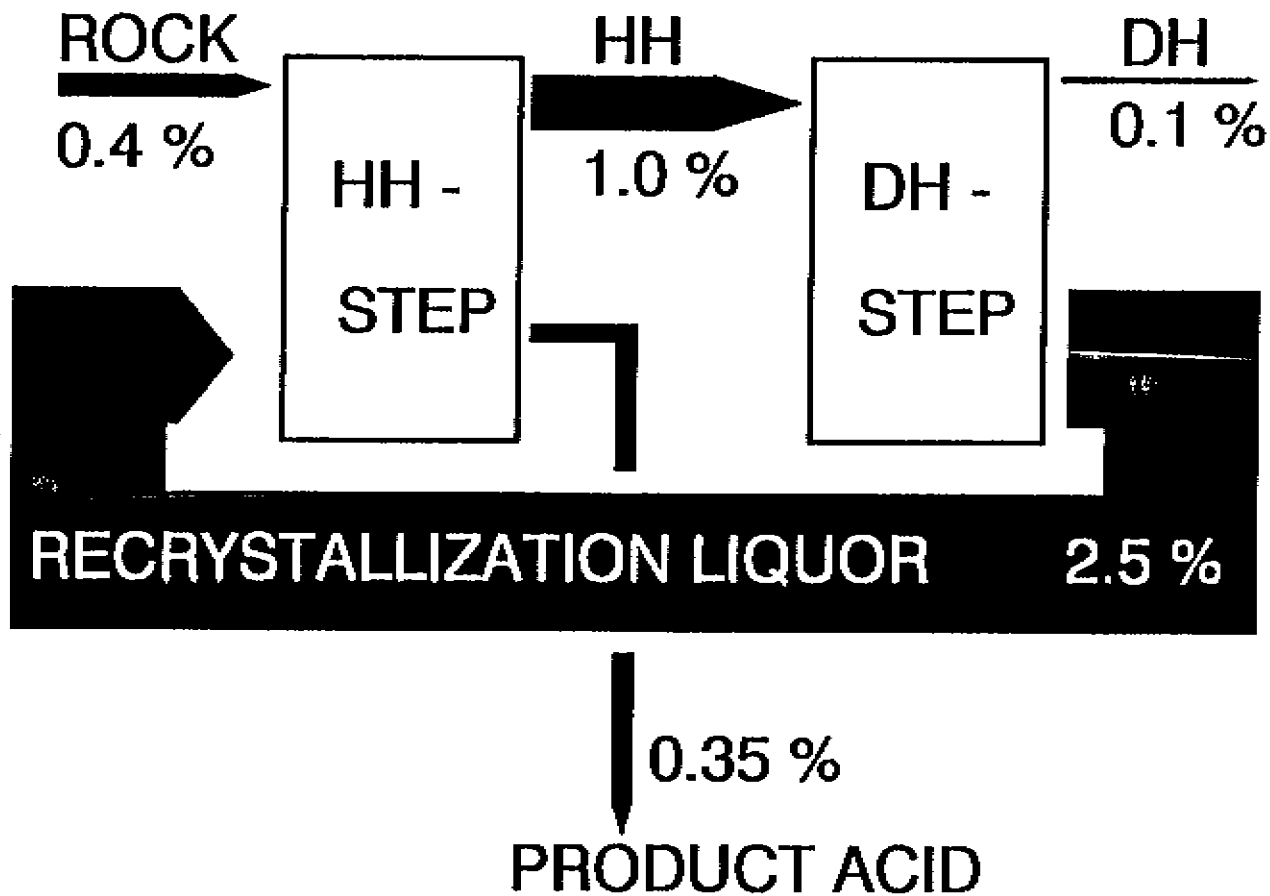
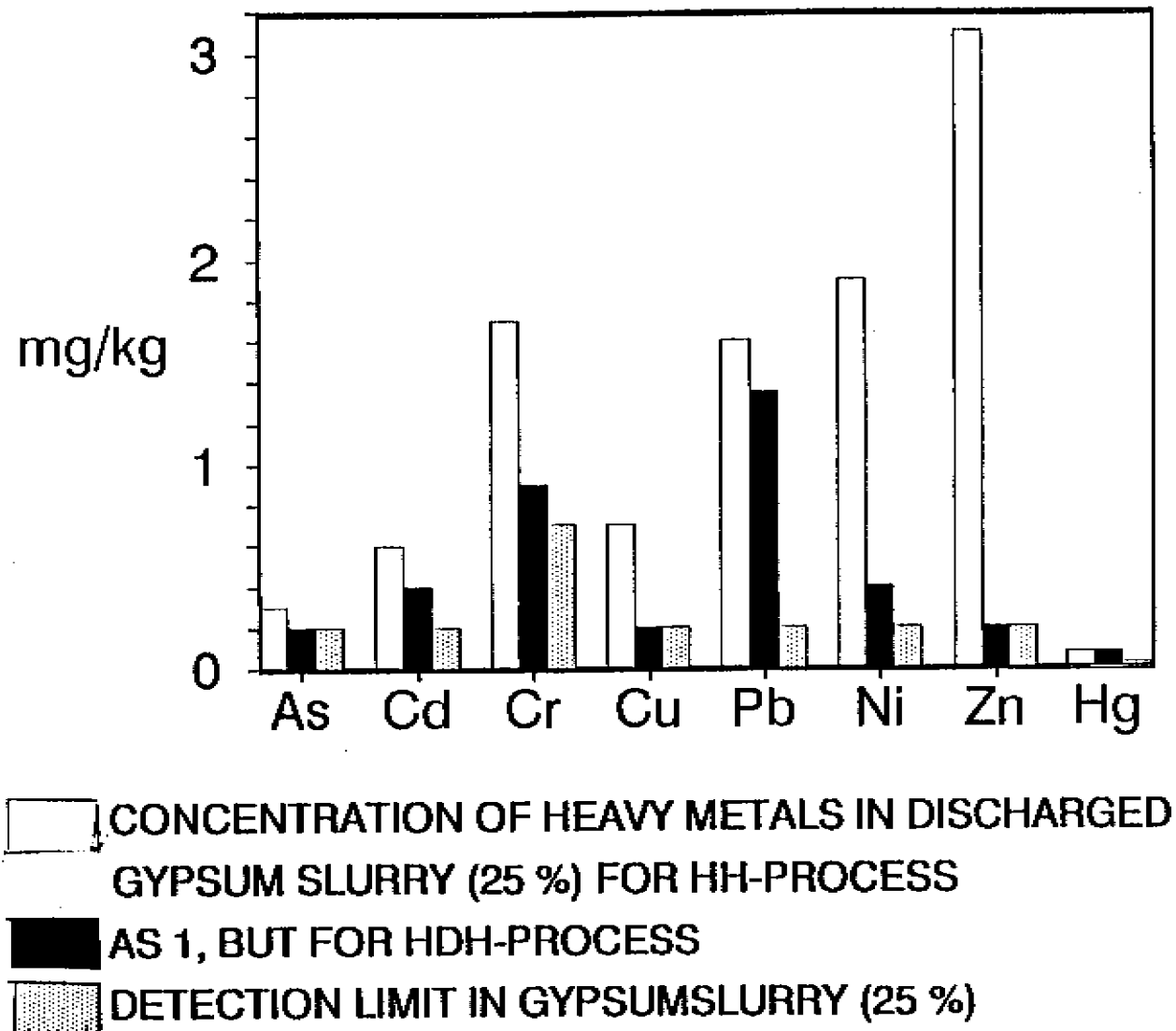
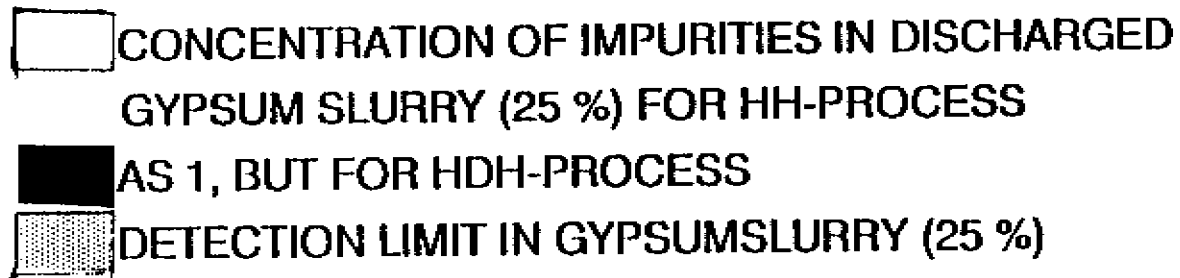
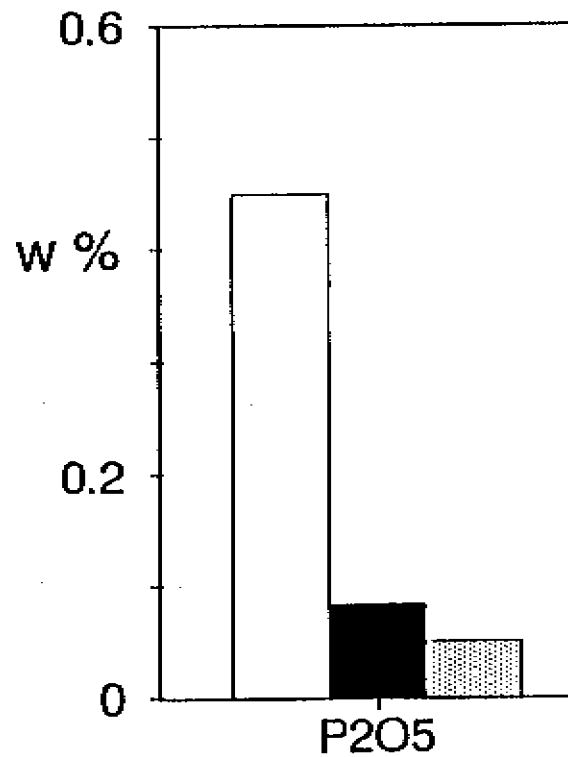


FIG.2

**FIG. 3**

**FIG. 4**

PAPER 22 **Developments in TSP production: Slurry versus powder route, the Chinese experience**
N.D. Ward and B.T. Crozier, Hydro Agri International Licensing, United Kingdom

Q - P.A. SMITH, Société Chimique Prayon-Rupel, Belgium

1. Your phosphate reactivity figures quoted are of great interest for SSP/TSP manufacture. Are such results useful for the evaluation for phosphates for phosphoric acid manufacture, the attack being by a sulpho/phosphoric acid with varying contents of sulphate?

Ans: As far as I know, and I am not an expert in phosphoric acid technology myself. Nothing can replace a pilot-plant test for the evaluation of a phosphate for phosphoric acid manufacture. The kinetics are linked with the characteristics of phosphate, the process and the reaction conditions including sulphate. The reactivity test is more useful in the evaluation of phosphates for superphosphate manufacture.

Q - 2. Although you say that Dayukou has a low reactivity and that the slurry route is not recommended for this phosphate could you please give the value of its Reactivity Index? We have tested the phosphate in the slurry route and have found it somewhat similar to Alkaim, Iraq which has a slurry process TSP unit in operation!

Ans: Actually we did not do a Reactivity Test for Dayukou, but I believe the client eventually selected a Den route due to the low reactivity.

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

Why have you omitted some of the important factors in the comparison of the Den and Slurry routes for TSP manufacture?

1. Grade of product: you infer, in your reference to Dayukou that it is possible to make higher grades of available P_2O_5 using Granulated - Run of Pile (Powder) compared with the Slurry - GTSP route. Surely this is the case for all phosphates. In the case of Dayukou the Chinese put an emphasis on the APA grade at any price.

Ans: Yes, the powder or Den route does tend to give a higher APA value. Also the Den route is better suited for phosphates of low reactivity. In the case of Anning, using thermal acid, the P_2O_5 APA content could have been higher but this was not required. There was a strict limit on Free Acid and the space available from the purer than normal acid was taken up by the neutralising agent, FCMP.

Q - 2. Hardness of the Granule: slurry GTSP is normally at least twice as hard as a Granulated Powder Granule which only has a crushing strength of about 1.5 kg. Thus the product quality of slurry route GTSP is superior.

Ans: It is well known that the granule hardness of GTSP is higher than Granulated Run of Pile.

Q - 3. Fume and Dust: the Chinese believe the Slurry Process GTSP, to be a more modern process due to the fact there is less emission of fluorine containing fume during the curing process and that there is less dust produced. For Dayukou they have chosen the Den route. Do you think that this may perhaps influence the future decision in China?

Ans: Probably the reason that the Chinese believe that the slurry process is more modern is that all the large plants they have seen in Florida were of the GTSP type. Personally I believe that in the future the Chinese will tend to use the slurry process wherever possible.

