IFA Technical Conference

Amman, Jordan

2-6 October 1994

International Fertilizer Industry Association - Secretariat: 28 rue Marbeuf - 75008 Paris - France
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JOINT VENTURE PHOSPHORIC ACID PROJECT IN JORDAN

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RESUME

L'une des nouvelles tendances de l'industrie des engrais est l'apparition d'un partenariat d'entreprise entre un fournisseur de matière première et un producteur d'engrais en vue de construire des unités de production.

L'Indo-Jordan Chemicals Company Ltd est une entreprise commune formée par Southern Petrochemicals Industrie Corporation Ltd (SPIC) de Madras, Inde et de Jordan Phosphate Mines Company Ltd (JPMC) d'Amman, Jordanie.

IJC a construit et fait fonctionner des unités d'acide sulfurique et d'acide phosphorique à Eshidiya en Jordanie. Le site de l'usine se trouve dans une zone franche jouxtant la mine de phosphate d'Eshidiya.

L'acide phosphorique produit doit être transporté à Aqaba d'où il sera expédié pour servir de matière première aux unités de DAP existantes de SPIC.

En 1993, IJC a signé un contrat avec la société d'engineering, Krebs et Cie, à Paris, France, pour la fourniture des unités d'acide sulfurique et d'acide phosphorique.

L'offre de Krebs comporte le procédé Hydro HH pour l'unité d'acide phosphorique et la technologie Hydro pour la concentration de l'acide phosphorique.

Cet exposé décrit le procédé utilisé pour la fabrication d'acide phosphorique, le contrôle du procédé et insiste sur les aspects économie de coûts et innovations dans le système de rejet de gypse et la conservation d'énergie en optimisant la répartition de l'offre et de la demande d'énergie entre les unités d'acide sulfurique et d'acide phosphorique.

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PHOSPHORIC ACID PROCESS DESIGN BASIS

Phosphoric Acid Plant Capacity

The plant is designed to produce 700 mtpd P_2O_5 as 42% P_2O_5 ex-filter (on 24 hours basis) which is then concentrated to 54% P_2O_5 acid.

The final acid will be merchant grade quality for shipping to India.

Phosphate Rock Feedstock

The plant will use Jordan phosphate rock. Three grades A, B and C will be available. The chemical analyses are given in **Table 1**. The plant will accept feeds of A or B, separately or blends of A and C or B and C.

Table 1

Chemical Analysis of Phosphate Rocks

Component % w/w	Grade A 73/75%	Grade B 70/72%	Grade C 70/72% (Fines)
	Grade A 73/75%	**Grade B 70/72%**	**Grade C 70/72%**
P_2O_5	33.4-34.32	32.00-33.00	32.00-33.00
CaO	50.00-51.50	50.60-52.10	50.60-52.10
SiO_2	3.00-5.00	4.00-8.00	4.00-8.00
CO_2	3.00-4.00	5.00-6.00	5.00-6.00
F	3.50-4.00	3.60-3.80	3.50-3.80
Cl	0.03-0.06	0.04-0.06	0.04-0.06
Fe_2O_3	0.15-0.40	0.20-0.50	0.20-0.50
Al_2O_3	0.20-0.40	0.30-0.60	0.30-0.60
Org. C	0.10-0.15	0.10-0.20	0.10-0.20
SO_3	1.00-1.50	1.00-1.50	1.00-1.50
Na_2O	0.40-0.60	0.40-0.60	0.40-0.60
K_2O	0.02-0.04	0.04-0.06	0.04-0.06
MgO	0.10-0.20	0.20-0.25	0.20-0.25
SrO	0.20-0.26	0.20-0.25	0.20-0.25
Free H_2O	2.00-5.00	14.00-18.00	1.00-3.00

PROCESS DESCRIPTION

This process description should be read in conjunction with the flowsheets given at the end of this section.

The plant is designed for a nominal 700 tpd P_2O_5 production capacity. The plant consists of a HH unit which produces 42% P_2O_5 acid, followed by a concentration unit producing the final 54% P_2O_5 acid.

Reaction Section

Reaction of phosphate rock with H_2SO_4 is carried out in the reaction vessels (Items 301, 303 and 312) of equal volume. Two "Reaction Zones" are required; distinguished by differing chemical operating conditions. In "Reaction Zone 1" comprising of Reactors 1A and 1B (301 and 303) an excess of CaO is maintained and in "Reaction Zone 2", comprising of Reactor 2 (312), an excess of SO_4 is maintained.

