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EXPERIENCES IN PHOSPHORIC ACID PRODUCTION
WITH THE NEW SIILINJÄRVI ROCK CONCENTRATE

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1 INTRODUCTION

Kemira Oy started the first West European apatite mine at the beginning of 1980 in Siilinjärvi, eastern Finland. After the expansion of the mine has been completed in late 1982 its output of apatite concentrate will be 500,000 tons a year.

After the mine and concentrator start-up the concentrate was immediately taken into use in the phosphoric acid plant, where several modifications had been made for handling and processing the new raw material.

The experiences of the two years' use of the new concentrate have been taken into consideration in designing the extension of the phosphoric acid plant. The extension has raised the capacity to 180,000 tons of P_2O_5 a year, which corresponds to the mine capacity.

The water recirculation systems of the phosphoric acid plant and rock concentrator are completely closed. This makes it possible to produce phosphoric acid even under the strictest regulations on environmental protection.

2 PLANT PROCESS

2.1 Background

The Siilinjärvi phosphoric acid plant was constructed in 1969 and it used the hemi-dihydrate process until the end of 1973. The nominal capacity was 75,000 tons of P_2O_5/a . Owing to increasing demand for phosphoric acid the process was altered into the dihydrate process in 1973 with a capacity of 120,000 tons of P_2O_5/a . The only raw material was Kola apatite until the end of 1975. After that several phosphates have been used.

A paper summarising the experiences in phosphoric acid production with hemi-dihydrate and dihydrate processes was presented in the Technical Conference of ISMA in 1978 (1).

The work's own ore mine was opened at the beginning of 1980 (2). Based on the use of this new concentrate the latest extension of the phosphoric acid plant was completed in March 1982. In addition a third concentration line was built. The capacity is now 180,000 tons of P_2O_5/a based on the use of Siilinjärvi rock concentrate. The extension is completely based on the company's own knowhow and engineering.

2.2 Process description

The process is a single-tank based dihydrate process comprising two reaction and filtration lines.

The old part of the plant consists of a single-tank reactor and a tilting-pan filter. In the extension a big single-tank reactor and a belt filter were built.

The principle of the process appears in the flowsheet in Appendix 1.

The moist rock concentrate is fed from a two-part silo via table feeders, belt weighers and belt conveyors into the reactors. In the reactors combined air-cooling and vacuum cooling are used. The cooling air is washed in floating bed scrubbers. Gas washing water is taken from the pond. Because of the environment protection the cooling is primarily by vacuum.

From the old reactor the slurry overflows to the belt filter feed tank and from the new reactor to the tilting pan filter feed tank. Filter fumes from both filters are collected, washed by scrubbers and sucked out by fan.

The gypsum cake is discharged as a dry cake and transferred by belt conveyor to the storage area.

The filter acid is pumped to the storage tanks and further to the concentration units.

Process water from gas washing, condensers and other sources is collected and after cooling reused in the process. For the circulating water cooling, ventilated cooling towers and plate heat exchangers are used.

2.3 Design parameters

The planned actual capacity for the Siilinjärvi rock concentrate is 400 tons of P_2O_5/d with the new reactor line and 200 tons of P_2O_5/d with the old one. The annual capacity depends on the annual running time, but a capacity of at least 180,000 tons of P_2O_5/a is expected.

The main design parameters are as follows:

	Line 1	Line 2
Reactor volume m^3	old 400	new 1000
Feed tank volume m^3	70	50
Filter type	belt	tilting-pan
Filter area m^2	32	110
Capacity t P_2O_5/d	200	400

3 SIILINJÄRVI ROCK CONCENTRATE ANALYSES

3.1 Chemical and mineralogical composition

Three groups of mean analyses of the Siilinjärvi rock concentrate are shown in figure 1. Group 1 presents the average analysis over 7-22 months and group 2 over 23-25 months after the start-up of the concentrator. Group 3 presents the mean analysis of the nine samples used in the bench scale phosphoric acid production tests. These samples originate from the pilot scale concentrator 1977-79 and from production scale 1980-82.

In addition to the apatite, calcite, phlogopite, and amphibole have been identified in the concentrate by petrographic and x-ray diffraction methods. The approximate content of these minerals as calculated from the chemical analysis is shown in figure 2.

	Group 1	Group 2	Group 3
P ₂ O ₅ %	35.8	36.8	35.0
CaO "	53.8	-	-
MgO "	1.4	1.1	1.6
Na ₂ O "	0.14	0.14	0.13
K ₂ O "	0.21	0.16	0.23
Fe ₂ O ₃ "	0.51	-	1.1
Al ₂ O ₃ "	0.29	-	0.32
CO ₂ "	5.3	4.1	4.8
F "	2.6	-	-
SiO ₂ "	1.5	1.4	2.3
SO ₃ "	0.2	-	-

x) Rare earths c. 0.4 %, chlorine c. 100 ppm and Cd c. 0.1 ppm.
- = not analysed

Fig. 1 Mean analyses of the Siilinjärvi rock concentrate (dry basis)

	Group 1	Group 2	Group 3
Apatite %	86.1	88.5	84.5
Calcite "	7.0	5.5	5.2
Dolomite "	4.3	3.8	5.4
Phlogopite "	2.1	1.6	2.3
Amphibole "	1.6	1.3	2.3

Fig. 2 Mineralogical composition of the Siilinjärvi rock concentrate (dry basis)

Of the accessory minerals calcite and dolomite are assumed to be totally and phlogopite mainly soluble in the conditions of the phosphoric acid reactor. Amphibole has been identified in the waste gypsum, so it is assumed to be mainly insoluble.

The main elements are distributed between the various minerals as follows:

- K₂O is assumed to be contained nearly completely in phlogopite.
- 2/3 of the total MgO exists in carbonates and 1/3 in phlogopite.
- 2/3 of the total Al₂O₃ exists in phlogopite and the rest in the other silicate minerals.
- 20-30 % of the total Fe₂O₃ originates in phlogopite and dolomite. The rest is distributed between the other silicate minerals and small amounts of magnetite and pyrite.

