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NPK PRODUCTION BY ION EXCHANGE  
OPERATING EXPERIENCE

By

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Chlorine-free NPK fertilizers are generally considered more expensive than chlorine-containing NPK fertilizers. There are technical reasons for this, the primary reason being that potassium chloride is the cheapest potassium source for NPK production. So, if you want to make chlorine-free NPK fertilizers you have to use a more expensive, chlorine-free potassium raw material. Or you had to until now.

On the 15th of December, 1971, the first industrial scale (400 tons per day) chlorine-free NPK production based on ion exchange was started in a plant built by Superfos in Fredericia, Denmark. The importance of this is that it invalidates the rule that chlorine-free NPK must always be more expensive than chlorine-containing NPK for technical reasons.

## THE PROCESS

The process is described in a previous paper, LTE/70/13, presented at the ISMA Technical Conference in Sandefjord, Norway, September 1970. The present paper deals with the experience gained during start-up and operation of the first industrial scale plant, but first we may recapitulate the basic features of the process as follows:

Phosphate rock is reacted with nitric acid, giving a slurry containing calcium, phosphorus and nitrogen, the calcium being present in the form of cations and phosphorus and nitrogen in the form of anions. This slurry is brought into contact with a potassium-loaded cation exchanger, whereby calcium and potassium change places. This means that calcium ions are removed from the nitro-phosphate slurry and potassium ions are brought into the slurry. The ion exchanger is regenerated with a potassium chloride solution, and the effluent from the regeneration is a calcium chloride solution.

Calcium originating from phosphate rock may be removed from the phosphate in connection with the NPK production by a number of different methods, the most commonly used being the removal in the form of gypsum by the

phosphoric acid production and the removal in the form of calcium nitrate by refrigerating a nitrophosphate solution (nitric acid + phosphate rock). The removal of calcium by ion exchange (K versus Ca) is another possibility. However, the fact that the Ca ions are replaced by K ions in the ion exchange process means that the resulting NPK fertilizer becomes chlorine-free. In the ion exchange process the Ca is removed in the form of  $\text{CaCl}_2$  instead of  $\text{CaSO}_4$  or  $\text{Ca}(\text{NO}_3)_2$ . Therefore, if the ion exchange process can remove Ca (in the form of  $\text{CaCl}_2$ ) more cheaply than the traditional ways of removing Ca (in the form of  $\text{CaSO}_4$  or  $\text{Ca}(\text{NO}_3)_2$ ), then the ion exchange makes possible the production of chlorine-free NPK fertilizers at lower costs than those of the traditional, chlorine-containing NPK fertilizers.

The simultaneous removal of the calcium and chlorine is the basic idea of the K-Ca-ion exchange.

#### THE PLANT

The construction of the ion exchange plant is based on experience obtained in a pilot plant.

The plant is erected next to an existing NPK plant, which operates according to the PEC phosphonitric process with spherodizers. The raw materials preparation (nitrophosphate slurry and potassium chloride solution) for the ion exchange takes place in the existing NPK building and the ion exchange equipment is installed in a new building (14 x 28 m) adjacent to the NPK building. Furthermore, a spray-concentrator has been erected for concentrating the product solution from the ion exchange plant from 60-65% water to about 30% water, before it goes into the NPK reactors and spherodizers for final NPK production. The production capacity of the spherodizers is about double that of the ion exchange unit, and therefore two 750 m<sup>3</sup> storage tanks for intermediate storage of the concentrated solution have been erected.

The general principle of the ion exchange method and apparatus appears from the diagram, Figure 1. The ion exchange resin inside the loop remains stationary, whereas the inlet and outlet places for the liquids are moved stepwise forward in the loop, thereby simulating a corresponding countercurrent movement of the resin. This system requires a number of valves, which are operated automatically according to a chosen programme.

The advantage of this patented ion exchange method is that it makes possible the construction of a rugged and reliable apparatus that permits the processing of large quantities of low cost chemicals with a reasonably low dilution of the product solution and a reasonably low consumption of chemicals for regeneration. In our opinion this is not possible with the hitherto known ion exchange arrangements, neither with the traditional fixed beds nor with the modern moving bed techniques.