Comment: We note that you report a 46% P_2O_5 APA grade for Jian Shan and Jinling phosphates. We assume that this is for slurry process GTSP. We cannot make 46% APA with 16% SiO_2 using Florida phosphate which is somewhat more reactive than the Chinese materials.

PAPER 23 Speichim / Rhône-Poulenc process: Main process features and the latest improvements of the phosphoric acid process
B. Satier, Speichim-Rhône-Poulenc, France and G. Apostoleris, SICNG, Greece

Q - F. DODEEN, JPMC, Jordan

1. You have described a system of gas cleaning with three stages of water scrubbing capable of attaining the EPA standards. Could you please compare this system with the system installed at Aqaba where the first two stages are water and the third stage is with sodium hydroxide solution?

Ans: 1. Sadly, I don't have the information at hand to reply to this question. The system presented in the paper was a "typical system" if required, a final stage with soda washing can be included. I have no additional details to those presented in the paper, perhaps I could eventually get information from the unit at Donau Chemie in Austria.

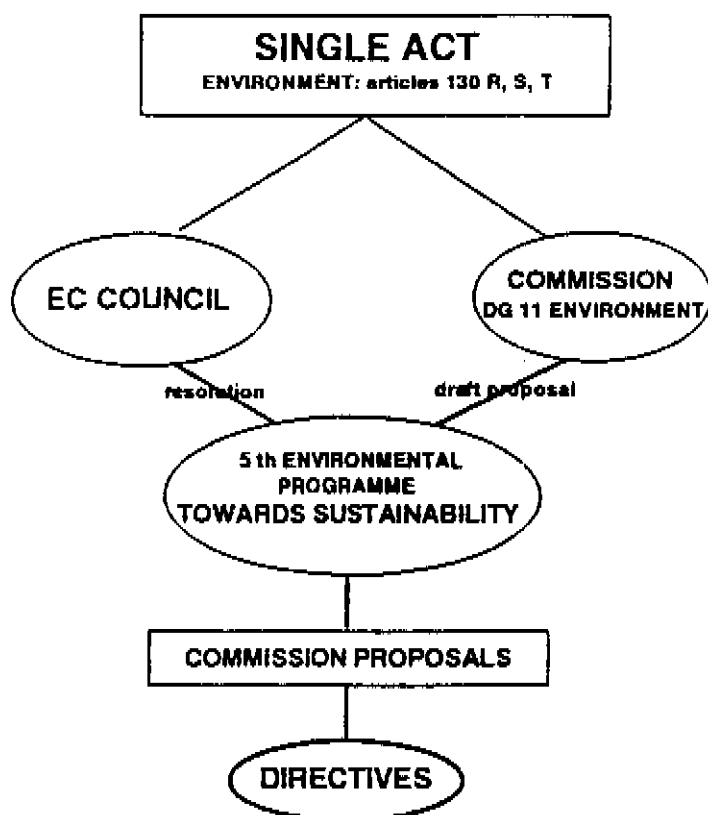
Q - 2. Could you please compare the heat exchangers used in the concentration of phosphoric acid, Polybloc versus Shell and Tube. Has Rhône-Poulenc/Speichim finally decided to use Shell and Tube units?

Ans: 2. We have made some tests comparing the block and graphite tube systems during the past few years at the Roches de Condrieu unit of Rhône-Poulenc and the results are extremely interesting especially in the fact that the use of tubes allows the reduction of surface area by a factor of 15-20%, depending on the case.

Q - M.S. SRINIVASAN, EID-Parry Ltd, India

What are the materials of construction used in the plate heat-exchangers of the closed-circuit cooling system linked to the condensers? What is the fluosilicic acid content of the circuit?

Ans: This is one more detail that we would have liked to have shown during the visit to the Rieme unit. At Rieme the metal used was Hastelloy C22, but this material depends on the local conditions of phosphate, water quality and water balance. The fluosilicic acid concentration also depends on these factors.



PAPER 13 Fertilizer granulation and growth through the fluidized bed drum technique - Experience and industrial results in the latest units built.
E. Vogel, Kaltenbach-Thüring S.A., France

The complete paper is enclosed.

PAPER 14 Operating experience with rule-based control of the CAN/NPK granulation
P. Bo Olsen and K. Sorth, Kemira Denmark, Denmark

Page 148: 5.3 The Rule Structure.... - Delete as it is the repetition of 5.3.

PAPER 15 Specialized liquid complex fertilizers in Lithuania
A.M. Sviklas, Azotas, Lithuania

The complete paper is enclosed.

PAPER 19(b) Utilisation of low grade rock phosphates in the manufacture of phosphoric acid
J.L. Thakkar and R.S. Vyas, The Dharamsi Morarji Chemical Company Limited, India

Page 202: 6.9. - 2) - Read 4.23% instead of 64.23%

PAPER 20 A new mineral process for clean commercial phosphoric acid production by simultaneous removal of sludge organics, cadmium and other heavy metals and recovery of rare earths
P. Becker, Duetag, France and OTP, Togo

Presentation of the slides:

Slide 1:

The objectives for the presented decadmiation technology are:

- . Operate directly with crude acid, (avoid a separate sludge removal stage),
- . Produce clean decadmiated acid,
- . Use simple, traditional crystallization technique,
- . Save costs.

Slide 2:

At its present state of development, the process offers three functions in one reaction/filtration operation:

- . Sludge removal,
- . Cadmium removal, (recovery),
- . Rare earth removal, (recovery),
- + organics, fluorides, if wanted.

Optional: Cadmium and rare earth recovery in a second crystallization filtration operation on small volumes.

Slides 3, 4, 5:

Sludge removal without additional process stage, just a reminder on sludge: there is no need to underline the advantage of an efficient sludge removal system; the only question is, what is the economical assignment to be given to sludge removal? To remind heavy cost effect, sludge can have - consequently - the advantage for an efficient sludge control.

Slide 6:

Rare earth recovery, if their concentration is high enough with the original phosphate ore, they will contribute to improve the economics for decadmiation. Crude rare earth oxide mixtures can be marketed at some US\$ 7/kg. It is to be expected that their potential for modern technologies will implement a growing market.

Slide 7:

The process is very simple. It consists in tailing a mini-phosphoric acid unit, (2% of the production plant), to the existing phosacid plant. The mini-plant is connected to the outflow of the concentration unit, 52-56% P_2O_5 .

Depending on the degree of decadmiation needed, only a part of the total production-flow may be processed.

The chemicals used are phosphate rock and sulfuric acid; producing some more acid, their costs can be considered nil. The customer is free from any chemical supplier.

For a 500 t P_2O_5 /day, decadmiation plant the reaction system will need a total reaction volume of some 35 m³

PSY will contain some 0.4% combined P_2O_5 .

Slide 8:

The reaction is presented on this slide; the solids precipitated, can be considered as a highly co-crystallized anhydrite, where PO_4^{--} and PO_4^{--} co-exist, replacing SO_4^{--} , Cd and rare earth replacing Ca^{++} . PO_4^{--} permitting ions with valencies up to three, like Yttrium, to be integrated.

Slides 9, 10:

Electronic microscope view of the precipitated crystals.

Slide 11:

Process development: 1988, first experimentation unit, batch system, 1/2 liter per hour systems; demonstrates how simple it is; (kitchen process).

Slide 12:

Minemet-recherche operation, 1989, batch system to study of the major factors governing the cadmium and rare earth distribution coefficients. There are 15 operating variables.

Slide 13:

Convertible, (Batch or continuous), Pilot unit, 10 liter/hour, operated with Hydro Agri Rotterdam; continuous but essentially batch operations. Togo acid from Grande Paroisse.

Slide 14:

Shows heavy loaded acid that was processed directly.

Slide 15:

Vacuum filter used for separation.

Slides 16, 17, 18:

Hydro Agri operating crew.

Slides 19, 20, 21:

Improved continuous reactor set up at Grande Paroisse, Rouen, France; capacity 19 liter/hour.

Slides 22, 23, 24, 25:

More efficient filtration system: improves cakes washing and recycle ratios: the Hoechst filter press. Size 1/20 of a square meter.

Slide 26:

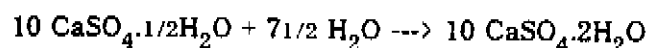
Cake shows consistent porosity, no cracks, low liquid concentration. For cadmium and rare earth recovery, this cake is then recrystallized in special liquor. Recrystallization resulting pure gypsum, (few ppm Cd), and mother liquor with cadmium and rare earth in solution; easy to be selectively precipitated.

Slide 27:

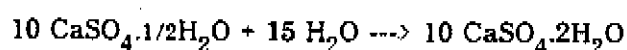
Recrystallization: mother liquor only 0.18 m³/ton P₂O₅ as acid. Liquor contains SO_4^- , Ca^{++} , Na^+ as major impurities.

PAPER 21 Mass balance of heavy metal ions in hemihydrate phosphoric acid processes
 E.T.M.J. Martynowicz, G.J. Witkamp, G.M. van Rosmalen, Delft University of
 Technology, Netherlands and N.W. Kolmeijer, Hydro Agri Rotterdam, Netherlands ..

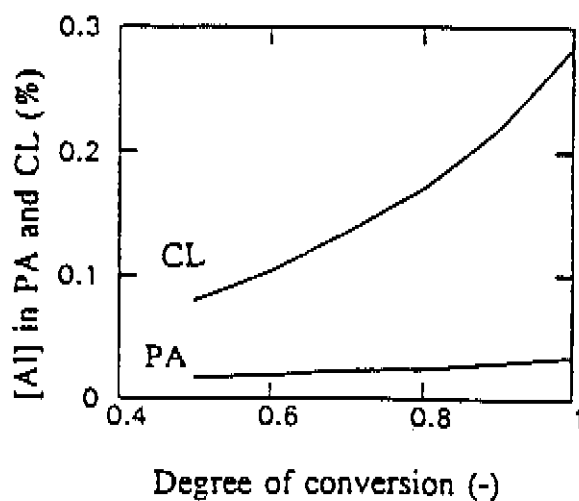
Page 224 last line:



should be:



page 234 figure 16 should be:



page 238 last line of "Conclusions"

The sensitivity analysis shows that the disposal via the calcium sulfate slurry is always lower in a HDH process compared to a HH process.

GRANULATION AND FATTENING OF FERTILIZERS USING THE KALTENBACH-THÜRING FLUID DRUM GRANULATION (FDG) TECHNOLOGY

E. Vogel
Kaltenbach-Thüring SA, France

LAST EXPERIENCES AND RESULTS FROM SOME PLANTS

1 - PRINCIPLE OF THE FDG PROCESS

The heart of the process is a horizontally-aligned cylindrical granulating drum, which rotates about its axis in the conventional fashion. The interior of the drum is fitted with special anticlogging lifters. But the main feature distinguishing it from conventional drum granulators is an internal fluidized bed. This comprises a flat perforated plate through which fluidizing air is blown (direct from the atmosphere or after conditioning, according to the product being granulated).

The granulator is supplied with seed material, which can be recycled off-size but might also be prills that it is desirable to enlarge or a compacted product that it is desirable to make smoother and rounder. In the granulator it is subjected to the dual operations of size enlargement and cooling or drying, as the case may be. This occurs progressively in the following cyclic sequence: the lifters raise the seed material to the upper part of the drum, where upon it falls onto the surface of the fluidized bed. This is where the product is cooled or (when the feed material is a slurry) dried. Product flows on the bed and falls into the lower part of the drum. As it falls, it is sprayed with the feed melt or slurry. The coated granule is then lifted back to the fluidized bed, where the new surface layer solidifies by cooling or evaporation of its moisture content. The same cycle is then repeated as many times as are necessary to reach the desired grain size.

Various additives, such as fillers, micronutrients or other specific additives, can be added with the sprayed product.

An external fan draws the air out of the granulator.

2 - PRACTICAL APPLICATIONS

2.1. Granulation of molten salts

The pre-eminent application of the FDG process is in melt granulation where the feed material is either completely anhydrous or contains so little moisture that the heat of crystallization is sufficient to vaporize it. Cooling is all that is needed to solidify the successive liquid layers deposited on the surface of the granules, so the fluidized bed is actuated by ambient air. The product collected at the granulator outlet passes straight to the screens and the on-size fraction undergoes final cooling before passing to store. The undersize and the crushed oversize are recycled to the granulation.

2.2. "Fattening" of urea or ammonium nitrate prills

Most prilling towers cannot produce prills with a mean diameter of more than about 2.5 mm, whatever the nature of the product. Ideally, from the point of view of handling or storage - and especially when it comes to bulk-blending with other granular materials, which are usually larger in size - many users would like larger prills. Without changing the usual recycle rate of a prilling tower (5 - 10%), the weight of the prills can be doubled and the mean diameter can be increased by 25%. The prills coming from the tower are simply introduced into the fluidized drum granulator as seed material to be fattened.

