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THE COMMERCIAL PRODUCTION OF 50 PER CENT P_2O_5 PHOSPHORIC ACID BY THE DIRECT SINGLE STAGE HEMIHYDRATE PROCESS

By

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1. INTRODUCTION

The hemihydrate phosphoric acid process discussed in this paper was developed at the Research and Development Station of Fisons Limited, at Levington (United Kingdom). Details of the new process are fully reported in previous papers outlining the research and pilot plant investigations which led to the building of the 40 mtpa P_2O_5 plant at King's Lynn in England in 1967 (1, 2, 3). The last of these papers noted that a 60,000 tpa P_2O_5 plant was to be built for Windmill Holland NV.

This paper presents the further development and exploitation of the process by discussing the design, startup and operation of the Windmill plant, which was designed to produce 45-54 per cent P_2O_5 phosphoric acid from Togo rock by a single reaction and filtration system. The reaction system operates under stable hemihydrate conditions, producing up to 54 per cent P_2O_5 acid directly from the filter without the need for further concentration. Only a single filtration unit is required to separate the acid from the hemihydrate cake. This cake may be removed as a 'dry' hemihydrate cake, as a hemihydrate slurry, or transformed to dihydrate according to the specific means of disposal.

The Fisons process was chosen for the new plant after a thorough technical and economic survey by Windmill of both the dihydrate and 'new process' routes available. The plant was built at Windmill's Vlaardingen factory near Rotterdam in Holland and commissioned in the summer of 1970. Detailed engineering, procurement and construction of the plant was carried out by Lurgi Chemie in close collaboration with Windmill and Fisons.

2. PROCESS DESCRIPTION

The overall chemical reaction for the hemihydrate process may be expressed as:

$$Ca_{10}(PO_4)_6F_2.CaCO_3 + 11H_2SO_4 + 5\tfrac{1}{2}H_2O \rightarrow 6H_3PO_4 + 11CaSO_4.\tfrac{1}{2}H_2O + 2HF + CO_2 + H_2O$$

This represents complete release of the P_2O_5 content in the rock feed as H_3PO_4. It is the aim of all commercial processes to get as near to this ideal state as possible. As is well known, some of the P_2O_5 can leave the system as unreacted rock losses, these being generally very small; and as lattice P_2O_5 which has co-precipitated in the calcium sulphate crystal in the form of HPO_4 ions. Additional physical losses also occur during filtration when acid (P_2O_5) is left in the cake as a result of inefficient cake washing. Mechanical losses resulting from poor filter pan drainage, pump leaks, line failures, etc, are other forms of P_2O_5 losses which are often ignored, mainly because they are difficult to measure and more often result from inadequate housekeeping and maintenance, rather than from faults in the basic process design.

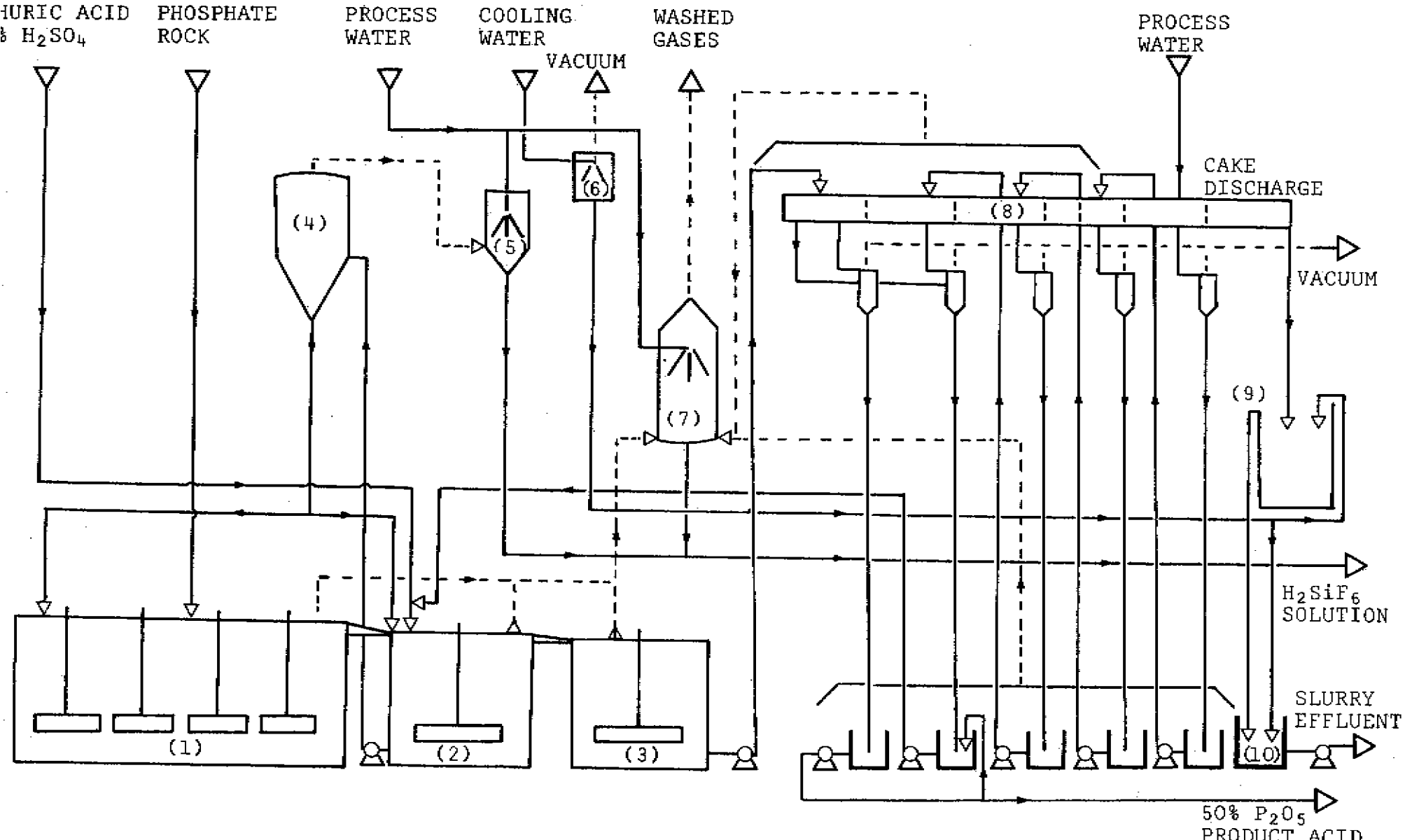
The physical resemblance of the hemihydrate process to the reaction and filtration section of the dihydrate process is immediately obvious by reference to Figure 1 - Simplified Flowsheet of Fisons Hemihydrate Process. There are, however, two important differences from the general requirements of most other phosphoric acid processes:

(a) The process requires only a coarse ground rock, 100 per cent passing through 10 mesh (approximately 1.6mm). Grinding costs are therefore reduced.

(b) The acid strength is already up to 54 per cent P_2O_5 and requires no further treatment. Intermediate storage, concentration and clarification equipment are not needed.

Phosphate rock, 100 per cent passing through 1.6mm, is fed to reactor No.1 via the rock weigh belt. This reactor has a reaction volume approximately twice that of reactor No.2. The slurry from reactor No.1 overflows to reactor No.2, to which the sulphuric acid and return acid are added. The temperature in both reactors is controlled within 98-102^{0}C by circulating the slurry through a flash cooler to remove the excess heat of reaction and dilution. Fluorine evolved from the flash cooler can be recovered in a fluorine scrubber.

The reaction conditions in both reactors are critical 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 on the full-scale plant during commissioning. In general, however, it is found that the free-sulphate level in reactor No.2 must be in the range 1.8-2.2 per



ACTOR ONE
ACTOR TWO
LTER FEED TANK

4. FLASH COOLER
5. FLUORINE SCRUBBER
6. CONDENSER
7. GAS SCRUBBER
8. FILTER
9. TRANSFORMATION TANK
10. EFFLUENT PUMPING TANK

FIGURE 1 - SIMPLIFIED FLOWSHEET OF FISONS HEMIHYDRATE PROCESS

cent SO_4. Slurry is pumped from reactor No.2 to the flash cooler. From the flash cooler the slurry is recycled to the first reactor at a controlled rate to maintain the correct SO_4/CaO level in reactor No.1. Any slurry in excess of the recycle requirement is returned to reactor No.2. This facility to recycle back to reactor No.2 is also essential for the correct control of the reaction conditions during plant shutdown and startup operations. The recycle slurry flow to reactor No.1 is controlled to give a sulphate deficiency in the reaction slurry to control the calcium sulphate precipitated in this tank. The effect of this on the final hemihydrate crystal shape and filtration properties is fully explained in the papers referred to earlier. Precipitation is completed in reactor No.2 at the higher sulphate level.

The design of both reactors was established after an extensive model testing programme, in which the flow pattern of all inlets and outlets was analysed and the efficiency of mixing optimized. Reactor No.1 is a fully baffled 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 reaction slurry overflows from reactor No.1 into reactor No.2 via a chimney and launder. The sulphuric acid is diluted with the return acid and mixed with the slurry in reactor No.2, which is also fully baffled and has a single six-bladed agitator pumping downwards.

The slurry then overflows to a third tank, the filter feed tank. This vessel allows further time for the slurry to mature before filtration by providing additional retention time during which the reaction is completed and the hemihydrate crystals are allowed to grow as the acid supersaturation is reduced. At startup slurry is normally fed to the filter when this tank is about one-third full. In this way the filter loading can be 'eased' up to its full rating whilst the tank continues to fill.