#### THE START-UP

The plant was ready for start-up at the beginning of October, 1971. During the first two months of operation the plant was only operated with recycling water. The reason for this was that we wanted to study the mechanical reliability and make the operating crew familiar with the running of the plant before introducing the nitrophosphate and potassium chloride solutions.

The plant is equipped with more than 100 automatically operated valves of different makes, butterfly as well as ball valves. To obtain acceptable running conditions in the system it is a necessity that these valves operate with a very low frequency of malfunction. During the initial two months period some of the valves were replaced due to insufficient reliability. Apart from that, no essential changes were made during this period.

After several days of continuous running without any faults in the functioning of the valves and of the plant as a whole, the nitrophosphate and potassium chloride solutions were introduced into the system on the 15th of December. The experience gained in some of the essential aspects during the operation of the plant is described in the following.

#### OPERATING EXPERIENCE

1. Reliability. From the beginning of the development of this method great importance has been attached to the elaboration of a rugged and reliable apparatus. This refers not only to the mechanical reliability but also to the possibility of keeping steady process conditions. The industrial scale plant has fully met this requirement.
2. Dilution of Process Solution. It was expected that the scale-up would give rise to relatively more

backmixing and thereby increased dilution of the nitrophosphate solution. Therefore, a correspondingly longer section for displacement of water by product solution was foreseen in the loop of the big plant. The result has been very satisfactory, and it has been possible from the beginning of the plant operation to get a product solution containing between 60 and 65% water, i.e. fully abreast of the results from the pilot plant.

3. Raw Materials Losses. Two sorts of losses occur: The rinsing losses and the regeneration losses.

The rinsing loss is a consequence of less than 100% displacement of nitrophosphate solution by water in the rinsing section of the loop. The result is the appearance of small amounts of nitrate and phosphate in the calcium chloride solution. At the beginning of the operation of the big plant surprisingly high rinsing losses were found, varying between 3 and 20% of the incoming nitrate and phosphate quantities. The rinsing losses varied along the loop, i.e. when the outlet place for the calcium chloride solution was moved around the loop the analyses of the calcium chloride solution showed a systematic variation of N- and P-content in accordance with this. It was established that the reason for the high rinsing losses was faulty design of the inlet arrangements for nitrophosphate solution and rinsing water. In January this was changed, and from then on the expectedly low and constant rinsing losses were obtained, i.e. less than 2% of the N- and P-content of the feed solution goes into the calcium chloride effluent and more than 98% goes into the product solution.

The regeneration loss comes from the fact that the ion exchange processes do not go to full completion. The result is that potassium ions occur to some extent together with the calcium ions in the calcium chloride solution. At the beginning of the plant operation it was found that about 12% of the incoming potassium was lost in the calcium chloride solution, whereas about 6% loss was expected based on the pilot plant results. It appeared to be more difficult to reduce the regeneration loss than to reduce the rinsing losses. Some improvements have been obtained, but so far we have not been able to run the plant at potassium losses significantly below 9%. However, we expect to be able to obtain further reduction of the potassium loss in the future work.

4. Precipitation. Even if the inlet solutions are quite clear some precipitation in the ion exchange bed is unavoidable. The nitrophosphate solution contains silico fluoride ions, and when it meets the potassium ions from the ion exchanger some precipitation of  $K_2SiF_6$  will occur. The change in acidity of the solution when hydrogen ions together with calcium ions replace potassium on the ion exchange resin may result in further precipitation (e.g. rare earth phosphates). Dilution of the process solution by rinsing with water may under certain conditions result in hydrolysis of calcium phosphates. Therefore, we do not attach much importance to having clear inlet solutions. The problem is to obtain and to keep such running conditions that the precipitation does not impede the liquid flow inside the resin bed. This was possible in the pilot plant, and it appeared to be possible also in the big plant.

The inevitable small amounts of solids precipitated in the resin bed are removed during operation by incorporating a backwash procedure in the loop. Thereby the resin bed is cleaned regularly once per full cycle of the loop. In addition to this some cleaning is necessary on the strainers that keep back the ion exchange resin in the loop at the outlet places for the liquids. The need for cleaning at these points has turned out to be pretty varied. At some points several months of operation without cleaning has been possible, whereas at other points cleaning has been necessary after only a few weeks. More experience is being gained for the time being on the frequency and the best method of handling this cleaning. Today we are of the opinion that a sufficient procedure will be a regular stoppage for inspection and cleaning once a week, say 6-12 hours.