3.2 Screen analysis

The average analysis during the last 12 months is shown in figure 3. The particle size is adjusted to obtain the flotation conditions.

μm	Weight %
-500	100.0
-297	98.8
-190	93.6
-140	86.5
-105	72.9
- 74	57.2
- 44	39.0
- 37	28.9

Fig. 3 Screen analysis of the Siilinjärvi rock concentrate.

The particle size distribution of the Siilinjärvi rock concentrate is approximately the same as that of Kola apatite.

4 BENCH SCALE STUDIES

4.1 General

Kemira Oy has developed comprehensive test methods for phosphate rocks and concentrates. This allows the company to evaluate the suitability of phosphate rocks for the production of phosphoric acid and fertilizers.

The suitability of the Siilinjärvi rock concentrate for phosphoric acid manufacture on an industrial scale was evaluated by the Company's Research Centre.

4.2 Test arrangement

The tests were carried out with the dihydrate process by using bench scale test equipment. The flowsheet of the test arrangement is shown in Appendix 2. The standard process conditions were as follows:

- The volume of the reactor was 10 litres.
- The temperature of the reactor was automatically maintained within a range of 76-78°C.
- The temperature in the agitated filter feed tank was maintained within a range of 75-77°C.
- The filter was a 0.1 m² batch-operating Büchner type filter.
- The concentration of the reactor acid was regulated by adjusting the density of the recycle acid.

- The volumetric amount of the recycle acid was kept constant to stabilize solid-liquid ratio in the reactor.
- The washing of the gypsum cake was performed with a two-stage counter-current wash. The amount of washing water was regulated, so that all the process water feed came from countercurrent washing.
- The filtration capacity was calculated from the sum of the filtering, washing and dewatering times and from the amount of the P_2O_5 fed during the filtration cycle.
- The rock concentrate feed was 660 g/h.
- The standard value for the excess H_2SO_4 was 2.4-2.6 % and that of the concentration of the reactor acid was 29 % P_2O_5 .
- The continuous test run took 120 hours.

The corrosion-erosion test was performed by determining the weight loss of an electrically insulated mixer impeller or of a fixed test coupon during the continuous test run. The role of the erosion is more pronounced in the case of the mixer.

In total, ten bench scale trial runs have been performed on the Siilinjärvi rock concentrate. The results are shown in Appendix 3. Some most important results are presented as contour maps as a function of two most significant accessory mineral contents (figures 4-6). The most differing sample (run No. 1) was not included in the regression model which was used as a base for the contour maps.

4.3 Filtration tests and P_2O_5 losses in gypsum

Phlogopite and dolomite are sources of chemical impurities that have a negative effect on the filterability and the lattice P_2O_5 losses of gypsum. Figures 4 and 5 show that one part of phlogopite has the same detrimental effect as about 3.5-4 parts of dolomite. This is due to the fact that nearly 50 % of phlogopite is dissolved as metal oxides in the reactor but only 17 % MgO is dissolved from dolomite.

The water soluble and unreacted P_2O_5 contents of gypsum are lower than in the production plant because of the more efficient wash in the Büchner type funnel and because of the higher shear forces between the mixer and baffles in the reactor. The correlation with the accessory mineral contents is restricted to the extreme cases.

4.4 Corrosion tests

The contents of phlogopite and amphibole were found to be significant factors which influence the corrosion properties - as silicon-containing minerals they are attacked by the free HF to form less corroding H_2SiF_6 . The ratio of the corrosion retarding efficiencies seems to be around 2.4:1 (figure 6) in favour of phlogopite. This difference is due to the fact that most of the phlogopite is again dissolved and the remaining silicic acid having a high surface area reacts rapidly with the free HF; from the amphibole only the outer surface layer reacts with HF. For the same reason amphibole can be found in the waste gypsum.

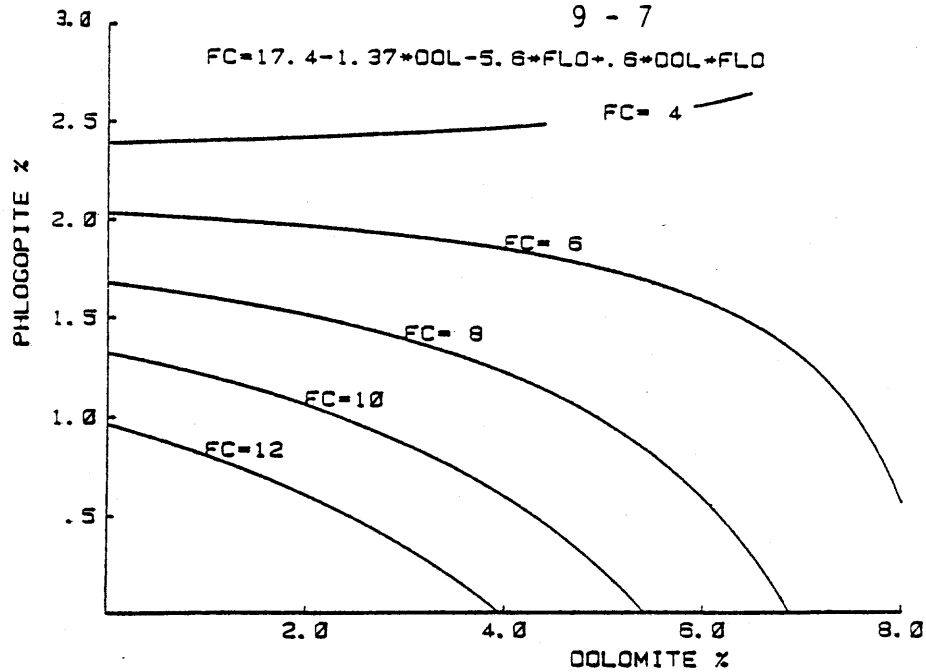


Fig 4. Contour maps of filter capacity FC (P205/m²/d) as a function of phlogopite and dolomite contents