The filter is a standard horizontal tilting pan vacuum filter with three or four counter-current wash stages. Process water at between 50 and 60^0C is used for the final cake wash.

The acid from the first wash stage together with some product acid from the first section of the filter is returned to the reaction system. This is the so-called 'return' acid which is used to dilute the sulphuric acid in reactor No.2. This return acid contains the water necessary to control the P_2O_5 concentration in the

system and also contains the recycle product acid to control the slurry solids content. The flow and concentration of this return acid is controlled so that any change in the filtrate composition and rate, resulting from fluctuations in filter operation, is corrected and the return acid delivered back to the reaction system is held constant. Clear product acid from the filter is pumped to the product acid storage tanks.

The hemihydrate can be removed as a 'dry' cake after discharge from the filter, as a hemihydrate slurry, or as a transformed dihydrate slurry. The exact form of disposal will depend on local facilities, legal requirements and marginally on the type of rock used. With Morocco rock for instance the stability of the hemihydrate is such that it can be safely handled as a 'dry' cake for up to 24 hours after filter discharge. If transformation to the dihydrate form is required it is necessary to feed the slurried hemihydrate to a transformation tank. For certain rock types chemical additives may be needed.

The exhaust gases from the reaction tanks pass via a water sprayed gas scrubbing duct to a counter-current void spray gas scrubber before being discharged to atmosphere. Fumes withdrawn through the ventilation hoods over the filtrate seal tanks and the filter are also washed.

Acid spillages are collected in a floor sump and returned to the reaction system. A wash tank is installed to allow a routine closed circuit wash for the filter equipment with hot water or chemical washing if needed. No regular washing is required for reaction slurry equipment.

3. WINDMILL PLANT DESIGN

The plant was built adjacent to an existing dihydrate process plant and it was planned to use as much of the existing equipment and buildings as possible. The existing control room was used to house the hemihydrate process control panel and the existing building was also suitable for installing the new filter and associated equipment. The reactors, flash cooling, fluorine recovery, transformation tank and gas scrubbing equipment were all erected outside without building cover. The available plot determined largely the plant layout which has in fact turned out to be very successful.

Simplicity, reliability and low cost were all of the utmost importance. Preliminary evaluations had shown that a single compartmented tank as used in the Fisons

dihydrate process, did not have the same advantages for the hemihydrate process. One of the main reasons for this was the low recycle ratio and the reduced cooling duty for the hemihydrate process. The slurry flow for both these duties was of the same order of magnitude and the flash cooler circuit served both purposes.

An extensive testing programme was completed, using Togo rock. The reaction and filtration characteristics were therefore well documented. In designing the plant great care was taken to minimise the potential trouble areas which had been experienced on the King's Lynn pilot plant; such as line blockages, inadequate instrument control, inferior materials of construction and slurry aeration.

The design capacity for the Windmill plant was 210 tpd P_2O_5, giving an annual capacity of about 60,000 tonnes P_2O_5. It is worth recording that the scale-up factors involved in the project were approximately as follows:

Levington Pilot Plant	:	1
King's Lynn Plant	:	700 (40 tpd P_2O_5 - Morocco rock)
Windmill Plant	:	3700 (210 tpd P_2O_5 - Togo rock)

The model of the hemihydrate process plant prepared by Lurgi Chemie is shown in Figure 2. A view of the Windmill plant showing clearly the sulphuric acid storage tank, reactor No.1 and the flash cooling equipment is shown in Figure 3.

3.1 Reaction System

Togo phosphate rock is used on the plant with sulphuric acid at about 96 per cent H_2SO_4. The mixing requirements and reactor volume ratios of 2:1:1, for reactors No.1 and No.2 and the filter feed tank, were previously established at Levington and later confirmed on the pilot plant at King's Lynn. The total reaction volume is generally of the same order as that used for the dihydrate process and varies from rock to rock. A range of between 2.5 to 3.5 cu.m. per tonne P_2O_5 per day is needed.

As a result of the King's Lynn experience it was realised that the recycle slurry flow from reactor No.2 to reactor No.1 is critical and this is controlled automatically to within 0.5 - 1.0 per cent of the set flow.

The reaction slurry, especially in reactor No.1 into which the rock is fed, is viscous and is slow to release the reaction gases or air drawn-in by the agitation. Cooling by air injection or impingement is therefore

unpredictable since cooling could be seriously affected by the aerated slurry. There is no doubt that the cooling capacity of the King's Lynn plant was well below design because of this condition. The degree of aeration can be controlled to some extent by steady operating conditions, proper rock dispersion and agitation flow patterns and also by the choice of reactor aspect ratio. Flash cooling was therefore chosen since by combining the cooling and recycle duties it was possible to withdraw the slurry from the base of reactor No.2, by which time the slurry was de-gassed. Any trace of gas remaining in the slurry would in any case be removed in the flash cooler. At no time has there been any suggestion of cavitation in the circulation pumps or loss of vacuum in the flash cooler which would result from excessive aeration in the circulation slurry.

Another potential trouble area was the gas offtake duct from the reactors to the gas scrubber. Up to 8 per cent of the total fluorine is evolved with the reactor off-gases. The gases leaving the reactors are saturated with water vapour and deposition of silica is almost immediate resulting in rapid build up in the duct.

$$3\ SiF_4 + 2H_2O \rightarrow SiO_2\downarrow + 2H_2SiF_6$$

To overcome this problem, the gas off-take duct is designed primarily as a horizontal gas scrubber. Sprays are installed along the total length of the duct to give complete wetting of the duct wall. After some experimentation to find the right type of spray and the correct positioning of these sprays, the duct now operates free from blockages and gives a high fluorine scrubbing efficiency.

During normal operation there is no difficulty in controlling the hemihydrate slurry in a stable form. However, it was not possible to predict exactly from the Levington and King's Lynn results the effects of operating the full scale plant on Togo rock under conditions which were not normally considered as safe for stable operations. Such cases could occur, for instance, during shutdown when the slurry might be held for prolonged periods at high temperatures under conditions which would favour the formation of anhydrite. Or, failure of the rock feed to reactor No.1 could result in a rapid rise in sulphate level and destroy the crystal form. Also a reduction in temperature in reactor No.1 could lead to a thixotropic mix with the gradual precipitation of monocalcium phosphate, destroying the agitation pattern and making rock dispersion very difficult on startup.

In actual practice, the plant has proved very insensitive

to the above mentioned abnormal operating conditions which have occurred from time to time since commissioning.

The plant is adequately instrumented to prevent or rapidly correct any deviation from the normal operating condition. Interlock actions ensure full chemical control of the reaction system in the event of failure of rock, sulphuric acid or recycle flows. The flash cooler is used to cool the slurry in reactor No.2 during shutdown to prevent anhydrite formation. Steam injection is used to increase the temperature of the slurry again for startup.

3.2 Filtration Section

A standard Eimco tilting pan filter type 790 is used with wet cake discharge. Four cake washes are used instead of the more conventional three washes, mainly to offset the low water/cake washing ratio which is only about half of that available for the dihydrate process. With only three washes, the filter is operating with very little margin for operational error and as was apparent from King's Lynn data, any reduction in the water/cake wash ratio results in a loss in washing efficiency of up to 1 per cent of the overall efficiency. When producing 50 per cent P_2O_5 direct, the approximate strengths of the acid filtrates are:

	per cent P_2O_5
Product	50
2nd filtrate	30-44 (return acid)
3rd filtrate	12-15
4th filtrate	5-8
5th filtrate	0.5 - 1.0

At the normal operating P_2O_5 strength the fumes from the filter and from the filtrate seal tanks are considerable and these sections are fitted with fume extraction hoods; the vapours being vented to atmosphere via the gas scrubber. All filtrate seal tanks are agitated and fitted with liquid level control units. The return acid flow is monitored and adjusted to maintain a controlled flow back to the reaction system.

The discharged cake from the filter is slurried with recovered cooling water from the condenser units and discharged to the transformation tank. In this tank the hemihydrate is transformed to dihydrate by seeding with dihydrate crystals already present in the tank. The transformed slurry overflows to the effluent disposal tank where it is further diluted with any surplus liquid effluent before being pumped to the final disposal location.