5. Resin Stability. During the first half year of operation the physical and chemical quality of the ion exchange resin has been very carefully checked. No alteration in the physical appearance has been observed. During the first months some reduction was observed in the total capacity measured in the laboratory, probably due to blocking of some tiny pores in the resin beads. This reduction did not go on and no reduction whatsoever has been observed in the useful capacity of the resin in the loop.

Based on the first half year operation we think that the figures we have given previously for

estimated resin consumption - e.g. 0,16\$ per ton 18-18-18 - are very much on the safe side. But of course more experience is needed to verify that.

6. Control System. Conductivity recording of the liquids leaving the loop and pressure recording of the liquid inlets to the loop are the most useful tools for supervising the system and have proved to give very good and reliable indication of any alteration in the running conditions. Flow of incoming liquids is controlled by means of magnetic flowmeters in connection with regulating valves.

The inlet and outlet valves in the loop are operated pneumatically (on-off) from a transistorized programming equipment. All the valves in the loop are equipped with position indicators and alarm is given in case of conflict between order and position.

In addition to this the system is equipped with the usual alarms relating to flow, pressure etc.

To improve the possibilities of chemically scrutinizing the system we have installed a Technicon Autoanalyzer, but this is only used periodically. The regular chemical analyses on inlet and outlet liquids are made on a shift or daily basis. A selective chloride ion electrode appears to be a good supplement to the conductivity meters.

7. Utility Consumption. Per ton of  $K_2O$  in the product solution the consumption today in the ion exchange plant - including the raw materials preparation and the evaporation unit - is as follows:

Fresh water	about 20 m <sup>3</sup> per ton $K_2O$
Electricity	" 190 kWh " " "
Fuel oil	" 350 kg " " "

In case of cheap steam available the fuel oil consumption may be replaced by about 2.5 tons of steam per ton of  $K_2O$  in a two or three stages vacuum evaporator.

Improvements in today's consumption figures are still possible.

The operating labour on shift is 1 man for supervising the ion exchange unit, 1 man for taking care of raw materials preparation and 1 man for the evaporator.

8. CaCl<sub>2</sub>-Effluent. The CaCl<sub>2</sub>-solution from the re-generation section is pumped into the sea through the pipe normally used for the gypsum slurry from our phosphoric acid plant.

A comprehensive investigation has been started to study the possible effect of this solution on the sea water.

9. Product Properties. The main product produced is 21-9-12 (N-P<sub>2</sub>O<sub>5</sub>-K<sub>2</sub>O) containing 3% MgO in the form of MgSO<sub>4</sub> and 0,2% Cu as CuSO<sub>4</sub>. The ion exchange process is carried through to a K<sub>2</sub>O/P<sub>2</sub>O<sub>5</sub>-ratio = 1,8-1,9 in the nitrophosphate slurry and the necessary additional P<sub>2</sub>O<sub>5</sub> is added to the slurry in the form of phosphoric acid in the neutralisation reactors in the NPK-plant. This means that about 30% of the P<sub>2</sub>O<sub>5</sub> in the end product comes from phosphoric acid, and this results in about 75% water solubility of P<sub>2</sub>O<sub>5</sub> in the end product. Also additional nitric acid and ammonia as well as MgSO<sub>4</sub> and CuSO<sub>4</sub> are added in the NPK reactors.

A typical analysis of the product solution as it comes from the ion exchange loop is 10,5% K<sub>2</sub>O - 5,7% P<sub>2</sub>O<sub>5</sub> - 4,0% N - 1,5% Ca - less than 0,2% Cl.

Tests on the decomposition properties of the end product have shown excellent results, mainly because of the very low chlorine content but probably also because more than 50% of the nitrogen is present in the form of nitrate.

Oxidising tests have shown significantly lower oxidising capacity for this NPK product than for the calcium nitrate and the calcium ammonium nitrate commonly used in Denmark today.

Therefore we expect no safety problems in this respect with the new fertilizer.

Preliminary investigations indicate lower caking tendencies during storage for this product than for the chlorine-containing NPK grades, possibly due to the absence of NH<sub>4</sub>Cl.

