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COMPOUND FERTILIZER TECHNOLOGY CENTRE

DP/CPR/85/012

CHINA

Technical report: Production of wet process phosphoric acid *

Prepared for the Government of China
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Barry Crozier, expert on granulation
of compound fertilizers from powder-or-slurry form materials

Backstopping officer: Igor Volodin, Chemical Industries Branch

United Nations Industrial Development Organization
Vienna

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ABSTRACT

Project for Compound Fertilizer Technology Centre at the Shanghai Research Institute of Chemical Industry (SRICI).

DP/CPR/85/012/11-04

The objective of this phase of the project was to furnish detailed information, practical knowledge and research activities on the up-to-date developments in wet process phosphoric acid production, particularly the hemihydrate (HH) and hemidihydrate (HDH) processes.

A series of lectures, seminars and discussion groups on the above subjects took place over the period 10-22 March 1987 at the SRICI.

The activities resulted in a greater appreciation among the staff at the SRICI for the features, benefits and practical application of the HH and HDH processes for phosphoric acid production; and the relevance of the use of this technology in China.

The main recommendation is that the foundations provided by these lectures, etc., be built upon by industrial experience of the technology. This can be achieved by visits to and training in industrial plants and research stations overseas. And that this knowledge be applied to the planning, evaluation and implementation of new fertilizer projects, by the SRICI.

CONTENTS

INTRODUCTION

The objective of this phase of the project was to furnish detailed information, practical knowledge and research activities on the up-to-date developments in wet process phosphoric acid production, particularly the hemihydrate (HH) and hemidihydrate (HDH) processes.

This was achieved by a series of lectures, seminars and discussion groups on the above subjects held at the Shanghai Research Institute of Chemical Industry (SRICI) over the period 10-22 March 1987. Included in the activity was a visit to the No.1 phosphoric acid pilot plant and a discussion on the design of the revamp of this plant.

The discussions were an especially valuable part of the activities, giving the opportunity of a wide exchange of views and detailed examination of the unit operations involved in the technology. These took up about one-third of the time period of the mission.

RECOMMENDATIONS

1. To consolidate and develop the activities of SRICI in the field of phosphatic and compound fertilizers it is necessary to gain wider industrial experience of production by visits to and training in industrial plants and research stations overseas.

2. In order to provide further detailed information and practical knowledge on the production of NPK compound fertilizers and phosphate intermediates, it is recommended that some or all of the following specialists be invited to give lectures at the SRICI:

DAVID IVELL : Specialist in the pilot plant investigation and industrial plant production of DAP and NPK compound fertilizers.

STEPHEN SMITH : Specialist in the design and operation of industrial plants for NPK compound fertilizer production.

NORMAN WARD : Specialist in the development and industrial plant application of processes to produce solid phosphate intermediates.

All can be contacted at:

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Levington Research Station
Ipswich
Suffolk IP10 OLU
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Telex: 98273 FERTS G

3. In order to evaluate and therefore optimise the fertilizer process production routes it is necessary to have agronomists more clearly define the type of fertilizer to be applied and indicate the capacities required.

4. In order to ensure that the developments and expansion of the fertilizer industry in China fully benefit from the activities and expertise of the Compound Fertilizer Technology Centre (CFTC) of the SRICI, then the CFTC should be more closely involved in the planning, evaluation and implementation of new fertilizer projects in China.

5. It would be helpful if in the future, visiting experts be given more details of the programme which they are to follow. The experts can therefore prepare lectures on the specific subjects and provide an outline in advance to the SRICI who in turn can plan to ensure the most efficient use of the expert's and SRICI staff time. Communications would be more effective if directly between the expert and the SRICI rather than through a third party.

I. MAIN DUTIES DURING MISSION

The specific duties of the mission were:

- To give lectures on the industrial technique of wet process phosphoric acid production, and give advice on the feasibility of using specific technologies in China.
- To describe industrial plant equipment and advise on the materials of construction used in phosphoric acid production.
- To introduce the activities of and new achievements made at the Norsk Hydro Levington Research Station.
- To discuss different routes involved in the production of compound fertilizers.