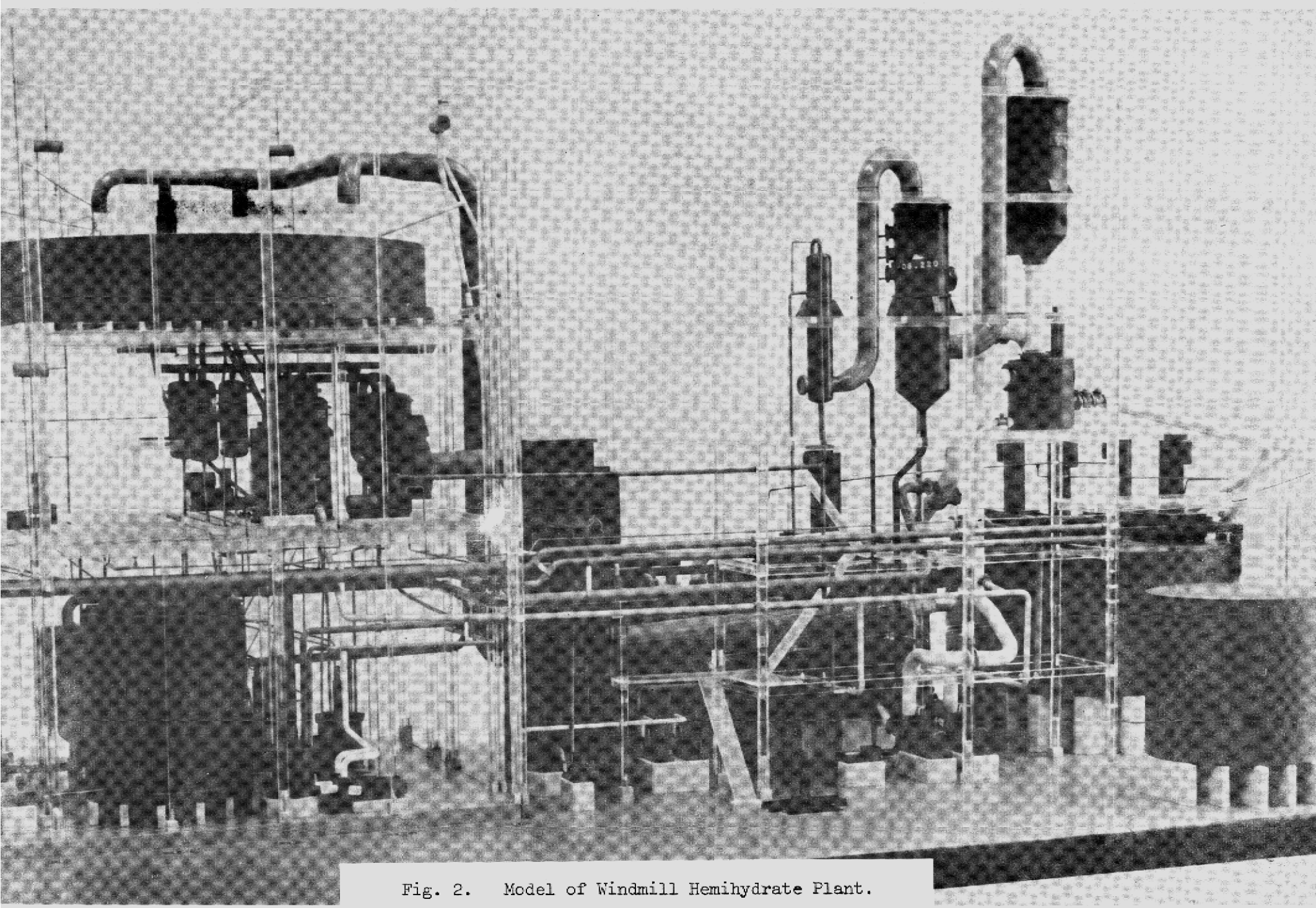


Fig. 2. Model of Windmill Hemihydrate Plant.

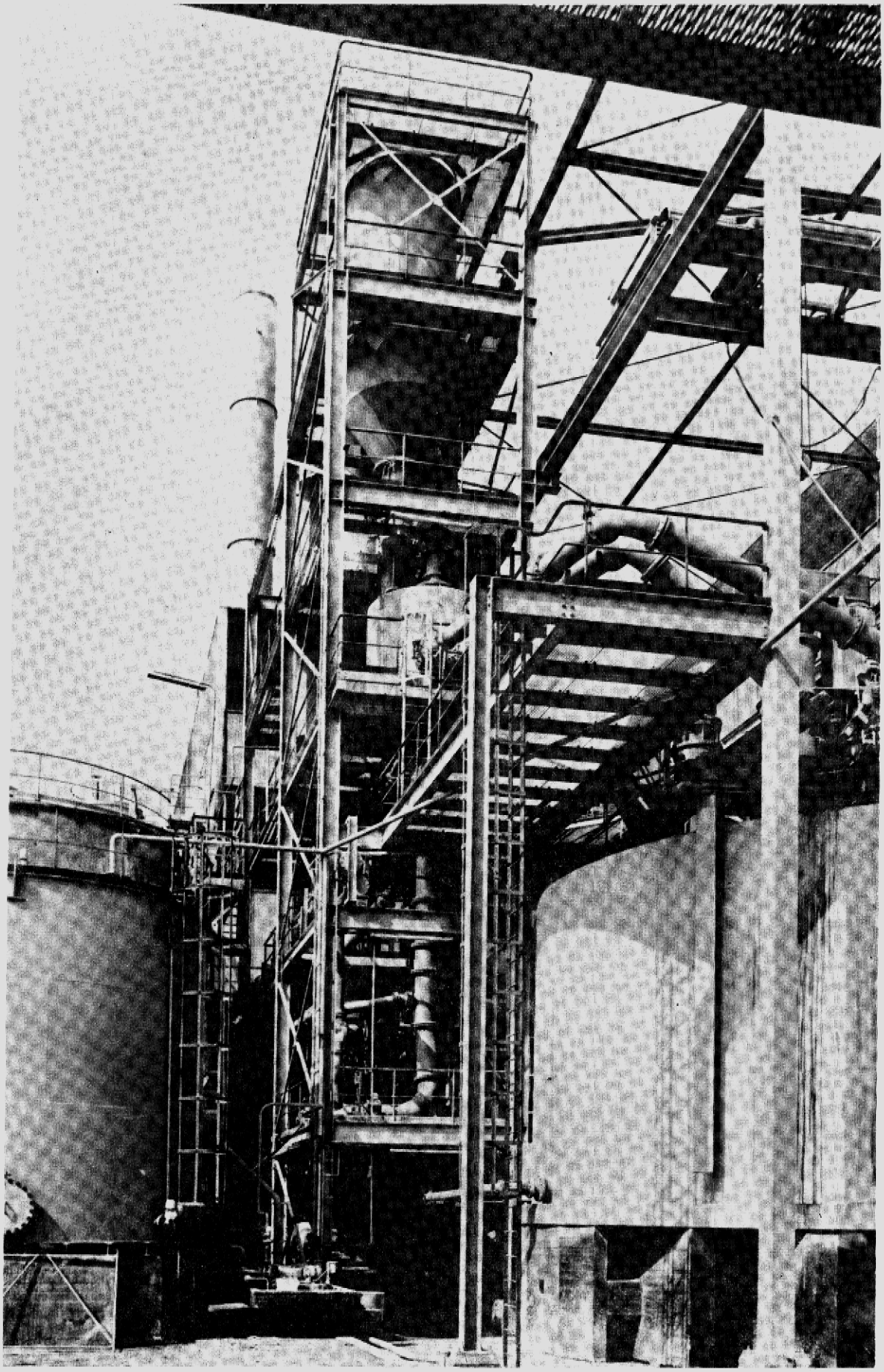


Fig. 3. View of Windmill Hemihydrate Plant showing Reactor No.1, flash

Because slurry from the adjacent dihydrate process plant is also removed by the same disposal system, it was necessary at the design stage to transform the hemihydrate to dihydrate before mixing the slurry effluents from the different plants. In this way the possibility of uncontrolled transformation of the hemihydrate at a later stage in the disposal system was avoided.

3.3 Materials of Construction

While using Togo rock in phosphoric acid processes both corrosion and erosion conditions are very severe, resulting from the high chloride and silica content of the rock. During the pilot testing stage with Togo rock, detailed dynamic and static corrosion tests were carried out on many stainless steels, rubbers and plastics.

For safety reasons and because of uncertain welding reliability, it was decided not to use plastic materials for any major item of equipment in which slurry at temperatures above 70^0C were involved. Polypropylene (reinforced where necessary) had been used at King's Lynn for both reactor and agitator design and was considered unsuitable as a result of the number of agitator weld failures and distortion of the reactor shell under the high temperature and agitator stresses. It is, however, very suitable for the construction of gas ducting, gas scrubbing units and for the smaller agitators which can be fabricated from solid sheet or bar.

The reaction tanks are of standard design for phosphoric acid plants but with a special rubber lining between the outer mild steel shell and the inner carbon brick lining.

From the range of rubbers tested, only two types of butyl rubber having adequate erosion and chemical resistance were acceptable for general use. This lining is used for most of the remaining slurry and acid handling equipment, including the flash cooler slurry circulation lines and the reaction slurry agitators. Because of the higher viscosity and density of the 50 per cent P_2O_5 slurry, erosion is less than with the dihydrate process.

A wide range of stainless steels are being used. The filter pans and the central valve are fabricated in 25Ni-20Cr-5Mo stainless steel. Similar high grade materials are used for reaction slurry pumping and lower grades such as 25:5 and 28:4 (Cr/Ni) are used for acid duties.

Most valves are either of butyl lined diaphragm type or Hastelloy C butterfly disc type.