2.3. Granulation of solutions or slurries

The process is applied to granulation of ammonium sulfate solutions or slurries resulting from scrubbing of the gas exhausting from power plants burning coal.

After granulation, the product is further dried in a rotary drum, screened and then cooled in a fluidized bed cooler.

Oversize product is crushed and returned to the FDG and the fines are recycled to the ammonium sulfate solution which is sprayed in the FDG.

Undersize product is recycled to the granulator for further size increasing.

2.4. "Rounding off" compacted products

One of the drawbacks of compacted products is that the particles have sharp brittle edges which break easily during handling, generating dust. The FDG process makes it possible to palliate this drawback by depositing a film of the same composition on the compacted product surface. The product obtained presents an aspect very similar to that of a granule. The edges are fairly rounded and the resistance to abrasion is significantly increased.

3 - CASE STORIES

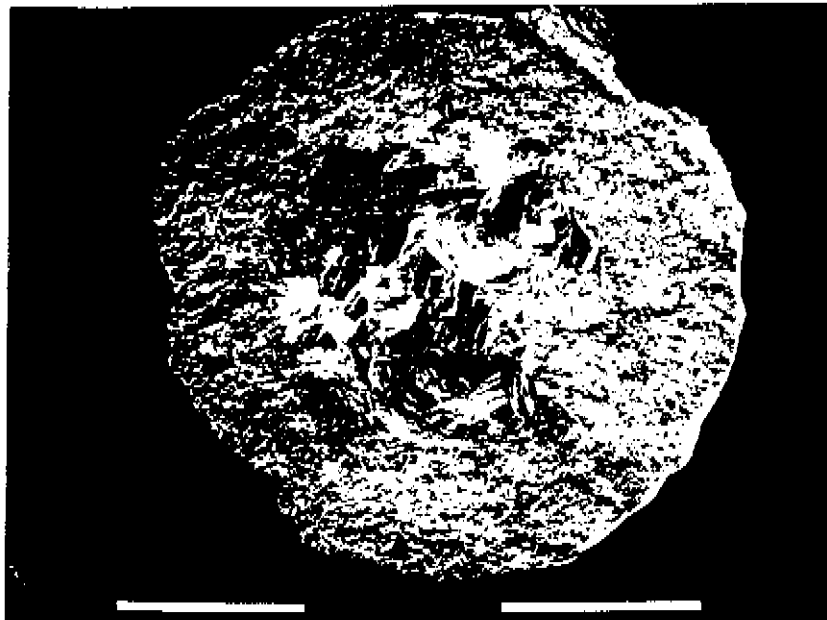
Nine plants have been built since 1985 using the KALTENBACH-THÜRING FDG technology.

This paper concentrates on five reference cases:

- a urea fattening plant with a capacity of 1200 T/D
- an ammonium nitrate plant with a capacity of 400 T/D
- an ammonium nitrate granulation plant of 250 T/D
- an ammonium sulfate granulation plant of 12 T/D
- a PK rounding off plant with a capacity of 1000 T/D



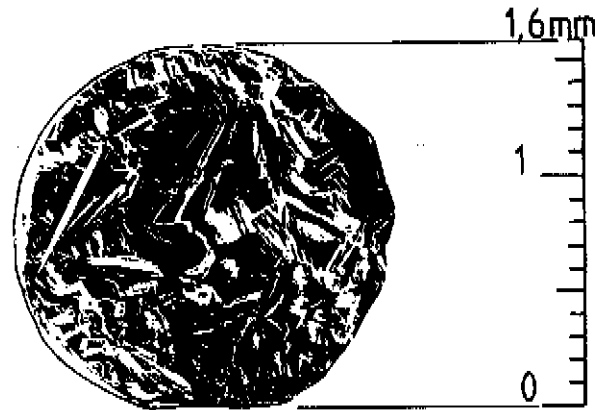
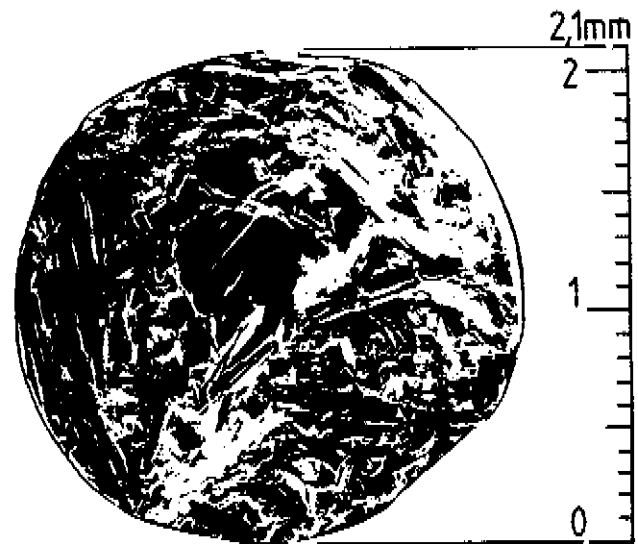
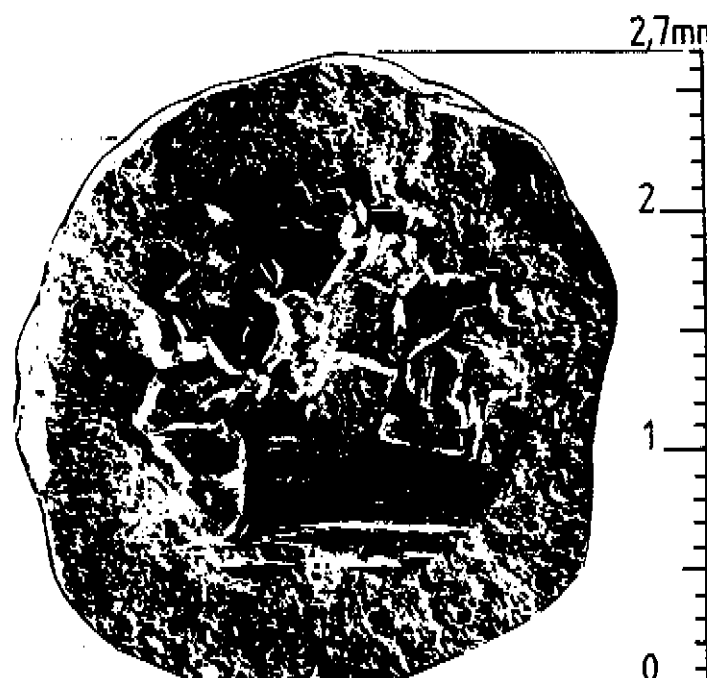
ORIGINAL PRILL - CROSS SECTION

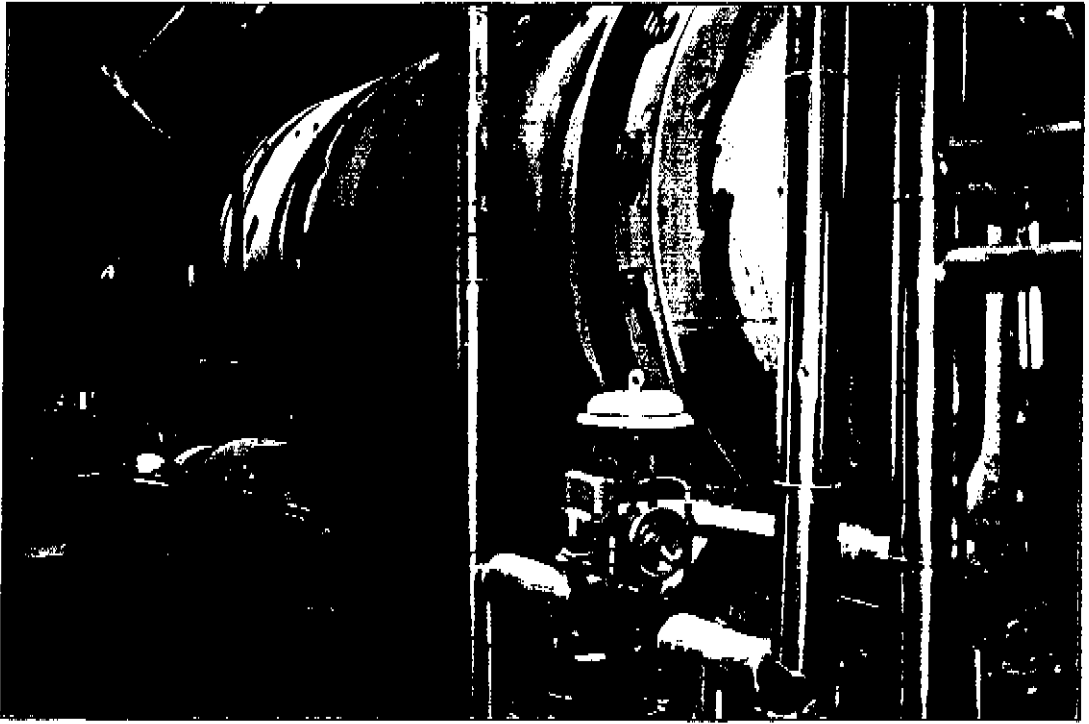


FATTENED PRILL - CROSS SECTION

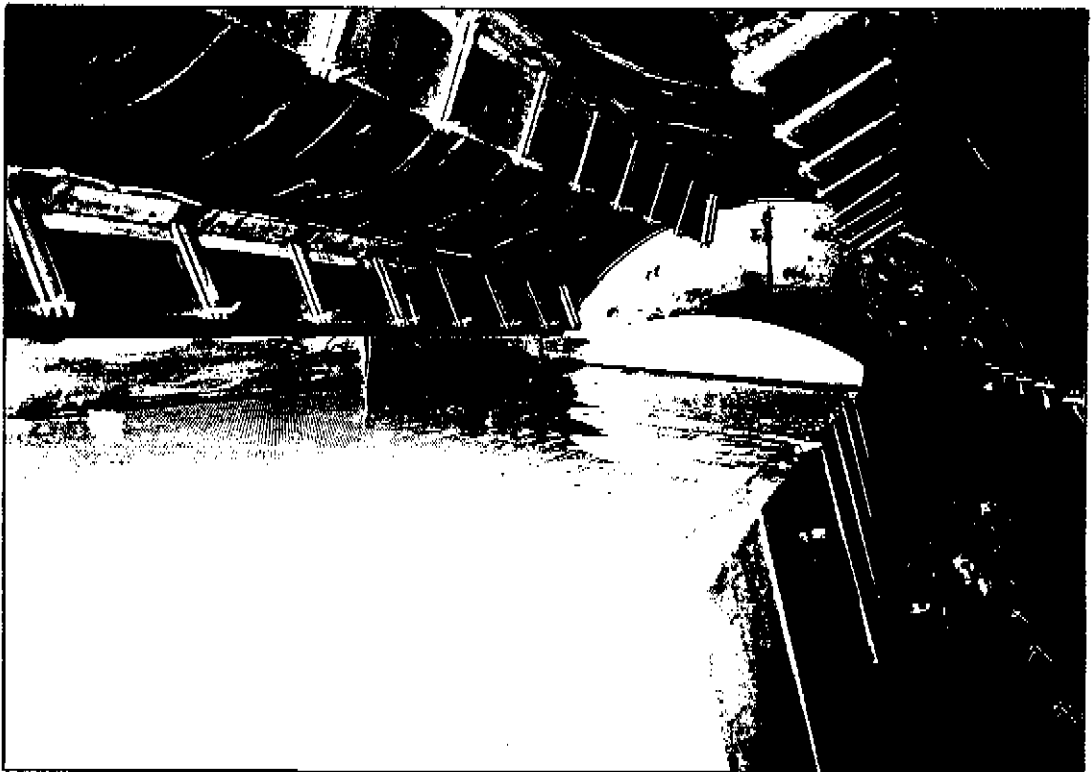
THE TWO STEPS OF THE PRILL FATTENING

1 - ORIGINAL PRILL

2 - FIRST STEP
Increase in the prilling tower3 - SECOND STEP
Fattening in F.D.G.