Phosphate rock is metered at the required rate to Reactor 1A (301). Reaction slurry from Reactor 2 (312) is recycled to Reactor 1A (301) via the Recirculation Pump (340). The quantity of reaction slurry recycled is such as to maintain the correct excess CaO in Reactor 1A liquid phase. The contents of Reactor 1A (301) overflows to Reactor 1B (303) (which operates under the same chemical conditions as Reactor 1A) which in turn overflows to Reactor 2 (312). Sulphuric acid is fed to Reactor 2 (312) to maintain an excess sulphate level. To prevent local high concentrations of sulphuric acid in Reactor 2 the sulphuric acid is pre-mixed in the Acid Mixer (330) with return acid from the Hemihydrate Filter (401).

The quantity of return acid is used to control the overall solids level within the reaction system.

Slurry from Reactor 2 (312) is transferred by the Flash Cooler Slurry Pump (341) to the Flash Cooler (344) which is used to maintain the reaction system at 98-100°C. From the Flash Cooler (344) slurry flows back directly to Reactor 2 (312).

The Flash Cooler (344) gases are drawn by the action of the vacuum system to the Flash Cooler Condenser (354) where the vapours are condensed under controlled conditions to create and control the vacuum in the flash cooler.

The Reaction system (301,303 and 312) is maintained under a slight negative pressure by the action of the Reactor Gas Scrubber Fan to prevent escape of fluorine gases to the plant atmosphere.

Due to the nature of the phosphate rock it is necessary to have provision to feed anti-foam agents to each Reactor (301, 303 and 312). For this purpose a Defoamer Tank (390) and pump (392) are included.

Hemihydrate Filtration

This section consists of two belt filters, operating in parallel. Slurry from Reactor 2 (312) is delivered to the Hemihydrate Filters (401A/B) by the Filter Feed Pumps (335A/B). The first off-take from each Hemihydrate Filter is "cloudy acid" which flows via the Cloudy Receivers (408A/B) to the Return Acid Tanks (431A/B). Product acid (42% P_2O_5) is taken next and flows via the Product Receivers (409A/B) to the Product Acid Tanks (432A/B) and is pumped to the storage tank with the Product Acid Pump (440A/B).

Used process water from the HH Filter Vacuum Pumps (419A/B), is collected in the Cloth Wash Tank (459). It is then pumped (461A/B) to the HH filter cloth and belt wash sprays. The spent liquor drains to the Plant Wash Tank (465). This recycled water is pumped by the Plant Wash Pump (466) to the discharge end of the HH Filters as further cloth wash. The used liquor from this section drains to the Cake Wash Tank (460) from where it is pumped (462A/B) to the HH Filters as final cake wash.

The cake wash water passes through the filter cake, removing residual P_2O_5, to the Weak Wash Acid Pumps (446A/B) via the Filtrate Receivers (412A/B). The weak acid is then pumped by the Weak Acid Wash Pumps (446A/B) back to the HH filters as the second counter-current cake wash.

After passing through the HH cake again it arrives at the strong Wash Acid Pumps (444A/B) via the Filtrate Receivers (411A/B). This liquor is then returned to the HH filters as the first counter-current cake wash.

The washed hemihydrate cake is discharged from the filters to a conveyor belt to take it to the main conveyor which transports it to the gypsum disposal area.

Fumes containing fluorine from the Hemihydrate Filters (401A/B) are extracted via the Filter Hoods (403A/B) to the Final Stage Scrubber by the action of the Exhaust Fan thus maintaining a clean working atmosphere around the filter.

An Anti-Scale Chemicals Tank (490) and Anti-Scale Pump (495) is included to deliver anti-scale agent to various points in the filter. In addition it is necessary to periodically wash the Hemihydrate Filter (401A/B) pipe-lines and this is achieved with the Plant Wash Tank (465) and Plant Wash Pump (466).

Concentration

Two identical units are to be installed. Each concentration unit consists of three principal operations:

a) Acid circulation and heating.

b) Acid boiling and removal of entrained acid from the released vapours.

c) Vapour defluorination, condensation and vacuum control.

42% P_2O_5 phosphoric acid ex-storage is fed to the flash chambers (601/602) of the evaporation units by the variable speed evaporator feed pumps. The feed rate to the flash chamber is automatically controlled as a function of the amount of water removed by evaporation. Acid is circulated continuously by axial flow pumps (605/606) through the flash chambers and heat exchangers (615/616) where it is heated by about 3°C by means of low pressure steam. The exchangers are arranged vertically at an elevation space level below that of the flash chambers such that boiling on the heating surface is eliminated and hence, scale formation is minimised. Reduction of the tendency to form deposits in the exchangers is also achieved by operation with low temperature gradients both along and across the tubes by the use of a high acid circulation rate. The material of construction of the exchanger tubes is impregnated carbon. Concentrated product acid is withdrawn from the flash vessels by means of the strong acid pumps (610/611); boiling point temperature control ensures that only phosphoric acid of the desired concentration is obtained.

