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SUBMERGED COMBUSTION EVAPORATION
RECENT EXPERIENCE AT THE BARREIRO WORKS

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1. INTRODUCTION

The general trend today in phosphoric acid production is to obtain highly concentrated acid without a concentration stage. The "direct acid" so obtained is cheaper since concentration plant is not needed.

However, despite this "new look" in phosphoric acid production, it is necessary to continue dealing with the concentration problems because of the development of high analysis fertilisers obtained from superphosphoric acid, which requires a new concentration step.

Generally speaking the basic principle of evaporation is still applicable in the case of phosphoric acid concentration.

In 1962 our Company decided to install a Nordac Concentration Plant with submerged combustion and this started up in 1964.

Based on our own experience, the aim of this paper is to explain how it operates and the difficulties encountered during and after starting up. As the main problems in a concentration plant are construction materials and recovery of the P_2O_5 mist in the waste gases, we also make special reference to the materials, and integration of the plant with the phosphoric acid plant.

2. THE GENERAL PRINCIPLE OF THE PROCESS

The Nordac Submerged Combustion Process uses the direct contact of combustion gases from fuel-oil with the phosphoric acid to evaporate water. Thermal equilibrium is reached at a given temperature and this corresponds to a given concentration of the acid. Thus, temperature is the main component to be controlled.

The direct contact eliminates heat exchange problems, i.e. scaling and materials, because the former has no influence on heat transmission (on the contrary it helps to protect the walls) and the problem of construction materials is not difficult to solve as will be explained later in the paper. This scaling, due to the decrease in solubility of some salts, depends on the quantity of solids in the diluted acid, which is saturated with gypsum, and this is connected with the weave of the filter cloth used in clarification of the acid. The fluosilicate salts, the gypsum scales on the evaporator surfaces and silica also adhere to it.

The hot combustion gases pass beneath the surface of the acid through a dip tube which is submerged in the acid. The

waste gases and water vapour leaving the evaporator carry a fine mist of phosphoric acid and a large quantity of fluorine. This phosphoric acid is recovered in the impingement scrubber which operates with diluted phosphoric acid from the phosphoric acid plant (see Fig. 1). Before being discharged to the atmosphere the waste gases are cooled by seawater in a three stage scrubber, in order to maintain a low concentration of fluorine.

The weak acid is first fed to a head tank and then through a pneumatically controlled valve to the evaporator. After concentration, it passes through one of the two product outlet lines. The main outlet and control valve are approximately 30 cm below the level of the liquid.

3. MAIN EQUIPMENT

Evaporator

The evaporator has a conical shape so that the gypsum may be collected in the sludge box (Figs. 1 and 2). This box may be emptied by a straight-through valve and is vented to make desludging possible without shutting off the burners. The vessel and box are designed so that there can be a long running period without the plant being stopped due to deposition of gypsum and scaling.

The acid level is maintained by mean of a float which controls the pneumatic valve of feeder acid.

Combustion gases and water vapour leave the evaporator through a rectangular duct with baffles, which avoids the entrainment of acid droplets.

Some waves are formed inside the evaporator (due to the injection of combustion gases within the liquid) and these may cause vibration in the plant. To avoid this, a special concentric tube is designed with the dip tube.

Oil burning equipment

Connected to the evaporator is the oil burning equipment which consists of two combustion chambers each designed to burn a maximum of 215 l/h, corresponding to a heat release of approximately 4.14×10^6 Kcal/h and 2.07 m. per chamber.

The atomizers spray the oil into the chambers and are fitted with nozzles. In order to control the oil flow, which is done manually, there is a relationship between burner

nozzle pressure and oil flow. There is the usual circuit for start-up and the flow can be checked with an oil integrator meter.

P₂O₅ scrubber

This is one of the most important pieces of equipment in a concentration plant. For overall economy satisfactory scrubbing is necessary and the recovered acid can be recycled to the plant or to the phosphoric acid plant.

This scrubber is situated in a section of the offtake duct and the removal of phosphoric acid mist is effected by high velocity impingement of scrubbing acid and gases (co-current). This system comprises :

- perforated plate made from keebush
- graphite spray boom
- a "D" plate to separate the liquid from the gases
- in addition, following the scrubber, there is a pair of cyclones to separate the gases from liquor spray
- a submerged pump (d'Ensival) to pump the diluted acid to the scrubber (Figs. 1 and 2).

Gas cooler

The plant has three-stage gas coolers which operate with seawater. The gases are washed and cooled to 30/35° C so that fluorine may be removed. The equipment is similar to that used in the P₂O₅ scrubber.

Control and safety system

The control system usually provided for this plant is combined with the safety system, which prevents start-up until all conditions are correct. The strength of the acid produced is controlled by a temperature recorder controller which operates the outlet acid valve and the acid level in the evaporator is maintained by a float.

The oil is always automatically shut off from the burners when one of the given conditions is not satisfied. A signal may be received from one of the following instruments :

- . Air pressure switch
- . Temperature recorder alarm
- . Temperature indicator alarm
- . Low liquid level differential pressure switch

The individual circuit safety system for each burner has a :

- . Primary air pressure switch
- . Secondary air differential pressure switch
- . Oil temperature alarm (Low temperature oil alarm)
- . Flame failure alarm

The system also has a hooter for flame failure and high level, and a light for high gas temperature.

After some months in operation a TIR temperature recorder indicator was placed on the top of one chamber, in order to allow better control of the burning of the bricks and cement.

4. OPERATING DATA AND PROCESS EFFICIENCY

The plant was erected in 1964 and is capable of concentrating 60 metric tons of P_2O_5 per day from 27% W/W P_2O_5 to 50% W/W P_2O_5 .

Operating data

In the following table, data obtained over the last three years are given :

Utilities/ton P_2O_5	Unit	Year		
		1967	1968	1969
Power	kWh	47.3	46.8	48.0
Fresh water	m ³	0.7	0.7	0.7
Seawater	m ³	38	39.7	41
Fuel Oil	kg	116	123	130
Steam	kg	32	31.4	34
Compressed air	m ³	0.5	0.5	0.5
Operating data				
ton H_2O (v)/ton P_2O_5	ton.	1.63	1.70	1.78
Production tons P_2O_5 /hr	ton.	2.88	2.79	2.56
H_2O (v)/hr	ton.	4.70	4.74	4.56

* H_2O (v) - evaporated water

If we consider a recovery of 99%, the Nordac Plant is capable of evaporating 1.72×2.5 tph P_2O_5 - 4.30 tons of water per hour. We have achieved the very satisfactory figure of 4.74.

The difference in the figure for fuel consumption in 1968 relative to that of 1967 is due to the more concentrated acid obtained. The figures for 1969 are nearer those specified by Nordac but this plant can improve on these, as in 1968 and 1967.

The plant can operate 330 days per year, producing about 20,000 t. P_2O_5 .

Process efficiency :

The efficiency of the process depends essentially on the scrubber system. Recovery figures are given in the following table :

Analysis of P_2O_5 content			Approximate Recovery %
Gases g/m^3	Cooling water g/l	Feed acid to the scrub. % P_2O_5	
0.584	0.084	Between 10 and 12%	99.22 to 99.4
0.651	0.105		
0.636	0.092		
0.643	0.110		

The above mentioned recovery is related to scrubber feed acid with about 10 per cent P_2O_5 , such as was used in 1964-1965 after erection of the plant, and it excludes the P_2O_5 collected in the scrubber system. The table also gives the P_2O_5 content of the flue gases and of the cooling seawater leaving the plant, and this is the only P_2O_5 which is really lost.