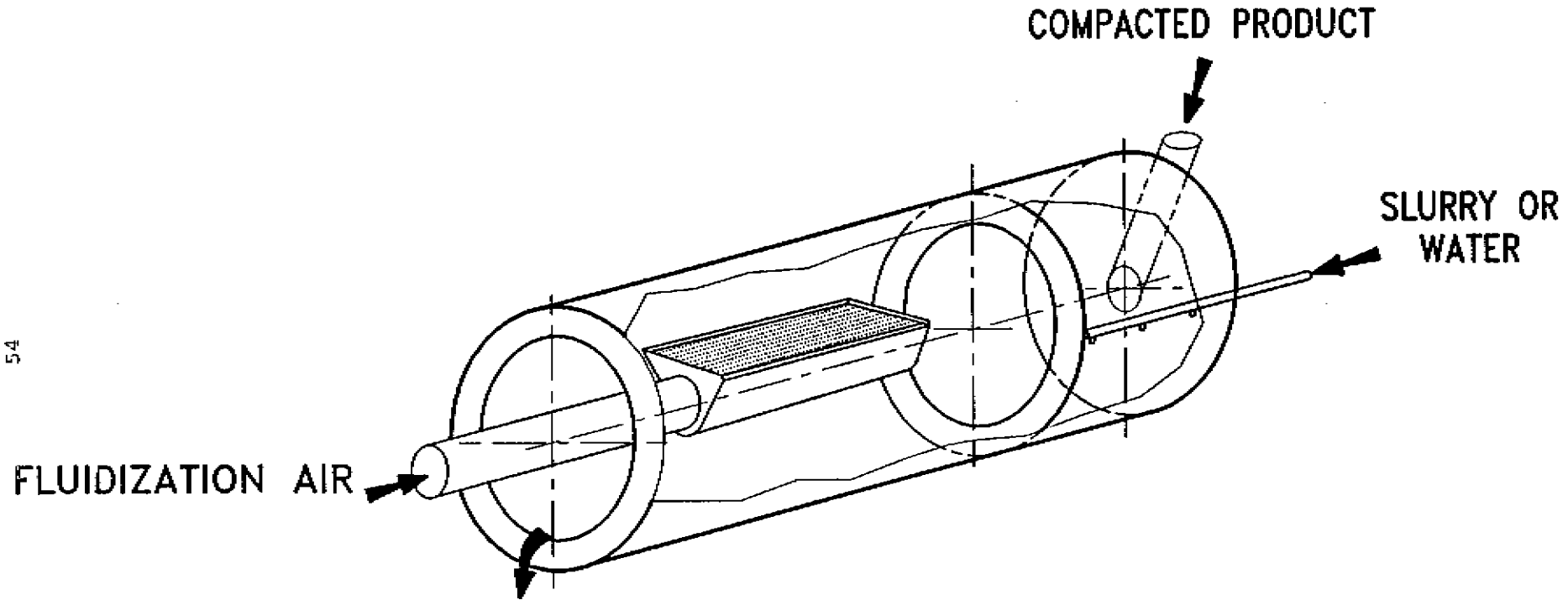
THE GRANULATING DRUM (EXTERNAL VIEW)



THE GRANULATING DRUM (INTERNAL VIEW)



KALTENBACH-THURING

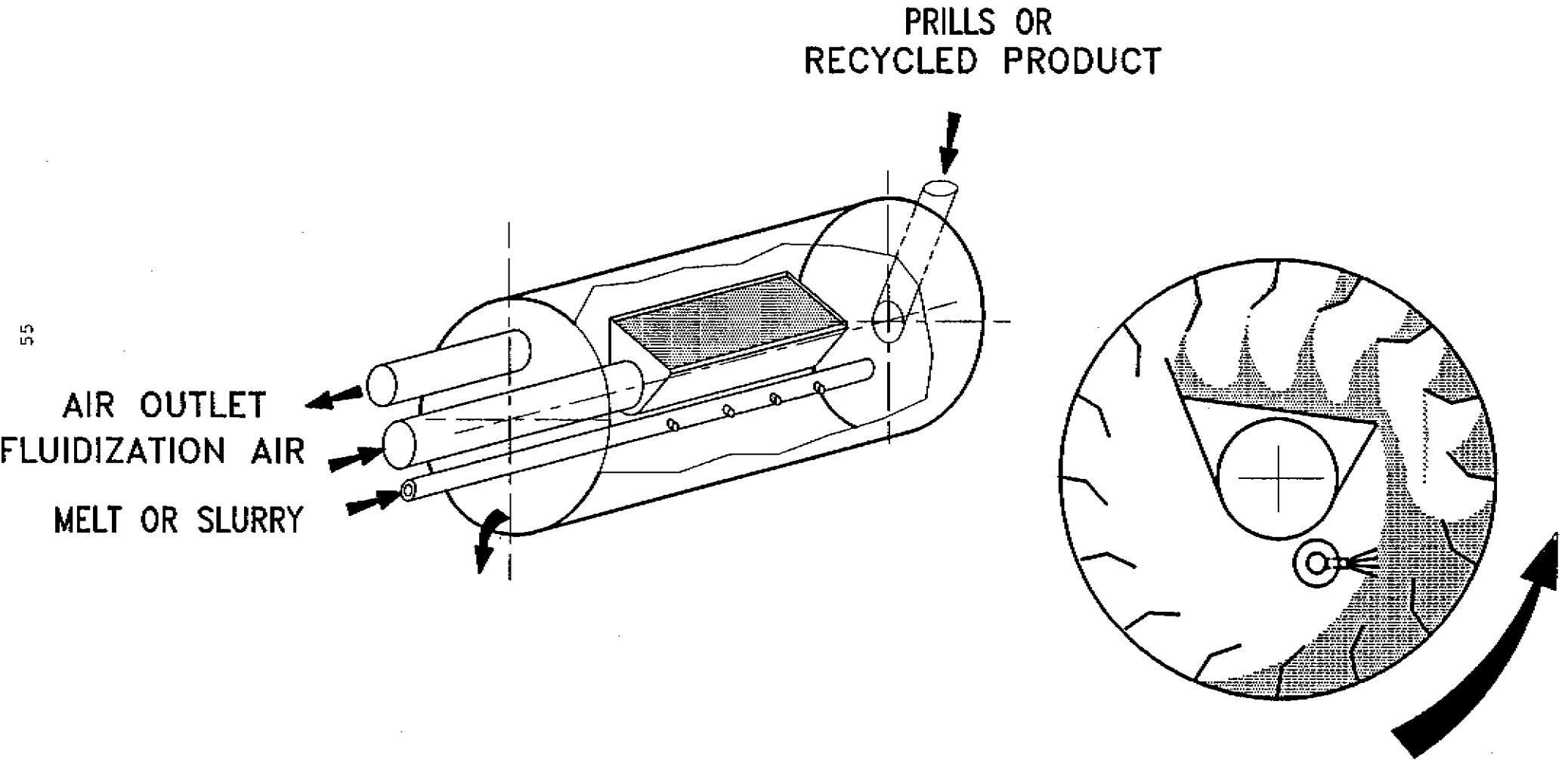


54

FLUID DRUM GRANULATOR
ROUNDING OFF - COMPACTED FERTILIZERS



KALTENBACH-THURING



FLUID DRUM GRANULATOR

REFERENCES

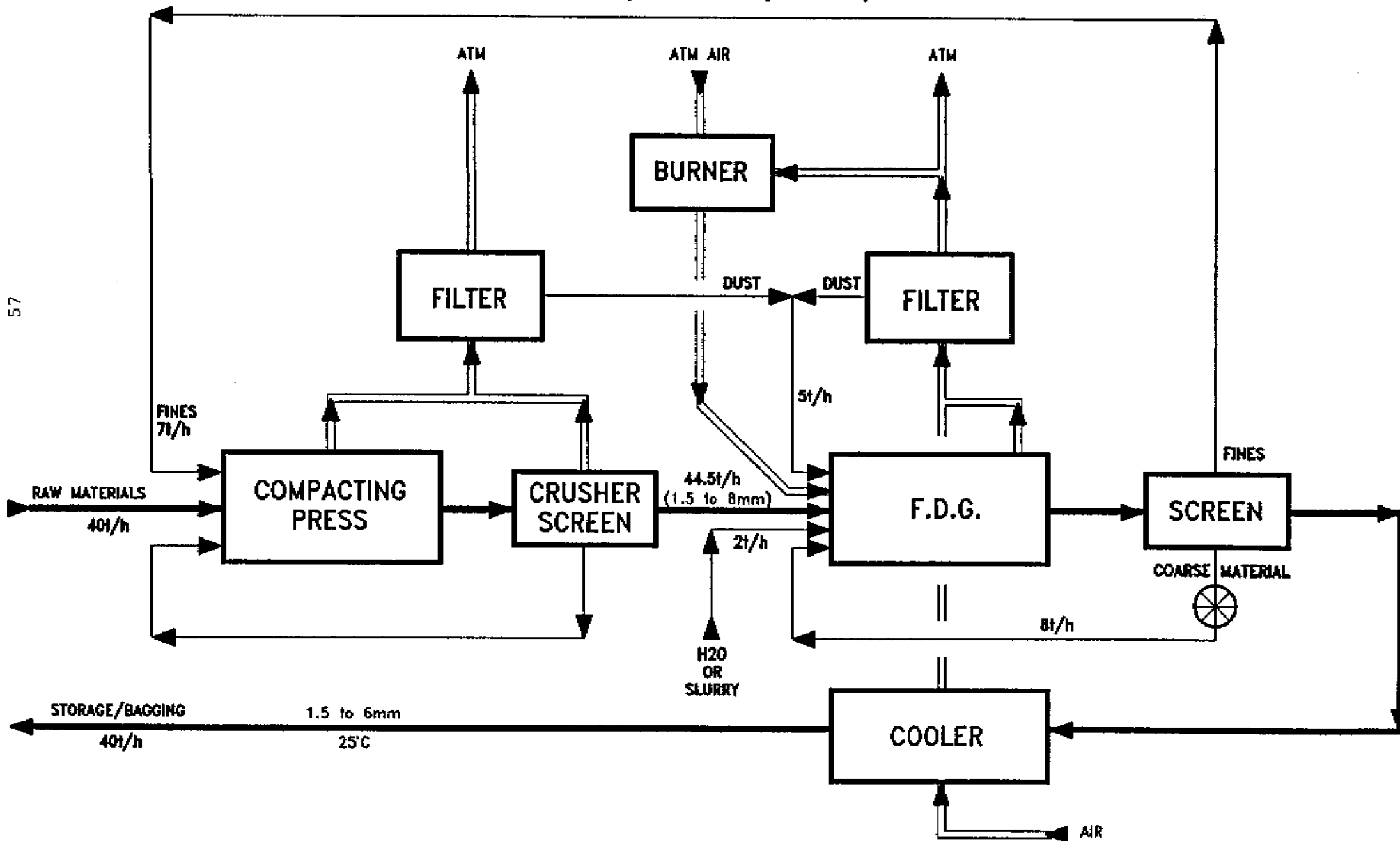
FLUIDIZED DRUM GRANULATION

FDG

NAME OF OWNER	LOCATION	TYPE OF PLANT	CAPAC. (t/d)	COMMISSION ING YEAR
SAPEC	SETUBAL (Portugal)	Compacted PK/NPK "rounding-off"	500	1986
TIMAC	SAINT-MALO (France)	Compacted PK/NPK "rounding-off"	1450	1987
CEDEST	THIONVILLE (France)	Compacted PK/NPK "rounding-off"	1000	1988
S.K.W	KARLSRUHE (Germany)	Ammonium sulphate granulation	30	1988
I.F.I.	CORK (Ireland)	Urea prills fattening	1100	1990
INCITEC (Eastern Nitrogen Ltd)	NEWCASTLE (Australia)	Ammonium nitrate granulation	250	1990
ENICHEM	TERNI (Italie)	Urea and/or calcium nitrate granulation	400	1991
ENEL	SULCIS (Italy)	Ammonium sulphate granulation	12	1991
SIMPLOT	BRANDON (Canada)	Ammonium nitrate fattening	400	1991

ROUNDING OFF AND COATING OF COMPACTED FERTILIZERS

CEDEST/DISTROF (FRANCE)



Plant owner	:	CEDEST
Location	:	Thionville (France)
Product	:	Compacted PK and NPK
Type of process	:	"Rounding off"

Specifications of the FDG inlet products

Solid

. nature	:	compacted PK (12-12-0) / NPK (13-11-15)
. particle size	:	2 % below 1 mm – 15% above 5 mm
. water content	:	0.5 to 1 %
. temperature	:	30 to 35°C
. flowrate	:	45 t/h
. dust index	:	2% to 6%

Liquid

. nature	:	water or slurry
. concentration (dry mater.):	:	0 to 50%
. temperature	:	ambient
. flowrate	:	2 t/h

Specifications of the final product

. nature	:	"rounded off" PK or NPK
. particle size	:	2% below 2 mm – 3% above 6 mm
. water content	:	0.5 %
. dust index	:	# nil
. temperature (to storage)	:	80°C
. flow	:	40 t/h