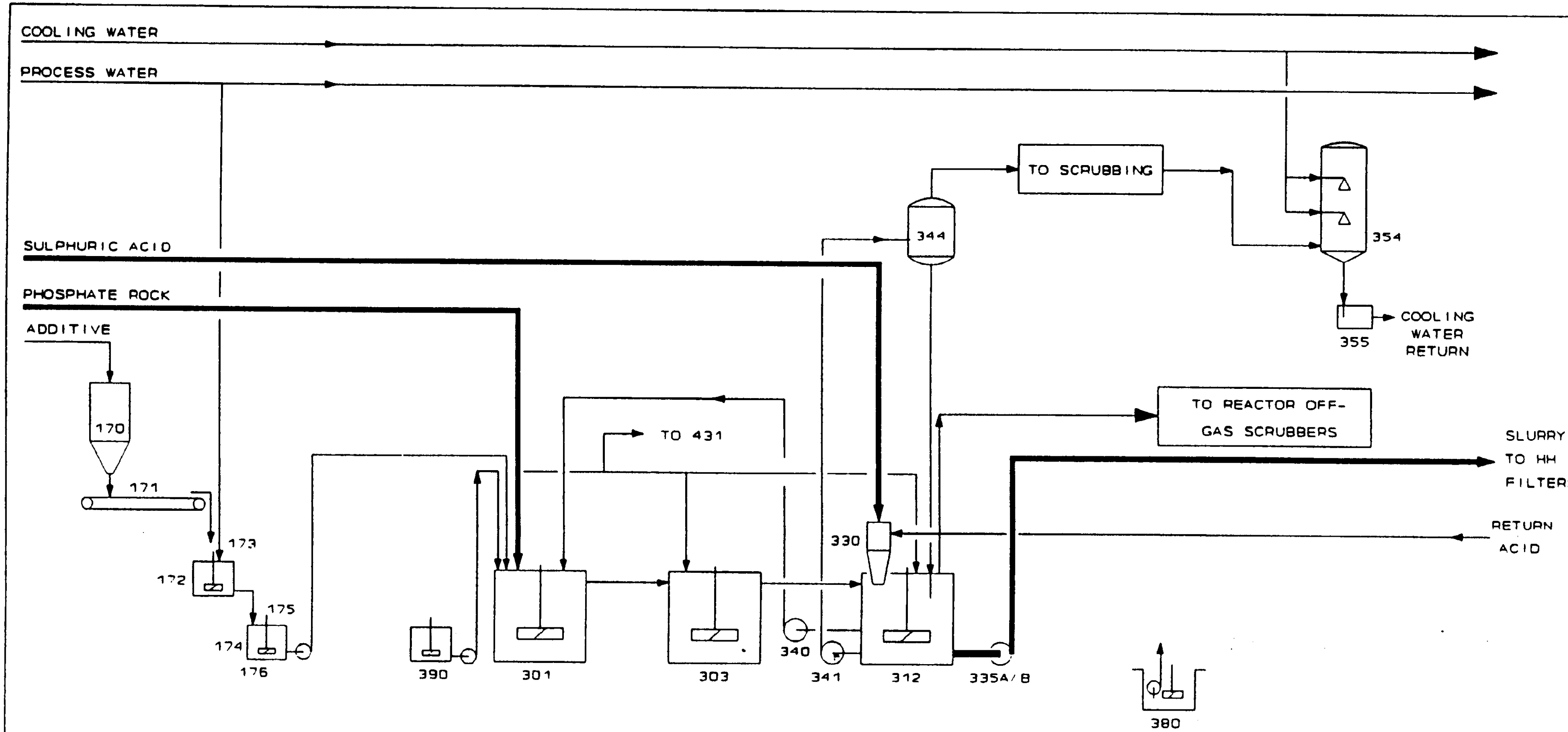
Vapours from the boiling phosphoric acid pass through entrainment separators (650/651) to remove small acid droplets, and then to the fluorine scrubbing units (660/661). From here the vapours pass into the condensers (670/671), where they are condensed by direct contact with water.

The condensate, mixed with cooling water, is collected in the condenser seal tank (690) and is then returned to the cooling water system. The pressure in the evaporator flash chamber is controlled by regulation of the cooling water flow to the condenser.

The steam used for heating the circulating acid in the heat exchanger is reduced to the design pressure prior to entry to the desuperheater and heat exchanger. Steam condensate is continuously pumped out from the plant by means of condensate pumps. A pH metering system monitors the steam condensate for the presence of phosphoric acid and gives a warning signal if it is acid contaminated.

The overall control system provided in the unit has proved highly reliable and permits practically automatic operation, thus the evaporation rate remains constant although during the operating cycle of the evaporator some scale forms on the heat exchanger tubes. As this occurs the steam pressure in the heat exchanger automatically slowly increases up to the design value to maintain the required degree of heat transfer.

