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FISONS HDH PHOSPHORIC ACID PROCESS OPERATING EXPERIENCE WITH A 160 TPD PLANT AT RMHK TREPCA (YUGOSLAVIA)

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1. INTRODUCTION

The development of the Fisons Hemi-Dihydrate (HDH) phosphoric acid process was carried out at the Levington Research Station of FISONS LIMITED, ENGLAND and followed the successful development and commercial installation (2) of the companies single stage hemihydrate process at WINDMILL HOLLAND BV in 1970.

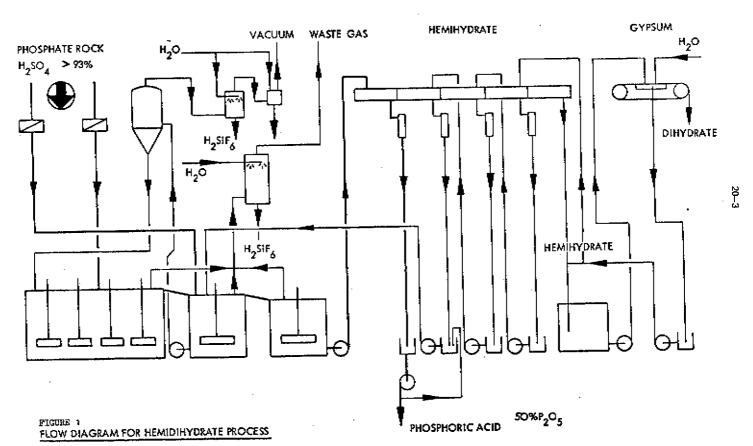
The results from pilot work at Levington and operation of the hemihydrate plant of WINDMILL HOLLAND BV have been used in the design of the production plant for RMHK TREPCA in Yugoslavia, the main features of which were reported to the 1974 ISMA Technical Conference in Prague.(1) The Trepca plant was designed to produce 50,000 tpa $P_2 O_5$ as 50 per cent $P_2 O_5$ phosphoric acid from Morocco rock and was constructed by LURGI CHEMIE UND HUTTENTECHNIK GmbH.

The main advantage of the HDH process is that 40-54% P_2O_5 phosphoric acid can be produced directly without the need for evaporation and with P_2O_5 recovery efficiencies higher than obtained in either hemihydrate single stage or the dihydrate processes. There are other advantages which were also considered by TREPCA in selecting the Fisons process and these are discussed in Section 3 of this paper.

The plant is sited within the chemical complex at Kosovska Mitrovica, approximately 200 kilometres from Belgrade and 500 metres above sea level. The factory is remote from a sea port and phosphate rock is transported to the site by rail from Salonika. Thus, cost of rock is high compared with the cost for plants which are situated at or near a sea port.

The area is comparatively isolated and this has presented additional challenges which are unconnected with establishing new technology.

Commissioning commenced in June 1975 and this paper describes the operating experience and results obtained during the period June to December when the first guarantee test was attempted.



2. PROCESS DESCRIPTION

The Fisons Two Stage Hemihydrate or HDH Process as constructed for TREPCA is for the production of 50-52% $\rm P_2O_5$ phosphoric acid at high efficiency - generally 98.5%. The process consists of a hemihydrate reaction system, hemihydrate filtration, a recrystallisation stage and dihydrate filtration. The process is illustrated in the flow sheet, (Figure 1).

Phosphate rock, 100% loss than 1.7mm, is fed to the first reactor via a rock weigh belt. The slurry from the first reaction overflows to the second reactor to which premixed sulphuric acid and "return acid" are added. The temperature in both reactors is controlled within 98° - 100° C by circulating the slurry through a flash cooler to remove the excess heat of reaction and dilution. A photograph of the reactors is shown in Figure 2.



FIGURE 2: VIEW OF HDH PLANT AT TREPCA SHOWING HEMIHYDRATE REACTORS

The reaction conditions in both reactors are important since the success of the process depends on the satisfactory growth of uniform crystals with a high filtration and washing rate. The actual conditions vary slightly from rock to rock and these are established initially by pilot plant testing and finally by optimisation during the full-scale plant commissioning.

