ISMA* Technical Conference

Sandefjord, Norway

8-11 September 1970

**In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).*

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CLOSED WATER CIRCULATION SYSTEM IN PHOSPHORIC ACID AND FERTILISER PLANTS

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SUMMARY

The article describes the water circulation system of the phosphoric acid and fertiliser plants at Siilinjärvi works of Rikkihappo Oy. Water arriving in and leaving the phosphoric acid and fertiliser plants has been connected into a single closed water circulation system, thus avoiding discharge of waste water into the environment.

The phosphoric acid plant operates on the hemihydrate-dihydrate system and has a capacity of 75,000 tons of P_2O_5 a year. The fertiliser plant produces 150,000 tons of monoammonium phosphate a year. Waste water containing nutrients coming from the plants is collected in one artificial basin which is about two hectares in area and two meters in depth. From the basin the water is pumped back to the plants for various purposes. The water in the closed circulation system is cooled by two cooling towers. The circulation system is also used to handle the run-off water from the storage area for the gypsum coming from the phosphoric acid plant, and from other areas which are liable to allow nutrients to enter the environment, such as unloading points for raw materials (apatite and ammonia) and product storage and loading areas. The circulation system handles also the nutrient containing water produced in the scrubbing of waste gases. These arrangements have made it possible to eliminate the environmental pollution effects of the whole works area almost completely.

INTRODUCTION

Protection of the environment, which has been a subject of growing concern with the expansion of industry, involves considerable restrictions in the location of new plants and the spread of pollution from old plants. Fertiliser factories are often in a specially difficult position with respect to nutrients, such as phosphorus and nitrogen, endangering the environment. In Finland, nutrients entering water result in the excessive stimulation of plant and animal life during the summer. This means a higher continuous consumption of oxygen in the water and can easily lead to a lack of oxygen especially under the ice covering the lakes and rivers in the winter. This may lead to catastrophic destruction of the fauna and fishes during the long winters. For this reason, every effort should be made to prevent the entry of nutrients into the water systems if the natural equilibrium is not to be seriously disturbed.

A good example of the measures nowadays taken in the design and construction of plants, with a view to their proper utilization of the water resources, are the water arrangements at the Siilinjärvi works of Rikkihappo Oy, which were started up last summer.

THE PHOSPHORIC ACID PLANT

The phosphoric acid plant process consists of hemihydrate and dihydrate processes. Kola apatite used as raw material in the hemihydrate reactors is dissolved in several stages with concentrated sulphuric acid. Cooling of the reaction slurry and removal of fluorine are carried out in vacuum flashing chambers whose vacuum and scrubber systems use sealing and wash waters. The results of the reaction are concentrated phosphoric acid with 40 to 43 per cent P_2O_5 and hemihydrate gypsum. The latter is filtered off and washed with a weak phosphoric acid fraction. The hemihydrate gypsum is slurried into a dihydrate reactor. This reaction stage also involves vacuum cooling and gas washing. The gypsum is filtered with an Eimco filter and washed with the circulating water, and in the final stage with pure fresh water, as far as this is possible without disturbing the water balance in the circulation system. The waste gypsum is removed by conveyor and transported to the gypsum area by trucks.

Gases containing fluorine compounds, coming from top of the filter and reactors, are removed from the plant premises to improve the condition of the air around the plants, and are washed with gas scrubbers. A total of 40,000 m^3/h of gases requiring treatment come from the phosphoric acid plant ; these are exhausted, after the scrubbers, through a collecting duct into a chimney, and the condensates are returned to the phosphoric acid plant.

The capacity of the phosphoric acid plant is 75,000 tons of P_2O_5 a year.

Temporary leakages and overflows from the installations and from the water used for washing the plant floors are drained into a floor sump, from which the water can be re-utilized. The only direct drains leading into the lake are those which are known to carry nutrient-free water only.