## ECONOMICS

Using phosphate rock as a raw material for NPK production means spending money in some way or another on calcium removal. Many cost comparisons have been made



to show which way is generally the cheapest, e.g. the phosphoric acid route or a nitrophosphate route. Such general cost comparisons may always give rise to discussions, not only on the unit prices but also on such items as for instance the value of the higher total nutrient content obtainable in the phosphoric acid route.

What is needed in each specific case is a separate feasibility study for this case. For instance, if an NPK manufacturer - based on a mixed acids process and producing the intermediate acids himself - wants to expand his NPK production capacity, he may first of all consider an expansion of his sulfuric acid, phosphoric acid, nitric acid and NPK units. This he may compare to other possibilities. In our opinion, he may today also consider the possibility of choosing the ion exchange route for the expansion, even if he attaches little or no value to the possibility of making chlorine-free NPK fertilizers.

In that case the investment comparison will comprise the following:

	Mixed Acid Route	Ion Exchange Route
New investment or Enlargement	Sulfuric acid Phosphoric acid Nitric acid NPK	Ion exchange Nitric acid NPK

Here, the NPK investment will be about the same in both cases. The nitric acid investment will be higher in the ion exchange case than in the mixed acid case because relatively more nitrogen in the end product is present as nitrate. However, the total investment needed will in most cases - depending on existing capacities and enlargement possibilities - no doubt be the lowest for the ion exchange route.

In addition to the above the running costs in the two cases are to be evaluated. Here, the most obvious differences are the sulphur consumption in the mixed acid route against the higher potassium loss and the higher evaporation costs in the ion exchange route.

What will turn out to give the lowest total costs (investment + running costs) will depend on local conditions and unit prices. The final difference in

total costs between the two routes will in most cases probably be fairly small, and in this case the ion exchange route has the additional advantages of giving chlorine-free NPK, high total nutrient content and a very high degree of flexibility when the existing sulfuric and phosphoric acid facilities are combined with the new ion exchange unit.

The above is mentioned as an example showing how the ion exchange possibility may go into feasibility studies regarding NPK production.

#### CONCLUSION.

A cheaper way of producing chlorine-free NPK fertilizers has been developed and has been tested in an industrial scale plant. The operating results as a whole have been very satisfactory, and the new plant has been put in regular operation for making chlorine-free NPK fertilizers for the Danish market. Further improvements in the running conditions are possible and will be aimed at.

Based on the results obtained we are of the opinion that feasibility studies in many cases will point out the ion exchange route as being the preferable way of producing NPK fertilizers.

DISCUSSION

MR. K.C. KNUDSEN (Superfos a.s., Denmark.)  
Chlorine-free NPK fertilizers are superior to those which contain chlorine. However, because they are more expensive they are only used to a limited extent and our aim was to find a cheaper way of producing chlorine-free NPK fertilizers.

The problem was to find a way of making chlorine-free NPK based on the cheapest potassium raw material, which is potassium chloride, i.e. how to retain the potassium and to get rid of the chloride at the lowest possible cost. Our idea was to combine this problem of chloride removal with the well known problem of calcium removal from the cheapest phosphate raw material, viz. the phosphate rock.

The aim was to remove in one operation both calcium and chloride in the form of calcium chloride. Calcium removal must be paid for in one way or another, for instance, in the form of calcium sulphate or calcium nitrate. So, if the calcium could be removed by means of chloride (from potassium chloride) at the same cost as by other means of calcium removal, then the chloride removal would obviously be at no cost.

The possibility of removing calcium by means of chloride from the potassium chloride arises from the fact that calcium and potassium are positive ions. This makes it possible, in principle, to use a technique - viz. the ion exchange technique - which is well known in water treatment and purification processes, and which allows the extraction of positive ions from a solution against a simultaneous addition of other positive ions without influencing the content of negative ions in the solution.

So, if you react phosphate rock with nitric acid you get a solution containing calcium ions as well as nitrate ions and phosphoric acid. By means of an ion exchanger loaded with potassium you may now extract the calcium ions from this solution and in return add potassium ions. The solution then contains potassium nitrate and phosphoric acid and can be used for making chlorine-free NPK. The ion exchanger has now become loaded with calcium instead of potassium.