$L = -.1349 + .0779*OOL + .2684*FLO - .0388*OOL*FLO$

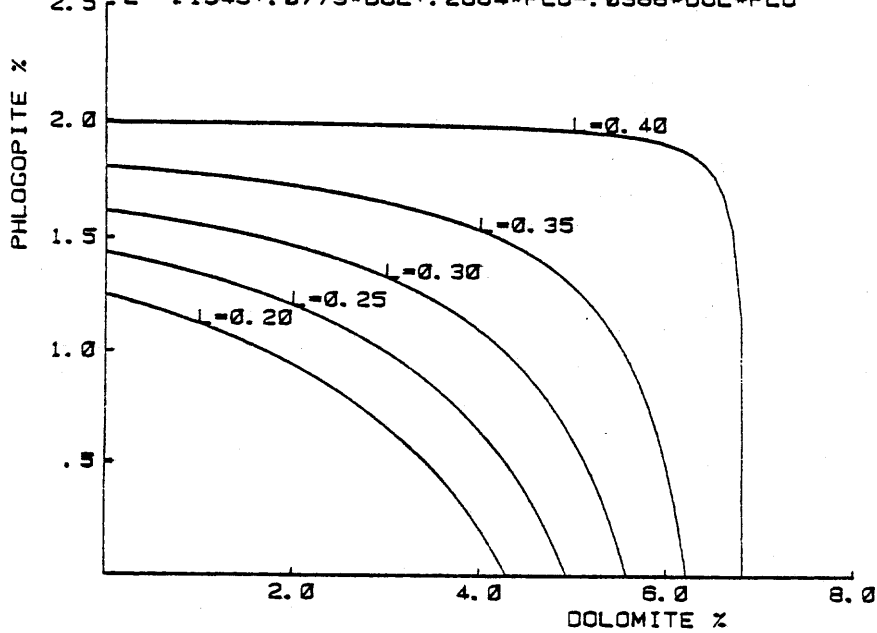


Fig 5. Contour maps of lattice P205 (L) in gypsum as a function of phlogopite and dolomite contents

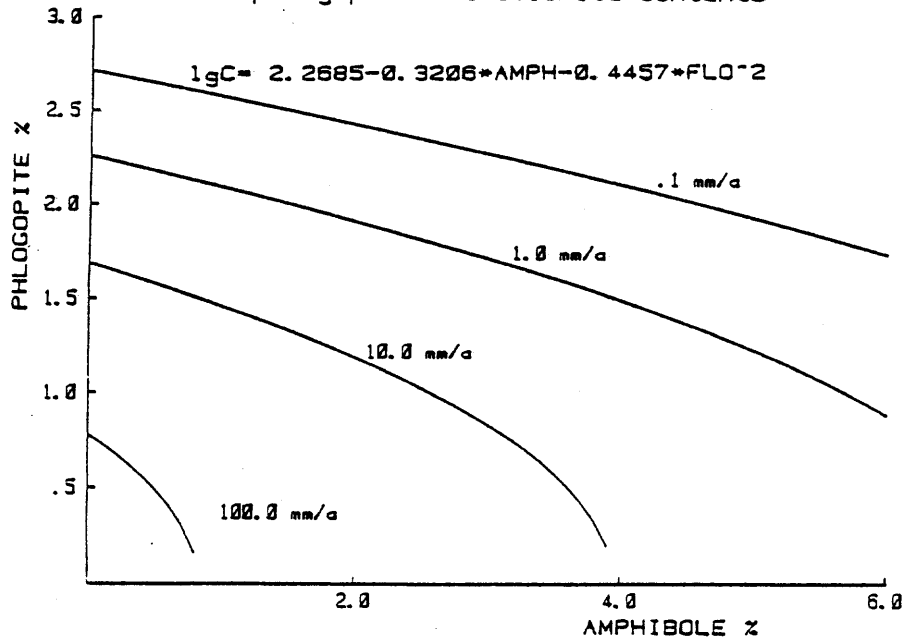


Fig 6. Contour maps of AISI 316 (mixer) corrosion as a function of phlogopite and amphibole contents

It is evident that phlogopite in the concentrate is desirable as regards corrosion prevention but very undesirable where the filter capacity or P_2O_5 -losses in gypsum are concerned.

Bierman and Long (3) have presented the oxidation requirement of the phosphoric acid as a corrosion affective factor. This is defined as meq (Ce^{4+})/l acid and is due e.g. to Fe^{++} present in the acid. When the oxidation requirement is low the metal in contact with the acid is in a passive state, and transfers to an actively corroding state when the oxidation requirement is high.

The effect of the oxidation requirement with the Siilinjärvi rock concentrate was tested in two one-week runs. The first was a control run without additive and in the second run H_2O_2 was used to lower the oxidation requirement from 60-80 meq/l as in the control run to 6-8 meq/l. The results are presented in figure 7.

	H_2O_2 additive	
	No	Yes
Filter capacity t $P_2O_5/m^2/d$	8.0	9.2
Slurry viscosity cP, 65°C	109	35
Corrosion mm/a:		
Mixer impeller Cr 17 Ni 12.5 Mo 2.5	26	9.3
" " Cr 20 Ni 25 Mo 4.5 Cu 1.5	10	0.2
Fixed test coupon Cr 17 Ni 12,5 Mo 2.5	5.1	3.0
" " " Cr 20 Ni 25 Mo 4.5 Cu 1.5	0.34	0.14
" " " Cr 22 Ni 44 Mo 6.5 Co, W	0.14	0.07

Fig. 7 The effect of H_2O_2 in reaction conditions

A clear decrease in corrosion was observed both in the mixer and in the fixed coupon. In addition, a higher filter capacity and a lower slurry viscosity were observed in the H_2O_2 -oxidized run. At this moment it is not possible to offer any reason for this.

4.5 Retention time tests

The effect of the retention time in the reactor was examined in the bench scale test by doubling the standard dosing rates into the reactor. The test was repeated using two grades of Siilinjärvi rock concentrate. The results are presented in figure 8.