Without baffles in the evaporator low velocity duct, recovery was about 98.5%.

Heat Balance : consumption of fuel to evaporate the water

With feed acid to the Nordac plant at 176° F and the outlet gases at 228° F, the consumption of fuel to evaporate the relevant water can be assumed to be about 80% of total fuel consumption.

5. INTEGRATION WITH THE PHOSPHORIC ACID PLANT

It is possible to integrate the concentration plant with the phosphoric acid plant using, for example, the gas cooling water for gypsum washing in the filters. Using this method the P_2O_5 lost in the scrubber can be recovered.

Another possible link between the two plants can be the use of several types of weak acid from the phosphoric acid unit in the scrubber system. If the cooling water is used in the filters one of the problems of the concentration plant (recovery of P_2O_5 mist in the waste gases) is resolved, because almost all the P_2O_5 lost in the scrubber is recovered in the filter.

When we started, in 1964, with the Nordac Plant the only link between it and the phosphoric acid unit was the feed of weak acid from the latter. In the first scrubber system, the liquor was recycled, with a submerged pump, to the scrubber plate until a content of P_2O_5 was reached which permits the use of the acid as a wash acid in the filter. Make-up water was added to the feed liquor to allow for evaporation and to maintain the acid content.

The acid so obtained should be taken up in the filter of the phosphoric acid plant but it causes some problems on account of the fluorine. After several months running we noticed that the stainless-steel of the Nordengren pans filters was corroded and some of them had to be welded.

We consider that the advantages of carrying out this operation using a weak acid of about 27% P_2O_5 are :

1. Heating the weak acid with the hot waste gases in the scrubber with correspondingly lower consumption of fuel needed to concentrate it.
2. Increasing the concentration of the acid by recovery of the P_2O_5 mist in the gases, with the same advantage as indicated above.
3. Avoiding damage of filter pans and the nearby reaction system with acid containing fluorine, because the new acid (27% P_2O_5) is fed to the concentration plant directly after coming from the scrubber.

Using this acid and the system referred to in the diagram, a little less recovery is obtained, but the problem is resolved and the characteristics of the feed acid are improved.

The advantages and disadvantages of the different types of scrubber wash acids can be summarised as follows.

Acid Type	Advantages	Disadvantages
Acid produced in the phosphoric acid plant	Lower fuel consumption No corrosion of the filter pans	Lower recovery A little more fluorine in the Nordac Plant. Possibly more corrosion in that plant
Acid with 7-10% P ₂ O ₅ (wash acid in the filter)	Hotter acid to wash the filter cake Less scaling in the Phosphoric Acid Plant Higher recovery in the Nordac Plant	Corrosion on filter pans Possible corrosion in reaction system
Recycle acid 20/22% P ₂ O ₅ fed to reaction system		Possible corrosion of reaction system Higher consumption of air to cool the slurry

A second link with the Phosphoric Acid Plant was effected by means of the sludge that is collected on the bottom of the evaporator. Initially the sludge fell into a small tank when water was added and was finally drained off, but since there is a desludging every two hours 4.0 tons per day were lost, which is equivalent to 1.0 tpd P₂O₅. To recover this acid the sludge is recycled by gravity to the third reaction tank which is 45 m³ in volume. The sludge volume is small compared with this and since the tank is agitated, no

problems of gypsum filtration or cloth life were encountered.

6. CONSTRUCTION MATERIALS AND MAINTENANCE

As is well known, the contaminants in the phosphoric acid are caused by the phosphate rock. In our factory we use Moroccan Phosphate Rock H.N. with an approximate composition as follows :

	1968	1969
P ₂ O ₅	32.62%	32.6 %
Oca	50.39%	50.0 %
O ₃ Fe ₂	0.30%	0.24%
O ₃ Al ₂	0.47%	0.72%
SiO ₂	4.12%	3.82%
Cl	-	0.01%
F	3.15%	4.77%

The most important contaminants are fluorine, chlorine, and silicate (F, Cl, SiO₂) which can form hydrofluoric acid, hydrochloric acid and fluosilicic acid when the slurry in the reaction tank system is obtained. The combination of corrosion-erosion is the reason for the corrosiveness of the wet process acid since some solids pass through the filter cloth.

With concentration the solubility of some salts like K₂SiF₆, Na₂SiF₆ decreases and they are deposited on the surfaces of the evaporator despite the increase in solubility on account of temperature. This scaling forms a protection layer against corrosion in the case of submerged combustion.

The evaporator can be divided into two parts, respectively below and above the liquid level, each with different corrosion conditions. The upper part, comprising the low velocity duct and cover of the vessel where the chambers are mounted, is subjected to more severe corrosion conditions on account of the high temperature of gases.

Some specific construction materials are given below and, at the end of this section, the frequency of repairs to the principal equipment is listed.

- Evaporator :

- lower part ; mild steel with a lead sheet (99.95% Pb). Additional protection with carbon bricks set in furane cement.
- upper part ; mild steel lined with homogenous lead. Additional protection with furane cement.
- Off-tack duct ; mild steel lined with rubber (up to scrubber plate) and carbon bricks.
- Cyclones ; mild steel lined with rubber.
- Gas cooler section ; mild steel lined with rubber.
- Dip-tubes ; 20 alloy lined with firecrete.
- Scrubber plate ; made from Keebush.
- Combustion chambers; are lined with refractory materials furnished by the Woodall Duckham Co. with high alumina and silica content. As a test, they also were lined with Portuguese refractory bricks, with good results to date.
- Pumps (submerged centrifugal d'Ensival pumps) ; Scrubber pump : Hastelloy C
Weak acid pump : HV-9
- Storage tank for concentrated acid ; lined with Flexi-Liner.

We should mention that the evaporator was designed by Nordac to be lined with rubber instead of lead sheet but, on account of technical difficulties, this was not possible.

Frequency of repairs table : next page

Frequency of repairs or replacement of the principal equipment

Equipment	Life (years)				
	1	2	3	4	5
. Evaporator brick and lead linings					x
. Low velocity duct				x	
. Combustion chamber refractory linings : roof	x				
walls					x
burner-cone		x			
. Combustion chamber (metallic part)				x	x
. Dip-tubes lining	x				
. Dip-tubes (metallic part)	x				
. Cyclones			x		
. Circulating tubes			x		
. Sludge box			x		
. Valve linings		x			
. Filters of the compressor	x				
. Baffles		x			
. Stack				x	

7. PLANT OPERATION

Normal running

After some initial problems, which were solved and which are mentioned below, the plant is performing well after some modifications, especially in the lining of combustion chambers.

It works absolutely normally with very steady feeding and output of product acid, indicative of excellent operation of automatic valves, burning and air equipment.

Every fortnight the plant is stopped for cleaning and desludging. Every three days one burner is replaced for cleaning, without stopping the plant.

Control is very easy and one man per shift is sufficient.

Principal problems

The most important problems encountered were connected with the combustion chambers and the low-velocity duct linings.

. Combustion chambers

The problem that occurred with the atomizers was due to a power failure which in turn caused the failure of the brick-lining with consequent over-heating of the atomizer housing.