Strong product acid is transferred by the evaporator product pumps (610/611) from the evaporators to the strong acid storage section.



MAIN PLANT ITEMS

301 REACTOR 1A
303 REACTOR 1B
312 REACTOR 2
330 ACID MIXER
335 FILTER FEED PUMP
340 RECIRCULATION PUMP
341 FLASH COOLER SLURRY PUMP
344 FLASH COOLER
354 FLASH COOLER CONDENSER
355 CONDENSER SEAL TANK
380 REACTOR SUMP
390 DEFOAMER TANK

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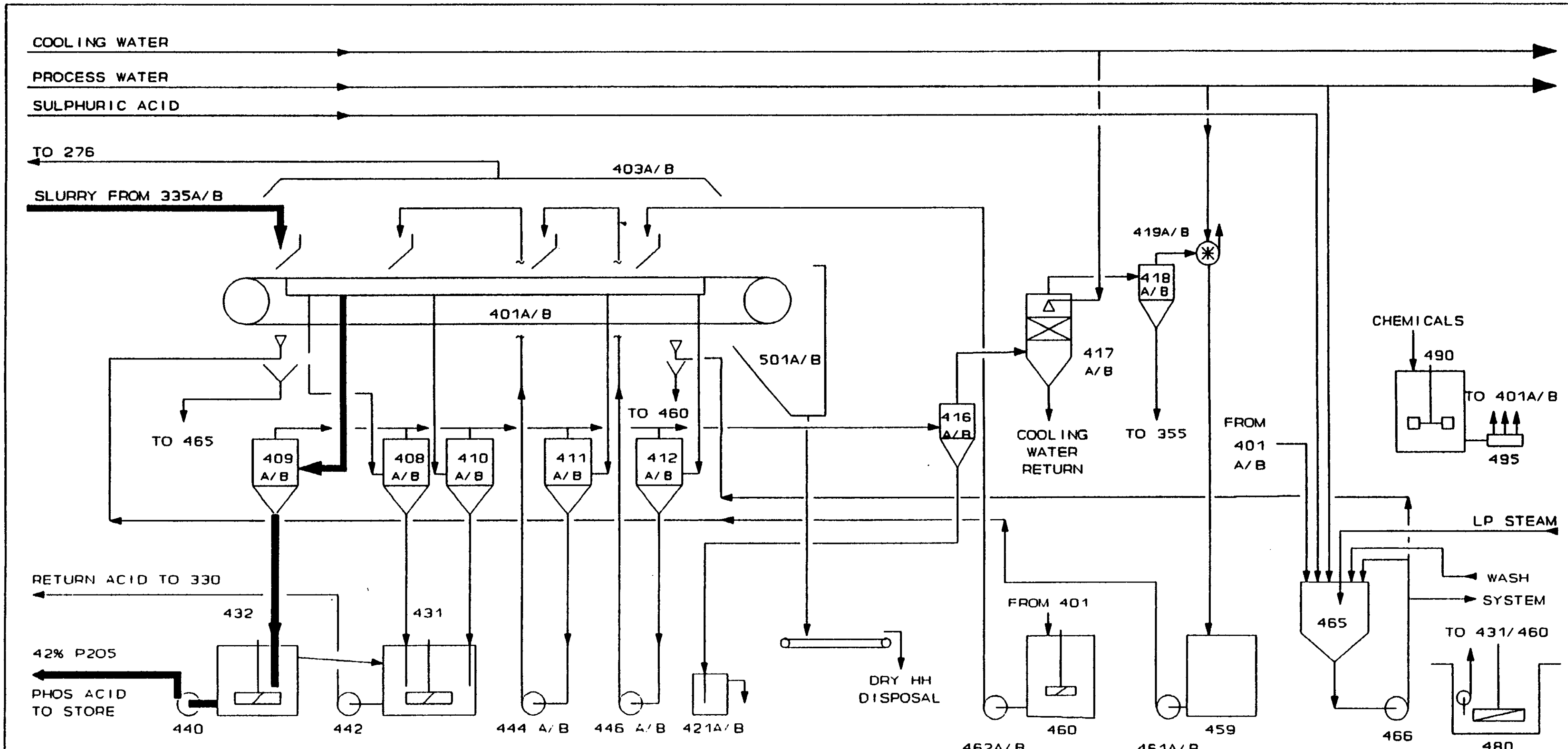
TITLE