	P ₂ O ₅ content in the concentrate			
	33.7 %		38.5 %	
	retention time		retention time	
	3 h	6 h	3 h	6 h
Filter capacity, t P ₂ O ₅ /m ² /d	2.5	3.5	5.6	6.4
WS - P ₂ O ₅ content in gypsum, %	0.65	0.06	0.17	0.06
Lattice "	0.62	0.44	0.40	0.32
Unreacted "	0.06	0.04	0.04	0.02
x) SDEV of free SO ₄ ²⁻ in reactor acid, %	0.25	0.20	0.26	0.17
Free H ₂ O in gypsum, %	41.3	33.0	27.8	25.1

x) measured at about 1/2 h intervals

Fig. 8 The effect of retention time on filter capacity and on P₂O₅ losses in gypsum

The short retention time leads to reduced filter capacity, higher P₂O₅ losses in gypsum, higher free H₂O content in gypsum and increased fluctuations in the free sulphate content of the reactor acid. The effect is more pronounced with low grade concentrate than with high grade.

The results of the bench scale tests were used in the design of the new reactor in the extension of the production plant. The retention time in the old reactor was 2 1/2 - 3 h and was approximately doubled in the new reactor system.

4.6 Concentration test

The concentration test is performed batchwise in a thin-film evaporator.

The results in Appendix 3 show that with high phlogopite and dolomite contents the viscosity, solid-content and tendency to postprecipitation in 50 % P₂O₅ acid increase rapidly.

5 PRODUCTION

5.1 Material handling

The concentrate is transferred to the plant by trucks and discharged to a screwfeed hopper and further by belt conveyors to the storage or directly to the two-part silo of the phosacid plant. It was necessary to rebuild the bottom of the silo, the hopper and the discharging equipment. The discharging equipment consists of a table feeder with variable speed control actuated by impulses from a belt weigher. No problems have been met with the moist concentrate feeding. When the flowability of the material is near to that of dry phosphate dedusting should be provided.

In practice the water content of the concentrate ranges between 6 and 10 %. Poor flowability and freezing in winter make the handling of moisture (over 10 %) concentrate problematic.

Intermediate storage of the rock concentrate is a future project to minimize the fluctuations in the rock concentrate quality.

5.2 Reaction conditions and efficiency

The average conditions of both reactors are as follows:

Temperature °C	78
Excess SO_4^{--} %	3.2
Acid density g/dm^3	1310
Reactor acid conc. % P_2O_5	27.0
Slurry density g/dm^3	1500
Liquid/solid ratio	2.2
Retention time h	4-6

During the first month of operation the yields were as follows:

Line 1	(old reactor - belt filter)	96.5 %
Line 2	(new reactor - tilting-pan filter)	95.5 %

The yield calculation includes the phosphor amount from the circulating water which is used as process water.

P_2O_5 losses in gypsum are presented in figure 9:

	Line 1	Line 2
Total	1.00 % P_2O_5	1.22 % P_2O_5
Watersoluble	0.66 % "	0.91 % "
Insoluble	0.34 % "	0.31 % "

Fig. 9 P_2O_5 losses in gypsum

The content of unreacted apatite in gypsum is obviously negligible.

The degree of whiteness of the by-product gypsum is very high as compared to gypsum originating from other commercial phosphate rocks.

5.3 Filtration capacity

The filter capacity of the production plant is estimated by increasing the feed rate until the surface of the gypsum cake dries just before the wash liquor is fed. The values obtained are as follows:

Line 1	Belt-filter	6.0 - 7.0 t P ₂ O ₅ /m ² d
Line 2	Tilting-pan	3.5 - 4.5 t P ₂ O ₅ /m ² d

Slurry is fed to both filters from separate reactors, and therefore the above mentioned capacity values should not be compared with each other.

The use of maximum capacity is often restricted by a bottleneck at some other phase of the process

P₂O₅ losses in gypsum have been stated in item 5.2.

5.4 Concentration

The plant has three similar concentration units each evaporating 330 t H₂O/d. These are forced circulation units with carbon block heat-exchangers. Process steam comes from the roasting unit and the nitric acid plant.

The surfaces of the heat-exchangers are cleaned every seven days with a weak H₂SiF₆ solution. Experience shows weekly cleaning to be necessary.

When concentrating weak acid produced from Siilinjärvi rock concentrate the capacity of the unit drops more quickly in a period of one week's running time than when using acid produced from other phosphates. This is because of the higher tendency to scaling on heat-exchanger surfaces.

5.5 Clarification

The plant has a clarification unit producing 50 % acid at 150-200 t P₂O₅/d. Our experience is that phosacid from Siilinjärvi rock is well suited to the clarification process. The situation is very different for example with Kola-apatite.

The clarification process uses flocculation agents at 4-6 g/t P₂O₅ to accelerate settling. Solid content of the product acid varies in the range 0.03 - 0.1 %.

5.6 Corrosion

Corrosion is one of the biggest problems when using Siilinjärvi rock concentrate. According to the research work done both in process conditions and in laboratories the factors affecting the corrosion are as follows:

- a) The low SiO₂/F ratio (1:2.6) causing the presence of free HF in phosacid.
- b) The low redox potential in phosacid (2).
- c) The high amount of excess H₂SO₄ (3.2 %).

The most problematical stages in the process can be listed as follows:

The corrosion of the Cr 17 Ni 12.5 Mo 2.5 steel mixers in the reactor has been about 10-15 mm/a depending on the location of the mixer. The highest rate is met at the point where the sulphuric acid is added into the reactor.

The corrosion rate has also been high in the circulation pump of the vacuum cooling system. The impeller of the pump has had to be replaced 2-3 times a year. The material of the circulation pump is Cr 20 Ni 29 Mo 3 Cu 3 steel.

The corrosion rate of the filter feed pump has, however, been quite moderate. This is mainly because of the variable speed control of the pump motor.