Sulphate control of the acid in reactor 2 is important, and a fixed proportion of the Slurry pumped from the second reactor to the flash cooler is recycled to the first reactor to control the precipitation rate of calcium sulphate in this reactor, and thus the crystal size. Any excess slurry is returned to the second reactor at 1.5-2.0% sulphate level and the slurry then overflows to the filter feed tank.

The first reactor is a fully balfled tank with four six-bladed agitators, two pumping up and two pumping down to achieve a highly efficient "turn-over" flow pattern of the slurry. The second reactor and the filter feed tank are equipped with single six-bladed agitators and are likewise fully baffled.

The Pisons process uses two independently controlled filters for the filtration of the slurries. This enables the adjustment of speed and cake thickness to be optimised on both filters separately.

Slurry from the filter feed tank is pumped to the hemihydrate filter - a standard horizontal tilting pan vacuum filter with three counter-current wash stages. "Dihydrate return acid" recycled from the second stage is used for the final cake wash and the pan wash.

The acid from the first wash stage, together with a part of the product acid, is returned to the reaction system. This is the "Hemihydrate return acid" which is premixed with 98% sulphuric acid and fed to the second reactor. The remainder of the product acid is pumped to the 50% $\mathrm{P_{o}O_{e}}$ acid storage tanks.

The hemihydrate, discharged from the 1st stage filter, passes via a chute into an agitated recrystallisation tank. In this tank the residence time, solids content and chemical composition are controlled to ensure complete recrystallisation of the hemihydrate to dihydrate. Co-precipitated lattice $\mathrm{P_2O_5}$ is released from the hemihydrate during the process. Sulphuric acid is added to the recrystallisation tank to fix the calcium ions so released, and to maintain optimum conditions for maximum $\mathrm{P_2O_5}$ recovery and for the growth of good dihydrate crystals.

The dihydrate slurry is pumped to a continuous belt filter where it is washed with incoming fresh water. The filtrate, the so-called "Dihydrate return acid" which contains the recovered lattice ${\rm P_2O_5}$ is recycled to the hemihydrate filter as the final cake wash and pan wash.

The dihydrate cake is discharged dry from the filter and reslurried in an agitated tank and pumped to disposal. Alternatively, a dry discharge could be used.

Acid spillage is collected in a floor sump and returned to the recrystallisation tank.

A wash tank is installed to allow a routine closed circuit wash for the filter equipment with hot water or chemical solutions if required. No regular washing is required for the reaction slurry equipment.

Some of the fluorine fed with the rock is evolved and this may be recovered as $\mathrm{H_2SiF_6}$. With standard grade Morocco rock, 40-50% of the fluorine, fed in the rock, is evolved in the flash cooler and the reactors.

The vapours from the flash cooler pass to a two-stage vacuum scrubbing tower. In the primary fluorine scrubber, the vapours are scrubbed with recirculating ${\rm H_2SiF_6}$. Fresh water is fed into the secondary fluorine scrubber and a total fluorine recovery in excess of 99% was guaranteed, as a 23% ${\rm H_2SiF_6}$ by-product.

The exhaust gases from the reactors and the hemihydrate filter are passed through a gas scrubber operated under atmospheric pressure, for which the same fluosilicic acid concentration and scrubbing efficiency is obtained.

3. SELECTION OF THE PROCESS

In selecting the process the principal economic factors influencing Trepca's choice were:

3.1 Price of Phosphate Rock

The lack of any domestic phosphate rock production and the remoteness of the factory site from a sea port results in a very high transport surcharge on all phosphate processed. During the period covered by this report the average price of rock into the plant store was approximately 85 US \$ per ton. This price reinforces the normal desirability for a high ${\rm P}_2{\rm O}_5$ recovery efficiency.