HEMIHYDRATE FLOWSHEET
REACTION SECTION



MAIN PLANT ITEMS:-

401A/B HEMIHYDRATE FILTERS
408A/B CLOUDY RECEIVERS
409A/B PRODUCT RECEIVERS
410-412A/B FILTRATE RECEIVERS
416A/B ACID TRAPS
417A/B FILTER CONDENSERS
418A/B ENTRAINMENT SEPARATORS
419A/B FILTER VACUUM PUMPS
431 RETURN ACID TANK
432 PRODUCT ACID TANK
440 PRODUCT ACID PUMP
442 RETURN ACID PUMP
444A/B STRONG WASH ACID PUMPS
446A/B WEAK WASH ACID PUMPS
459 HH CLOTH WASH TANK
460 HH CAKE WASH TANK
461A/B FILTER CLOTH WASH PUMPS
462A/B FILTER CAKE WASH PUMPS
465 PLANT WASH TANK
490 CHEMICALS TANK
501A/B HEMIHYDRATE CHUTES
550 HEMIHYDRATE CONVEYOR

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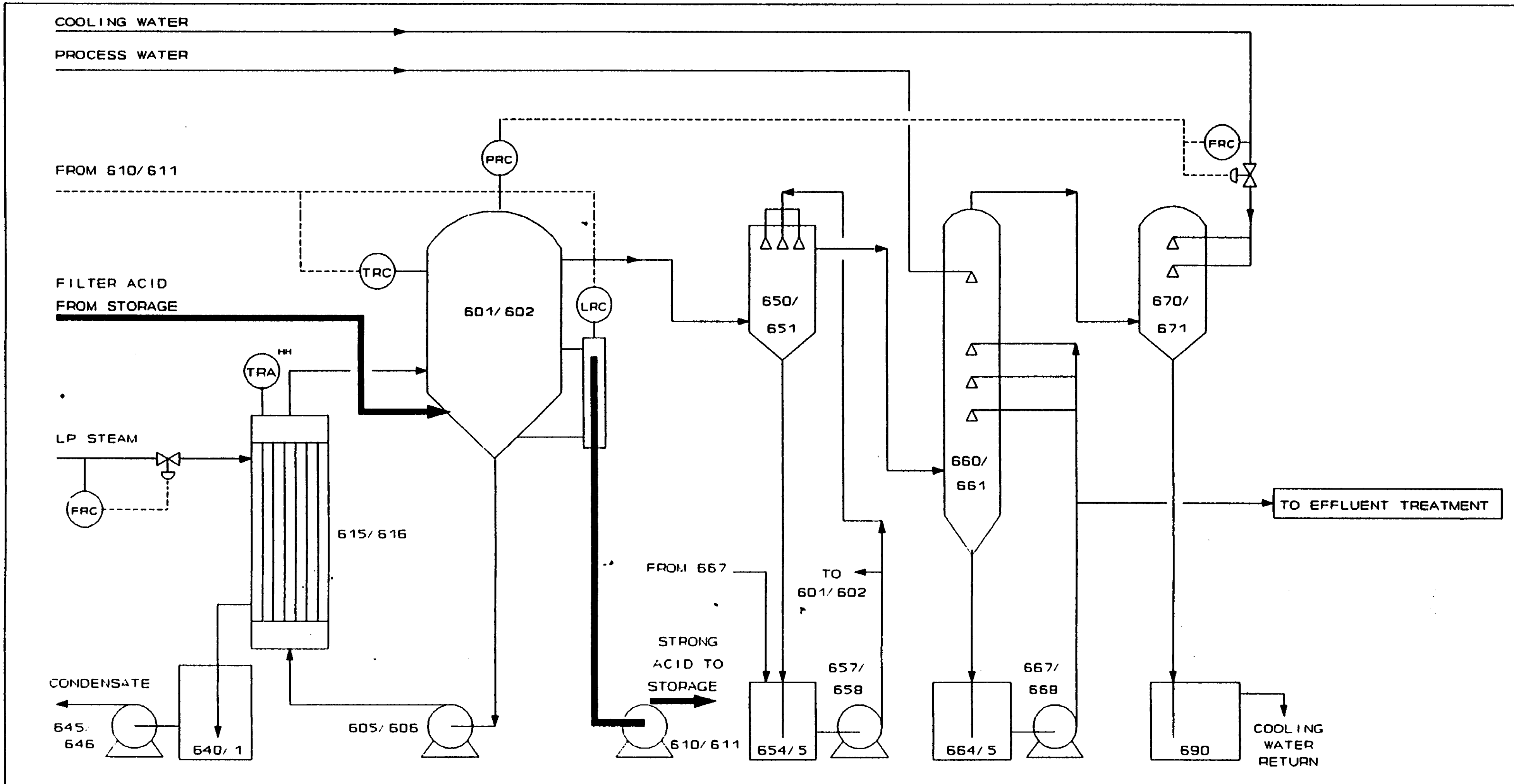
TITLE
HEMIHYDRATE FLOWSHEET
HH FILTRATION SECTION



MAIN PLANT ITEMS:-
601 FLASH CHAMBER
645 CONDENSATE PUMP
605 RECIRCULATION PUMP
610 PRODUCT PUMP
615 HEAT EXCHANGER
650 DROPLET SEPARATOR
657 DROPLET SEPARATOR RECIRCULATION PUMP
660 FLUORINE SCRUBBER
664 FLUORINE SCRUBBER SEAL TANK
667 FLUORINE SCRUBBER RECIRCULATION PUMPS
670 CONDENSER
675 VACUUM PUMP
690 CONDENSER SEAL TANK

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TITLE
CONCENTRATION UNIT FLOWSHEET
INCLUDING FLUORINE SCRUBBING

PROCESS CONTROL

The success of any phosphoric acid process depends on the satisfactory growth of gypsum crystals with a high filtration and washing rate.