Expressed qualitatively, the water balance at the phosphoric acid plant is as follows :

Water entering the plant

1. With sulphuric acid (93% H_2SO_4)
2. To the filter to wash the gypsum
3. To the filter to wash the cloths
4. For the scrubbing of the reactors' exit gases
5. To the vacuum pumps as sealing water
6. To the condensers
7. As sealing water for pumps
8. For floor-washing
9. As purge water for instruments
10. For laboratory use
11. As steam for heating the gypsum cake wash water

Water leaving the plant

1. With the phosphoric acid
2. With the 20% fluosilisic acid
3. With the gypsum
4. With the waste gases
5. From the vacuum pumps
6. From the condensers
7. From the scrubbers
8. Sealing water from the pumps
9. From the plant's internal floor drains

THE FERTILISER PLANT

The process of the fertiliser plant consists of two stage neutralization of phosphoric acid with ammonia, crystallisation, drying and cooling. The end product is crystalline monoammonium phosphate.

Water vapor is released at the reaction stages and this, together with the drying and cooling air going through the processing equipment, forms 40,000 m^3/h of waste gases which have to be scrubbed.

The plant's production capacity is 150,000 tons of monoammonium phosphate per year.

The water balance in the plant is as follows :

Water entering the plant

1. With the phosphoric acid
2. To wash the waste gases
3. Condensate from the NH_3 vaporizer
4. As sealing water for pumps
5. For floor washing

Water leaving the plant

1. With the waste gases
2. With the product (3 to 6%)
3. As condensates from waste gases
4. Sealing water from the pumps
5. From the plant's internal floor drains

OTHER PLANTS

In addition to the plants described above, the Siilinjärvi works contain a pyrrhotite roasting plant, a sulphuric acid plant and a steam power plant. Impure waste water temporarily coming from sulphuric acid plant is connected to the water circulation system of the two plants mentioned above.

SIILINJARVI WORKS AREA

The works area is situated on the upper reaches of an extremely clean waterway. This means that an ample supply of clean water is available, but possibilities for release of waste water are extremely limited. As examples of the purity of local water there can be mentioned an overall hardness 0.5 to 1.5 DH^{o}, a phosphorus content of 0.02 mg/liter, a nitrogen content less than 0.1 mg/liter, and a water change rate of 6 m^3/sec. maximum and 0.01 m^3 minimum per second in the near-by lake.

In the neighbourhood of the works there are very good forests ; therefore, the waste gases must be washed as well as possible, which of course increases the load on the water circulation system. The waste gases of the phosphoric acid plant and monoammonium phosphate plant, about 80,000 m^3/h, are washed in gas scrubbers almost free of nutrients and to less than 15 mg of fluorine per m^3.

Storage tanks and stocks, loading and unloading installations for raw materials and products, have also been built on the area to serve the plants described. There is a danger that nutrients from these will escape into the environment, and finally, with rain into the waterways. The most sensitive areas have therefore been ditched so that the rainwater from these can be collected separately.

The waste gypsum of the phosphoric acid plant is dumped in high piles on its own waste area. In all, a 20 hectare area has been reserved for the gypsum, and it is separated from its surroundings by ditching. The gypsum area has been divided into smaller sections from which rainwater with too high a nutrient content can be collected separately, and harmless water is led into the waterway system. About 9 hectares of the gypsum area is now being used.

The total surface area of the locations which are considered critical, including the gypsum area, the unloading, loading and storage sites for nutrient-containing materials, is about 24 hectares, and rainwater coming from this area must be adequately controlled.

The mean annual rainfall in Finland is 600 mm, and the natural evaporation is about 300 mm. Thus, half of the rain forms run-off water which must be collected and treated separately with process water when it comes from critical areas with excessive nutrients.

CLOSED WATER CIRCULATION SYSTEM

The starting point for preventing nutrients entering the environment was the principle that water containing nutrients should be gathered into one collection basin and led back to various points in the phosphoric acid plant where water is needed. The water collection basin has been dimensioned so that the water from snow melting in spring over a 25 hectare area can be collected and used over the course of the year. This water is led to an artificial basin two hectares in area and two meters in depth, with an effective volume of 40,000 m^3. The location of the basin was chosen so that the water flows into the basin by gravity from the points from which it is collected. The basin was, of course, made water-tight.

The closed water circulation system follows the appended flow diagram.

The amount of water circulating is decided by the quantities needed at the various points and by the cooling requirement. The total water circulation is designed for 700 to 750 m^3/h. The water is heated in the phosphoric acid process equipment and a corresponding cooling takes place in the circulation system.