The corrosion rate of the Cr 17 Ni 12.5 Mo 2.5 steel slurry pipes has been on average 15 mm/a. Because of such a high corrosion rate it has been necessary to replace these pipes with plastic or rubberlined ones.

All the gas and fume pipes must be either plastic or rubber-lined ones.

The corrosion rate of the carbon steel vacuum pump of the filter has been high. After replacing carbon steel parts with Cr 17 Ni 12.5 Mo 2.5 steel material and with epoxy pulverized carbon steel no more corrosion has been met.

The corrosion rate of the filter pans and the filtrate pumps has been moderate. This is because of the low solid content, the lower velocity and the lower temperature of the acid.

5.7 Scaling

In addition to corrosion problems big scaling problems have also been met with when processing Siilingjärvi rock concentrate. The general opinion is that scaling is due to low Al_2O_3 content. Because of very low Al_2O_3 content there will appear soluble AlF-complexes, but also slightly soluble sodium and potassium silicofluoride compounds in the acid. These compounds have a tendency to deposit with gypsum e.g. on pipelines, pumps and filtrate receivers to form scale.

Typical pipe scaling is shown in figure 10.

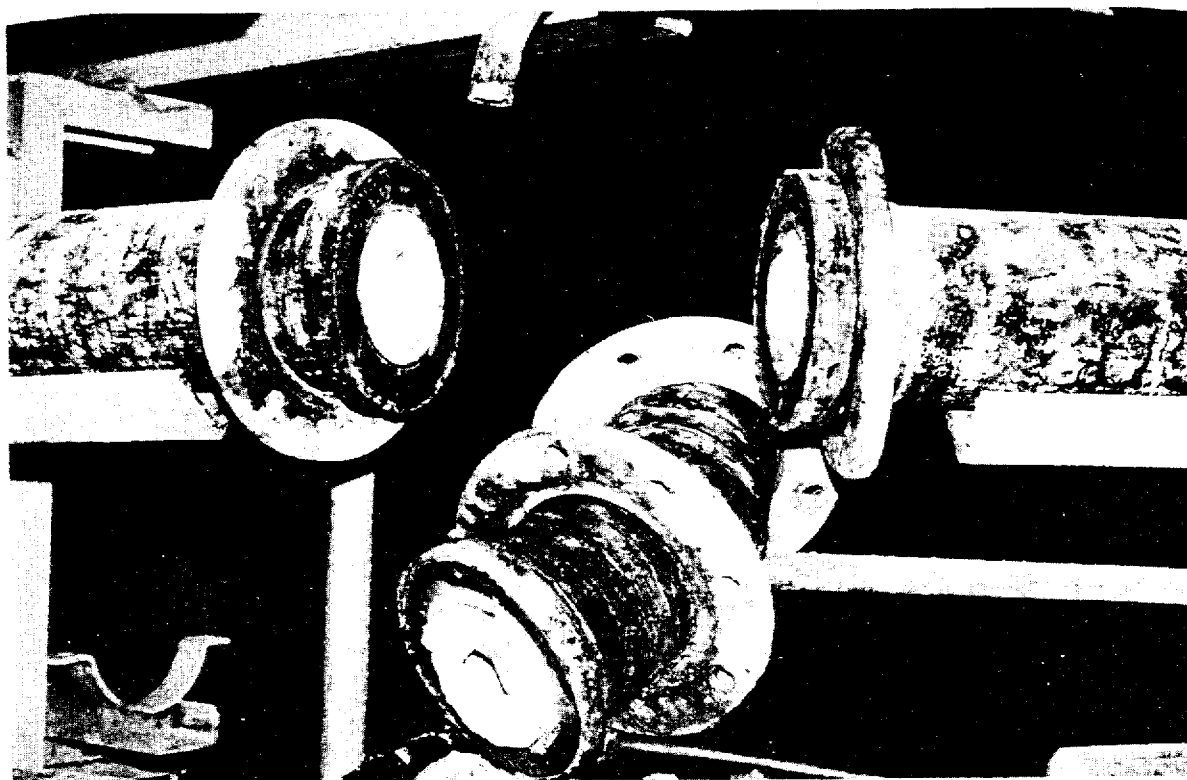


Fig. 10 Typical pipe scaling in the filter pipelines

The following possibilities have been found to solve the scaling problems:

- a) Al_2O_3 content of the rock concentrate should be raised to the same level as in the other phosphates using e.g. bauxite or anorthosite. However the costs of this extra anorthosite, for example, would be about 1 % of the total production costs of filter acid.
- b) Care should be taken not to exceed the solubility limit of $(Na, K)_2SiF_6$ in the filtrate lines. The temperature of the filtrate tends to drop by heat losses through the pipewall and by boiling of the filtrate in the vacuum. To avoid this steam is injected into the filtrate lines. By this method the rate of scale formation is reduced to the same degree as with other phosphates. The results of steam injection are presented in figure 11.
- c) Because the rate of scaling depends a lot on the roughness of the pipewall and the velocity of the acid in the pipeline it is necessary to construct pipelines so that sharp benches causing high turbulence are avoided and smoothest pipe materials are used.

Equipment	Steam injection			
	No		Yes	
	Number of cleanings per week	Scaling	Number of cleanings per week	Scaling
Vacuum divide box of the tilting-pan filter	3	Blocked	2	30 mm/week
Suction side of the prod. acid pipeline	1.5	120 mm/week	0.3	10 mm/week
Prod. acid pump	6	Blocked	1	Blocked
Pressure side of the prod. acid pipeline	3	150 mm/week	0.5	10 mm/week

Fig. 11 The effect of steam heating on scaling

5.8 Impurities interfering with the process

The most important impurities have been described under items 5.6 and 5.7. Furthermore it can be mentioned that the observations presented under the item Bench scale studies concerning the filtering capacity and P_2O_5 -losses correlate with operating experiences.