3.2 Rock Grinding

The ability of the Fisons Hemihydrate process to use unground phosphate directly without any efficiency penalty was of considerable advantage to Trepca, in reducing capital requirement and eliminating grinding costs. The use of coarse phosphate, normally specified as:

100% through 10 BSS (1.68 mm) 75% through 30 BSS (0.5 mm) 25% through 50 BSS (0.25 mm)

is easier to handle and meter than the substantially < 100 BSS or finer grinds normally used for the dihydrate process.

3.3 Steam Consumption

The production of 50% $\rm P_2O_5$ phosphoric acid directly without need for concentration was perhaps the overriding consideration for TREPCA. Because of the local situation there was no surplus steam available from sulphuric acid production. Provision of steam for a dihydrate phosphoric plant would have involved additional energy and capital cost — (approximately 15,000 kg/hr steam capacity).

3.4 Acid Product Quality

The client wished to be able to export a significant proportion of the P_2O_5 produced as merchant grade acid. The low level of solids in hemihydrate acid and subsequent absence of post

precipitation are important advantages that avoid the need for expensive clarification equipment.

It was also planned to use the product acid for sodium tripolyphosphate manufacture which requires acid with low levels of impurities, particularly sulphate.

Fluosilicic acid produced from the process off gases is to be used for the manufacture of aluminium fluoride, and a plant is currently being constructed. A high yield and recovery efficiency are therefore important, apart from the normal pollution control considerations.

3.5 Gypsum Quality

Space limitation precludes the storage in the immediate area of the factory for more than 2-3 years production of by-product gypsum. Ultimately gypsum will be disposed of to the plaster and cement industries and the high quality of the gypsum product produced in the HDH process was an important factor.

3.6 Overall Economics

The approximate capital costs for units for the production of 50,000 tons per annum of $P_{\gamma}O_{\gamma}$, as 50% phosphoric acid are:

Dihydrate Process : 7.5 Million US \$
Hemi-Dihydrate Process: 7.0 Million US \$

The capital cost savings in rock grinding, intermediate 30% $\rm F_2O_5$ storage capacity, boiler capacity and acid clarification possible in the Hemi-Dihydrate process are partially offset by the increased cost of the larger reaction volume and filter area, and the recrystallisation reaction tank and filter. The overall effect is a reduction in capital cost of about 7-10% for the Hemi-Dihydrate process compared to the Dihydrate process.

The manufacturing costs for the two processes are shown in Table 1, for the TREPCA situation. Overall, the Hemi-Dihydrate process shows a cost saving of 17.5 \$ per ton. This is due to savings in:-

Fixed costs of 4.2 \$ per ton due to lower capital costs.

Material costs of 6.1 \$ per ton due to higher material efficiencies,

Variable costs of 7.2 \$ per ton due to lower steam consumption.

The high price of phosphate rock and steam at TREPCA particularly favours the Hemi-Dihydrate process. However, with the general world wide increase in the price of money, phosphate rock and fuel, the cost advantage of the Hemi-Dihydrate process is likely to increase.

TABLE 1

COMPARATIVE PRODUCTION COST: PHOSPHORIC ACID BY THE DIHYDRATE AND HEMIHYDRATE PROCESS ROUTES

Basis 50,000 tonnes P₂0₅/annum

Capital Costs: Dihydrate Plant

7.5 Million US \$

Hemi-Dihydrate Plant 7.0 Million US \$

		Dihydrate Process		remi- Dihydrate Process	
	Unit Cost US \$	Units per tonne P2 ⁰ 5.	Cost per tonne P ₂ O ₅ US \$	Units per tonne P205	Cost per tonne P ₂ O ₅ US \$
Fixed Costs				· · · · · · · · · · · · · · · · · · ·	
Depreciation (1) Interest (2) Rates (3) Return on Investment (4) Maintenance (5) Operating Labour (6)	- - - - - 8/hr	- - - 1.8 hr.	10.5 18.0 4.5 22.5 7.5 14.4	- - - - 1.8	9.8 16.8 4.2 21.0 7.0 14.4
Total Fixed Cost	-	-	77.4	,	73.2
Material Costs					
Phosphate Rock (7) Sulphuric Acid Chemicals	85/tonne 22/tonne -	3.09 tonnes 2.57 tonnes	262.6 58.7	3.02 tonnes 2.57 tonnes -	256.7 56.5 2.0
Total Material Cost			321.3	,	315.2
Variable Costs Cooling water Process water Steam Electricity	0.005/m ³ 0.05/m ³ 5.0/tonne 0.05/KWH	40.0 m 5.0 m ³ 1.7 tonnes 110 KWH	0.2 0.3 10.2 5.5	20 m ³ 4.0 m ³ 0.2 tonnes 150 KWH	0.1 0.2 1.2 7.5
Total Variable Cost			16.2		9,0
Potal Manufacturing Cost			414.9		397.4