Recycle

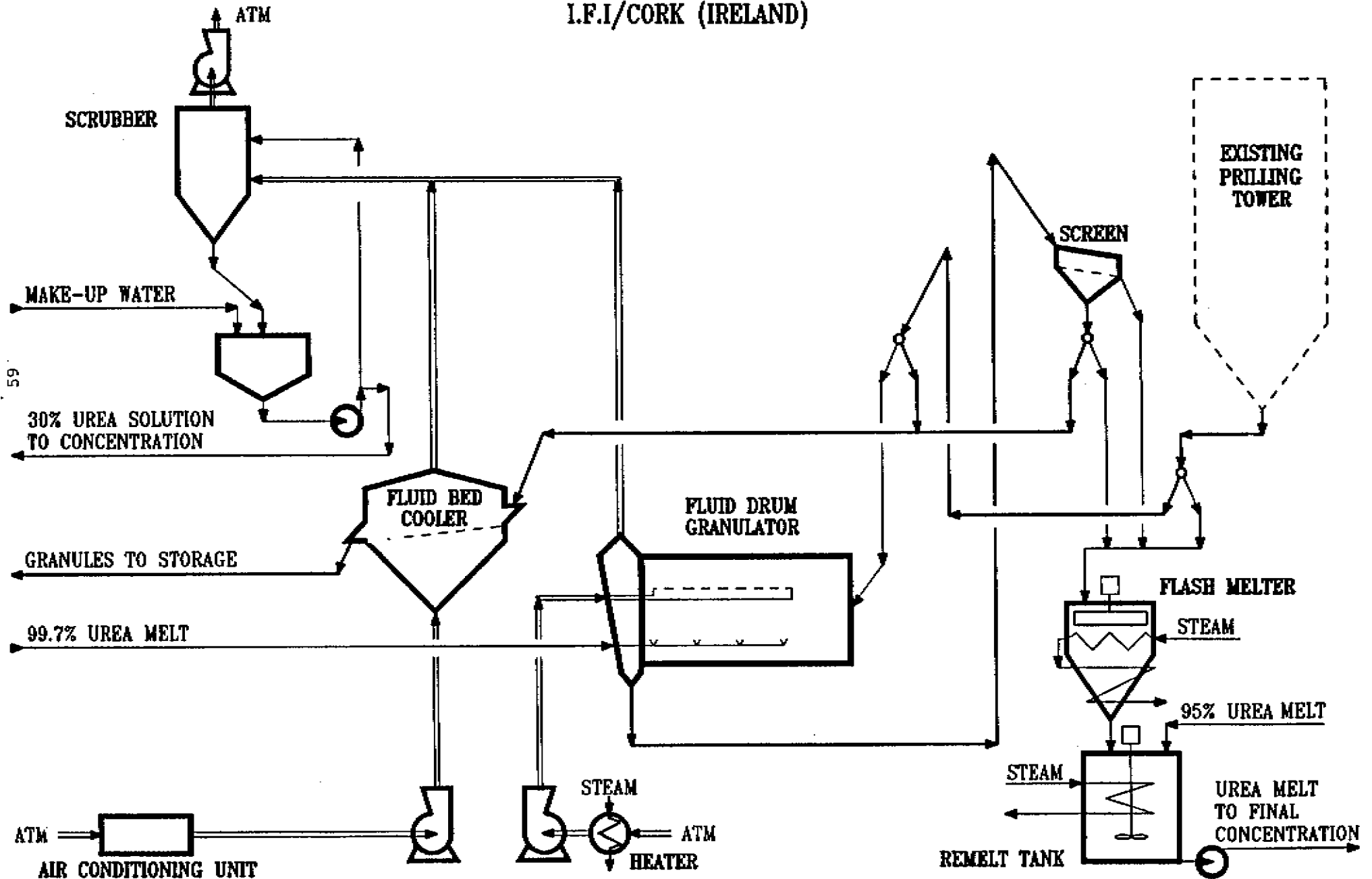
Solid

. nature	:	undersized product and crushed oversize
. ratio	:	0.125 / 1

Liquid

. nature	:	water or PK or NPK slurry
. ratio	:	0.05 / 1

UREA FATTENING I.F.I./CORK (IRELAND)



Plant owner	:	I.F.I.
Location	:	Cork (Ireland)
Product	:	Urea
Type of process	:	Fattening

Specifications of the FDG inlet products

Solid

. nature	:	urea prills
. mean particle size	:	2.1 mm
. water content	:	0.3 %
. temperature	:	75°C to 85°C
. biuret content	:	1%
. crushing strength	:	0.75 kg
. resistance to abrasion (% breakdown)	:	5 %
. flowrate	:	22 t/h

Liquid

. nature	:	urea melt
. concentration	:	99.7 %
. temperature	:	140°C
. flowrate	:	23 t/h

Specifications of the final product

. nature	:	"fattened" prills
. particle size / mean diameter	:	2.7 mm
. water content	:	0.2 %
. biuret content	:	1 %
. crushing strength	:	2 kg
. resistance to abrasion (% breakdown)	:	0.3 %
. temperature (to storage)	:	45°C to 50°C
. flow	:	45 t/h

Recycle

Solid

. nature	:	nil
. ratio	:	nil

Liquid

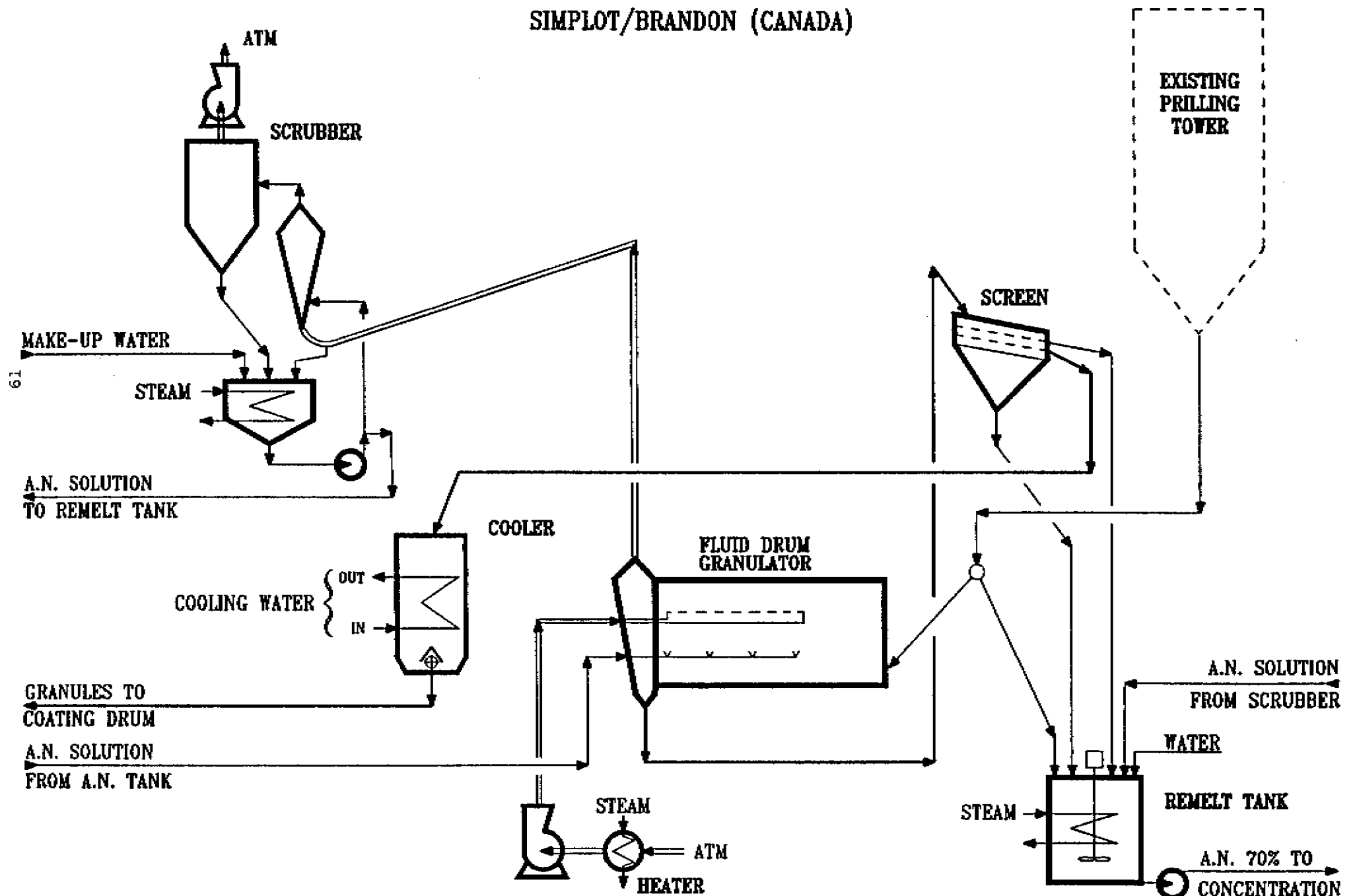
. nature	:	Urea solution
. ratio	:	0.025 / 1 to 0.050 / 1

Emissions

. Prill tower to atmosphere	:	
- before installation of FDG	:	38 kg/h
- after installation of FDG	:	18 kg/h
. Scrubber of FDG to atmosphere	:	1 kg/h

A.N. PRILL FATTENING

SIMPLOT/BRANDON (CANADA)



Plant owner	:	SIMPLOT
Location	:	Brandon (Canada)
Product	:	Ammonium Nitrate
Type of process	:	Prill fattening

Specifications of the FDG inlet products

Solid

. nature	:	AN prills
. mean particle size	:	2 mm
. water content	:	0.05 %
. crushing strength (resistance to abrasion)	:	1,6 kg 48%
. temperature	:	111°C
. flowrate	:	10 t/h

Liquid

. nature	:	AN melt
. concentration	:	99.9 %
. temperature	:	190°C
. flowrate	:	8 t/h

Specifications of the final product

. nature	:	"fattened" AN prills
. particle size / mean diameter	:	2.5 mm
. water content	:	0.05 %
. crushing strength (resistance to abrasion)	:	2.9 kg 82 %
. temperature (to storage)	:	24°C
. flow	:	16 t/h

Recycle

Solid

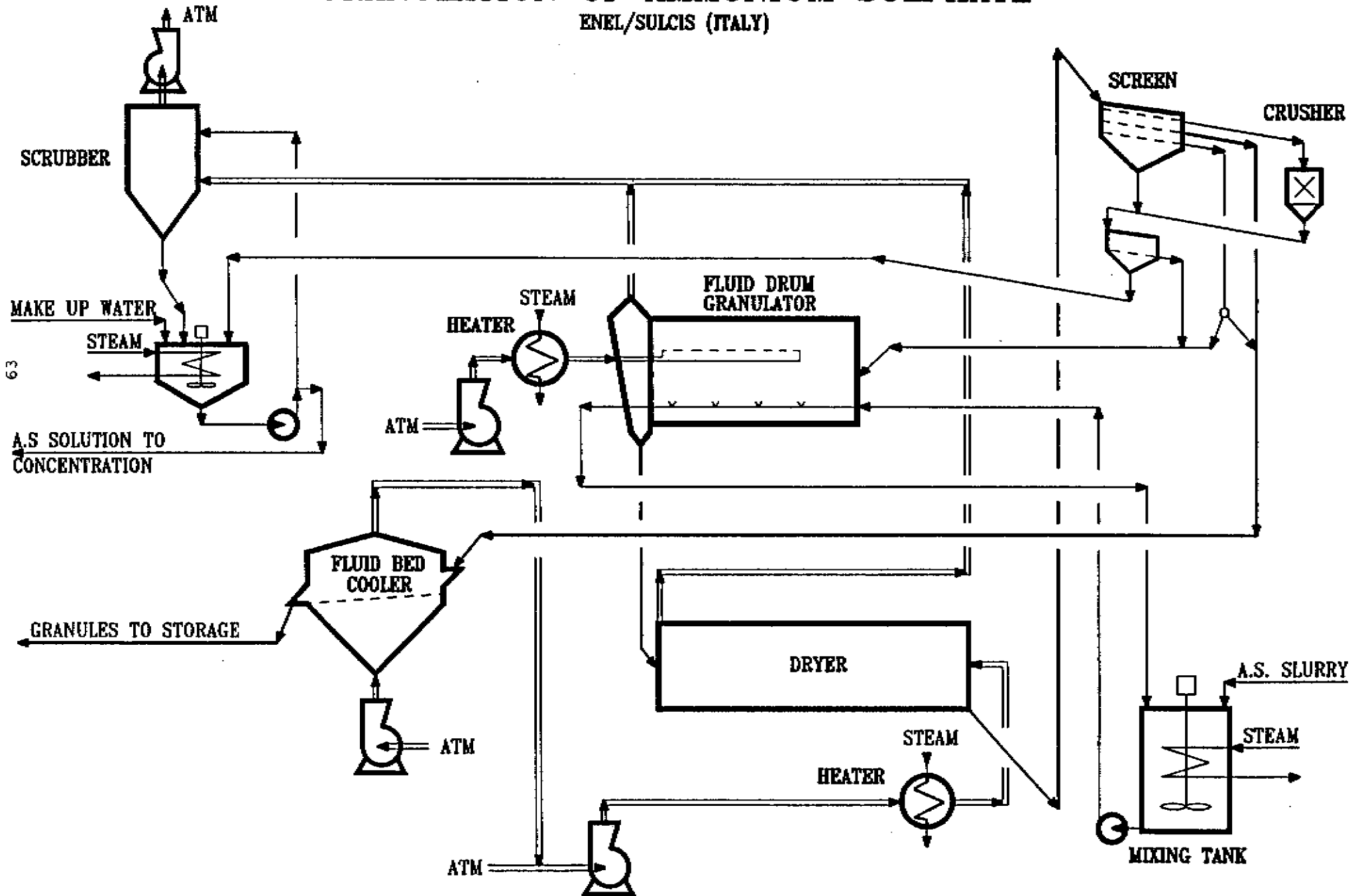
. nature	:	nil
. ratio	:	nil

Liquid

. nature	:	AN solution
. ratio	:	0.125 / 1

GRANULATION OF AMMONIUM SULPHATE

ENEL/SULCIS (ITALY)