As already mentioned the tops of the chambers are now lined with a refractory cement which is replaced every year, but initially they were lined with bricks. The breaking of this brick, especially of the quorl bricks which surround the atomizer housing, might be due to several reasons :

- badly manufactured bricks
- vibration of the plant
- over-heating of the roof chamber

We tend to favour the first possibility because some of the bricks had cracks before installation.

Following the damage to the quorl bricks there was a twelve hour power-failure, which permitted over-heating of the burner housing. We then stopped the number one chamber and continued with number two. After some days we noticed that, through having the secondary air-valve in No. 1 chamber completely open, the dip-tube cone, and even the side walls were wet with acid.

Inspection of the burner cone of that chamber showed that its layer of firecrete had been reduced by about two inches and the bricks were corroded. We decided to reline only this part and the cover, keeping the damaged bricks in the side wall. Because we did not have another chamber, the damaged chamber had been working for more than one year.

This second problem had been the long-term result of running the factory with only one chamber because the velocity of gases with the burner off is several times less than with the burner on.

Basically the power failure was the main trouble and the others were the consequence of it. As it was not possible to provide sufficient stand-by electrical power for the secondary air compressor, in the event of a power failure, we considered installing a stand-by unit to provide 30% of the normal air flow. However, in view of the cost and the likelihood of a power failure like the previous one, this idea was not followed up.

. Low-velocity duct

It has been mentioned that this part of the vessel is subject to more severe corrosion, with, consequently, a shorter life than the evaporator body (4 years as opposed to 5). The vertical walls of this duct were corroded because the lead lining does not resist cracks in the furane cement. In the near future we shall have to replace the lead with a rubber lining.

As happened in the chambers, one problem is often followed by another, and the first damage in the low-velocity duct was responsible for corrosion of the evaporator body. The evaporator body life is normally more than five years.

. Vibration

The injection of combustion gases within the liquid causes waves which, on account of their amplitude and frequency, can result in strong vibrations in the evaporator, chambers and supports. With the starting-up, in 1964, the circulating tubes were rectified to avoid vibration.

8. PLANT COST

A distribution of plant cost between the main sections of the plant and equipment is as follows :

	Percentage of total plant cost
Engineering	12.0
Metallic equipment	35.0
Auxiliaries	6.0
Electrical equipment	5.0
Civil engineering operations	17.0
Brick and rubber linings	5.0
Erection	15.0
Start-up	5.0

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Slides to be shown during the meeting

- 1 - Process flow-sheet
- 2 - General view of Nordac Concentration Plant
- 3 - Evaporator body
- 4 - Sludge box
- 5 - Dip-tube
- 6 - Corroded dip-tubes for welding
- 7 - Damaged brick lining (chamber cover)
- 8 - Chamber cover lined with firecrete
- 9 - Damaged combustion chamber lining
- 10 - Undamaged " " "

DISCUSSION

Mr. R. MARÇAL (Companhia Uniao Fabril, Portugal) :
The Nordac Concentration Plant using the submerged combustion process started up in 1964. It can concentrate 60 t. per day of P_2O_5 , from 27% to 50%, producing about 20,000 tonnes P_2O_5 per year.

The aim of this paper is to describe how it works, what were the difficulties during and after starting up. There is a special reference to the materials, and integration of the plant with the phosphoric acid plant.

(Figures were then shown as slides).

Figure 1. As you know, the Nordac Submerged Combustion Process uses direct contact with the phosphoric acid of combustion gases from fuel-oil to achieve the evaporation of water.

The weak acid is first fed to a head tank and then to the evaporator. After concentration it goes through one of the two product outlet pipes.

The hot combustion gases pass beneath the surface of the acid by means of a dip tube which is submerged in the acid.

Leaving the evaporator, the waste gases and water vapour are carrying a fine mist of phosphoric acid and a large quantity of fluorine. This phosphoric acid is recovered in the impingement scrubber which operates with diluted phosphoric acid from the phosphoric acid plant.

Before being discharged to the atmosphere, the waste gases are cooled with seawater in a three-stage scrubber, in order to maintain a low concentration of fluorine.

Figure 2. The evaporator has a conical shape in order that the gypsum may be collected in the sludge box.

The vessel and box are designed so that there may be a long running period without stopping. Down-time 15%.

OPERATING DATA AND PROCESS EFFICIENCY

The operating data and efficiency are given on pages 5 and 6.

The efficiency we are obtaining now is about 99% in relation to the scrubber feed acid and the plant can evaporate 4.7 t. H_2O (v)/hr.

INTEGRATION WITH THE PHOSPHORIC ACID PLANT

With the first scrubber system we recycled the liquor with a submerged pump to the scrubber plate until a content of P_2O_5 was achieved which permits the use of the acid as a wash acid in the filter.

The acid so obtained should be used in the filter of the phosphoric acid plant but it caused some trouble on account of the fluorine. After several months of running we noticed that the stainless steel of the Nordengren pans filters was corroded and we had to repair some of them.

We considered that it was possible to effect this with some advantages by using weak acid with about 27% P_2O_5 and these are referred in the paper.

Figure 3. Evaporator body, low velocity duct and combustion chambers, during erection of the plant.

Figure 4. Bottom of the evaporator with valve and sludge box, water pipe, vent pipe.

Figure 5. One of the two dip-tubes where the "firecrete" lining and the circulation tube can be seen.

Figure 6. These are dip-tubes for welding in order to recover them.

The upper part is not damaged, thus showing the corrosion line due to the level of the liquid.

The most important and interesting problems were connected with the combustion chambers and low-velocity duct linings.

The plant has however been running normally with very steady feeding and output of product acid, indicative of excellent operation of both automatic valves, burning and air equipment.

Every fortnight the plant is stopped for cleaning and desludging. Every three days one burner is replaced for cleaning without stopping the plant.

Figure 7. Combustion chamber top with the damaged bricks.

One can see the quarl bricks with carbon, which probably caused poor air distribution.

Breaking of these bricks might be due to several reasons :

- 1 - badly manufactured bricks
- 2 - vibrations in the plant
- 3 - over-heating of the atomiser housing

Figure 8. Top lined with firecrete.

Some cracks can be seen but these are not important and they are common after some months running.

The little tubes are for air injection.

Figure 9. A perfect combustion chamber lining. First and second layers.

Figure 10 Damaged chamber side-walls. The causes are referred to in the paper.

The lining of the cone was reduced by about 2" and it was possible to see the second layer through the cracks in the first brick lining.

The chamber with some large cracks in the first layer has been working for more than a year.

Mr. M. FROCHEN (Pierrefitte-Auby, France) : For various reasons, the most important being that many plants where phosphoric acid is produced have low pressure steam, most phosphoric acid concentration plants use vacuum evaporation and steam heating.

It is this which forms the subject of Uniao Fabril's presentation on submerged combustion evaporation, which not only describes the process used, but which takes account, with many details, of the operational conditions and furthermore of the difficulties encountered and means employed to resolve these difficulties.

In every concentration system, the principal difficulty arises from the scaling properties of solutions of phosphoric acid and it is to try to provide an answer to this problem that NORDAC uses a submerged burner, on the basis of the idea in SLACK's book that there cannot be scaling on the walls of a gas-bubble.

It is true that with this system the transmission of heat is practically constant over time, but it is effected in spite of all the scaling on the walls of the concentrator and of the various deposits in the apparatus. Mr. MARÇAL's paper describes means used to eliminate the scaling and

deposits with the impression that all goes well, but we all know that the importance and nature of deposits vary considerably with the phosphate used to make the acid.