The diagram shows that all the water coming from the phosphoric acid plant and fertiliser plant, except the vacuum pump sealing water, has been successfully included in the closed system comprised by the inner circle in the flow diagram. The amount of vacuum pump sealing water is, in fact, relatively large. However, the nutrient content of the water in question has been kept extremely low by means of efficient drop eliminators.

The diagram shows roughly the distribution of the various water quantities. The largest quantities circulate through the evaporator.

The heat taken up by the water from the phosphoric acid plant is utilized to evaporate water. For this purpose, the water coming from the plants goes through two cooling towers on the edge of the circulation basin. The temperature of the water is decreased by evaporation which is about 4 to 15 m^3/h, and a mean value over a year is about 8 m^3/h.

As much pure lake water as can be taken into the system is used in the final gypsum wash. Naturally, the more of this extra water that can be utilized, the smaller is the amount of water soluble phosphorus remaining in the gypsum and the better is the total yield of phosphorus.

If there is heavy rainfall, or very rapid melting of the snow in the spring, or if it proves impossible to keep the plant running continuously at nearly full capacity, then the closed water circulation system is not able to handle all the water load. There is a water treatment plant located at the circulating water basin in which the phosphorus and fluorine in the circulating water can be precipitated with lime or iron compounds. Treated water can be led into the waterways.

Water from the phosphoric acid plant, which is part of the water circulation system, is collected into a tank and pumped to cooling towers through its own pipe line. Water coming from phosphoric acid plant floor sump, sulphuric acid plant and from the tank and storage area is drained through its gravimetric pipe line to the basin.

The circulating water system involves a fair amount of pipes, pumps, valves etc. All pumps are made of acid and fluorine resistant alloys, plastics or are rubber-lined. The pipes working at higher temperatures (up to 80°C) and pressures (up to 5 atm) are made of polypropylene and pipes working in cooler conditions are of polyethylene. The big connection pipes between the basin and plant are all underground.

Selection of material for the equipment in the phosphoric acid and monoammonium phosphate plants has been extremely critical because of the acidic and very corrosive circulation water.

To avoid sudden catastrophes, the circulating water system has been quite effectively instrumentized. Warnings reach the plant control rooms from all the critical points if the nutrient content of water entering the environment rises above a certain limit. The circulating systems themselves also have meters and alarm devices giving various indications.

The circulating water system has been built to be as flexible and complete in action as possible. It can be operated in the following ways :

A. Normal case :
Water coming from phosphoric acid plant is pumped through the cooling towers straight back to the phosphoric acid plant via a pump station.

B. When the turpidity of the water needs to be reduced :
Through cooling towers to basin.

C. In the case of trouble in cooling towers :
From phosphoric acid plant straight to the basin.

For the present, no chemical balance has been achieved in the water circulation system and this will in fact vary according to the time of the year. The impurity content will fall when the snow melts in the spring and large amounts of run-off water fill the basin. The circulating water may contain 6 to 10 grams of phosphorus per liter and 2 to 4 grams of fluorine, and these values may be reduced to half in springtime when the snow is melting.

Direct investment in the water circulating system was nearly 1 million dollars.

EXPERIENCES OF OPERATION

The plants have been in operation a little less than a full year. The water circulation system has resulted in scarcely 4 to 6 kg of phosphorus entering the waterways per day, a figure that must be considered extremely low.

The effect of the water circulation system on the yield in phosphoric acid plant is two-fold. On the one hand, since only a very limited amount of pure water can be used to wash the gypsum, there is a tendency for the amount of water soluble phosphorus in the gypsum to rise and thus reduce the yield. On the other hand, the phosphorus in the circulating water is constantly returned to the process, which would not be the case in an open water system, and this has the effect of improving the yield of the phosphoric acid plant. It is still impossible to say what the final effect of the closed water circulation system will be on the yield.

It has been estimated that the closed water circulation system costs almost 1/4 million dollars extra a year in capital costs, supervision, maintenance, pumping and repair costs. However, the system has made it possible for the plants in question to operate in surroundings and under conditions in which the requirements are very high from the point of view of protection of the environment.

DISCUSSION

Mr. E. UUSITALO (Rikkihappo Oy, Finland) : Due to the special environmental conditions in the Scandinavian area, and especially in Finland, fertiliser plants in our country used to be located in every case on the sea, as can be seen from the first slide.