Plant owner	:	ENEL
Location	:	Sulcis (Italy)
Product	:	Ammonium Sulphate
Type of process	:	Granulation

Specifications of the FDG inlet products

Solid

. nature	:	AS granules (recycle)
. mean particle size	:	2.1 mm
. water content	:	0.2 %
. temperature	:	55°C
. flowrate	:	700 kg/h

Liquid

. nature	:	AS slurry
. concentration	:	52 %
. temperature	:	80°C
. flowrate	:	450 kg/h

Specifications of the final product

. nature	:	AS granules
. particle size / mean diameter	:	3.1 mm
. water content	:	0.2 %
. hardness	:	3.5 kg
. temperature (to storage)	:	30°C
. flow	:	230 kg/h

Recycle

Solid

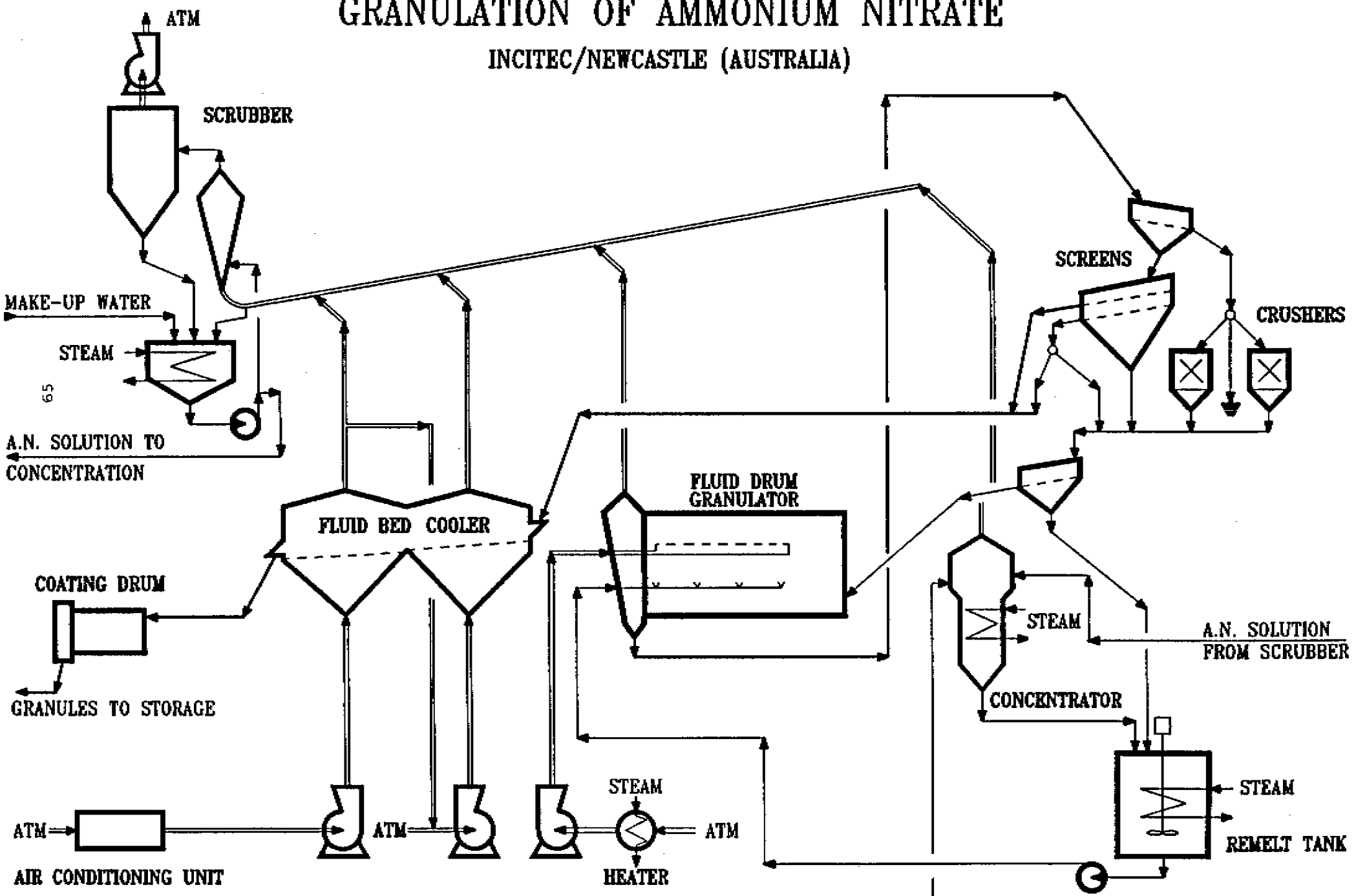
. nature	:	undersized and crushed AS
. ratio	:	3/1

Liquid

. nature	:	AS solution
. ratio	:	0.05 / 1

GRANULATION OF AMMONIUM NITRATE

INCITEC/NEWCASTLE (AUSTRALIA)



Plant owner	:	INCITEC
Location	:	Newcastle (Australia)
Product	:	Ammonium nitrate
Type of process	:	Granulation

Specifications of the FDG inlet products

Solid

. nature	:	AN granules (recycle)
. mean particle size	:	2 mm
. water content	:	0.2 %
. temperature	:	102°C
. flowrate	:	13 100 kg

Liquid

. nature	:	AN solution
. concentration	:	99.3 %
. temperature	:	173°C
. flowrate	:	12 500 kg

Specifications of the final product

. nature	:	AN granules
. particle size / mean diameter	:	98% between 2.5 mm and 3.8 mm
. water content	:	0.15 % to 0.18 %
. hardness	:	2 to 4 kg
. temperature (to storage)	:	27°C
. flow	:	10 000 kg/h

Recycle

Solid

. nature	:	undersize and crushed oversize AN
. ratio	:	1.3 / 1

Liquid

. nature	:	AN solution
. ratio	:	0.25 / 1

SPECIALIZED LIQUID COMPLEX FERTILIZERS IN LITHUANIA

A.M. Sviklas,
Azotas, Lithuania

ABSTRACT

The main standard fertilizers: urea, ammonia, liquid ammonia, ammonium nitrate, superphosphate, ammonium phosphate, are produced in large scale at two Lithuanian chemical plants - Kedainiai and Jonava.

The new research objective of the last years was seeking technology for the production of specialized liquid fertilizers and these were developed at Jonava "Azotas" state enterprise-nitrogen factory.

According with results obtained, many grades of specialized liquid fertilizers were developed at Jonava "Azotas" state enterprise-nitrogen factory.

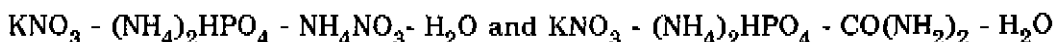
Liquid fertilizers process is based on flow type technological scheme. It involves the production of potassium nitrate, ammonium nitrate and ammonium phosphate solutions as well as the preparation of chelated microelements and mixing all components. The compounds mentioned above are produced simultaneously.

1. INTRODUCTION

Specialized complex fertilizers destined for greenhouse plants, flowers, horticulture are usually produced by dissolving and mixing salts in water.

This paper describes the NPK process using only liquid ammonium phosphate, ammonia and potassium nitrate solutions. Corresponding laboratory investigation was carried out to ensure an extraction of the compounds mentioned from by-products and intermediates of large scale nitrogen and phosphoric fertilizers production.

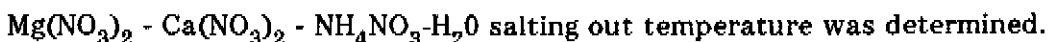
Crystallization temperature and solid phase of:



systems were determined by visual -polythermic method. Chemical composition of the solutions with salting out temperature at 0°C was defined.

Production involves processes to obtain potassium nitrate, ammonium nitrate and ammonium phosphate solutions by the neutralization of potassium carbonate with nitric acid as well as ammonia water solution by nitric acid and phosphoric acid simultaneously. By mixing all the components, 10-7-5, 7-7-5, 5-7-7 and 5-3-7 NPK liquid grades are produced.

Magnesium-nitrogen liquid composition was investigated:

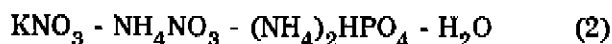


Liquid magnesium-nitrogen fertilizers technology consists of:

- 1) magnesite decomposition by nitric acid,
- 2) ammoniazation of acid magnesium nitrate solution,
- 3) liquid fertilizers standardization and packing.

2. LABORATORY SCALE EXPERIMENTS ON COMPOSITION OF LIQUID NPK

These experiments were carried on using visual-polythermic method to determine crystallization temperature of:



systems. The results are summarized below:

2.1. Polythermic crystallization data of 9 different initial compositions of (1) and 8 compositions of (2) systems were obtained. The graphic view is presented in Figure 1.

2.2. Compositions of liquid and solid phase in critical points of each curve were determined. These results are given in Table 1.

Table I - Chemical composition of critical points of systems (I and II)

N° of System	Section of System	Composition of liquid phase (w)				Crystallization temperature, °C	Composition of solid phase
		KNO ₃	CO(NH ₂) ₂ or NH ₄ NO ₃	(NH ₄) ₂ HPO ₄	H ₂ O		
1	I	8,76	30,00	8,76	32,48	- 6,0	KNO ₃ ↓ CO(NH ₂) ₂
	II	7,36	26,50	11,04	55,10	- 12,0	KNO ₃ ↓ CO(NH ₂) ₂
	III	9,86	34,30	6,57	45,07	- 0,2	"
	IV	12,03	38,50	9,23	40,24	16,6	"
	V	7,80	4,61	18,44	69,15	- 9,8	ice ↓ KNO ₃
	V	18,00	4,10	16,40	61,50	21,0	KNO ₃ ↓ CO(NH ₂) ₂
	VI	8,00	9,20	13,00	69,00	- 12,6	ice ↓ KNO ₃
	VI	13,00	8,70	13,05	65,25	3,5	KNO ₃ ↓ CO(NH ₂) ₂
	VII	7,70	4,61	23,07	64,62	- 10,8	ice ↓ KNO ₃
	VII	13,90	4,30	21,32	60,27	11,2	KNO ₃ ↓ CO(NH ₂) ₂
2	VIII	8,70	4,56	13,69	73,05	- 8,5	ice ↓ KNO ₃
	VIII	17,40	4,13	12,39	66,08	14,6	KNO ₃ ↓ CO(NH ₂) ₂
	IX	6,50	4,67	28,05	60,77	- 12,9	ice ↓ KNO ₃
	X	15,00	4,25	25,50	55,25	23,4	KNO ₃ ↓ CO(NH ₂) ₂
	I	8,9	23,8	8,9	53,4	- 4,8	KNO ₃ ↓ NH ₄ NO ₃
	II	7,7	62,30	11,6	57,7	- 8,3	"
	III	11,9	20,8	7,9	59,4	2,2	"
	IV	16,0	20,0	12,0	52,0	24,4	"
	V	6,8	4,7	13,6	69,9	- 10,5	ice ↓ KNO ₃
	V	15,0	4,3	17,0	63,7	14,0	KNO ₃ ↓ NH ₄ NO ₃
VI	6,4	9,4	14,0	70,2	- 12,7	ice ↓ KNO ₃	
VI	13,5	8,7	13,0	64,8	8,4	KNO ₃ ↓ NH ₄ NO ₃	
VII	5,0	4,7	23,8	66,5	- 10,5	ice ↓ KNO ₃	
VIII	15,0	4,3	21,3	59,4	20,6	KNO ₃ ↓ NH ₄ NO ₃	
IX	7,7	4,6	13,9	73,8	- 10,0	ice ↓ KNO ₃	
X	17,5	4,1	12,4	66,0	18,5	KNO ₃ ↓ NH ₄ NO ₃	

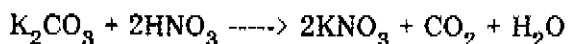
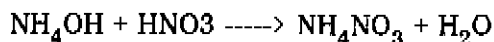
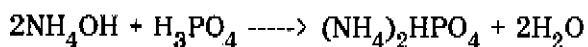
2.3. Composition of solutions in crystallization point at 0°C, obtained from polythermic investigations by extrapolation, is presented in Table 2. It was suggested to be used as liquid complex fertilizers. The manufacturing of 10-7-5, 7-7-5, 5-7-7 and 5-3-7 grade were chosen according to agrochemical requirements.