Now you solubilize potassium chloride in water and by means of the calcium-loaded ion exchanger you extract the potassium ions from this solution and replace them by calcium ions from the exchanger, i.e. you get a calcium chloride solution and the ion exchanger becomes potassium-loaded again. This means that this ion exchange operation removes at the same

time not only calcium from the phosphate rock, but also chloride from the potassium chloride.

Application of the ion exchange technique in this way is very different from traditional applications. This created a number of problems, which were studied first in the laboratory and then on a pilot plant. Based on experience obtained on the pilot plant we constructed an industrial scale ion exchange unit next to our existing NPK plant which has a capacity of about 800 tons per day while the ion exchange plant was designed for about half that. This means a scale-up factor from the pilot plant of 1:400.

Production in the ion exchange plant started in December last year, and operating experience with this unit is described in the paper presented today.

Based on the results obtained we may conclude that by ion exchange it is possible to produce chlorine-free NPK fertilizers much more cheaply than by the traditional methods. In fact ion exchange is so much cheaper that the question must now be asked in each specific case: is it more costly to produce chlorine-free NPK fertilizers or those containing chlorine.

We have been told that the future prospects for the ion exchange process will depend upon the progress of the market for chlorine-free NPK fertilizers. Obviously, the reverse is the case. There is only one reason for a farmer to buy NPK containing chlorine, viz. that it is cheaper. So, if by technological advance the price difference can be eliminated, then the market is there.

Therefore, the progress of the market for chlorine-free NPK will depend upon the development of new processes for making chlorine-free NPK.

DR. F. ZANETTI (Montecatini Edison): We have heard with much interest the paper of Superfos and the good results at the industrial plant.

I warmly congratulate the author and would like to ask him the following question:

1) Evaporator

What kind of evaporator do you use? Can you give some information about the corrosion risk in your evaporator coming from chlorine and fluorine? You reported the chlorine concentration (0.2%), but you did not tell us the fluorine percentage.

May we know why you use a water concentration of 30% and put so much water into the drying system?

2) Losses of  $K_2O$

Does your figure of 9% of losses include the loss as fluorosilicate and what is the amount of this? Might the losses of 9% be reduced by a longer regeneration? Why do you not use countercurrent flow in regeneration?

3) Losses of N and  $P_2O_5$

I think the losses depend on an imperfect piston flow in the washing system before regeneration. What losses would you forecast in a larger plant?

MR. KNUDSEN: The first question was on the kind of evaporator we use. It is a spray concentrator of Danish design. The combustion gas from an oil burner is brought into direct contact with the spray of the liquid to be evaporated, in a vertical chamber.

You ask whether there are any corrosion problems. Not in the evaporator itself but in the piping for the off-gases after the evaporator where condensation can take place. It is necessary to use a stainless steel containing molybdenum, say 316. In the evaporator itself this is not necessary due to the inhibiting effects of the nitrate; you could use 304 there.

You ask why we do not concentrate to more than 70% solids evaporation. That is because the slurry is intended for granulation in a spherodizer and therefore we want to end up with a fluid. When we have evaporated down to 30% water and added ammonia and the other constituents, the resulting fluid at the spherodizer inlet is a slurry containing the normal amount of water for spherodizing.

Then you ask about the losses of potassium, whether potassium fluorosilicate is included in the losses? It is. We register total potassium loss and we do not distinguish the losses. The potassium loss in the potassium fluorosilicate probably amounts to between 1 and 2% depending on how much fluorine goes into the product solution and how much into the calcium chloride solution. Normally it is about 50-50.

You want to know if a longer regeneration section would reduce potassium losses. A longer regeneration section alone would not necessarily reduce the potassium losses, but a longer loading section together with a longer regeneration section would in principle do so. This is a matter of evaluating higher investment against the production cost.

You want to know why we do not regenerate countercurrent. In the first place, the resin and the regeneration liquids are moving countercurrent to each other. However, in the ion exchange terminology, countercurrent regeneration means that the regeneration liquid moves in the opposite way to the liquid during loading and this would, in our case, be a very complicated arrangement and furthermore we would not get the normal benefit of countercurrent regeneration because our regeneration section is split into a number of small consecutive parts and you can only move forward one step at a time.