5.9 Utilization of product acid in fertilizer production

After two years experience no problems have arisen when using Siilinjärvi rock concentrate phosphoric acid in NPK-production. The acid is used at 40 % P_2O_5 concentration.

6 PRODUCTION COSTS

The most important factors in production costs are as follows:

	Filter acid	Concentrated acid
Raw materials	94.6 %	95.5 %
Energy	2.4 %	3.7 %
Maintenance	1.3 %	0.5 %
Transportation	0.5 %	-
Other	<u>1.2 %</u>	<u>0.3 %</u>
Total costs	100.0 %	100.0 %

The production costs of clarified acid are about 0.5 % of the total costs of concentrated phosphoric acid.

7 SUMMARY

The start-up of the Siilinjärvi apatite deposit has reached our expectations as regards both production and costs. On the basis of operational experiences of the extension of the phosphoric acid plant it can be stated that from the annual output of 500,000 tons of concentrate it is possible to produce phosphoric acid at the efficiency and with the consumption figures characteristic of the dihydrate process. Furthermore, the phosphoric acid produced with this process is an excellent raw material for the production of concentrated clarified phosphoric acid and fertilizers.

Plant operations will be coordinated with those of the mine and concentrator to achieve optimum overall results.

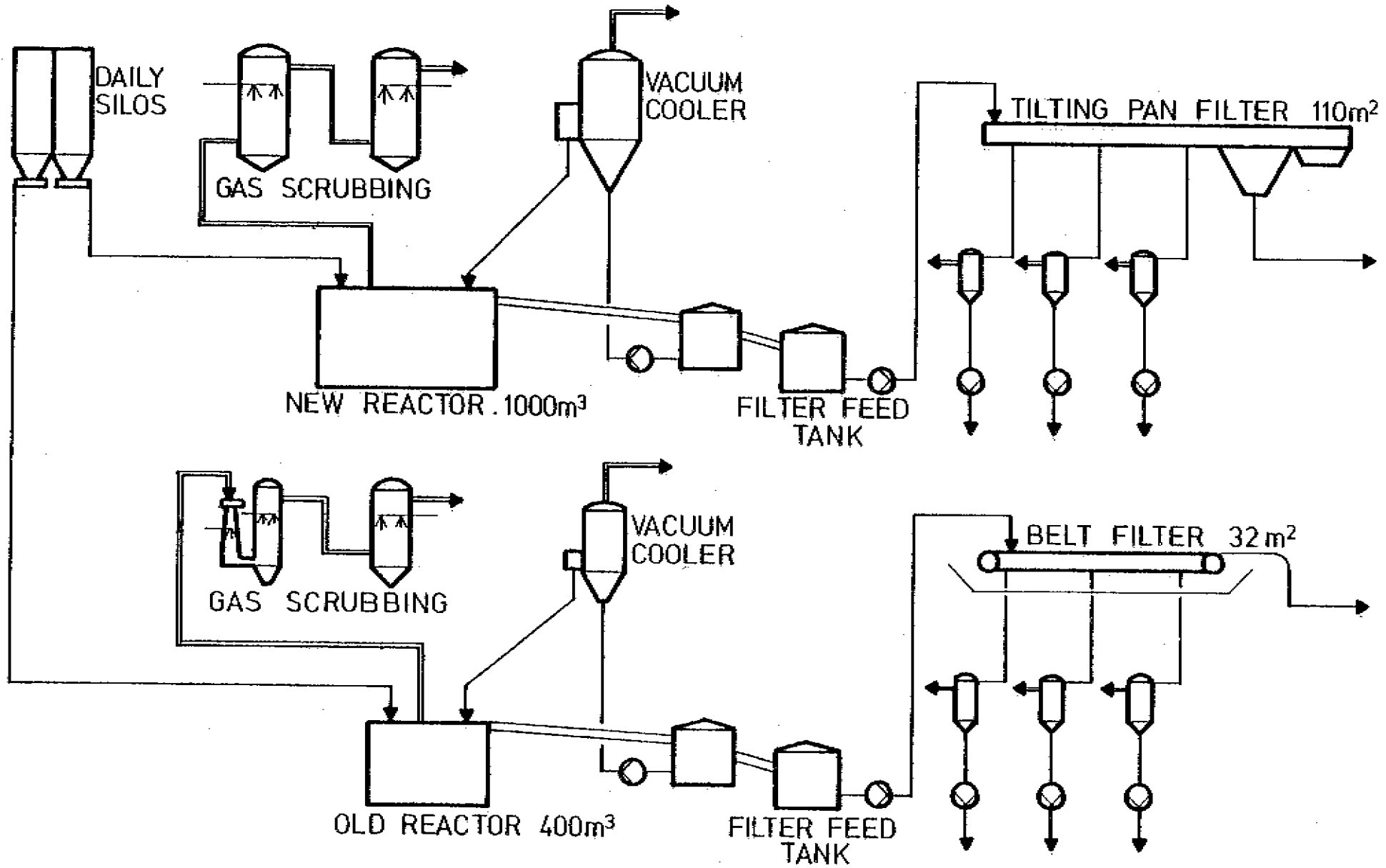
This provides the optimization of the whole process of phosphorus production. A condition for this is that the factors which easily affect the yield are stabilized and kept under constant surveillance. Such factors are:

- The dependence of the phosphorus yield on the P_2O_5 content of the concentrate at the concentrator.
- The dependence of the phosphorus yield on the P_2O_5 content of the concentrate at the phosphoric acid plant.
- The operating costs of the mine and concentrator
- Capacity of the new phosphoric acid plant