Not all these factories are fertiliser plants, but many of them are. One of the oldest factories is in South East Finland on the sea. We have two in South West Finland, one in Mid-West Finland. However, recently it proved advisable to locate a fertiliser plant project inland, because of raw materials availability and the marketing area. The location which was chosen was very difficult in respect of environment protection. Stringent requirements were imposed by the very pure water courses, the flourishing forests in the area and the long cold winter period. The fertiliser plant is in mid-Finland and you can see that our most beautiful lake country begins there. The water there is very clean in spring when the snow is melting. Then this water is like distilled water. Water hardness there is only 0.5. It does not help us very much that the water courses whose source is in this area go through Russian country and then back to the Finnish Gulf. That means that the requirements in respect of water purity are terribly high. We have very clean water to use but for wastage this type of waterway is very difficult. We also built water circulation systems and other systems to protect the environment in our earlier plants. But in the inland case, particular attention has been paid to the waste products and to environmental aspects right from the beginning of project planning.

The layout of the plants can be seen in the following slide. You see the waterway which begins there and flows southwards. First there are roasting plant and sulphuric acid plant, then further to the left power plant and, in the mid-centre, there are phosphoric acid and monoammonium phosphate plants. On the left there are the social buildings and there are other buildings for storage etc. Down in the right hand corner you can see a little water. This is the beginning of our circulating water system, and this is an artificial lake. The total area is about 2 hectares and the depth is about 2.5 metres. Further to the right is the gypsum deposit area of nearly 20 hectares and a little further down there is an extra purification station. This is there because if we have too much water and if the water balance is not reached then we can purify the circulation water and put it in the lake.

The distance between these factories which are drained into the circulation system pond is about one km., and all the connecting pipes are built underground. The draining of rain water and of the cooling waters of the plant area is to the water course, and from this to the lake.

However the waste waters, condensates and process waters, and also floor and wash waters from the plant, i.e. all waters that can carry nutrients, have been led to a common circulating system. We never direct contaminated process or similar waters above ground to the pure lake. The rain water from the acid containing area and waste gypsum area, which cover together a little more than 20 ha., are also taken into the circulation water basin and thereby to the process water. I want to point out in this connection, that in Finland there is about 600 mm of rain yearly and evaporation in the very short summer is only 300 mm. Thus we get 300 mm every year from this drained area into our circulation pond.

The circulation system of the waters can be seen from slide 3 which is also included in the paper. The incoming waters are shown there in the open and the outgoing waters underground. This circulation system is running at a little more than 700 m^3 per hour and you can see that the condensers are the biggest water circulators. On the right hand side of this circulation system we have shown one cooling evaporator which can be used to some extent to control the water balance, i.e. within the limits where there is enough heat for the phosphoric acid to evaporate extra water out. New water for the system is taken into the scrubbers or to the reactor gases and to the circulation basin. Water is removed from the system with gypsum, in the product acid, as fluosilicic acid solution, in the cooling and operating towers included in the circulation system, and, if needed, through the purifying plant for the circulation waters. The last mentioned has not yet had to be used. Water is leaving the system in monoammonium phosphate fertilisers and with waste gases. The contribution of the monoammonium phosphate plant to this water system is minor. Its only contribution to the system is primarily and temporarily from the gas scrubbers and from the ammonia evaporator at 7 m^3/h.

This circulation system for waters has now been in operation for a little more than 1½ years and the results have been very good. With this system we have achieved a state where the amounts of nutrients getting into the environment, into the waterways and atmosphere are only some few kgs daily, which must be considered an excellent result. It has made it possible to build and to run plants in a region where the plant environment is highly susceptible to the effects of waste products. But it has all cost quite a lot of money. The investment costs we have calculated for this system are nearly one million dollars and the extra yearly costs about $ 250,000. This means an addition to the production costs.

Mr. L.G. NILSSON (Förenade Superfosfatfabriker, Sweden) : I want to congratulate Mr. UUSITALO on a most interesting paper. They have really succeeded in reducing air and water pollution to a minimum from their new plant in Siilinjärvi.

The loss of phosphorus from your factory is about 5 kg/day and if I have calculated correctly it is only 60 ppm of the phosphoric acid you produce in your plant. It is really seldom that it is necessary to calculate in ppm in cases like this.