Finally you had a question about nitrogen and  $P_2O_5$  losses, the so-called rinsing losses, and you asked whether they would increase in a plant with a higher capacity. In principle, they should. However, surprisingly enough, in the 400 t.p.d. plant the rinsing losses, (N and  $P_2O_5$  losses) are no higher than those which we had in the 1 t.p.d. plant. I might add that the rinsing losses could probably be reduced as low as you wanted to make them if you are prepared to pay the higher evaporation costs. If you use more rinsing water, you have higher evaporation costs and lower rinsing losses.

MR. R. CALMEYER (Norsk Hydro, Norway): You say that you operate with losses of potassium of the magnitude of 9% which you possibly hope to decrease, but, if this is about the average you are operating with, it must mean a lot in the economy of the process. I made a very rough calculation. The economic advantage of the process should be the price difference between potassium sulphate and potassium chloride. If you then lose 9% it would take away roughly between 30 and 40% of the economic advantage which would be obtained with zero loss. And when you then add the production loss to the additional cost for evaporation, it seems to me that there will not be very much left of the price advantage gained from the difference between the two potassium sources. Is this the case or not?

MR. KNUDSEN: This depends of course on how much you pay for your potassium sulphate compared with your potassium chloride. It is generally considered that potassium sulphate is between 50 and 60% more expensive per unit of  $K_2O$  than potassium chloride. If you can obtain it very much more cheaply, then of course you may put your own figure in. You may compare how much more you pay for the potassium sulphate than for the potassium chloride. Since you use 9% more  $K_2O$  in the ion exchange process if you can get potassium sulphate at a price which is only 9 or 10% more per unit of  $K_2O$  than potassium chloride,

then of course there is a balance. But this does not mean that you have detracted from the economics of the ion exchange process because our contention is that you must pay for removing your calcium. We remove the calcium by ion exchange and, if we can do that at the same cost as you can remove calcium in traditional ways, then it does not matter what the price of potassium sulphate is compared with that of potassium chloride. It does not enter into the matter.

MR. P. VERSTEEGH (U.K.F., Netherlands.) If I read the paper correctly some 2 to 2.5% of CaO remains in the product solution. How does this influence the quality of the product? I see two possibilities: the first is a higher pH value. This may mean a lot in water solubility of  $P_2O_5$  as you yourself mention in your paper. But in a lower pH value there is the possible formation of potassium nitrate which might endanger the hygroscopicity of the product.

You mention that your calcium chloride effluent is washed away to sea. This effluent contains quite appreciable amounts of potassium and presumably  $P_2O_5$ . What have your studies on environmental effect shown?

MR. KNUDSEN. The first question concerned the calcium or CaO content of the product solution. It is indicated in the paper that we add phosphoric acid to obtain the grade we want. If we want a grade corresponding to the product, we make 21-9-12. If you only put so much potassium during ion exchange into the solution that you get this ratio between  $K_2O$  and  $P_2O_5$ , a lot of calcium is left. So we go higher and then we have to add phosphoric acid; we then get a ratio between CaO and  $P_2O_5$  corresponding to the water solubility required. If you insist on higher water solubility, you can accept less calcium, which means you must add even more potassium and then a correspondingly higher amount of phosphoric acid in the reactors.

The second question was on calcium chloride effluent. We said in the paper that we have started a comprehensive investigation to study the possible effect of this solution on sea water. In fact nobody would expect much effect from calcium chloride on sea water, but there are these other constituents: the potassium which has a minor effect, the nitrate and the  $P_2O_5$ , the acidity, the small fluorine content which is the only toxic component, and there might be also some problems with sediments. Everything is checked by a large number of analyses, samples are taken from the sea and the investigation is to be

extended over more than one year after which we will make a report to the Danish authorities.

So far we have observed no effect at all on the sea water.

MR. VERSTEEGH. I would repeat my question about the quality of the product and whether you have noticed any effect on caking tendency, or hygroscopicity because of the calcium content of the product.

MR. KNUDSEN. The calcium content of the product does not end up as calcium nitrate or anything like that. In normal procedures, when you ammoniate the solution, the calcium content ends up as dicalcium phosphate, and studies so far of the caking tendency have only indicated that it might be less in these products, possibly due to the absence of ammonium chloride, because there is no chloride in the product. That is the only difference as far as caking goes.