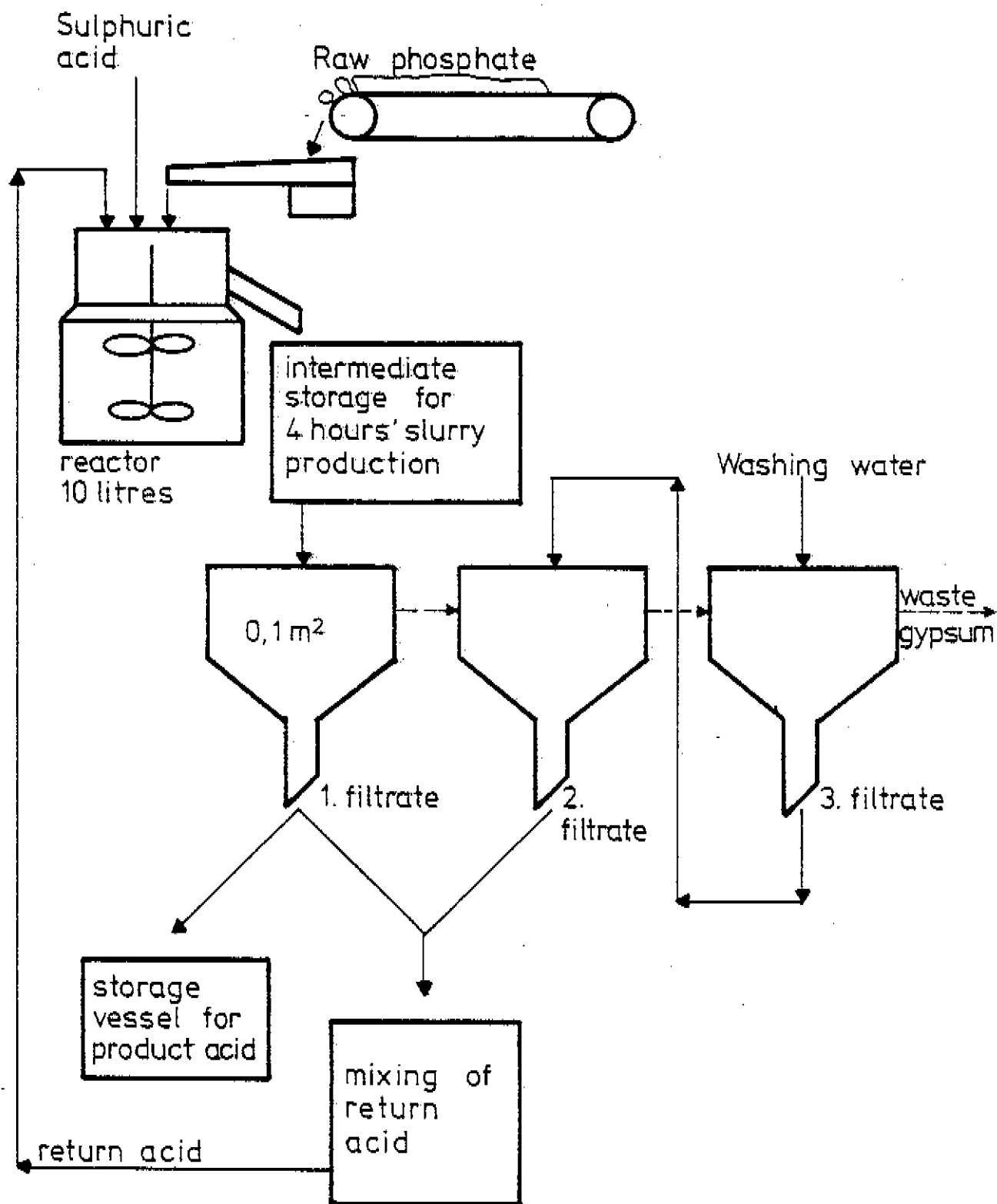
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Dihydrate process Kemira Oy Siilinjärvi Finland



FLWSHEET OF TEST ARRANGEMENTS



	Run No.									
	1	2	3	4	5	6	7	8	9	10
Chemical analyses, %										
P ₂ O ₅	29.2	31.9	32.7	33.7	36.1	36.5	37.9	38.5	38.9	39.8
CaO	47.0	-	51.7	52.8	50.2	52.8	51.5	53.4	53.9	-
MgO	3.2	2.2	2.2	1.6	1.6	1.1	0.8	0.9	0.9	0.5
Na ₂ O	0.10	0.13	0.13	0.13	0.12	0.16	0.15	0.15	0.14	-
K ₂ O	0.75	0.11	0.19	0.25	0.20	0.08	0.21	0.16	0.12	-
Fe ₂ O ₃	3.0	0.75	1.0	0.74	0.86	0.64	1.3	0.46	0.90	-
Al ₂ O ₃	1.0	0.20	0.30	0.34	0.34	0.10	0.24	0.22	0.17	0.10
F	2.1	1.9	2.0	2.0	2.6	2.1	2.6	2.2	2.2	2.5
SiO ₂	10.2	0.65	1.4	1.1	1.7	0.9	3.1	0.9	0.6	0.2
CO ₂	4.8	9.2	7.5	7.0	3.5	4.6	1.1	3.2	2.5	2.0
Mineral composition, %										
Apatite	70.2	76.7	78.6	81.0	86.8	87.7	91.1	92.5	93.5	95.6
Calcite	2.5	10.0	7.1	10.7	1.3	5.4	0.7	4.6	2.5	1.5
Dolomite	8.0	10.3	9.4	5.0	6.4	4.8	1.7	2.5	3.0	1.5
Phlogopite	7.5	1.1	1.9	2.5	2.0	0.8	2.1	1.6	1.2	1.0
Amphibole	12.6	0.3	1.1	0.1	1.5	1.0	4.0	0.4	0.1	0
t H ₂ SO ₄ (100 %/ t P ₂ O ₅ in feed										
	2.49	2.77	2.74	2.69	2.46	2.53	2.36	2.42	2.49	2.35
Filter capacity, t P ₂ O ₅ /m ² /d										
	3.0	4.3	5.0	3.5	5.2	8.0	6.4	6.4	8.0	11.9
P ₂ O ₅ content in gypsum, %										
water soluble	0.16	0.08	0.04	0.06	0.05	0.05	0.07	0.05	0.05	-
lattice	0.75	0.56	0.43	0.44	0.38	0.27	0.45	0.31	0.22	-
unreacted	0.57	0.06	0.04	0.04	0.03	0.03	0.02	0.02	0.02	-
50 % P ₂ O ₅ acid										
viscosity, 70°C, cP	169	21	-	-	-	-	9	-	-	8
% F	1.2	-	-	-	-	-	1.4	-	-	-
solids after conc, %	5.7	3.0	-	-	-	-	1.6	-	-	1.6
postprecipitation	5.8	2.7	-	-	-	-	1.5	-	-	0.1
Ammoniated acid (pH 5.5)										
water soluble P ₂ O ₅ from total, %	75	90	-	-	-	-	95	-	-	95
Corrosion, mm/a										
Cr 17 Ni 12.5 Mo 2.5 mixer blade	0.04	-	0.28	0.22	4.5	31	0.09	38	26	16
Cr 17 Ni 12.5 Mo 2.5 fixed blade	0.01	-	0.13	0.17	1.0	-	0.04	2.7	5.1	-
Cr 20 Ni 25 Mo 4.5 Cu 1.5 fixed blade	-	-	-	-	0.04	-	0.03	0.41	0.34	-
Cr 15.5 Ni 54 Mo 16 Co 2.5 fixed blade	-	-	-	-	0.05	-	0.03	0.10	0.20	-
Cr 26 Ni 31 Mo 3.6 Cu 1.0 fixed blade	-	-	0.19	-	-	-	0.02	-	0.15	-
Cr 22 Ni 44 Mo 6.5 Co 2.5 W fixed blade	-	-	-	-	0.02	-	-	0.04	0.14	-