Uniao Fabril uses Moroccan HN which is not a frequently used phosphate. Have they experience of other phosphates such as Moroccan 75, Florida 72 or richer phosphates such as Taiba or Togo phosphates ?

Another source of particular difficulties in submerged combustion concentration systems results from the production in substantial quantities of phosphoric acid aerosols and P_2O_5 steam which are difficult to stop with normal washing apparatus, and which can give rise to relatively serious losses and to risks of atmospheric pollution. NORDAC and Uniao Fabril seem to have resolved the problem of losses and the yields given are completely satisfactory, but is it the same with atmospheric pollution ?

The fact that the losses are low proves nothing in this respect, since very low quantities of very finely dispersed acid are sufficient to contaminate the atmosphere.

In his paper, Mr. MARÇAL explains that, in order to obtain the best yields possible, they have linked the filtration plant closely with the concentration plant, using one of the acids leaving the filter to wash the gases coming from concentration. At present, they are using the production acid for this purpose, which causes no problems apart from some lowering of yield, but when they use very weak acid from the last washing of the filter do they risk putting the filter out of order, unless this filter is over-size, which would allow one to suppose that they can return the slurry from concentration to filtration ?

Mr. MARÇAL :

1. As I said in the paper, the plant is stopped every fortnight to effect desludging through the bottom of the evaporator. We open the two doors, one in the upper part in the low velocity duct and the other near the bottom. This desludging is very easy because the phosphate we use, Moroccan phosphate HN (Hatane Nord) 72 BPL does not give as hard a scaling as Moroccan 75 BPL. Under these circumstances we can clean the evaporator every fortnight with cold water only to remove the sludge of gypsum and acid from the concentrator. There are, of course, some difficulties with the valves which may need cleaning every 2 or 3 days. Only the outlet valve and the feed valve are automatically controlled valves. Regarding the scaling, we can

divide the evaporator in 2 parts :

- The lower part, below the level of the acid.
- The upper part, the low velocity duct and cover of the evaporator.

Especially in upper part, the scaling is a protection of the walls, brick and furane cement linings.

We have experience with Moroccan phosphate rock and one type of American phosphate. The American phosphate does not give as many problems as the Moroccan phosphate but we have some problems with the P_2O_5 content on account of the aluminium and iron phosphates. With the American phosphate we can achieve greater production in the phosphoric acid plant and less scaling.

2. The second question, dealing with pollution problems, is related to the scrubbing system. We have no air pollution because only a fine mist of P_2O_5 and water vapour is lost through the stack and we can see a clear water vapour. This fine mist depends on the distance between the last cooler stage and the stack and on the scrubber efficiency. The cooler system is a three stage cooler and the last one is 3 to 4 metres away from the end of the plant but we have only a fine mist of P_2O_5 . The total loss is about 1% P_2O_5 but only part of it in the stack.

3. Concerning the use in the filter of the diluted acid from the phosphoric acid plant. We now use 27% acid and there is no trouble, with that acid, in dealing with the reduction of recovery of the filter, because the 27% acid, as you can see in figure 1, is now fed from the phosphoric acid plant directly to the scrubber and is collected in the small tank with 2 submerged pumps. At first we used more dilute acid, with about 7 or 10%. Now this percentage is a little less on account of recovery. We use this acid in the concentration plant, and we can control the filter as all variables are known, and can obtain steady running. We can add a little more water but, as I said, the acid has been used for 3 or 4 months only and we had to return it as a circulating acid to the reaction tank. Of course, we can solve the problem of the filter by introducing a little more water and getting a circulating acid with a content of P_2O_5 which it is possible to feed into the first reaction tank. If there is a large volume with this acid we can compensate for it with a little more phosphate, to maintain the relation between the circulating acid and the phosphate rock. We can correct the circulating acid with the product acid as is usual in a phosphoric acid plant.

Dr. B. RAISTRICK (Albright and Wilson) : Were you short of steam when you chose this particular kind of evaporator ?

Mr. MARÇAL : Yes, we did not have enough steam available and we had to choose this type of submerged combustion. Now we have another factory which can produce 500 t/day of sulphuric acid and we are erecting another with 600 t/day capacity, with a new process. We have to face the problem of keeping this concentration plant which works well or replacing it by concentration that uses steam. It is an economic problem.

Mr. A. DAVISTER (Prayon, Belgium) : I should like to ask for some details on the operation of the submerged burner. We have seen details of the tube outside the burner and assembled. What is the level of the acid in relation to the lower level of the burner ? At what distance from the submerged burner is the external cylindrical protection and how much of this protection itself is submerged in the acid ?

Mr. MARÇAL : There are some data that I cannot give you because we did not design the plant. The engineering firm was Woodall Duncan and these are data connected with the design of the chamber and the dip tubes. I can say, however, because the figure is well known, that the dip tube is submerged in the liquid concentrator by about 40 cms. I cannot tell you the right size of tube.

Mr. G.B. WHITE (Scottish Agricultural Industries Ltd., U.K.) : The storage of concentrated acid creates many problems with cleaning of tanks and the build-up of solids in the bottom of the tanks. On page 10 of the paper mention is made of a storage tank for concentrated acid lined with Flexy-Liner. I would be glad to have more details of this liner and to know if it is effective in minimising scaling and build-up in the storage tank.

Mr. MARÇAL : The Flexy-Liner lining of the concentrate storage tank was placed in that tank to replace the old lead lining and we have good experience with that material over 1½ years. There is no scaling on the Flexy-Liner but there was also no scaling on the lead. Corrosion was the most important problem. The Flexy-Liner that we have now is a good lining, better than the lead, but the cost of installing this lining is very high because we had to prepare the

tank surface. We have no corrosion problems although the acid is fed to the tank at a temperature of about 85-90° C.

Dr. RAISTRICK : I think Mr. WHITE was asking what is the material of construction of the Flexy-Liner. What is it and how does it work ?

Mr. MARÇAL : I can give you some details because I have here a booklet on the Flexy-Liner. It is a modified PVC. I think it is a dichloride PVC.

Mr. PEARCE (Fisons, U.K.) : I am still not clear on the P₂O₅ losses from the process. Could Mr. MARÇAL say what percentage of the P₂O₅ fed is lost out of the stack, and what percentage is lost in the discarded wash liquors that are not fed back into the process ?

Mr. MARÇAL : It is necessary to make some calculations, but as one can see on page 6, we had a recovery of about 90.2-90.4% with the first acid used in the plant. You can also see there, all the other values you asked for. Now, with the more concentrated acid we obtain about 99%. And of course we lose 1% of the acid fed to the evaporator. It is about 1% of 2.7 tons of P₂O₅ fed to the evaporator.

Mr. PEARCE : This is of course a very significant cost element. Do you still feel that this process is economic compared with alternative processes in view of this substantial loss ?

Mr. MARÇAL : I have no experience of steam processes and I can only compare with information from published papers i.e. SLACK's book. This gives us a broad comparison with the other processes, but I think it is a good recovery.

Mr. PEARCE : With a steam evaporator, one would normally expect to lose not more than 0.2% of the P₂O₅ fed. So you are losing an extra 0.8% which, at say \$ 100 per ton for raw materials, is almost a dollar a ton as compared with the overall cost of steam evaporation of perhaps \$ 1.5 per ton.