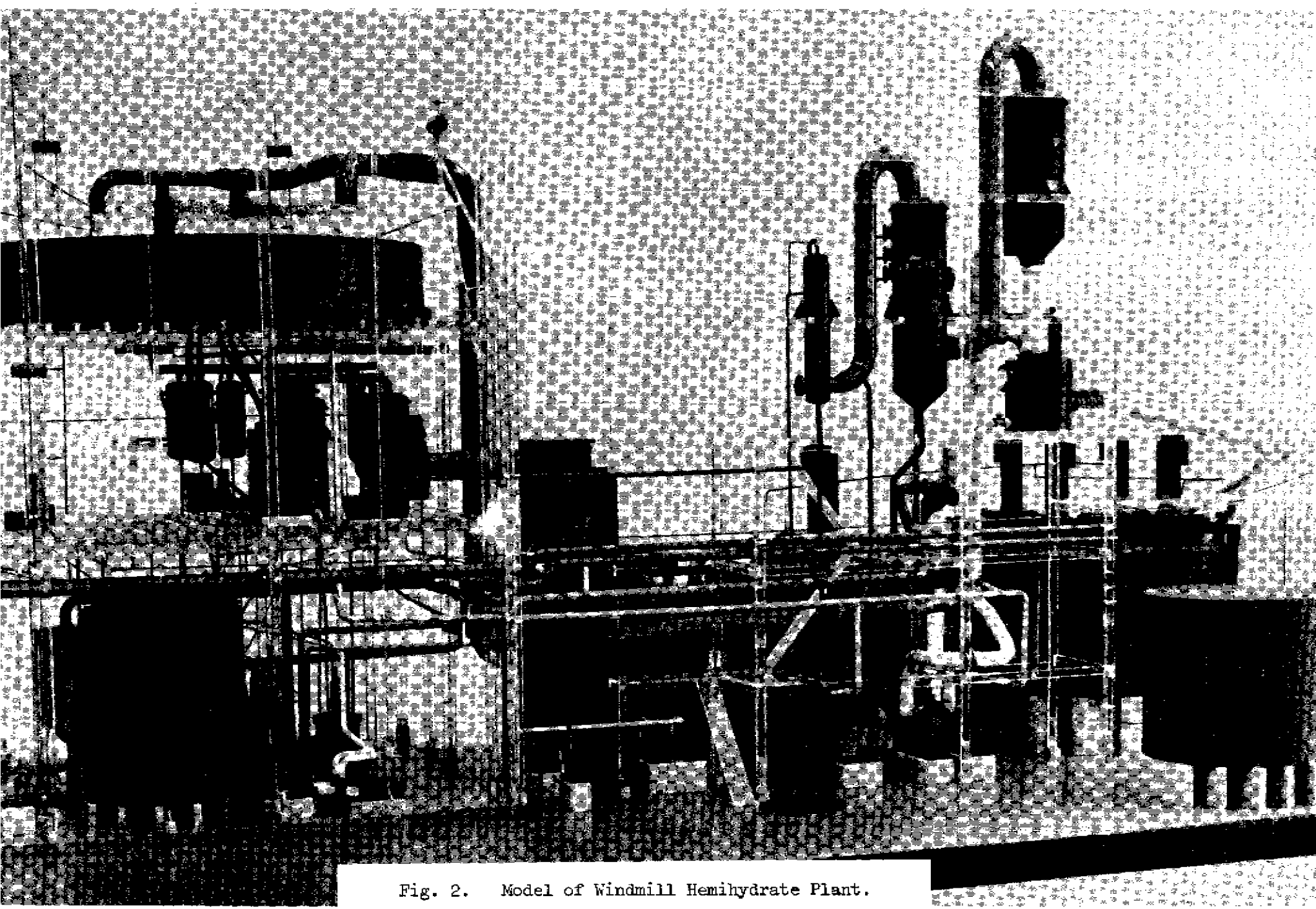


Fig. 2. Model of Windmill Hemihydrate Plant.

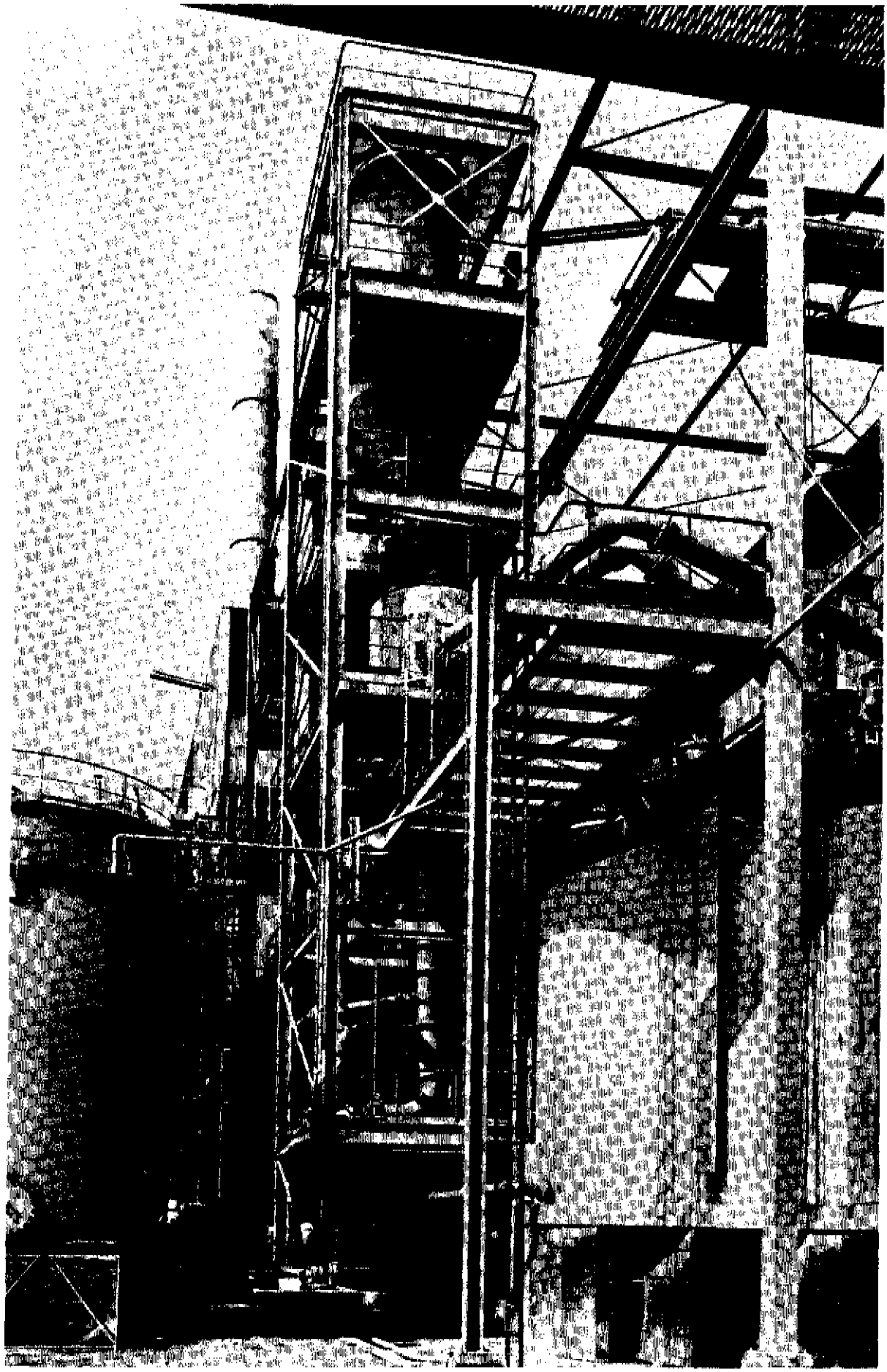


Fig. 3. View of Windmill Hemihydrate Plant showing Reactor No.1, flash

3.4 Pumps

Slurry pumps are open bladed impeller centrifugal or mixed-flow types. Acid pumps are standard centrifugal open-impeller pumps.

3.5 Instrumentation

Instrumentation is basically simple and uses equipment now normally accepted as standard for all types of phosphoric acid plant control. The main control zones are:

1) CaO level in reactor No.1
2) SO_4 level in reactor No.2
3) Reactor temperature control
4) Filter control

Rock feed to reactor No.1 is by continuous extraction weigh belt feeder and sulphuric acid feed to reactor No. 2 is controlled by magnetic flux meter. The SO_4/CaO feed to reactor No.1 is controlled by adjusting the recycle slurry flow from reactor No.2 to reactor No.1, also by magnetic flux meter. The sulphate addition to reactor No.1 can in this way be controlled very accurately. Failure of any of the above three control flows automatically stops the remaining two flows. Thus, in any emergency, the interlock actions keep the reaction slurry in its normal operating condition, ready for immediate restart.

The reaction temperature is maintained between 98-102^{0}C. The control action is by variation of the absolute pressure in the flash cooler in response to a signal from the temperature recording probes in the reaction system.

Filter control is standard; the control variables being filter speed, reaction slurry and cake wash water flow controlled by variable speed gear box, magnetic flux meter and orifice meter respectively. All the seal tanks are fitted with level controllers to ensure steady operation. A bleed control is fitted from the product acid line to the return acid tank to control the strength of the return acid to the reaction system. The return acid flow is also controlled by magnetic flux meter.

Rubber-lined diaphragm valves and stainless steel butterfly disc type valves are used throughout the plant and sizes range from 50mm to 600mm.

3.6 Fluorine Recovery

Between 30 and 40 per cent of the fluorine in the rock

feed is released during the flash cooling operation. A fluorine scrubbing unit is installed after the flash cooler to recover the fluorine from the vapours as fluosilicic acid.

4. PLANT STARTUP AND OPERATION

Because of the importance of starting-up a new process, designed from data obtained from pilot plant work only, a very high priority was given to reliability of equipment such as pumps, instrumentation, electrics and general mechanical soundness of all items before the chemical commissioning of the plant was started. A period of about two weeks was allocated to water testing. During this period every item on the plant was checked, calibrated and run continuously to locate and correct any defects.