Therefore the control of operating conditions in the reaction system is very important.

The IJC plant process control is by DCS, but the principles are those which Hydro has proven and refined in operation over many years.

Sulphate Control

The operating conditions in Reactor 1 are controlled indirectly by controlling:

(i) the SO_4 content in the liquid phase in Reactor 2,
(ii) the phosphate rock feed to Reactor 1 and
(iii) the recycle slurry flow from Reactor 2 to Reactor 1.

In this way we control the CaO to SO_4 ratio in the feeds to Reactor 1 and so create the optimum conditions for producing good filtering crystals.

The control of the SO_4 in the liquid phase in Reactor 2 is maintained by taking a sample of slurry from Reactor 2 once per hour. This sample is filtered in the control laboratory and the liquid phase is titrated against barium chloride with a sulphanazo or rhodizonate indicator to determine the SO_4 content.

If the sulphate content is within the limits set for the process, say 1.9 to 2.1%, then no action is taken.

However if the SO_4 content falls outside these limits then corrective action is taken by adjustment of the set point on the sulphuric acid controller.

It is normal Hydro practice to adjust the set point for 10 minutes to provoke swift change in the SO_4 level in the reactor followed by small adjustment of the set point to give a long-term corrected sulphuric acid feed.

Control of Phosphate Rock Feed

The feed of phosphate rock (A or B or a blend) to Reactor 1 is controlled by a weighbelt, and a series of conveyors takes the rock to the plant.

An integrator is provided to enable the total rock feed to be recorded over any period of time.

This is the primary control parameter for the plant, against which all other feed flows are ratioed.

Recycle Slurry Control

The recycle flow from Reactor 2 to Reactor 1 is controlled by adjustment of the flow controller in the recycle line. Normally this is fixed for a given rock feed rate.

Solids Content Control

The solids content in the slurry in Reactor 2 is controlled by adjusting the flow of return acid from the filter(s) to Reactor 2.

A sample of slurry is taken from Reactor 2 every two hours and the solids content determined. If the result is outside the control limits, corrective action is taken by adjusting the set point of the return acid flow controller.

P_2O_5 Concentration Control

Too high P_2O_5 strength causes a deterioration in the hemihydrate crystals and high slurry viscosity which leads to poor filtration. It also increases the lattice losses in the hemihydrate cake.

The P_2O_5 content of the acid phase of the recycle slurry is determined by the flow rate of cake wash water to the hemihydrate filter.

The P_2O_5 analysis is recorded every 2 hours and, based on the results, adjustments to the cake wash liquor flow rate are made.

When re-setting the cake wash water flow rate no correction to the return acid and filter feed flow rates is required unless a correction to the slurry solids content is required.

Temperature Control

The control of the temperature of the slurry in Reactor 2 is performed by adjustment of the vacuum in the flash cooler.

This is controlled by adjustment of a valve which controls the flow of air to the flash cooler.

As the temperature response of the reaction system is slow, adjustment of the air ingress is done in small increments so as not to disturb the equilibrium conditions drastically.

COST SAVING FEATURES

Low Investment Cost

- No grinding of the phosphate rock is required.
  Therefore the capital cost of a rock grinding mill is avoided.

- Smaller flash cooler equipment (compared with the dihydrate route) because :
  - heat of reaction is significantly less
  - the decrease of slurry temperature in the flash cooler can be much higher without causing scaling.

    Flash cooler equipment includes :
    - the flash cooler vessel
    - the recirculation pump
    - condenser and cooling tower associated
    - piping and ducts

- Smaller equipment for the concentration unit, because the acid needs only to be concentrated from 42% to 54% P_2O_5. The steam consumption is about 0.6 ton per ton of P_2O_5.

- No clarification is required for the 42% P_2O_5. The storage tanks are only raked and a small quantity of sludge returns to the filtration section.

Integrated Water Usage

Savings of process water are maximized with the hemihydrate process because:

- the reduced free water content of phosacid ex filter; the saving is slightly more than 1.0 t/t P_2O_5.
- lattice and free water in the filter cake, saving is about 1.0 t/t P_2O_5.
- lower heat of reaction involves less water evaporated in the flash cooler: the saving is about 0.4 t/t P_2O_5.