Mr. MARÇAL : The recovery in this plant can be improved and there is a paper that appeared early this year by

Mr. ATKINSON of Woodall Duncan* which gives a loss with this plant of about 0.2 to 0.4% putting the process in the same range as other processes. I think we can reach more than 99.5 if we use another acid and if we correct the scrubber plate, reducing the holes of these plates and increasing the pressure drop through it. So I do not agree with the difference of 0.8%.

Mr. A.H. ALAMI (D.C.P., Morocco) : You have said that other qualities of phosphate have been used. Would you give us some precise figures concerning yield, scaling and corrosion.

Mr. MARÇAL : This question is difficult to answer of course. With the American phosphate I cannot give a figure and can only say that we improved the production of the phosphoric acid plant by about 10-15% with American phosphate. It is not sufficient to compare over one or two months the corrosion properties between phosphates. With Moroccan HN and Moroccan 75 BPL I think corrosion is more or less the same but, as I said at the beginning, the Moroccan phosphate 72% BPL gives a hard scale. Of course we made some experiments with special stainless steel pipes, and I can give you these. One of them is a comparison between different types of stainless steel using Moroccan HN.

Mr. R.D. YOUNG (T.V.A., U.S.A.) : Do you consider the recirculating tube to have any advantages other than decreasing vibrations in the evaporator ?

Mr. MARÇAL : Since we did not design the plant there are some points we do not know very well. This is one of the most important points concerning the contact of gases and acid in the evaporator. I think that one of the purposes of the circulating tube is to avoid vibration of the plant, to break the waves inside the evaporator. At first we had some trouble with the design of the circulating tubes. I think it is difficult to make a perfect scale-up because this plant is the largest in Europe and in the world - 60 t/day P₂O₅ (but we can produce a little more). I think that the other purpose is to obtain a better contact between the gases and the liquid. I think these were the two main purposes when they designed this circulating tube.

Mr. YOUNG : You mention the use of fuel, does this give a particularly dark colour to the acid due to partial burning ?

* British Chemical Engineering, April 1970, Vol. 15, No. 14.

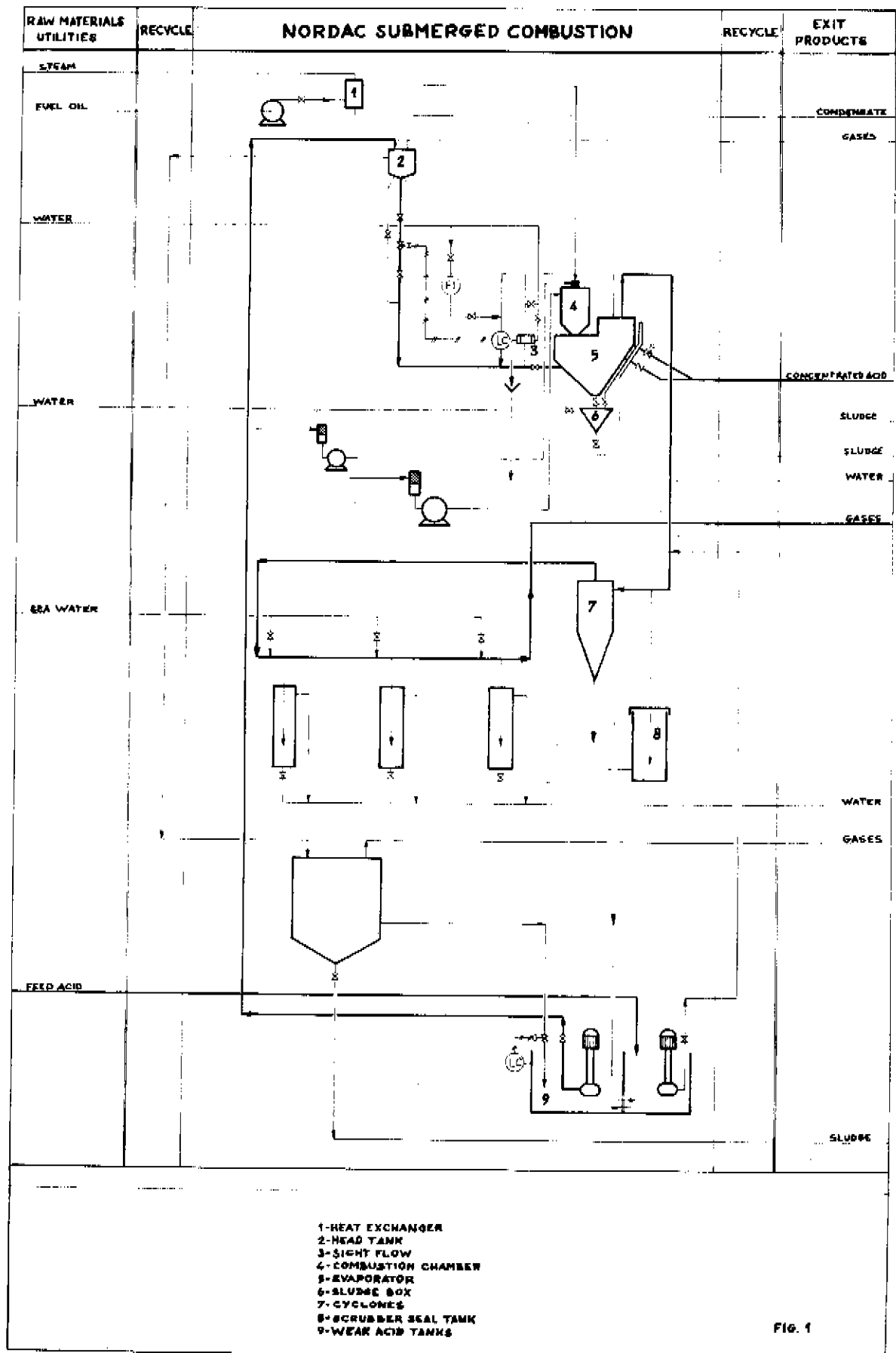
Mr. MARÇAL : No, provided there is relative control of the two materials, fuel and secondary air flow, and especially if this control is an automated as in OSARK MAHONING Plants*, I think you can obtain a clear, a green acid. Of course I have seen that sometimes carbon appears in the acid and it gives a dark colour.

Mr. M. BOORGAARD (Windmill, Holland) : I should like to come back once again to the matter of recovery of P_2O_5 . Apparently in your plant you have more installation for recovering the silico fluoride acid from the gases. If an installation is there for recovery of H_2SiF_6 , which might give an important contribution to the economics of the evaporation section, then recovery with the recirculated acid from the filter cannot be done and most of the 1% P_2O_5 you mentioned will be lost into the recovered hydrosilicic acid. This looks to me to be another distinct disadvantage of this method of evaporating.

Mr. MARÇAL : We do not have this type of equipment, nor the system for the recovery of hydrofluosilicic acid. At first, when we erected the plant, it was proposed by Nordac Ltd. that this equipment should be installed and I think it would give a higher loss of P_2O_5 , as you said.

Only Woodall Duncan can give you the right figure but I think that it was more or less 1-2% less recovery.

* See SLACK's book.