Togo rock, screened through 1.6mm, was first fed to reactor No.1 on June 11, 1970, at 50 per cent design loading. The hold up in the system is several hours and some time was spent checking various items as slurry reached them for the first time. There were no major snags however and slurry was fed to the filter the following afternoon. Filtration was immediately seen to be satisfactory and the plant was kept at 50 per cent loading for the next five days to allow final adjustment of instrumentation and to allow the operators to familiarise themselves with the new process control conditions and analytical control methods which differed slightly from the dihydrate process.

The plant was then shutdown for a first inspection and the slurry filtered off. Slight modifications were made to the filter central valve settings and a damaged agitator was changed in reactor No.1.

When the plant was restarted the rock input was increased to full load within two days.

4.1 Reaction and Filtration Control

Control of the reaction system proved better than expected. Having set the main control flows, the plant runs automatically with only minor reset adjustments necessary to maintain the set control conditions in the reactors, at the following levels:

Reactor No.2 : Per cent SO_4 (1.8-2.2 per cent)
Per cent P_2O_5 (50 per cent)
Per cent Solids (-)
Temperature (100^0C)

The per cent CaO in reactor No.1 is set at the level determined from the pilot plant trials and is controlled by the flow of recycle slurry from reactor No.2. Quite wide deviations from the set control limits were experienced on occasions but these had little effect on the plant performance.

The response to changes in the SO_4/CaO feed to reactor No.1 was so predictable and consistent that it was possible to carry out a series of tests during commissioning to optimise this ratio and also the SO_4 level in reactor No.2. The final operating conditions chosen were only slightly different from those predicted from the Levington pilot plant testing. The relationship between crystal surface area versus SO_4/CaO feed to reactor No.1 is shown in Figure 4 for Togo and Morocco rocks.

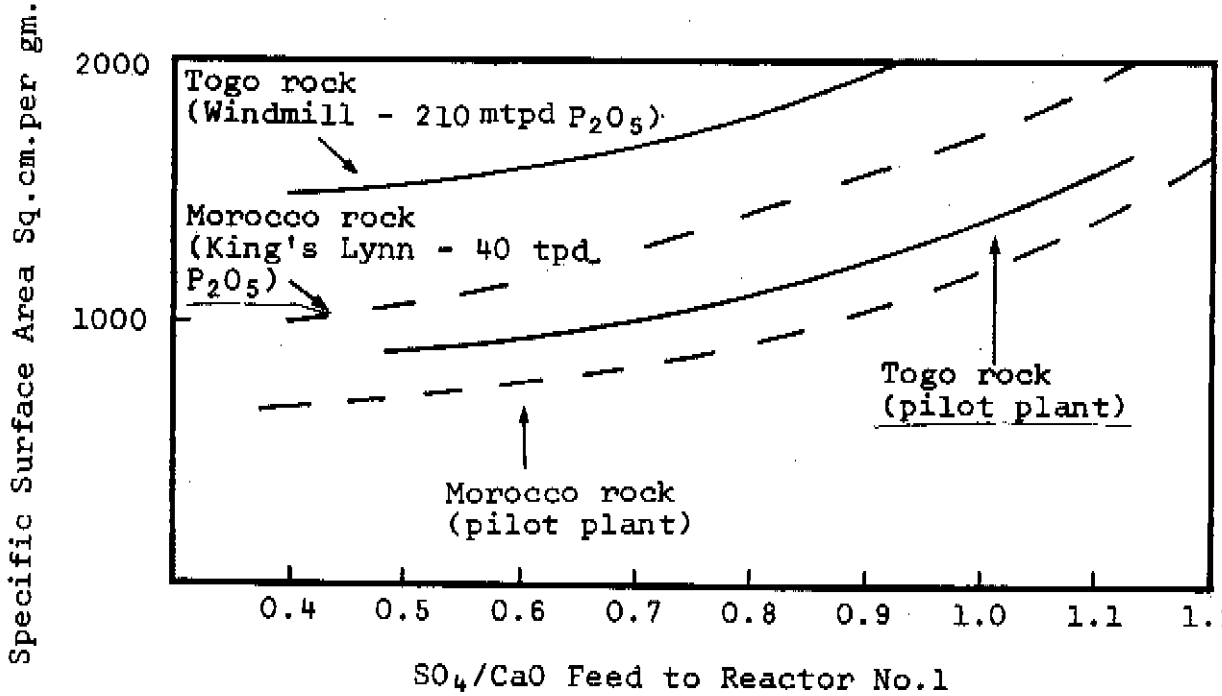


FIGURE 4

Relationship Between Specific Surface Area and SO_4/CaO Feed Ratio

The increase in surface area for Togo rock was as predicted from results of the scale-up on the King's Lynn plant. The

filtration rate and hence filter loading at Windmill was as expected. This accuracy of scale-up has now been proved by both the Morocco rock trials at King's Lynn and with Togo rock on the Windmill plant. The filter normally runs at a loading of 4 tonnes P_2O_5 per day per square metre of active filter area. This may not seem high when compared to the claims of 6-8 tonnes P_2O_5 being handled on some dihydrate plants, but it should be remembered that the viscosity of the acid filtrates is generally higher than for the dihydrate process and the filter is using one additional wash stage. The hemihydrate filtration rate varies less with different types of rock, is more predictable and less likely to daily variations in filterability than for the dihydrate process.

The filter cloth supports are of polypropylene grid construction and give rapid drainage from the cake and cloth. The cloths are also polypropylene, monofilament construction, and have a life of one to three months. The cloth specification is still being investigated and the actual life of the cloth depends on the particular type used and the frequency of removal for inspection. The need for replacement is due entirely to mechanical damage and no cloths have yet been replaced because of blinding.

4.2 Scale Deposits

The reaction system has remained completely free from any scale deposits. When inspecting equipment it is normally only necessary to wash-down the rubber or brick lining and to remove any sedimentation from the base of the equipment.

By producing 50 per cent P_2O_5 acid directly from the filter, the product and return acid sections are operating under stable hemihydrate conditions and the rubber lined receivers and filtrate equipment remain clear to the rubber.

If no special precautions are taken, the remaining filtrate strengths are low enough to allow transformation to dihydrate to take place, causing scaling in the equipment. With the use of special chemicals, scale formation is prevented and also in these stages the fi filtrate equipment remains clear to the rubber.

4.3 Fluorine Distribution

The distribution of fluorine predicted from pilot trials and the actual plant data are given in Table 1, as

percentages of fluorine in the rock feed. For comparison, data are also given for the distribution in a typical dihydrate plant. It is seen from Table 1 that the available fluorine which can be recovered is about the same for both processes. However, it is not normally considered worthwhile to attempt recovery from the gas scrubber of the dihydrate process but this is certainly worthwhile for the hemihydrate process by combining the recovery circuits of the flash-cooler scrubber with the reactor off-gas scrubber. The recovered fluosilicic acid from the Windmill hemihydrate process plant is used on-site for the production of fluorine compounds.

TABLE 1

Fluorine Distribution From Hemihydrate and Dihydrate Processes Using Togo Rock

(per cent w/w)

	Pilot Plant Hemihydrate	Windmill Plant Hemihydrate	Typical Dihydrate
Acid	12	12	15
Cake	50	50	45
Reactor off-gases	38	8	5
Flash cooler		30	-
Concentration unit	-	-	40

4.4 Effluents

Gaseous: The Dutch authorities allow a maximum emission of 0.4 kg F per hour for this plant. All vapours from the reaction system, filter and seal tanks are collected and thoroughly cleaned in order to meet this requirement.

Liquid: There is only one liquid effluent stream since all waste liquors are pumped to the effluent disposal tank, to dilute the dihydrate slurry. This is then transferred by pump to the disposal system.

Solid: Although the hemihydrate cake is removed at Windmill as a transformed dihydrate slurry, a typical hemihydrate cake analysis from the filter, on a dry basis, corresponding to a product acid strength of 50 per cent P_2O_5, is:

	per cent w/w
P_2O_5 (total)	1.50
(water soluble)	0.10
(rock)	less than 0.1
CaO	36.40
SO_4	59.00
F	1.20
Fe_2O_3	0.08
Al_2O_3	0.65
SiO_2	1.60
Free H_2O	20

5. PRODUCT ACID QUALITY

The acid is very clean with less than 1.0 per cent suspended solids and lower sulphate and aluminium contents than the dihydrate acid. Typical analyses of hemihydrate acids from Togo and Morocco rocks are given in Table 2. For comparison the analysis is also given for Togo acid from a typical dihydrate process after concentration and clarification.

TABLE 2

Typical 50 Per Cent P_2O_5 Acid Analyses

(Per cent w/w)

Characteristic	Togo Rock		Morocco Rock
	Hemihydrate (Ex Windmill)	Dihydrate (Ex Windmill)	Hemihydrate (Ex King's Lynn)
P_2O_5	50.0	50.0	50.1
CaO	0.5	0.4	0.2
SO_4	2.0	4.0	2.0
SiO_2	0.1	Nil	0.4
F	0.7	0.8	0.7
Fe_2O_3	1.6	1.5	0.3
Al_2O_3	0.3	0.9	0.1
MgO	0.2	0.3	-

The product P_2O_5 concentration can be controlled between 45 and 54 per cent P_2O_5 by adjusting the cake wash water flow. No other changes are necessary other than to adjust the return acid flow and concentration to compensate for the changed water flow.