I think this paper will be a great help to many companies who have to deal with pollution problems and not only from phosphoric acid plants. To clarify some points I want to put the following questions :

1. On page 6 you mention that you have an effective control system for the circulating water. Could you please describe what type of equipment is used for this control ?

2. You have a basin of 40,000 m^3 for the circulating water. Do you get any sedimentation of solid material on the bottom ? If so, in what way and how often do you have to clean the basin ?

3. The waste gypsum is dumped in piles in a special area. How does this gypsum behave ? Will it stabilise so you can drive with trucks on the surface or is it necessary to have special conveyors to get a sufficiently high pile ? What in your opinion is the maximum height for storing gypsum in this way ?

4. When it is raining some of the water soluble phosphorus in the gypsum must be washed out. Do you have any idea of how much phosphorus you will get back into the production in this way ?

5. How often do you use the purification plant for the circulating water ? Do you separate the precipitated phosphorus before the water is led into the waterways ?

Mr. UUSITALO : In your first question you asked what kind of control system we have for this circulation water system. Perhaps the most important equipment in this system is the conductivity cells. With them we first check the quantity of ionic impurities in the waters which are circulating and leaving the system. These conductivity cells are also used to give several warnings. If the conductivity is increasing over some limit, eg. 7,500 microsimons, they give an alarm and, depending how contaminated the water is, automatic valves are closed and opened so that the contaminated water only comes into the circulation water system. Regarding other instruments for effective control of this type of system we have several flowmeters which show the size of waterflow in condenser, washers etc. There are also level indicators in floor sumps etc. in several places, and also some thermometers etc. This has meant quite a lot of extra work, equipment and instrumentation in the whole phosphoric acid and fertiliser plant.

In your second question you ask if we have had some sedimentation in our basin and how we have removed it. The sedimentation has been very small and we expected that it would be much more. We have now worked the system for nearly two years and it has never been necessary to take out gypsum or silica or other material coming from fertiliser or phosphoric acid plant. The only material we have removed from our circulation system is iron oxide which has entered during washing periods from roasting and sulphuric acid plants. We built this circulation system with dimensions which allow us to scrape gypsum from the bottom if necessary but it seems that this will be only perhaps once in 2 or 3 years. We have not yet had enough experience to know precisely.

Your third question was to do with our waste gypsum. You ask if it is stabilising so that we can drive trucks on it. In this phosphoric acid plant we have the so-called hemi-hydrate-dihydrate system and our waste gypsum hardens in a few days. It is no problem to drive on it with trucks and heavy equipment. The height of the heap is currently about 15 or 20 m but we cannot see today the limitation. It could be 30 or 40 m.

Your fourth question was that, when the rainwater is returned from the gypsum area, how much phosphorus is being recovered. When we are heaping up to 20 m the surface which is in contact with the rainwater is very small compared to the amount of gypsum. This means that the amounts recovered in this way are very small. We have taken some measurements of phosphoric content in this drain water and observed values from 5 to 10 mg per litre. We have also calculated the recovery of phosphorus in this way. It is very small, only some hundred kg per year.

Your last question was how often we have used the purification plant. As I said we have not yet used it at all. But we have this purification plant, which can be used if required to sediment out precipitated phosphorus if necessary, thus avoiding putting it into waterways.

Mr. Y. DETUNCQ (Péchiney-Saint Gobain, France) :

1. What is the material of construction used in your cooling tower and have you measured the quantity of fluorine entrained into the atmosphere at this point ?

2. In your water circulation diagram, the 5 m^3 per hour which go through the MAP plant pass direct into the bay of Kuustahti. Is it that there is no significant fluorine emission in this plant ?

3. Can you indicate the concentration of the H_2SiF_6 solution which you obtain with the indicated flow of 1 m^3 per hour and give us approximately the distribution of the fluorine contained in the phosphate between this solution, the gypsum, the phosphoric acid, discharged waters and the atmosphere ?

Mr. UUSITALO : You asked first what material we are using in our cooling tower. It is wood and it has proved an excellent material. It has prevented corrosion totally and also the scaling problem has not been bad.