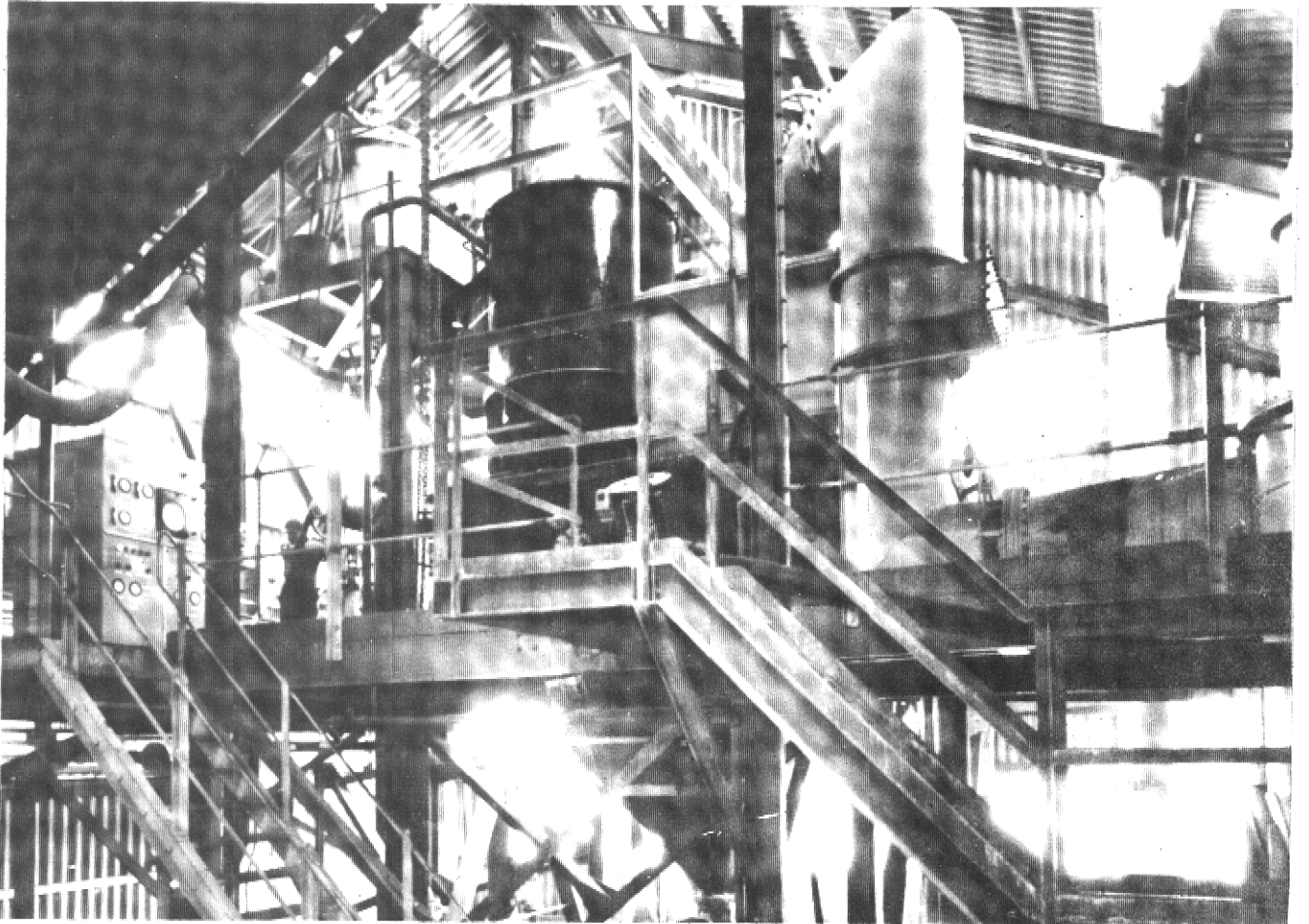


fig. 2

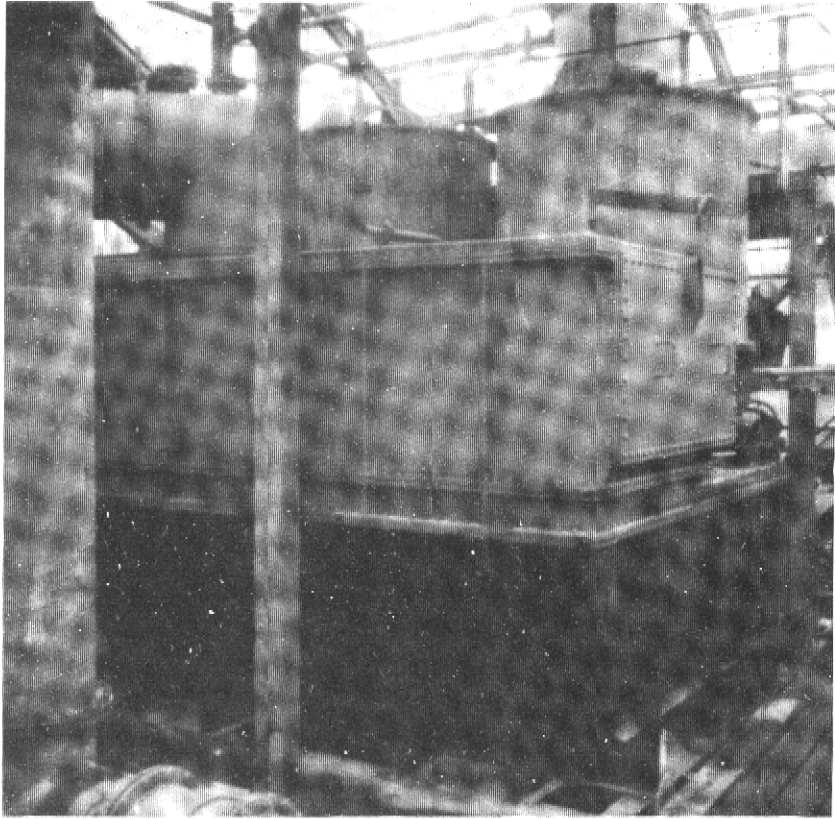


Fig: 3

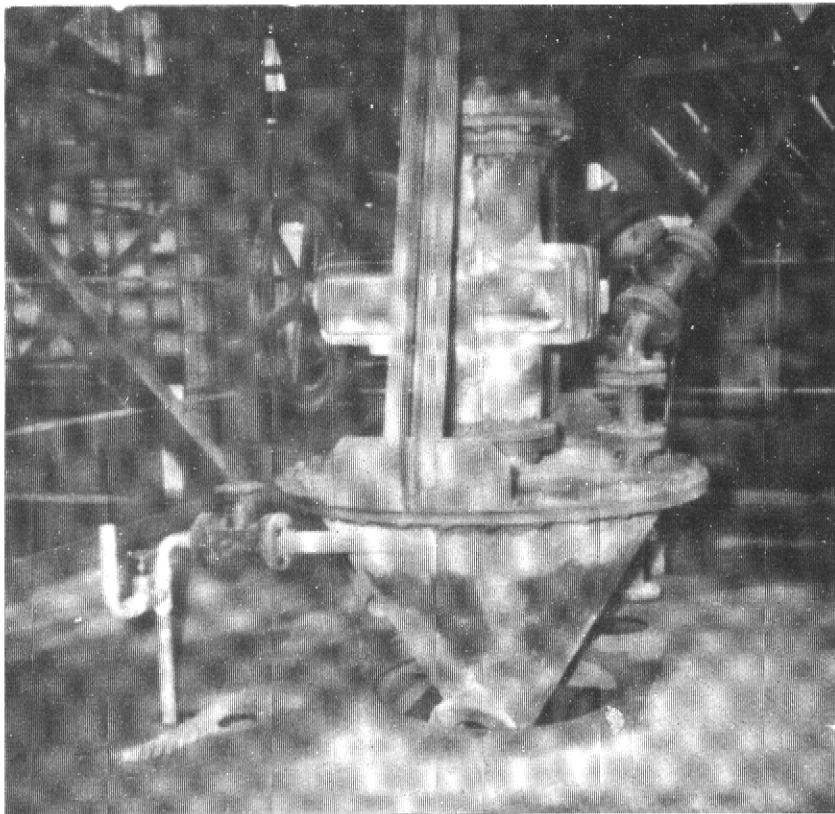


Fig: 4