MR. P. ROLFSEN (Norsk Hydro. a.s., Norway.) In your last paper we asked about the stability of the catalyst, and how long it actually lasts. You were very optimistic at that time and we would be very interested to know the results in practice. Is the catalyst, the ion exchanger, lasting as well as you expected? It is probably a matter of temperature and may I presume that you are using a temperature of about 50-60°C which is the temperature to be expected when phosphate rock is dissolved in nitric acid?

MR. KNUDSEN As regards the stability of the resin and its consumption, I do not think that we can add much to what is in the paper, which gives an even more optimistic view than we had two years ago. So far, indications are that resin consumption will be lower than the 20% per year which we expected, based on the pilot plant experiment.

The second question was about the temperature and it is correct that this is about 50°C.

DR. M. PEARCE (Fisons Limited, U.K.) I would like to ask two questions. The first is - what is the range of phosphate rocks that can be used in this process? The second question is in relation to the economics of the process; it is said in the paper that the process is comparable in economics with the mixed acid process even though no credit is given for the freedom from chloride of the product. Now in such an equation one must take a figure for depreciation and return on capital and I would like to know what



MR. KNUDSEN First question - which phosphate rock could be used? On the full scale plant we have so far used only Kola phosphate rock. In the pilot plant, we used only Kola and Morocco phosphate rock. We see no reason why it should not be possible to use other phosphate rocks. Secondly, the economics. I think you want to know what figure we use for depreciation and return on capital. I think I should state that two years ago we made some studies of the economics of the process in which we assumed some figures on unit costs and then got into trouble because everybody else said this was unreasonable costing because the unit costs were different in their case. So what we have done this time is to say - for each specific case, make your own feasibility study. You have the operating costs in our case and you can compare them with the other operating costs and you may put in whatever you want for return on capital and so on. If you want a very low investment then you put in the figure in your feasibility study. If you have a mixed acid process and want to compare it with ion exchange, you may put in the figure which is relevant for you: 20%, 10%, 30%, 40% return on investment, whatever you want. Then you will see whether or not it is advantageous for you to use the ion exchange. Generally speaking, ion exchange would have a lower capital cost than the sum of sulphuric acid and phosphoric acid and then it depends on whether you place great emphasis on having low investment or whether you prefer high investment and low running cost, low potassium loss for instance.

DR. M. PEARCE My question is answered in that the capital cost of this process is said to be lower than that of the mixed acid process. I have just one supplementary question: in the running costs the price of sulphur is obviously important. Do Mr. KNUDSEN's comments refer to present conditions of low sulphur prices?

MR. KNUDSEN If you put in, say, \$27 per ton of sulphur delivered in Western Europe, and, say, \$40/ton of potassium chloride you will find that, with a 9 or 10% potassium loss, the cost of potassium lost is lower than the cost of the corresponding amount of sulphur.

MR. M. NOIRBENT (Docks Industrials S.A., France) The fact of not having any potassium or ammonium chloride in the solutions could present definite advantages for subsequent processing and prilling of NPK products. What are the speaker's views on this?

MR. KNUDSEN We have no experience of prilling.