Table 2 - Composition of solution with crystallization temperature 0°C

N° solution	Composition of solution (w)				Composition of nutrients (w)				Ratio of N:P ₂ O ₅ :K ₂ O
	KNO ₃ (NH ₄) ₂	HPO ₄	CO(NH ₄) ₂	NH ₄ NO ₃	H ₂ O	N	P ₂ O ₅	K ₂ O	
1	8,4	8,4	33,0	-	50,2	18,3	4,5	3,9	4:1:0,9
2	8,7	10,1	32,6	-	50,6	18,3	5,4	3,1	3,4:1:0,6
3	10,1	6,7	32,8	-	50,4	18,2	3,6	4,7	5:1:1,3
4	9,9	6,6	34,2	-	49,3	18,7	3,5	4,5	5,3:1:1,3
5	11,2	17,8	4,4	-	66,6	7,4	9,6	5,0	0,8:1:0,5
6	12,0	13,2	8,8	-	66,6	8,5	7,2	5,4	1,2:1:0,7
7	10,6	22,3	4,5	-	62,6	8,3	12,0	4,8	0,7:1:0,4
8	11,8	13,2	4,4	-	70,6	6,5	7,0	5,6	0,9:1:0,8
9	8,7	8,7	-	30,3	52,3	13,7	4,7	4,1	2,9:1:0,9
10	7,4	11,2	-	25,7	55,7	12,4	3,5	6,0	3,5:1:1,7
11	10,3	17,9	-	4,5	67,3	6,8	9,7	4,8	0,7:1:0,5
12	10,6	13,4	-	8,9	67,1	7,4	7,2	4,9	1:1:0,7
13	8,3	22,9	-	4,6	64,2	7,2	12,3	3,9	0,6:1:0,3
14	11,2	13,3	-	4,4	71,1	6,0	7,2	5,2	0,6:1:0,3

3. CHEMISTRY AND FLOW SHEET OF LIQUID NPK PROCESS

Liquid complex fertilizers process is represented by equations as follows:



Referring to the liquid NPK process (Figure 2), ammonia water solution as by-product from tank (4) is reacted simultaneously with nitric acid from tank (1) in reactor (3) as well as with phosphoric acid from tank (5) in reactor (6). Potassium carbonate is reacted with nitric acid in reactor (2). The products obtained are diluted by water in appropriate reactors at 60-80°C. Ammonia nitrate, ammonia phosphate and potassium nitrate solutions are mixed in reactor (8), which is supplied with chelated microelements. These are prepared in reactor (7). The final product is pumped by (9) to standardization (10) and packing (11).

Physical and chemical properties of liquid NPK are presented in Table 3.

Table 3 - Physical and chemical properties of liquid complex fertilizers

Grade of liquid complex fertilizers	Temperature (°C)	Viscosity (sst)	Electro-conductivity (sm)
5-7-5	20	2,50	4,8
	40	1,91	4,2
	60	1,72	3,8
5-7-7	20	1,63	5,0
	40	1,35	4,5
	60	1,26	4,1
10-7-5	20	1,84	5,5
	40	1,73	4,9
	60	1,54	4,1
5-3-7	20	2,10	5,1
	40	1,96	4,6
	60	1,85	4,1

Liquid NPK contains six microelements: Cu, Zn, B, Mn, Co, Mo usually in chelated form free of chlorine, heavy metals and other toxic substances. Agrochemical research illustrates that liquid NPK obtained decreases nitrate amount in vegetables and fruits as well as increases amount of sugar and lemon acid. The equipment desired was used from the other plant necessary for restructuring. The capacity of plant complex is 10 000 tons per year.

4. LABORATORY SCALE INVESTIGATION OF LIQUID MAGNESIUM-NITROGEN FERTILIZER

These experiments were carried out on by visual-polythermic method too in order to determine crystallization temperature of $Mg(NO_3)_2 - Ca(NO_3)_2 - NH_4NO_3 - H_2O$ system. The results are as follows:

4.1. Polythermic crystallization data of 5 different initial compositions are presented in Figure 3. Each curve consists of two parts corresponding to crystallization of $Mg(NO_3)_2 \cdot 6H_2O$ and NH_4NO_3 .

4.2. Composition of solutions corresponding to crystallization temperature at 0°C obtained by extrapolation is presented in Table 4.

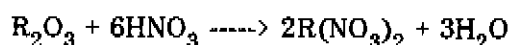
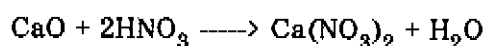
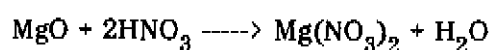
Table 4 - Composition of magnesium-nitrogen solutions

Composition of solutions %		Nutrient concentration %					Solid phase
Mg(NO ₃) ₂	Ca(NO ₃) ₂	NH ₄ NO ₃	H ₂ O	MgO	CaO	N	
33,20	3,00	11,50	52,20	8,97	1,06	10,83	MgO(NO ₃) ₂ ·6H ₂ O
28,63	2,67	23,66	45,04	7,78	0,91	14,16	NH ₄ NO ₃
28,67	1,91	23,66	45,80	7,78	0,65	14,01	NH ₄ NO ₃
32,50	1,67	16,67	49,16	8,83	0,57	12,26	MgO(NO ₃) ₂ ·6H ₂ O
30,47	1,56	21,88	46,09	8,28	0,53	13,68	NH ₄ NO ₃
31,82	2,07	17,36	48,75	8,65	0,70	12,26	MgO(NO ₃) ₂ ·6H ₂ O
29,39	1,91	23,66	45,04	7,98	0,66	14,16	NH ₄ NO ₃

It shows the possibility of production of 14-7 and 10-9 N:MgO grade liquid magnesium-nitrogen fertilizers.

5. CHEMISTRY AND FLOW SHEET OF THE LIQUID MAGNESIUM-NITROGEN FERTILIZERS

The chemistry of the liquid magnesium-nitrogen fertilizers technology is presented by following chemical equations:



The magnesium-nitrogen liquid fertilizers process is described below by reference to Figure 4.

Magnesite (storage 2) is flowed by pneumotransport to reactor (4) fed by nitric acid (5) and condensate from set. The mixing continues about 30 min. at 85°C. Magnesium nitrate solution is pumped (11) to settler (6) and depositives are sent to (12). A clear solution is pumped for neutralisation (7) with ammonia water solution while pH value 4,5 is reached. Liquid magnesium-nitrogen fertilizer manufactured is sent to storage tank (9) and into cartank or bottles.

This process is mastered in pilot plant scale.

CONCLUSIONS

1. The chemical compositions of specialized liquid complex and magnesium-nitrogen fertilizers are chosen as result of visual-polythermic investigations of multicomponent systems.
2. Flow sheet of liquid complex fertilizers is proposed and pilot plant process is mastered.
3. Chemical and physical properties of liquid fertilizers are determined.

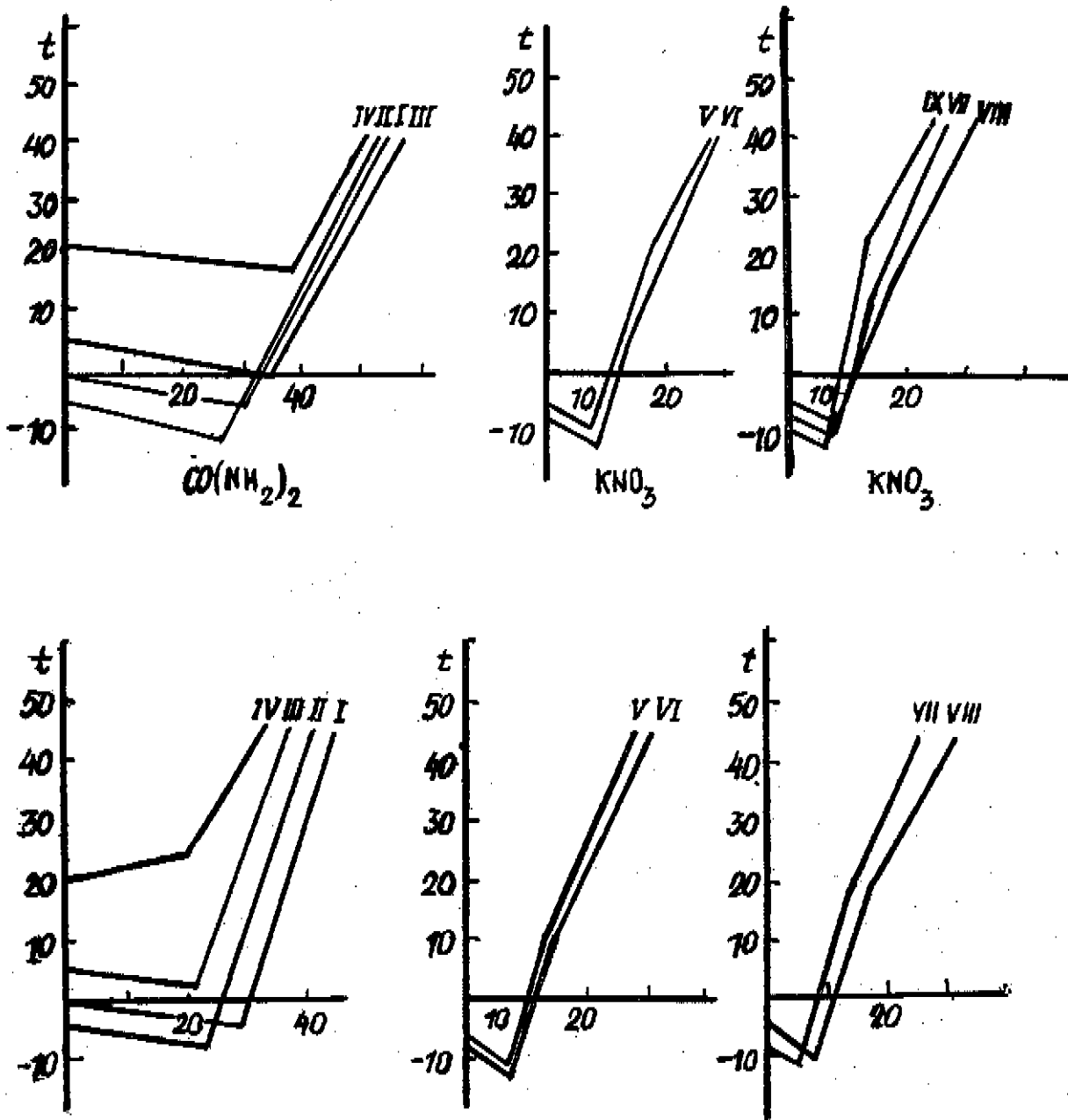


FIGURE 1

Polythermic curves of systems 1(a) and 2(b)

t - temperature ($^{\circ}\text{C}$)

Section (% w): a) I - $[12.5 \text{KNO}_3 + 12.5(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{CO}(\text{NH}_2)_2$,
 II - $[10 \text{KNO}_3 + 15(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{CO}(\text{NH}_2)_2$, III - $[15 \text{KNO}_3 + 10(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{CO}(\text{NH}_2)_2$,
 IV - $[20 \text{KNO}_3 + 15(\text{NH}_4)_2\text{HPO}_4 + 65 \text{H}_2\text{O}] \rightarrow \text{CO}(\text{NH}_2)_2$,
 V - $[20(\text{NH}_4)_2\text{HPO}_4 + 5 \text{CO}(\text{NH}_2)_2 + 75 \text{H}_2\text{O}] \rightarrow \text{KNO}_3$, VI - $[15(\text{NH}_4)_2\text{HPO}_4 + 5 \text{CO}(\text{NH}_2)_2 + 80 \text{H}_2\text{O}] \rightarrow \text{KNO}_3$,
 b) I - $[12.5 \text{KNO}_3 + 12.5(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{NH}_4\text{NO}_3$, II - $[10 \text{KNO}_3 + 15(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{NH}_4\text{NO}_3$,
 III - $[15 \text{KNO}_3 + 10(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{NH}_4\text{NO}_3$,
 IV - $[20 \text{KNO}_3 + 10(\text{NH}_4)_2\text{HPO}_4 + 75 \text{H}_2\text{O}] \rightarrow \text{NH}_4\text{NO}_3$, V - $[20(\text{NH}_4)_2\text{HPO}_4 + 5 \text{NH}_4\text{NO}_3 + 75 \text{H}_2\text{O}] \rightarrow \text{KNO}_3$,
 VI - $[15(\text{NH}_4)_2\text{HPO}_4 + 10 \text{NH}_4\text{NO}_3 + 75 \text{H}_2\text{O}] \rightarrow \text{KNO}_3$,
 VII - $[25(\text{NH}_4)_2\text{HPO}_4 + 5 \text{NH}_4\text{NO}_3 + 70 \text{H}_2\text{O}] \rightarrow \text{KNO}_3$, VIII - $[15(\text{NH}_4)_2\text{HPO}_4 + \text{NH}_4\text{NO}_3 + 80 \text{H}_2\text{O}] \rightarrow \text{KNO}_3$.