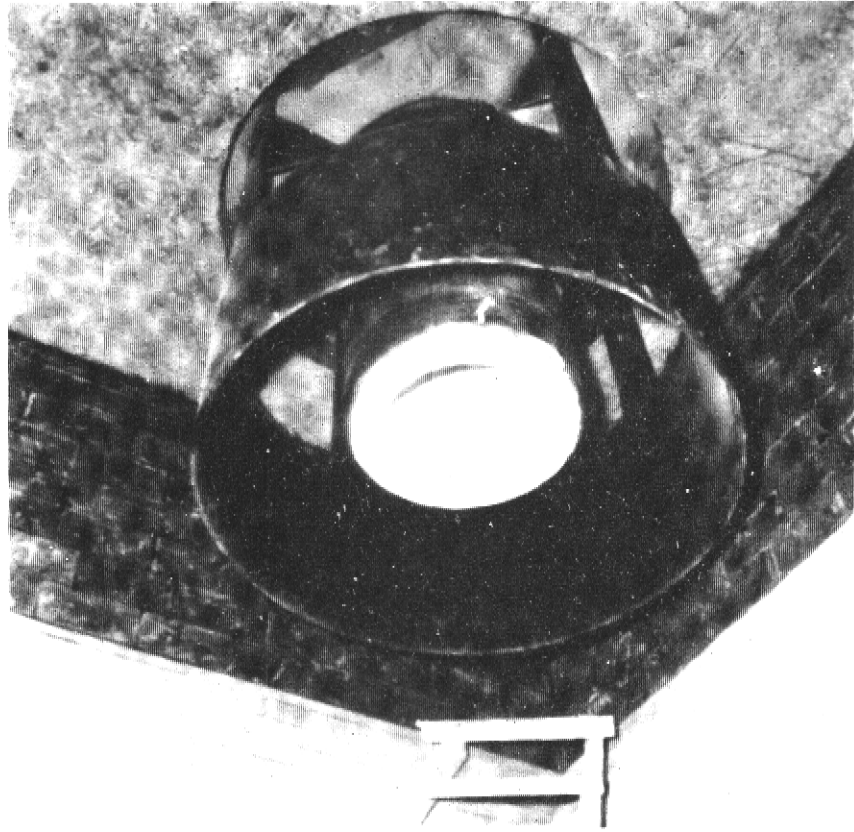


Fig: 5

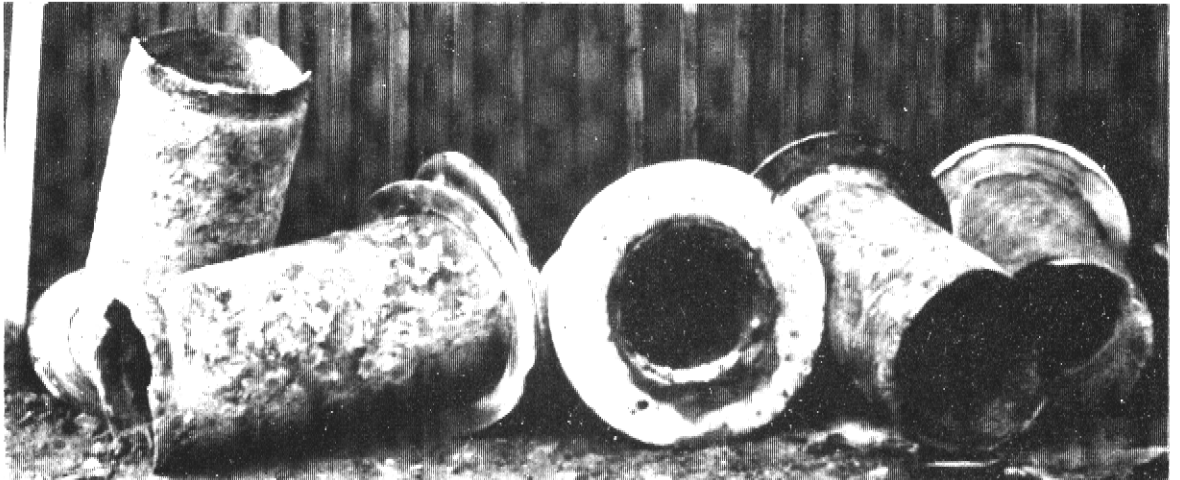


Fig :6

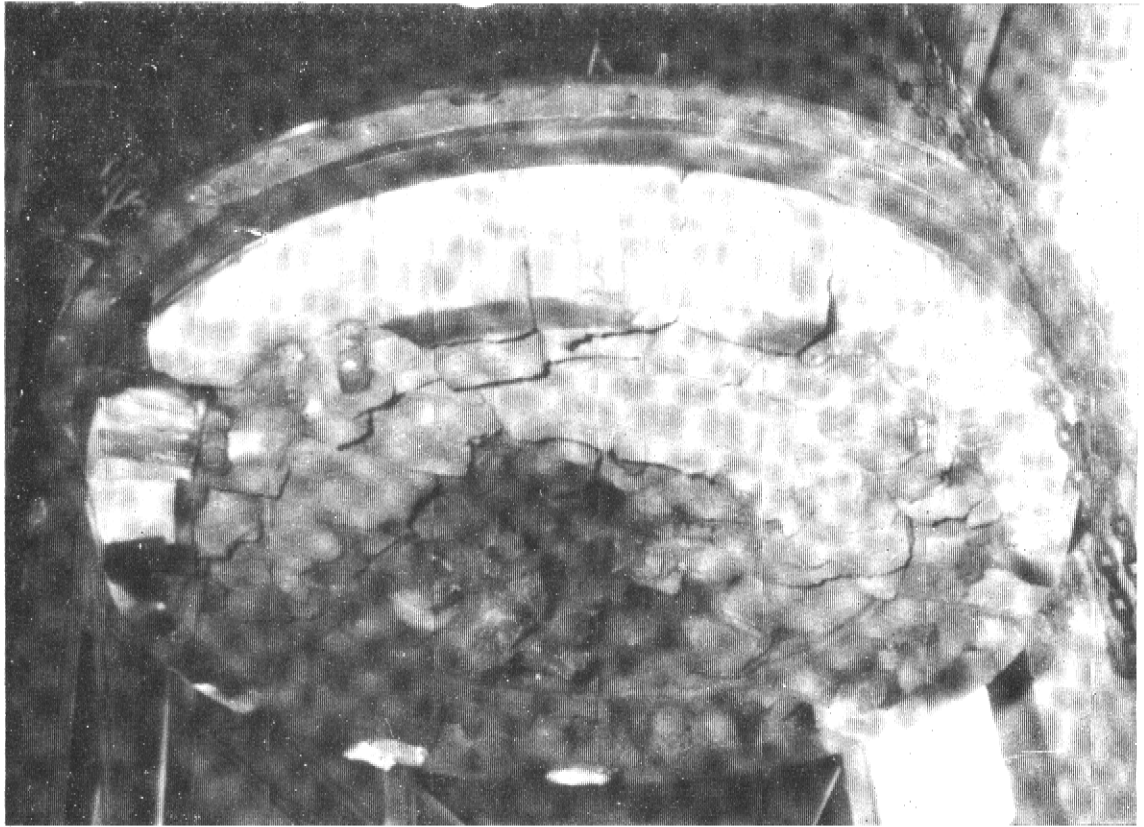


Fig: 7