Therefore the total savings of process water are about 2.4 t/t P_2O_5 which is equivalent to 70 t/h for the unit.

In the sulphuric acid unit connected with a turbogenerator, the quality of the raw water is such that a large blow-down from the cooling towers is required.
So, savings of water are not as high as generally expected for such a plant.

However savings are maximised in the phosphoric acid unit. A block diagram of water distribution shows that the use of this blow-down is maximized in the phosphoric acid plant and that only the vacuum pumps (and probably some stuffing boxes) will be fed with fresh water.

So, the maximum water consumption of this plant will be easily less than 3,000,000 m^3 per annum.

<u>Optimized energy balance</u>

A preliminary energy balance of the I.J.C. Complex is included in this paper. It shows that the complex will export a large quantity of electricity. This is due to energy savings of the HH process.

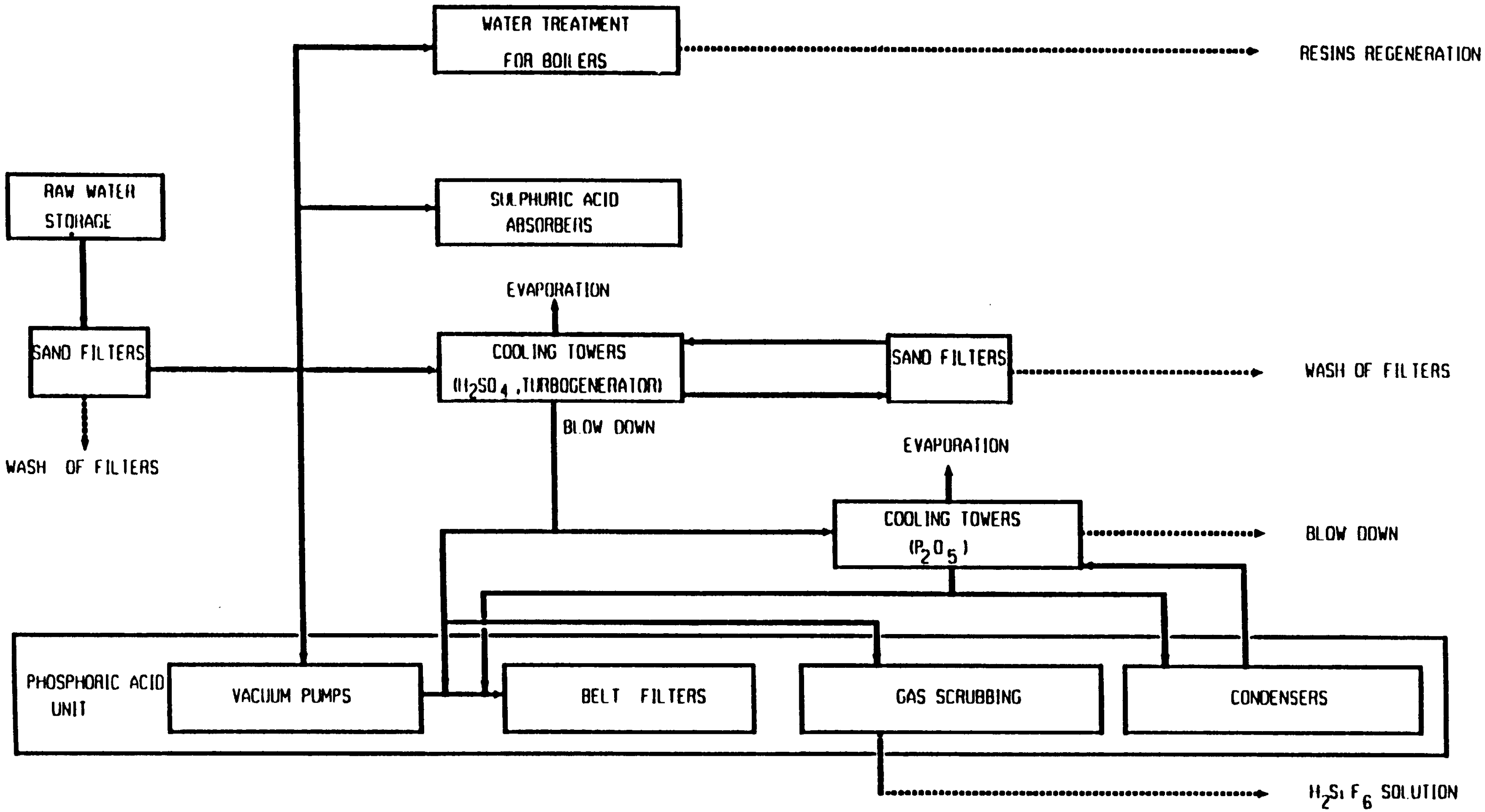
- no grindings, savings about 2000 kWh/h electricity.
- low steam consumption: savings about 1.5 t of steam per t of P_2O_5 i.e. about 44 t/h.