Notes

- 1. 7% of capital cost/annum
- 2. 12% of capital cost/annum
- 3. 3% of capital cost/annum
- 4. 15% of capital cost/annum
- 5. 5% of capital cost/annum
- 6. 4 men/shift
- 7. 98.5% efficiency for Hemi-Dihydrate Process 96.3% efficiency for Dihydrate Process

4. OPERATING EXPERIENCE

4.1 Start-up

The plant was first started up on the 10th June 1975, using 50% $\rm P_{20}_{5}$ acid from a dihydrate plant at Prahovo to partially fill the hemihydrate reaction system. Standard grade Morocco rock was used for the first two weeks operation.

Due to the high level of organics in the Frahovo acid, foaming problems in the hemihydrate reaction system were encountered during initial operation but this effect rapidly diminished as the organics were displaced from the system. Apart from this minor problem, start up went very well and the initial impressions were that the hemihydrate and the dihydrate filters had reserve capacity. The filter cakes on both filters were discharged at lower moistures than had been expected.

As expected from the pilot plant results, none of the potential process problems, encountered by others, such as non-conversion of the hemihydrate to dihydrate, setting of the hemihydrate on the filter, blockages in the gypsum disposal line and scaling in the dihydrate filtration system were experienced. In fact, even though the start-up was plagued by short power failures, no setting on the hemihydrate filter occurred.

Both the two fluorine recovery systems were operated but the product was not regularly recovered as the erection of the AEF plant to process the product fluosilicic acid had only just started and was not scheduled for completion until mid 1976.

Initially, the plant was run at 60% capacity but the load was increased to 100% after a few days when the operators had gained more confidence. The start up procedure was as developed in 1967 at the Fisons experimental 40 tpd ${\rm P_2O_5}$ hemihydrate plant at King's Lynn, England. This was found to be very satisfactory, giving hemihydrate crystals which filtered extremely well immediately on start-up.

The first two weeks operation were used to correct minor design faults and obtain plant data in preparation for a guarantee test run. After these two weeks, the supply of standard grade Morocco rock was exhausted and alternative rocks had to be processed in the plant. The guarantee run was postponed until standard grade rock became available in December 1975.

4.2 Routine Operation

4.2.1 General

Throughout the period from the end of June to the beginning of December, the plant operated using mainly low grade Morocco rock with short periods processing standard grade Morocco rock and Youssoufia rock. The plant operated with few process problems using the alternative rocks although capacity was generally limited to 80% or 90% of design by hemitydrate filtration. Production was also reduced by the lower $\rm P_2O_5$ contents of these rocks although product strength was maintained without difficulty. Complete recrystallisation of the hemitydrate to dihydrate was maintained throughout, in the recrystallisation tank.

No gypsum scaling was apparent in the hemihydrate filter circuit due to the use of a recrystallisation inhibitor. It had been anticipated that there could be a build up of sodium fluorosilicate scale in the dihydrate filtration system. Up to the present time no scaling whatseever has been found in the dihydrate filtration section.

4.2.2 Filtration

Very few problems were experienced in the operation of the filtration sections. It is worth noting that the hemihydrate crystal size, shape and filtration characteristics were virtually identical to the pilot plant results. For comparison, photographs of the hemihydrate crystals produced on the pilot plant and the TREPCA plant are shown in Figures 3 and 4.