Results of the bench scale tests and the composition of the samples used in the tests.

TA/82/9 Experiences in phosphoric acid production with the new Siilinjärvi rock concentrate, by E. HANNINEN, E. AALTO, H. SIRVIO (Kemira Oy, Finland)

DISCUSSION : (Rapporteur: C.H.M. Vinke, Windmill Holland B.V., The Netherlands)

Q - Mr. A. NICOTRA, ANIC SpA, Italy

Because of the low content of SiO_2 in your rock, did you never utilize addition of active silica in your corrosion and filter capacity tests?

A - We have used active silica in laboratory tests. The addition of active silica has slightly decreased the corrosion but it has had a decreasing effect on the filter capacity. In industrial scale the use of active silica is too expensive in our case.

Q - Mr. P. BECKER, COFAZ SA, France

What kind of silica is the 1.5% SiO_2 ?

A - About 2/3 of the silica is in phlogopite and 1/3 is in amphibole. Most of the phlogopite is dissolved in the process, and the remaining silicic acid, having a high surface area, reacts rapidly with the free HF. From the amphibole only the outer surface layer react with HF.

Q - 5.3. Filtration capacity: what are the for line 1 and line 2 filter cycle times (or filter speed)?

A - The average filter speed for line 1 (belt-filter) is 12 metres/min and the cycle time for line 2 (tilting-pan filter) is 5 minutes/round. The vacuum box length of the belt filter is 14.4 metres.

Q - 5.5. What type of clarification do you use?
How much sludge do you have in 50% acid?

A - The clarification process consists of a conventional tank system with slow rotating raking mechanism. The acid made of the Siilinjärvi rock concentrate clarifies very easily and the solids go down very quickly. The solids content in the 50% P_2O_5 acid is below 0.5%, usually 0.2-0.3%.

Q - Mr. F.T. NIELSSON, IMC, USA

Does your rock contain barium as is the case with Kola?

A - Our rock does not contain any barium.

Q - Mr. E.G. PULLEN, The Phosphate Co-operative Company of Australia Limited, Australia

In figure 10 you refer to typical pipe scaling in the filter pipe lines due to principally sodium and potassium silico fluoride.

Page 9-13 Section 5.7b you state, "care should be taken not to exceed the solubility limit,"... "steam is injected into filtrate lines".

As this procedure transfers the scaling problem elsewhere in the process, has any attempt been made to selectively remove the silico fluorides by partial cooling of the intermediate filter wash liquors? For example:

Run 1 shows 0.1% Na_2O , 0.75% K_2O , 10.2% SiO_2

a rock of which we would particularly like to learn about your scaling problems and how you propose to overcome scaling at this level of impurities.

A - Mr. Pullen is quite right in assuming that the scaling is transferred to another place in the process.

However, as we can see in figure 11, the need of cleaning has decreased quite considerably in the pressure side of the product acid pipeline. The next place where scaling can occur, is the product acid storage tank, where crystallization and scaling certainly occur, but it has not caused problems in practice.

So far we have not tested any other methods in plant scale; likewise we have not tested the partial cooling of the intermediate filter wash liquors. By the way, we have not met any scaling in the second and third filter lines.

The sample tested in the laboratory run number 1 is from the beginning of the production of the concentrator and therefore it is not representative. However, I have understood that Mr. Pullen is interested in handling this kind phosphate concentrate. I must say that we too would be very happy if we had the knowledge.

Q - Item 5.7 - page 9-13. Could you expand your reasons for the use of Al_2O_3 to prevent scaling which we presume is the sodium silico fluoride?

A - We have studied the use of aluminium oxide only to prevent corrosion and only in the laboratory scale. It is however not easy to test scaling in the laboratory.

In our case, the addition of aluminium oxide in the production scale would be too expensive.

Q - Mr. S. ORMBERG, Norsk Hydro, Norway

Is your production of phosphate rock only for domestic use or have you plan to be an exporter in the future?

A - Our phosphate rock is only for domestic use. (There was misunderstanding during the conference; the author had understood that the question concerned phosacid, which is exported).

Q - Have you done trials with your rock in nitro-phosphate processes like the Odda process and in case with what result? Does the rather high MgO-content create any viscosity problem?

A - We are using Siilinjärvi rock concentrate continuously in our nitro-phosphate process in our Siilinjärvi plant. There have been no problems, nor any problems in viscosity. On the contrary, the concentrate has proved to be very suitable for the fertilizer process.