According to the design of turbogenerator, the production of electricity can be increased between about 2,000 and 4,000 kWh/h.

Normally, the superheated steam is expanded in the turbogenerator from 42 bar g to 0.16 bar g. However in this case to decrease the investment cost of this project, the superheated steam only works from 42 bar g to 1.3 bar g. So the production of electricity is reduced. Nervertheless with the use of HH process, it is possible to generate 2,000 kWh/h more than in DH process.

So, all the electricity export is due to the energy savings of the selected process.

WATER DISTRIBUTION OF IJC COMPLEX



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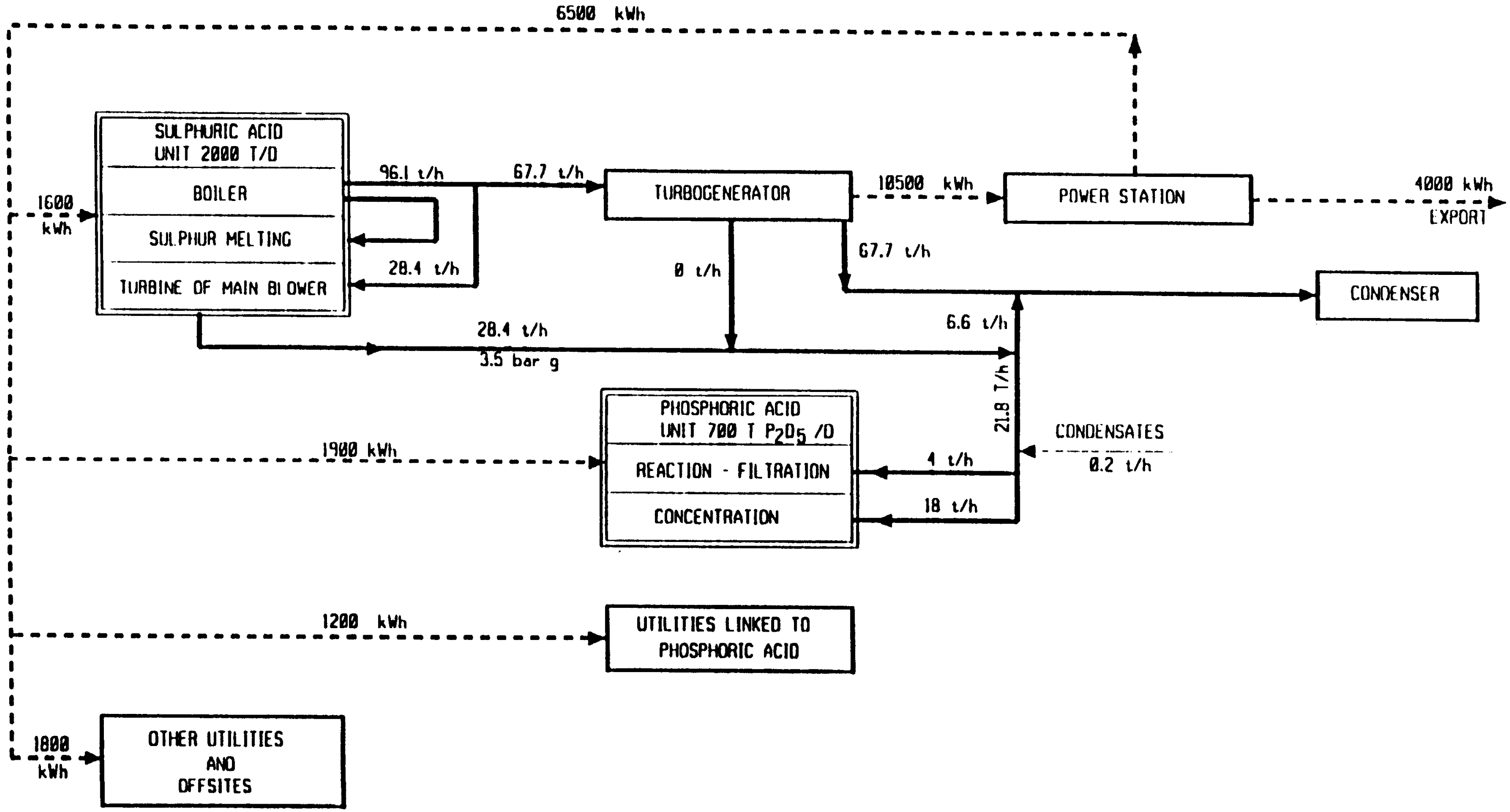
ENERGY BALANCE OF I.J.C COMPLEX



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