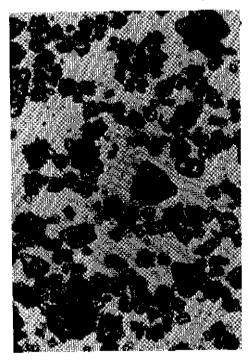


FIG 5: HEMIHYDRATE CRYSTALS PILOT PLANT

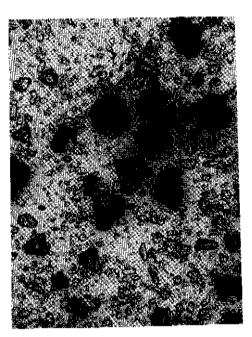


FIG 4: HEMIHYDRATE CRYSTALS TREPCA PLANT

The dihydrate crystals are shown in the following photographs (Figures 5 and 6).

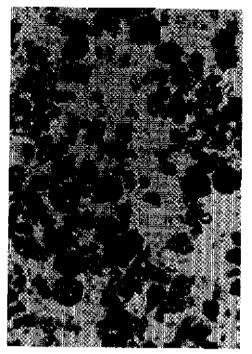


FIG 5: DIHYDRATE CRYSTALS PILOT PLANT

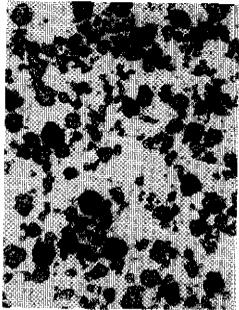


FIG 6: DIHYDRATE CRYSTALS TREPCA PLANT

The dihydrate crystal shape was also as expected but the size and filtration characteristics were considerably better resulting in a lower than predicted moisture content of the dihydrate cake discharged from the belt filter; the photograph (Figure 7) shows that the wash section of the belt filter is dry for almost all of its length, due to the high rate of filtration.

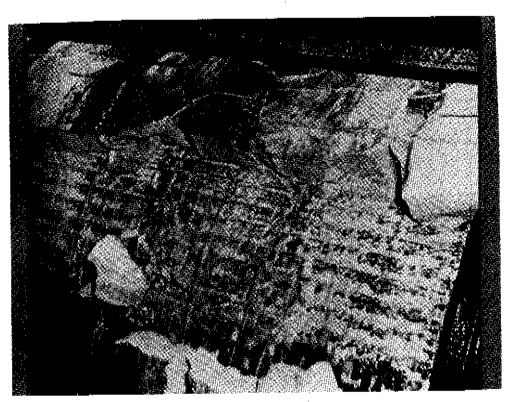


FIG 7: CAKE DISCHARGE FROM THE DIHYDRATE FILTER

The size (as indicated by Specific Surface Area measurements), shape and filtration characteristics of the hemihydrate crystals were similar for the two plants. The shape of the dihydrate crystals from the TREPCA plant was also as expected but size and filtration characteristics were considerably better than expected. This improvement resulted in a lower than expected moisture content of the dihydrate cake discharged from the belt filter. The photograph (figure 8) of the filter shows that the wash section of the belt filter is dry for almost the entire length of the section; this indicates the excellent filtration properties of the dihydrate.

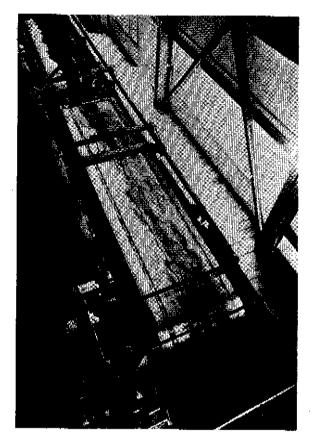


FIG 8: DIHYDRATE BELT FILTER IN OPERATION

During the period when low grade rocks were processed the physical characteristics of the hemi and dihydrate crystals remained unchanged. Filterability was affected by the higher carbon content of low grade Khourigha and Youssoufia rocks causing blinding of the filter surface.