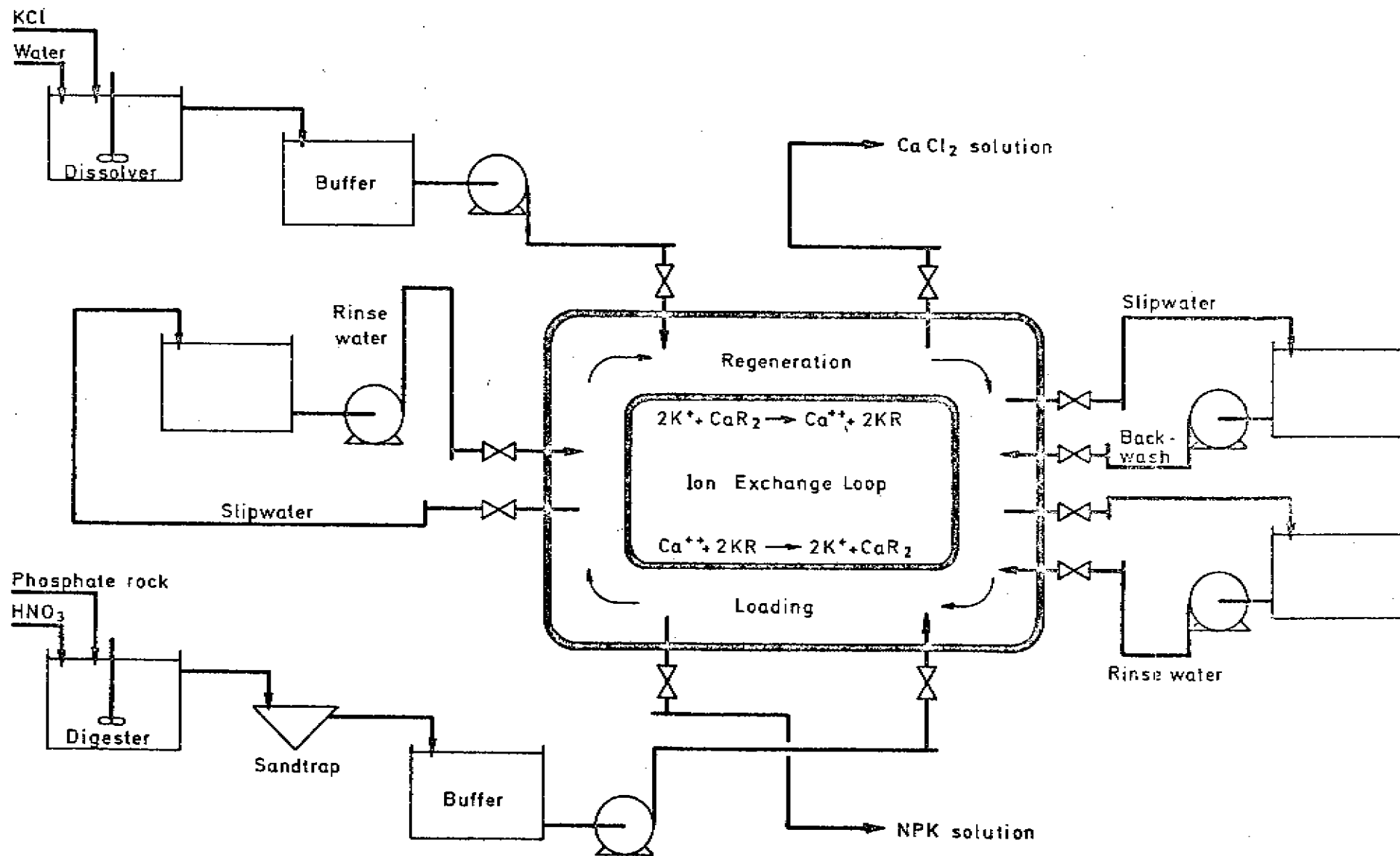
MR. O.M.LIE (Norsk Hydro, Norway) I have two questions to ask Mr. Knudsen. The first is technical. Is it normal for the potassium chloride to contain a certain amount of sulphate? When you prepare your regeneration solution obviously you have some sulphate in solution. When you then regenerate your ion exchanger this brings calcium into solution and it seems to me likely that the solubility product of calcium sulphate will be such as to precipitate gypsum on the ion exchange resin. Is the removal of the sulphate from the potassium chloride necessary in the process? My second question is on the complexity of the process. In the paper, I read that you have at least a hundred control valves of various types. I am sure that, during the starting phase, the plant was saturated with bright engineers and I assume that you must have had at least one man per valve. It seems to me that in this particular case, the risk you run when these people leave the plant is somewhat greater than in most fertiliser processes, since the economy of the process is obviously influenced by delicate manipulation of the control valves. Does this happen?

MR. KNUDSEN To answer briefly the first question on sulphate content: this is of no importance when you use 60% K<sub>2</sub>O potassium chloride, because it contains so little sulphate, but when you use lower grade potassium chloride such as 50%, you may get into trouble if you do not specify a reasonably low sulphate content, or if you do not dissolve so quickly that the magnesium sulphate does not have time to go into solution.

The second question refers to the plant availability or running factor, and I can give you some plant availability figures expressed as a percentage of total time for the last month. The highest obtained during one week was 99.7% and the highest during 16 consecutive days was 98.7%; there were no engineers in the plant except during the day and then only one in total and not one per valve.

Fig.1

Chlorine-free NPK fertilizers by ion exchange



The inlet and outlet places for the liquids are moved stepwise forward in the loop thereby displacing the loading and regeneration sections around the loop.