The product hemihydrate acid is stored without further treatment and has presented no problems in storage or during transport. Using this acid also gives a significant upgrading of intermediate products such as monoammonium phosphate, when used in place of unclarified dihydrate acid. The analysis of monoammonium phosphate is given in Table 3.

TABLE 3

Analysis of Monoammonium Phosphate (On dry basis)

	(per cent w/w)	
	N	P_2O_5 Total
1. Using Unclarified Dihydrate Acid	10.5	53
2. Using Hemihydrate Acid	11.5	58

6. PLANT MAINTENANCE AND AVAILABLILITY

It should be borne in mind that the Windmill plant is the first commercial hemihydrate process plant in operation. Maintenance costs, based on materials and direct labour including some alterations in the plant equipment, were approximately 10 per cent and 7 per cent of the installed equipment costs for the first and second years of operation. For a dihydrate process plant, including the high maintenance concentration units, maintenance costs are also 6 per cent - 8 per cent of the installed

equipment cost. Since the actual capital investment for a hemihydrate process plant is approximately 30 per cent less than an equivalent dihydrate process plant, including rock grinding, concentration and clarification, the maintenance costs are actually less for the hemihydrate plant.

Planned downtime comprises an annual shut-down of 14 days and one day every 4 weeks for cleaning and some repairs.

Allowing an additional 10 per cent for unforeseen downtime, the plant is available for production for over 300 days per year.

7. PRODUCTION COSTS

The predicted efficiency for the Windmill plant was 93.4 per cent based on the measured cake losses from Togo rock, when producing 50 per cent P_2O_5 acid directly. The efficiency on the full scale plant is between 93.4 - 94.0 per cent.

The following evaluation is based on 73/75 BPL Morocco rock. The efficiency for Morocco rock is well proven from Levington pilot plant and the King's Lynn production data. When making 50 per cent P_2O_5 the efficiency for Morocco rock is 94 per cent.

The following evaluation compares the hemihydrate process with a high efficiency 'classical' dihydrate process.

Basis of Evaluation

Rock	:	Morocco 33.5 per cent P_2O_5
Sulphuric Acid	:	93-98 per cent H_2SO_4
Product Acid Strength	:	50 per cent P_2O_5
P_2O_5 Efficiency	:	94 per cent for hemihydrate unit based on measured cake losses. 96.3 per cent for dihydrate unit based on measured cake losses. (96.8 per cent less 0.5 per cent for intermediate storage and concentration unit losses).
Capital Costs	:	Battery limit costs for phosphoric acid plant only, excluding rock, sulphuric acid and product acid storage. Fluorine recovery is not included.

Capital Costs (cont'd..) : Costs for the dihydrate process include estimates for rock grinding and clarification.

Overhead Charges : These vary considerably from site to site. Only direct operating labour and maintenance costs are included.

TABLE 4

Comparative Production Costs of Phosphoric Acid by the Dihydrate and Hemihydrate Processes

Basis: 50,000 tonnes per year P_2O_5 as 50 per cent P_2O_5 from Morocco rock

A. VARIABLE COSTS		Dihydrate		Hemihydrate	
Extraction Efficiency Based on Measured Cake Losses, per cent		96.3		94.0	
	Unit Cost Dfl	Units per Unit of P_2O_5	Cost Dfl per Unit of P_2O_5	Units per Unit of P_2O_5	Cost Dfl per Unit of P_2O_5
Phosphate Rock (tonne)	55.00	3.10	170.50	3.17	174.35
Sulphuric Acid 100 per cent (tonne)	55.00	2.73	150.15	2.77	152.35
L.P. Steam (tonne)	8.50	2.00	17.00	0.20	1.70
Fresh Water (tonne)	0.35	7.00	2.45	6.00	2.10
Cooling Water (tonne)	0.04	37.50	1.50	22.00	0.88
Power (KwH)	0.05	115.00	5.75	125.00	6.25
Total per tonne P_2O_5			347.35		337.63
B. CAPITAL COSTS (Dfl)		10,000,000		7,000,000	
Depreciation, interest, taxes and Insurance charges @ 20 per cent		2,000,000		1,400,000	
Maintenance @ 7 per cent		700,000		490,000	
Operating Labour		200,000		120,000	
Total per tonne P_2O_5			58.00		40.20
C. PRODUCTION COST PER TONNE P_2O_5 Dfl			405.35		377.83

Notes

1. The above costs include only those costs which are relevant for a comparison between the two processes. Additional expenditure such as off-site costs, supervisory labour, overheads, capital charges for working capital, etc. which are common to both processes are not included.

2. Operating Labour requirements:
   Dihydrate Process : 4 men per shift
   Hemihydrate Process : 2 men per shift

3. No allowance has been made for the use of defoaming agents which may be required for certain rock types.

8 FUTURE DEVELOPMENTS

Joint efforts to optimise the Windmill plant by carrying out a development programme both on the plant and at the Levington Research Station of Fisons Limited are continuing. Investigations are in hand to test a wide range of rock types and a Two Stage Hemihydrate/Dihydrate Process is now available for certain rock types such as Moroccan and other Mediterranean rocks. The two stage process offers a higher efficiency and a very pure by-product calcium sulphate (gypsum).

At present it is possible to design single stream units of up to about 700 mtpd P_2O_5. Above this capacity the cost of downtime and loss of flexibility makes the economy of single stream units rather uncertain.

9 CONCLUSIONS

(1) After more than 24 months continuous operation the plant is now proven as a viable unit, operating under stable hemihydrate conditions. It offers the following advantages:

(i) Capital investment is reduced because:

(*) Only a coarse rock is required eliminating expensive grinding equipment

(*) Up to 54 per cent P_2O_5 acid is produced directly, eliminating the need for concentration and clarification units and intermediate acid strength storage tanks.

(ii) The product acid contains less than 1.0 per cent suspended solids, up to 2.0 per cent SO_4 and substantially less aluminium impurities than can be obtained from the dihydrate process. The acid can be stored and shipped without further treatment.

(iii) The process is flexible in that it can produce acid of any concentration between 45 and 54 per cent P_2O_5 without equipment modification.

(iv) The flowsheet is simple with only the same equipment as normally required for the reaction and filtration section of a dihydrate process. Only one filter is used.

(v) The unit cost of P_2O_5 is less for most rock types than that of acid produced by the dihydrate process.

(2) The pilot plant rock testing techniques used for rock evaluation are comprehensive giving sufficient data for accurate design of full-scale units.

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DISCUSSION

MR. C.H.M. VINKE (Windmill Holland, N.V. Netherlands): At the ISMA Technical conference in Brussels in 1968, a paper was presented by Fisons Ltd. U.K., describing a process for the production of concentrated phosphoric acid without evaporation and with hemihydrate as a byproduct from the filter.

At that time an evaluation was made of work on a pilot plant and on a small production unit of 40 tons per day P_2O_5 using Moroccan rock.

In the early days of 1969 Windmill Holland decided, after thorough investigation of the available processes, to select this one for the extension of their phosphoric acid production at Vlaardingen in Holland.

This paper describes our experience with the plant, which came on stream in July 1970.

It was designed for a capacity of 60,000 tons of P_2O_5 per year. The main rock used is Togoland rock and an extensive pilot plant run was carried out in the Fison research facilities at Levington to establish the correct design features for this type of rock.

All the important data collected during these pilot runs were confirmed in commercial plant operation, which proves that Fisons pilot plant rock testing techniques give sufficient data for the accurate design of commercial units.

The most important advantages of this process are:

1. It requires only a coarse ground rock, 100 per cent passing through 10 mesh (approximately 1.6 mm). Grinding costs are therefore reduced.

2. Sulphuric acid is used at 96 per cent. No dilution, cooling and intermediate storage of diluted sulphuric acid is required. Compared with a dihydrate process and starting both from 96% sulphuric acid, the overall heat balance over the process shows a reduction in the excess heat to be dissipated of about 30%.

3. The phosphoric acid from the filter has a concentration of 45 to 54 per cent, depending on the concentration required for further use. No evaporation is necessary. So an important process step has been eliminated.

4. The quality of the acid is superior to acid made from the same rock in a dihydrate process. Sulphate content is only 2 per cent, enabling end products to be made of higher plant food concentration. Also the aluminium content is reduced appreciably. With Togo rock the aluminium oxide content is reduced to 0.3 per cent as compared with about 1 per cent in dihydrate acid.

   During the concentration of dihydrate acid calcium sulphate and silica fluoride are precipitated. During storage, additional iron and aluminium phosphates precipitate. To obtain pure acid, a curing time of two weeks is required before the impurities can be removed.

   No additional post precipitation occurs with the hemihydrate acid and no curing time or impurity removal system is required.

5. The capital investment in a hemihydrate plant is about 30% lower than for a dihydrate plant.

Slide - Description of the plant (picture of the model). After two years experience, I will now review some important features of the plant.

1. Product concentration

Slide - Concentration graph.

The main feature of this process is the production of concentrated acid straight from the filter. The minimum concentration required for further use by Windmill Holland is 48 per cent.

As you will see from the graph the concentration produced has always been according to this spec. and in fact the plant is operating with great stability and concentration is easy to control.

2. Efficiency

One of the drawbacks of the single stage hemihydrate process is its fairly low efficiency.

Slide - Cake efficiency graph.

Based on the measured cake losses, efficiency can be controlled between 93.4 and 94.0 per cent. As you will see, it took us a few months to bring efficiency to the level predicted by the pilot plant work.

The fluctuations in efficiency are caused by several tests which were conducted in order to optimise the process conditions.