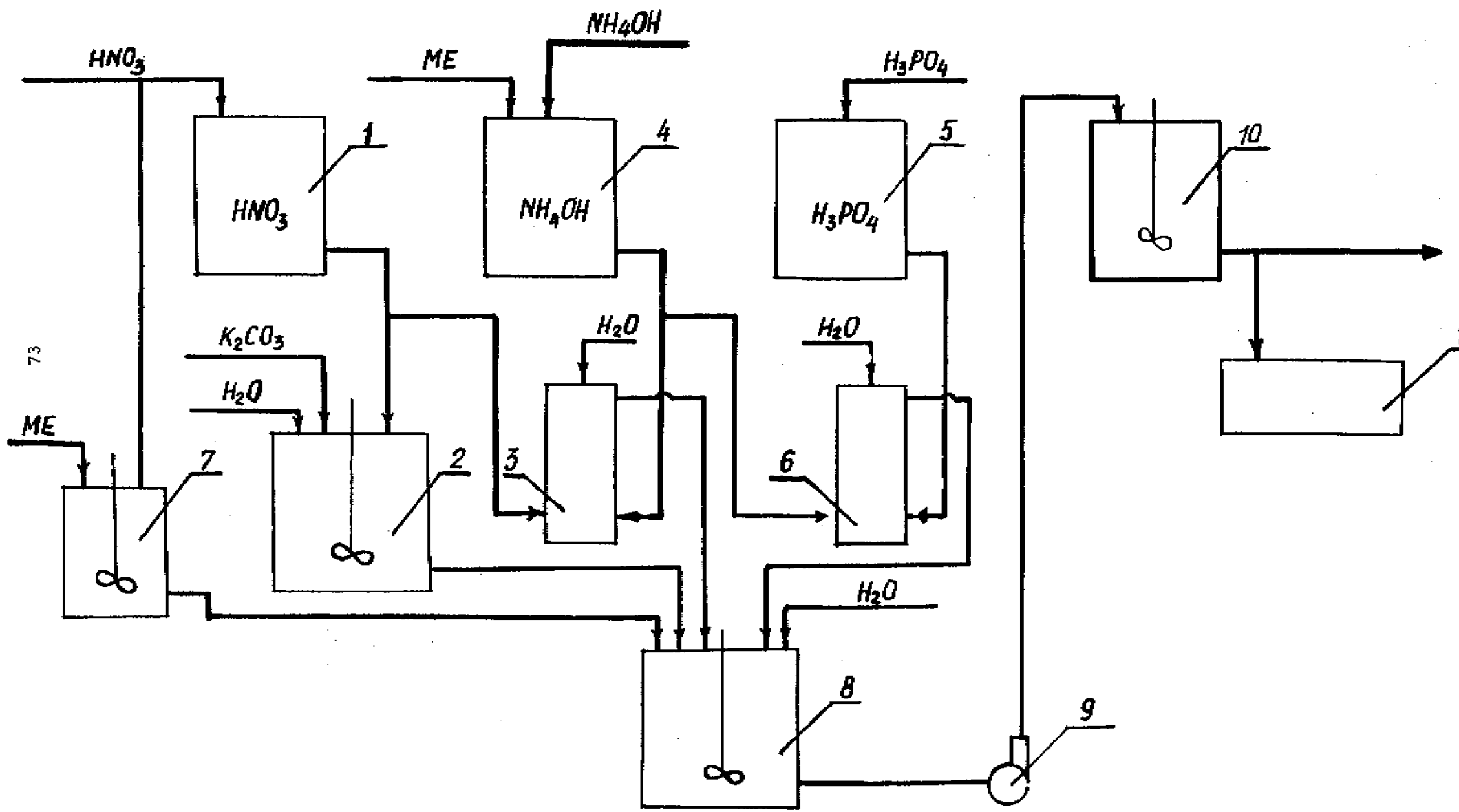


FIGURE 2 - FLOW SHEET OF LIQUID COMPLEX FERTILIZERS PRODUCTION

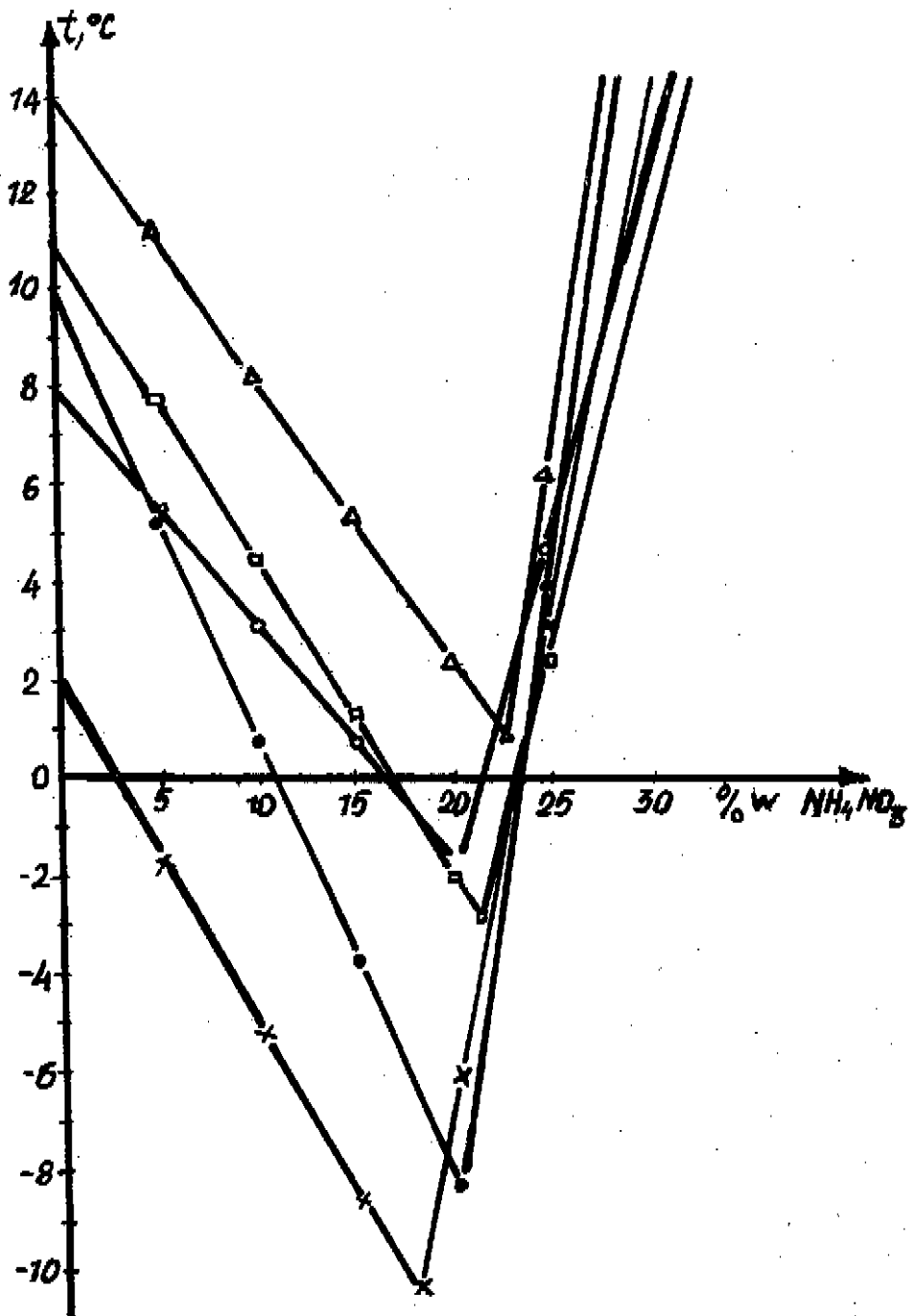


FIGURE 3

Polythermic curves of system
 $Mg(NO_3)_2 - Ca(NO_3)_2 - NH_4NO_3 - H_2O$

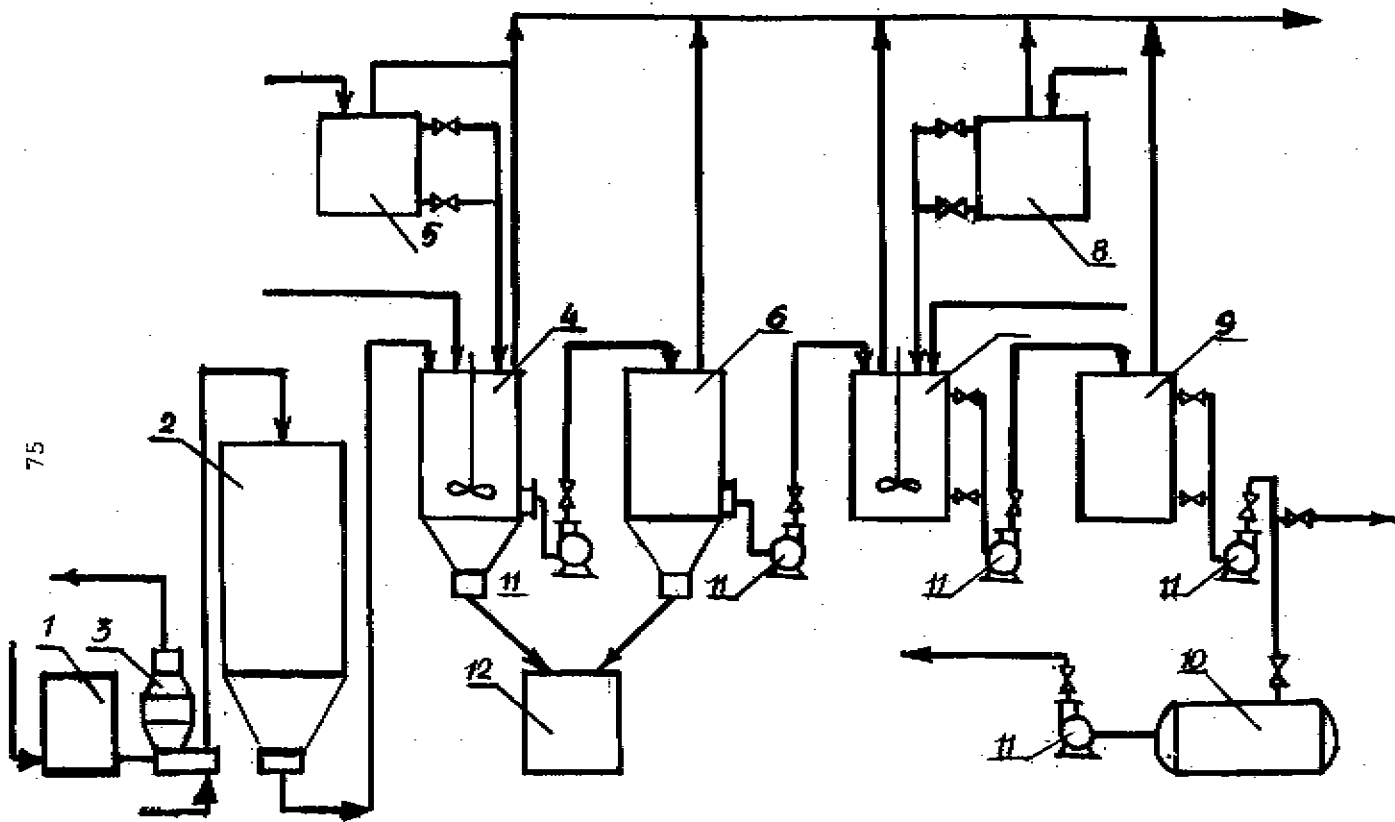


FIGURE 4 - FLOW SHEET OF LIQUID MAGNESIUM - NITROGEN FERTILIZERS