Concerning the fluorine evolved into the atmosphere. In the beginning, when we started up this circulation system and the phosphoric acid plant we had quite a lot of dust and small droplets emitted through this evaporator, but now we have changed the system so that these droplets are removed and not only gas fluorine impurities are emitted and the amounts are very small. The amount that is emitted into the atmosphere is about 2 to 5 kg/day.

The second question you asked concerned the 5 m^3 water passing through the monoammonium phosphate plant. This water is going through the ammonia evaporator and it has nothing to do with process waters etc. It is therefore very clean and contains no fluorine and so it can be led directly to the waterway. In this connection I want to point out that the waste gases from the monoammonium phosphate plant are washed not using this circulation water system but with phosphoric acid, in a similar way as was presented in Mr. YOUNG's paper. So it is a separate circulation system not shown in the water circulation system.

Your last question was what is the concentration we have reached in our washing system by washing fluorine from our waters and phosphoric acid. The fluosilicic acid content at 1 m^3 per hour has been between 15 and 20%. Then you ask about the fluorine balance in the whole system i.e. the distribution of fluorine content in the phosphate and how it is divided between solution, gypsum, phosphoric acid, liquid discharge and to the atmosphere. The values vary. In spring we must empty our circulation pond as much as possible to be ready to take all the melting water from the snow, and this means that we have much more diluted circulation water than in winter or summer. I can give a rough idea as to how the fluorine is divided. As fluosilicic acid we are recovering about 25-30% of the total amount of fluorine which we are getting from the raw phosphate. In the gypsum there is 20-25% of the fluorine and it varies according to the fluorine content in circulation water. The remaining fluorine, some 40-50%, goes into phosphoric acid and in that way to the monoammonium phosphate plant. In liquid discharge it is very small, some kg daily. Into the atmosphere we calculate that it can be some 10 kg daily and thus by present standards insignificant amounts.

Mr. W.R. SCURR (Bosveld, South Africa) : My company has been operating a phosphoric acid plant at Phalaborwa, in a very dry part of the country, for 5 years and we have had to adopt a very similar type of water balance, but for completely different reasons. There is a limited amount of water available and there is no large body of water into which to dispose of liquid effluents. In fact the only liquid effluent we can dispose of is the water which is imponded with the gypsum in a similarly hydraulically disposed dump. One of my questions has already been answered - I wished to know whether any deterioration of the water cooling towers had occurred. Our experience here is that with timber water cooling towers there has been considerable deterioration due to the fluorine in the circulating water, but our fluorine level is considerably higher than the author has in his case. We have 5 g per litre. In fact I should correct that. We have to limit it to 5 g/l of fluorine to prevent excessive deterioration of the timber cooling towers. I should also like to state that we have reached a height of 70 feet with our gypsum dump with no sign of collapsing or unstableness of the heap.