4.2.3 Process Data and Plant Efficiencies

Plant efficiency and Product Analysis data for the months July-December are shown in Table 2 below. The data is averaged over each individual month and covers plant performance for both low and standard grade Morocco and Youssoufia rocks.

TABLE 2
PLANT PROCESS PERFORMANCE

Month	Average Monthly Product Acid Analysis		Average Monthly Dihydrate Cake Analysis			Overall Average Efficiency	
Month	° Р2 ^О 5	% SO ₍₎	% solids	Acid P2O5	I/S P2O5	Tot P ₂ O ₅	2 1016110y
July 1975	_	-	-	0.13	0.34	0.47	97.8
August	49.3	-	-	0.30	0.28	0.58	97.2
September	49.1	2,5	1,2	0.72	0.33	1.05	95.0
October	49.9	3.3	0.5	0.61	0.45	1.06	95.0
November	48.0	2,1	1,2	0.16	0.20	0.36	98.3
December	50.2	2.3	0.8	0.11	0.29	0,40	98.1

Operation during July and August was affected by somewhat irregular plant operation which affected plant efficiency performance. However, during this period efficiency results of over 98.5% were regularly achieved during consistent periods of operation.

The low efficiencies for September and October, mainly due to the high acid P_2O_5 losses in the dihydrate, were caused by a problem with the dihydrate filter feed automatic control; the filter feed was manually operated throughout this period. The problem was rectified during a shut-down at the end of October. Another contributory factor was poor washing of the hemihydrate filter cloths which caused

blinding. This has since been improved by redesigning the spray system.

Performance in November and December was very encouraging particularly as low grado rocks were being processed. Efficiencies greater than 99% were obtained at design load on several occasions.

It must be noted that although these efficiency figures are based on gypsum cake losses they are effectively the overall efficiency. As the recrystallisation step occurs in a low $^{\rm P}_2{}^{\rm O}_5$ regime and a belt filter is used on the second stage the mechanical losses are minimal.

4.3 Maintenance

Major causes of shut-down maintenance on a phosphoric acid plant are corrosion and erosion. At TRIPCA, only two pump impellers on the slurry recycle pumps have been changed in nine months operation and all other slurry and acid pump impellers will exceed their expected life. The material used for most of the pump casings and impellers is Ferralium (25/5/2/3.Cr,Ni,Mo,Cu) which has proved satisfactory in service.

Rubber lining failures have occurred on minor items of equipment and these would be judged normal by comparison with dihydrate plant experience.

High density polyethylene used for the fluorosilicic acid pumps is quite satisfactory, however, several mechanical seal failures have occurred, possibly due to the ingress of silica and/or dirty seal water. It is planned to replace these pumps with a vertical submerged type.

Glass fibre reinforced polyester pipes have been used extensively on the plant for acid lines, and these have given good service.

Reactor slurry lines are constructed from mild steel with a double rubber lining. These have given no problems apart from two areas where high grade stainless steel has now been successfully used as a substitute.

The filter cloth life on the hemihydrate filter is about two months, and one to two months on the dihydrate filter. Mechanical damage of the dihydrate cloths is the sole reason for cloth change and proposed modifications to the cloth guiding system are expected to give a longer life. Polypropylene cloths have given longer life than polyester cloths.

4.4 Scaling

4.4.1 Reaction System

There has been a complete absence of sodium fluorosilicate scaling in the hemihydrate reaction system. This may be compared with the build-up of thick layers of scale in dihydrate reactors. Slight scaling has occurred in the recrystallisation reactor in the form of a 5 mm layer of glass-like sodium fluorosilicate.

4.4.2 Memihydrate Filter System

Scaling has only occurred in the heminydrate filter system and analyses have shown that this is predominantly sodium fluorosilicate scale, not transformed heminydrate. These deposits have mainly occurred in the first filter wash, or return acid section. Scale of the same type has also been formed, but at a much lower rate in the product and cloudy acid sections of the filter. Both scales have been easily removed manually, being a soft amorphous type material unlike the normal hard, layered and glassy fluorosilicate scale which is produced in a dihydrate process. However, it is now planned to instigate a regular filter wash system which will produce an easier solution. This wash, which will be carried out at Trepca every 3 weeks, will also prevent the gradual build up of hard fluorosilicate scale in the hemihydrate filter pan arms that has occurred over nine months operation.