The main part of the losses is so called lattice P_2O_5, which cocrystallized as dicalcium phosphate in the hemihydrate crystal. To reduce this loss, the hemihydrate has to be transformed into dihydrate in an additional transformation and filtration stage.

In the pilot plant such a second stage has already been developed for Moroccan and other Mediterranean rocks. Development work is continuing on a wide range of rock types.

3. Crystal size and filtrability

There is no doubt that one of the main reasons which has so far prevented the successful development of a concentrated acid process is that the right process conditions to promote satisfactory crystal growth have not yet been established. In this process crystals are formed that filter well.

Slide - Picture of crystals.

The filter normally runs at a loading of 4 tonnes P_2O_5 per day per square metre of active filter area. This may not seem high when compared with dihydrate plant filtration rates, but it should be remembered that the viscosity of the acid filtrates is higher than for the dihydrate process and the filter uses one additional wash stage.

4. Crystal stability

One of the risks of running a hemihydrate process is the uncontrolled recrystallization into dihydrate, causing scaling of equipment, pumps and pipelines.

In our plant the reaction section has always been free of any scaling. In the filtration section the lower filtrate strengths are low enough to allow transformation to dihydrate to take place, causing scaling in the equipment.

After plant start-up we went through a period which caused us quite a lot of headaches. Frequent removal of scaling by mechanical cleaning and washing was required in the lower strength filtrate section. However, this problem has been successfully tackled, and scaling no longer occurs. It should be mentioned that no scaling or clogging of filter cloths was noticed either by gypsum or by silica compounds.

3. Production rate

Slide - Production rate graph.

With a planned production rate at 100%, you will see that this level was reached in January 1971, and has increased ever since. The fluctuations are partly caused by trial runs to optimize the plant.

6. Plant control

The plant proved to be very obedient to the control system installed and any undesired deviation from the required process condition is very easy to redress. Having set the main control flows, the plant runs automatically with only minor reset adjustments.

Slide - Picture of control panel.

The operator sits at the electric switch panel, which serves two phosphoric acid plants and the automatic rock feed system from rock storage. In front of him is the control panel of the hemihydrate plant, which is compactly arranged and easy to survey.

7. Materials of construction and plant maintenance

In addition to the more severe conditions in a hemihydrate plant compared with a dihydrate plant, we had to take into account the relatively high chlorine level in Togo rock.

Because of this chlorine content we did not choose stainless reaction slurry agitators, which are perfectly satisfactory for the more usual rock types, but installed instead rubber lined steel. Erosion of this rubber lining is still too high which forces us to renew the lining of the blade tips every 6 months of operation.

In addition, the choice of material for the slurry pump impellers has not yet been solved to our full satisfaction. All the other equipment has a life time according to our expectations or better.

It is always rather difficult to find a proper yardstick for maintenance costs comparison, as every company has its own system of allocation and calculation of labour costs.

We feel that a fair estimate of materials and direct labour costs is 7% of the installed equipment costs.

8. Stream factor

As quoted in the paper the stream factor of the plant is 83%. Sometimes stream factors are calculated taking into account only unplanned or unexpected downtime.

In this case 83% is defined as the number of hours

per year that raw materials are fed into the plant divided by the total number of hours per year.

One has to bear in mind that this plant is the first of its kind and it is quite obvious that the design of the next plantwill be somewhat different. By changes in the design this stream factor can be improved by another 3 or 4 per cent.

9. Economics

In the paper, a comparison is given of the production costs of the hemihydrate and the dihydrate process. It is obvious that such a comparison can vary widely in different cases, depending on unit costs, local circumstances, availability of steam, etc.

MR. J. FROCHEN (Compagnie Française de l'Azote): Since the manufacture of phosphoric acid started, much research and testing has been carried out with the object of devising a simple and economic method of obtaining a product with as high a concentration as possible. To sketch in the background, I shall merely recall the work of Larson and Nordengren, who both achieved some results but results which did not seem to lead anywhere. What they did, above all, was to determine accurately the operating conditions required in order to achieve results. They showed, in particular, that in order to exceed the normal 30-32% concentration it was necessary to work at high temperatures, precipitating the calcium sulphate in the form of hemihydrate or anhydride. But it was when the principles had been established that difficulties began. First of all the technological difficulties when working at high temperatures with high P_2O_5 concentrations, the problems of resistance of materials, lining reactor tanks, metals for agitators and pumps, fabrics for filters etc., were virtually insoluble at the time. Then there were working difficulties in the process itself. In order to obtain the right filtration speeds, the crystals had to be large enough, particularly as the viscosity of the acid increases considerably with concentration. And at that time, although the method of crystallizing dihydrate was well known, this was not true of hemihydrate and filtrations speeds were very low.

For all these reasons, processes which provided a direct means of obtaining acid at high concentration were abandoned, and for many decades technicians dealing with phosphoric acid concentrated on developing the dihydrate process.

We must therefore congratulate the Fisons/Windmill team for tackling a problem generally considered extremely

difficult if not insoluble, and for achieving results which seem to be of interest. However, I shall raise an initial question: when we examine the Windmill flow-sheet, nothing fundamental distinguishes it from the old hemihydrate installations or even the traditional dihydrate installations. What, then, is the secret of its success? Is it just a better knowledge of the operating conditions and more accurate adjustments, or are we to assume that not everything has been set out on the flow-sheet? Another question: if Fisons consider their process to be particularly economical, why have they not converted their existing installations or built a new unit? A last general question. The Windmill workshop uses Togo phosphate. The pilot tests were also conducted with Morocco 75. Have other tests been carried out with different phosphate rocks, and if so, what have the results been?

Having said this, there are a few matters of detail: What is the temperature of the slurry on reaching the filter? What happens in the case of a long shutdown? Do the risks of conversion of hemi-hydrate to dihydrate in the slurry not make it necessary to take precautions such as diluting or heating the slurry? Do not such precautions result in the manufacture, on resumption of working, of an appreciable quantity of more dilute acid? Is it possible to know what type of chemical is used to prevent scaling of the filtration circuits? I am not casting doubt on the analyses of the acids produced by the two processes, but I cannot believe that a difference in impurity content of less than 3% in the acid results in a gain in purity in excess of 8% in the monoammonium phosphate. In fact Fisons contradict themselves, for in their publications concerning the manufacture of Minifos with ordinary acid, they give 11.5 - 55.5 and not 10.5 - 53.

May we know how the SO_4/Cao ratio is measured?

After these questions and critical assessments of detail, I come to the economic aspect of the process. The cost comparison of the two processes calls for some comment.

Fisons say that, with their process, grinding of the phosphate is not necessary, but they need a tank volume of between 2.5 and 3.5 m 3 per tonne of P_2O_5, and it is certain that with such a volume, whatever the process, grinding is superfuluous. In addition, the filter is almost twice as large, more washes are required, and therefore more pumps than are required in a traditional installation. Under these conditions a large part of the saving in investment, equipment maintenance and electricity disappears.

The same applies to operating personnel. If we take into account the comment made above, there is no saving on grinding. We must therefore consider only concentration, and in most plants this is carried out without any increase in the labour force by the personnel making the 30% acid.

As for the stated yield of 94%, if this is really obtained industrially, all losses included, I warmly congratulate Fisons and Windmill. I did not think it possible with such a process, whereas 96.3% for a traditional process in a unit which is not overloaded is possible.

In this connection can Fisons give a breakdown of losses into

water soluble,
citrate soluble,
insoluble
and mechanical losses?

Having said this, for the 3 reasons set out briefly above I do not think the difference in cost is very appreciable, nor is it greater than that accounted for by the imprecision inherent in this type of calculation.

There remains of course the simplification of the installation, and a consequent reduction in the number of problems which may arise, and I am far from regarding this as negligible. You will, therefore, certainly be in agreement with me in congratulating Fisons and Windmill on the importance of their work and the results achieved. It is a long time since we have seen, in phosphoric acid manufacture, an industrial set-up which is both successful and off the beaten track. I have been lengthy, but the time I have used shows the extent of my interest in examining Fisons' process. I trust I have induced you to share it.

MR. VINKE: This a joint presentation by Windmill and Fisons and a number of your questions will be answered by Mr. Robinson whom I will now ask to deal with your first three questions which concern the general background of the hemihydrate process.

MR. N. ROBINSON (Fisons Ltd. U.K.): It is true that there is very little difference in the flow-sheet between the hemihydrate and dihydrate processes. To my mind this does not imply a disadvantage but means that the process is simpler. You ask the reason for our success. Could I answer this by giving some answers you yourself have already given: a better control of reaction conditions

which affect the process variables. We have done a considerable amount of pilot plant work which led up to this development and two papers were presented at various stages. We feel that it was by doing all this preliminary work and really understanding how variables affect the process that we were able to decide upon our operating plans and this is the real reason why we decided to go ahead with full scale work and is the reason for our commissioning and operating success.

You ask why Fisons did not convert existing installations. This is very simple. Since we developed this process we have not increased our capacity. We did in fact consider modifying one of our existing processes at Immingham to the hemihydrate system but this was just not economic because it would have meant changing all the materials of construction, altering all the reaction volumes, changing patents etc. It was just not on. This was the situation as I understand it.

With regard to the different rocks, we initially developed the process on Moroccan Rock, and this was then taken to the stage of the 40 ton/day plant which has been described.