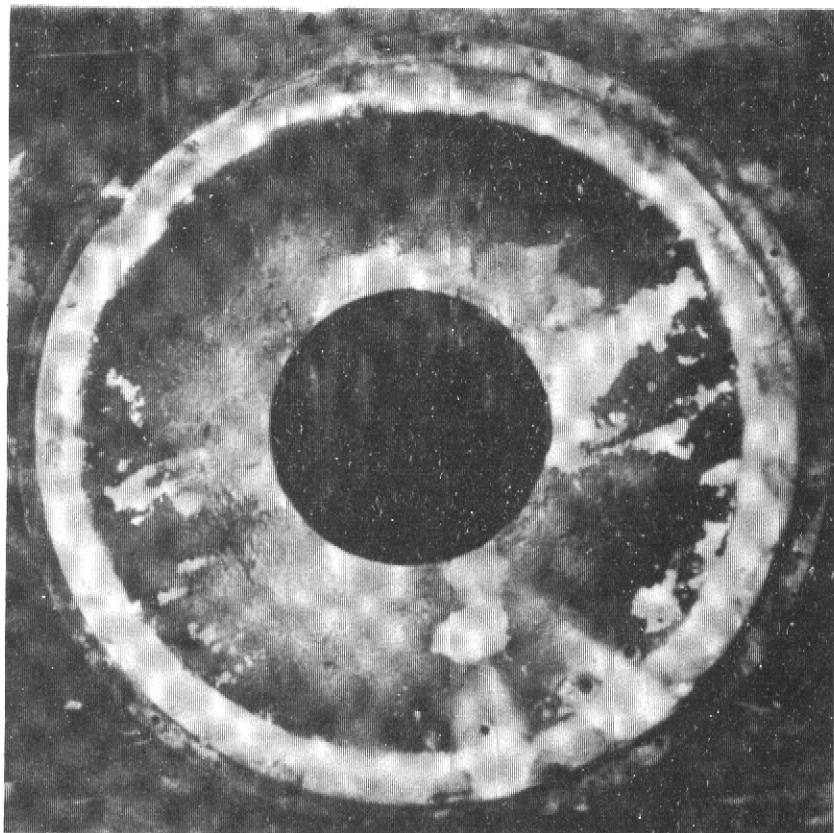


Fig: 8

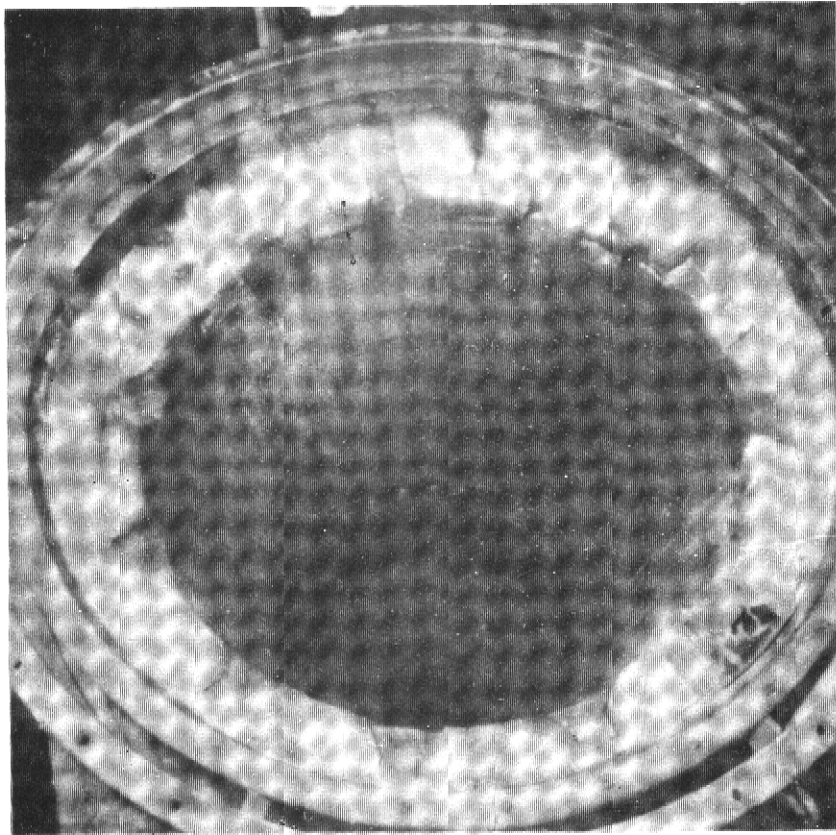


Fig : 9

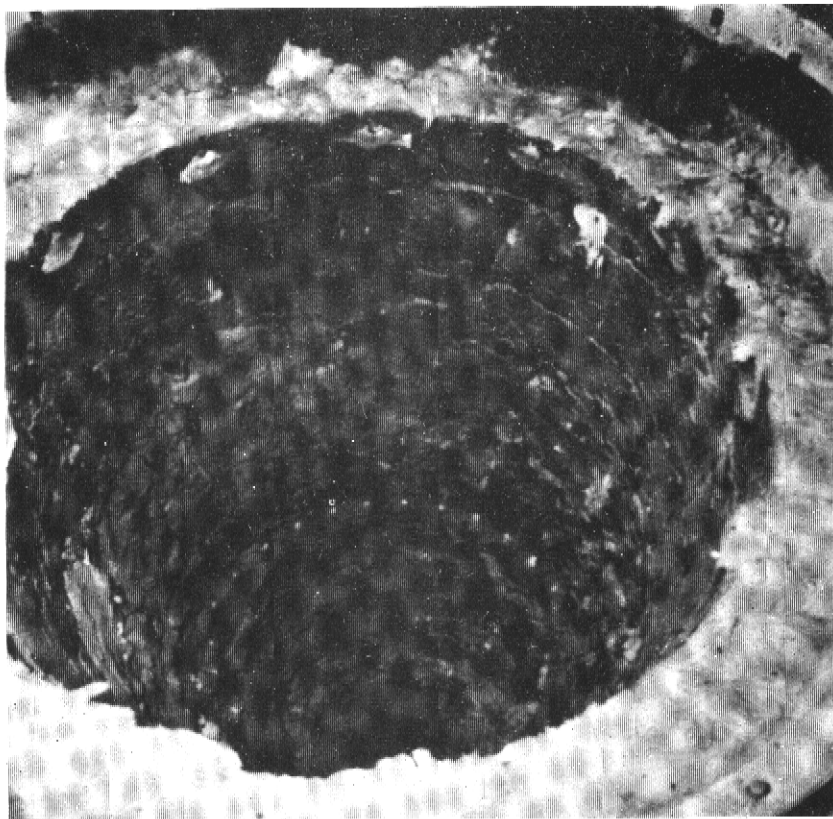


Fig:10