No scaling has occurred in the hemihydrate filter pens nor the last two wash sections of the hemihydrate filter.

4.4.3 Dihydrate Filtration System

In the dihydrate filter system where high scaling rates were anticipated, no scale has been formed.

The use of a chemical recrystallisation inhibitor to prevent scaling in the hemihydrate filter circuit has not affected conversion in the recrystallisation tank,

4.5 Comparison of Actual and Predicted Performance

A guarantee run was started in December 1975. On the sixth day of operation, a hole appeared in one of the two 1,600 tonne $^{\rm P}2^{\rm O}5$ product storage tanks. The guarantee run was abandoned, due to lack of storage space, the use of the second storage tank was not possible because of incompletion.

The data given in Table 3 compares the performance achieved with that expected from the pilot plant trials, and the expected full scale performance on standard Morocco Rock.

The average plant production rate was 100% and availability 84% compared with the expected of 88%, over the guarantee run period. The slighly low availability was due to a replacement of mechanically damaged dihydrate filter cloth. This reduced the availability from 93% to 84%.

The ${
m P_2O_5}$ efficiency, which was 97.5% based on the total ${
m P_2O_5}$ measured in the dihydrate cake discharged from the dihydrate filter. This is 1% below the expected figure.

Both free acid and insoluble $P_2 O_5$ losses were high, accounting for the lower than expected efficiency. Principal cause of these higher losses was inefficient cloth washing of the hemihydrate filter. This caused the $P_2 O_5$ concentration in the recrystallisation tank to reach a higher level than expected, which resulted in a lower recovery of lattice losses and a higher $P_2 O_5$ concentration of the free acid discharged with the dihydrate. The mechanical defect in the spray system has now been rectified and the filter washing is now effective. Product $P_2 O_5$ concentration of 50.9% average is well above the expected 50%, although the solids content is slightly higher than expected. Sulphate analysis is essentially that predicted.

20-21TABLE 3

COMPARISON OF PREDICTED AND ACTUAL PERFORMANCE

			Actual Performance	
	Pilot Flant Results	Predicted . Performance	Test Period*	Typical [‡] Ranges
Production Rate mtpd P ₂ 0 ₅		-160	156	100-170
P ₂ O ₅ Acid Efficiency (DH cake analyses) %	99,0	98.5	97,5	97.0-99.0
Product Acid Analysis P ₂ O ₅ % w/w SO ₄ : % w/w Solids % w/w	50.0 2.0 0.8	50.0 2.0 1.0max	50.9 2.0 1.4	47.5 -52. 0 1.6-2.4 0.9
Dihydrate Cake analysis Total P ₂ O ₅ % dry basis W/W P ₂ O ₅ % dry basis Insoluble P ₂ O ₅ % dry basis F % dry basis H ₂ O % dry basis	0.21 0.10 . 0.11 0.4-0.6 25.0	0.32 0.15 0.17 0.4-0.6 25.0	0.51 0.19 0.32 0.4 21.5	0.20-0.65 0.05-0.20 0.15-0.45 - 20.0-25.0

^{*} December 5th-11th 1976, 75/77 BPL Morocco Rock

 $^{^{\}frac{1}{4}}$ November, December 1976, 70/72 and 75/77 BPL Morocco Rock

PRODUCT QUALITY

5.1 Phosphoric Acid

The ${\rm P_2O_5}$ concentration of the product acid has been in the range 48 to 52% and the unclarified acid has been used in the fertilizer complex for the manufacture of Triple Superphosphate and directly in the NPK Granulation Plant. Solids content has been generally slightly higher than the 1% expected and modifications to the hemihydrate filter central valve have been made to correct this. However, the plant is frequently run with damaged filter cloths in order to obtain absolute maximum life, and this has also increased product acid solids.