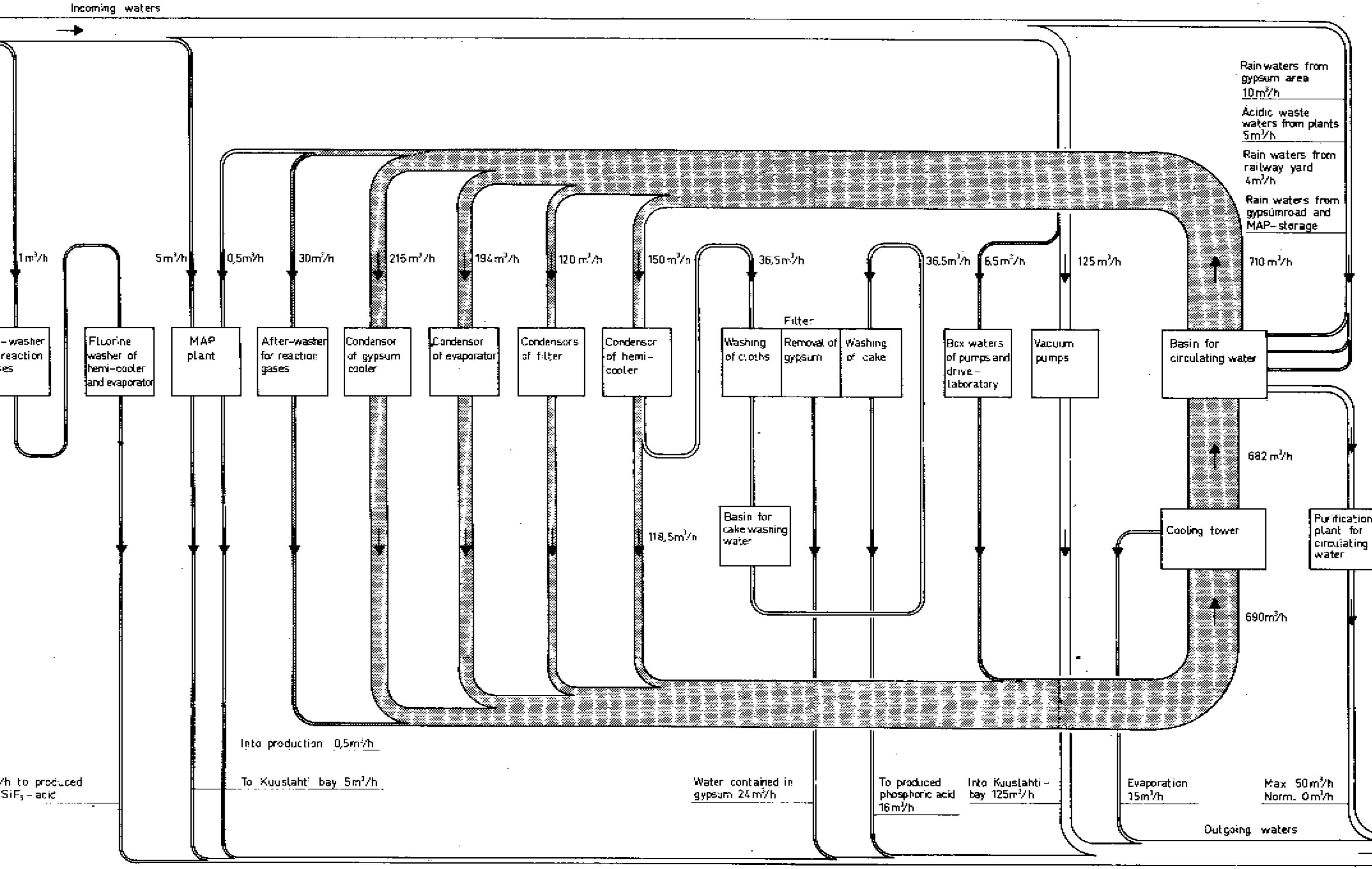
I have one other question for the author. Because of our reliance on underground water supply the authorities have required us to sink monitoring bore holes all around our gypsum disposal area to monitor for underground pollution of water. I would like to ask the author whether his company has had to adopt any precautions to prevent the pollution of underground waters from the circulating basin or the gypsum disposal area ?

Mr. UUSITALO : It was very interesting to hear you have the problem, but opposite to ours. We have too much water and you have too little. It could be a good combination. You asked if we have taken precautions in respect of underground leakages and really we have done very much. It has been very expensive to put all these heavy plastic pipes underground and protect them so that they are very safe and would not freeze. All precautions were preceded by a study of the ground where we have built the pipeline system and where we have built the circulation water system. We wished to find the best possible location and have planned very carefully where we could put the water ponds and the pipelines. They are then made watertight and sealed and also there are extra precautions so that, if something leaks, it leaks in every case into our circulating water pond. This water pond is the narrowest place in the whole area so that if something goes wrong in this area contaminated waters first go into our water circulation system, and there is no possibility of allowing them to escape into the pure waterway system.

Mr. PEARCE (Fisons, U.K.) : Mine is a very simple question, also concerning the flow sheet for water disposal. I notice that fresh water is used in the vacuum pumps and is discharged to the lake. This implies that there is no pollution in the gases going to the vacuum pumps. I wonder if Dr. UUSITALO could comment on that point.

Mr. UUSITALO : These condensers were at first one of the most difficult places to deal with. We had nearly continuous alarms from too high phosphorus content in the water going to the lake. But after we built new drop and dust eliminators we had no more alarms. Also the corrosion stopped after their installation.

Water circulation schema of the phosphoric acid and monoammonphosphate plant at Siilinjärvi



Balance calculations based on production 75,000 tons of P_2O_5 a year

RIKKIHAPPO OY FINLAND 1970