We tested Togo rock on the pilot plant and Windmill uses Togo Rock. On the pilot plant we have also processed Kola rock, Taiba, several types of Florida rock including calcined and uncalcined from different sources and all these have their own characteristics. We cannot assume in this process that one rock, just because it has an identical analysis will behave in exactly the same way as another. We make a point in fact of testing every rock fully on the pilot plant before we make predictions on this.

MR. FROCHEN: Did you obtain the same efficiency?

MR. ROBINSON: No. Efficiencies vary. For example, with Morocco rock, we expect 94%; Togo 93.4, Florida depends on the source and whether it is calcined or uncalcined as I mentioned. This can be anywhere between 90 and 93, Kola 93.2-93.5 and Taiba 91-91.5. And this assumes 50% phosphoric acid because the efficiency depends a great deal on the P_2O_5 concentration of the acid.

MR. VINKE: The next question was about the temperature of the slurry feed to the filter. This is about 95°. It is only a few degrees lower than the

temperature of the reactors.

On the subject of shutdown, in the case of stops of up to, say, 48 hours, you just leave the plant as it is and it slowly cools down to about 70°C. At this point we re-heat the slurry before re-starting the plant with steam.

You asked about the quantity of dilute acid, which is delivered on re-start. This is presumably based on the assumption that one of the precautions would have been to dilute the slurry. As this is not done, the acid is again 50% or 48% immediately after start up.

Your next question was on the nature of special chemicals used in the plant to prevent scaling in the last filter section. We cannot reveal this. We can only say that they are very common chemicals, very cheap and readily available on the market and are not chemicals which could upset the economics of the process.

As to the difference in impurities between the hemi- and dihydrate acids, you account for 3% and you ask how it is possible to arrive at an increase of 8% in plant food in the MAP product made out of it. In the analysis of the MAP given in the paper, we took an MAP made from unclarified dihydrate acid, which means that we also took the solids present in that acid because there had been no clarification. We also wanted to compare with an acid which had not had after-treatment, since the hemihydrate does not require any after-treatment. Again, the sulphate level is about half and the aluminium content is also low. On that basis, you arrive at the analysis shown, 10.5 - 53 for the unclarified dihydrate acid and 11.5 - 58 for the hemihydrate acid. The SO_4/Cao ratio is measured by the analysis of the calcium oxide in the first reactor and also the amount of SO_4 entering the first reactor after recycle and then quite a simple equation gives you this ratio.

You then moved to the economics of the process. We have not said in this paper that the same reaction volume was required for the dihydrate process; we have compared here a dihydrate process where rock grinding is familiar and quite a normal technique with the hemihydrate process. So I agree with you that you should also allow for this rock grinding in going for a much larger reaction volume than in a dihydrate process, but we feel that the difference in investment will not be the same as is suggested in this paper. Secondly on the labour required; in a dihydrate process we say 4 men per shift and in the hemihydrate process 2 men per shift. I agree with you that the difference is too great, and is more or less based on the Windmill situation where the evaporation in the dihydrate system we also operate is in a separate building.

Therefore we have to use additional manpower. Apart from that I would say that, in our opinion, at least one additional operator will be required to handle the evaporators and the system for removing sludge from the acid.

Your last question was on the efficiency of the process. You have seen the slides. I agree that these are all measured cake losses and any mechanical loss which may occur is not included in them, but they do seem to us to be comparable for the dihydrate and for the hemihydrate plant. For the process itself the major loss is really the cake loss and it is not too difficult to remain in the 94% efficiency range as we have shown you. The breakdown of the various losses as set out in the paper is 1.5 in total, of which by far the major part is co-crystallized dicalcium phosphate, water soluble is up to about 0.4 or 0.3% and rock losses on unattacked phosphate are of the same order or less, 0.1 or 0.2%.

MR. F.W. EDWARDS (Humphreys & Glasgow, U.K.): I would like to pursue the question of losses. Referring to your table, could you state clearly what the overall loss is on the Windmill plant, that is total P_2O_5 losses? Certainly on the normal dihydrate plant there are losses not only in the filter cake but also arising from pump gland leakage and pipe line washing. What is the overall P_2O_5 loss as compared to cake loss? In regard to the hemihydrate cake being transformed to the dihydrate slurry, you can, in fact, introduce a second reaction system. Would you like to comment why Windmill introduced a second reaction system but did not take advantage of the possibility of additional P_2O_5 recovery?

MR. VINKE: First your question about overall efficiency. It is rather difficult to answer that because we have a system in which we operate two plants, a hemihydrate plant and a dihydrate plant, very close together. They are actually in the same building, have the same disposal system and the disposal streams of dihydrate are combined. It is very difficult to distinguish exactly between losses from the dihydrate (I mean the losses additional to cake analysis) and those from the hemihydrate plant. As far as we can tell, they are of the same order and we have no reason to suppose that one has a much greater loss than the other.

The other question concerned recrystallization from the hemihydrate into the dihydrate system, and why we do not try to recover the P_2O_5 from that system? As I told you, this recrystallization is merely for disposal reasons and to prevent any risk of blockages down the

disposal line. For recovery of P_2O_5, an additional filter is required and a different overall process control. It may be an answer to your question that we are still very interested in this second stage but at the time that this plant was built such a two stage system was not yet available for Togo rock. We are still interested in this second stage.

Owing to pressure of time, it was not possible to take further questions at this stage. The following questions were the subject of correspondence:

MR. EDWARDS: Thank you, Mr. Vinke, for your answers. I am, however, not fully satisfied with your reply on the subject of P_2O_5 losses. I understand the layout of the hemihydrate plant in relation to your old dihydrate plant may make it difficult to measure total P_2O_5 losses from each plant. However, if you are going to compare production costs of the two processes as you attempt to do in Table 4 of your paper, it is necessary to do this on the basis of total P_2O_5 loss and not just the P_2O_5 loss in the gypsum cake. It is not necessarily true that the non-cake P_2O_5 losses are about the same for the two processes.

As some 80% of the total variable production cost is accounted for by raw materials I believe it to be important that you base your cost figures on total P_2O_5 loss which comprise:-

a) P_2O_5 in the cake

b) P_2O_5 in the filter effluents

c) P_2O_5 in scrubber effluents

d) Mechanical losses - filter and pipeline washing, gland leakage, drainage, etc.

I should much appreciate answers to the following specific questions:-

1. What are the total P_2O_5 losses for the hemihydrate process for conditions as posed in Table 4 of your paper, i.e. cake P_2O_5 loss 6%, Morocco rock, 50% P_2O_5 acid, and how do these losses vary when the acid strength is ± 4% P_2O_5.

2. The cake water soluble P_2O_5 loss of around 0.4% (based on P_2O_5 fed to the plant as rock) when producing 50% P_2O_5 acid is very low. What is the water soluble P_2O_5 loss in the slurry arising from

the filter cake mixed with other filter effluents? Does this include filter pan wash water - if not what do you do with it?

3. What is the difference in % P_2O_5 between the acid fed to the filter and the first filtrate, i.e. what dilution is taking place?

MR. VINKE: Mr. Edwards is of course correct, total P_2O_5 loss is the essential criterion in assessing phosphoric acid cost. There is, however, some difficulty in establishing accurate figures for total P_2O_5 losses from the dihydrate and hemihydrate plants at Windmill. In comparing one process route with another, overall P_2O_5 efficiency is not necessarily the best criterion for judging plant performance. The difference between P_2O_5 loss in the filter cake and total P_2O_5 loss, normally referred to as mechanical loss, can vary widely even between two plants operating the same process. The figure for mechanical loss depends largely on the accuracy of measurement of input and output, plant loading and plant management. To incorporate a figure for mechanical loss can be misleading and is always open to argument.

Loss of acid from the hemihydrate plant is of course more serious than for the dihydrate because of the higher P_2O_5 strength but this effect is more than balanced by the simpler plant design with fewer sources for mechanical leakage. The elimination of weak acid handling, storage, evaporation and clarification, when producing merchant grade acid, are the more important benefits.

The normal weekly wash cycle of the dihydrate slurry piping and filter unit is avoided in the hemihydrate process showing a significant saving in P_2O_5 loss, and also of production downtime. The Windmill Hemihydrate Plant operates on a 2 month wash/maintenance cycle.

The underlying thought behind Mr. Edward's question is, I am sure, of P_2O_5 loss from the filter. This problem was well known during the Windmill commissioning period when high levels of P_2O_5 were found in the cloth washing water. Modification in operating technique has eliminated this problem.

There is no reason to assume that mechanical losses from the filter will be higher in a hemihydrate process than in a dihydrate process.

To answer the specific questions asked:

1. As stated before, mechanical P_2O_5 losses for any

phosphoric acid plant depend largely on factors which are not inherent in the type of process used. So I have no answer to your question regarding total P_2O_5 losses for the hemihydrate process.

The hemihydrate process is not recommended for operation at acid strength above 52% P_2O_5, due to lack of water available for washing the filter cake. At 46% P_2O_5 product acid strength the P_2O_5 efficiency increases to 96% based on cake losses.

2. The 0.4% water soluble P_2O_5 loss is a realistic figure when using three wash-stages, and is based on hemihydrate from the filter pan. The discharged filter cake is slurried with the filter pan wash water, mixed with a similar effluent from the dihydrate plant and discharged to sea by barge.

   Some water soluble P_2O_5 is found in this effluent. However, there is no reason to calculate a higher loss from the hemihydrate process than from the dihydrate process.

3. There is a dilution of acid strength across the filter of about one per cent.