A comparison of expected and actual product acid analysis is made in Table 4.

TABLE 4

EXPECTED AND ACTUAL PRODUCT ACID ANALYSIS

	Analysis (% w/w on solids free basis)		
	Pilot Plant	RMHK TREPCA	Typical Dihydrate Plant After Concentration
P ₂ 0 ₅	50.0	50.9	50
\$O ₄	2.0	2.03	4 to 5
Fe ₂ 0 ₃	0.36	0.35	0.35 to 0.45
Al ₂ O ₃	< 0.10	0.12	0.45 to 0.55
MgO	0.65	0.28*	0.4 to 0.5
F	0.80	0.67	0.5 to 0.7
Solids	1,00	1.41	4 to 5 ex evaporator

^{*} Low value due to change in rock analysis

5.2 Triple Superphosphate Manufacture

No problems have been encountered with TSP manufacture using the acid produced in the hemi-dihydrate process.

5.3 Fluosilicic Acid.

Fluosilicic acid has been produced continuously since the plant start-up. The product has been sent to drain as TREPCA cannot yet accept acid for processing into aluminium fluoride. Little data has been gathered other than to confirm that acid of the expected strength (23% $\rm HSiF_6$) can be produced. $\rm P_2O_5$ in the product was predicted at 0.5% maximum and all our data so far have given results below 0.1%. The achievement of the expected fluosilicic acid production rate is not likely to present any problems.

5.4 Dihydrate

The dihydrate effluent from the plant is transported as a slurry to a lagoon where it is available for recovery at a later date.

The gypsum crystals produced in the process possess better filtration characteristics than expected from pilot plant work. It is possible that the free acid losses in the gypsum could be even lower than the pilot plant prediction, following optimisation of the dihydrate filter operation.

A comparison of dihydrate analysis with pilot plant results is given in Table 5.

TABLE 5
PILOT PLANT AND ACTUAL GYPSUM ANALYSIS

<u>Component</u>	Analysis (% w/w dry basis)		
	Pilot Plant	RMHK TREPCA	
 ·			
P ₂ O ₅	0,2 to 0,22	0,52	
so _u	54 to 55	53.28	
CaO	32 to 33	31.42	
. Fe ₂ ○ ₃	< 0.02	. 0,0043	
Al ₂ O ₃	0,3 to 0,4	0,20	
MgO	< 0.02	0.0020	
F	0.4 to 0.6	0.40	
% Dihydrate	95 to 100	98 to 100	

6. CONCLUSIONS

- Overall the process and the plant have performed well and on occasions the expected performance has been exceeded. Failure to obtain the expected performance on a regular basis has been, generally, for non process reasons.
- Variations in rock quality has led to a reduction in plant performance and operating difficulties. However, these difficulties would have been even more pronounced in a conventional dihydrate process.
- Inefficient filter washing caused the plant to marginally fail
 its efficiency guarantee. However, plant performance was generally
 satisfactory producing acid at the guaranteed output rate and
 product quality.
- 4. Fluorosilicate scaling is less than anticipated in a dihydrate process. No problems have arisen from dihydrate scaling in the hemihydrate filtration system and complete conversion of hemihydrate to dihydrate in the recrystallisation reactor has always been attained.
- 5. There has been a significant lack of mechanical problems due to corrosion and erosion and Ferralium has proved to be an excellent material of construction. The plastic pipework, extensively used, has not given erosion problems.
- 6. The close agreement between the results predicted by the pilot plant tests and the full scale results reaffirms the excellent correlation obtained during the commissioning of the single stage process and reported to the ISMA Conference in Seville 1973. Fisons has complete confidence in its ability to predict the operating parameters of other phosphate rocks for a full scale plant. Further scale up from 50,000 tpa size plant is only limited by availability of proven items of equipment. The critical factor would undoubtedly be the hemihydrate filter which is likely to restrict maximum single train plant size to 150,000-180,000 tpa capacity at this time.

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