The objectives behind these duties were to expand the knowledge and understanding of particular techniques for wet process phosphoric acid production, specifically the HH and HDH processes. These processes have been developed by Norsk Hydro Licensing UK and though implemented industrially by many operating companies any discussion of the technology cannot avoid recognising Norsk Hydro's ownership of the technology.

Because phosphoric acid is an important intermediate in the route to producing compound fertilizers, it is essential that any scheme aimed at strengthening the know-how and activities of the SRICI in compound fertilizers, should include a comprehensive appreciation of the production of intermediate products such as phosphoric acid.

The above duties were fulfilled by giving a series of lectures, and holding seminars and discussion groups. These were attended by, on average, 30 staff members. The names and specialisation of senior counterpart staff are given in Annex 1. The programme followed is given below.

Project of Compound Fertilizer Centre, SRICI
March 1987
Programme

10/3	am	Arrive in Shanghai.
	pm	Meeting with Mr Dai Yuan Fa and Mr Zhang Guo Xin to discuss schedule and objectives and finalise programme.
11/3	am	Visit SRICI, meeting with Mr Xiao Xi Zhen, Vice-President, introduction to SRICI activities, tour of SRICI facilities.
	pm	Lectures on basic principles of dihydrate (DH) and hemihydrate (HH) processes, chemistry of HH process.
12/3	am	Discussion on principles of HH process. Lectures on basic principles of hemidihydrate (HDH) process.
	pm	Discussion on HDH process principles. Lectures on features and benefits of HH and HDH processes, discussion. Lectures on raw material and utility consumptions in HH and HDH processes, discussion. Lectures on production costs of HH and HDH processes, discussion. Lecture on implications for electricity co-generation of HH and HDH processes.

13/3 am	Lectures on conversion of existing DH plants to HH/HDH process, industrial experience and case studies, discussion.
pm	Lectures on performance of Florida and Morocco phosphates in HH and HDH processes, discussion.
14/3 am	Detailed presentation of P&I diagrams of HDH process, discussion.
pm	Industrial plant design, discussion. Materials of construction. Equipment selection. Discussion.
17/3 am/pm	Visit to SRICI Pilot Plant, techniques for phosphate rock evaluation.
18/3 am	Discussion of laboratory and pilot plant techniques for phosphate rock evaluation. Scale-up of results.
pm	Lectures on calcium sulphate crystal characteristics, discussion. Control of operation conditions in industrial plants. Methods of chemical analysis. Discussion.
19/3 am	Industrial plant operating procedures. Plant maintenance. Down-time analysis. Discussion.
pm	Discussion on application of HH/HDH technology in China. Lecture on disposal of phosphogypsum.

20/3 am	Activities and projects at Levington Research Station. Discussion. Evaluation of project of Compound Fertilizer Centre: Next step. Granulation and DAP technology.
pm	Report preparation. Agree objectives for the future.

The lectures were presented (through an interpreter) with the aid of overhead projector slides, copies of which are provided in Annex 2. In addition, 35mm slides were used to show actual factory layout and specific equipment type. Further documentation in the form of written reports and manuals, heat and mass balances and piping and instrument diagrams were provided to support the lectures. Copies of these are given in Annexes 3, 4 and 5.

The lectures were designed to give initially an appreciation of the basic principles of the process routes, with their main features and benefits. However, the majority of the presentations centred on the industrial application of the technology. They covered items such as calculation of heat and mass balances, equipment design, equipment selection, materials of construction, plant layout and P&I diagrams. Following this, methods of full scale plant operation and control were presented. The importance of relating R&D activities to industrial production was stressed.

The seminars naturally included explanation of laboratory and pilot plant techniques for phosphate rock evaluation, and process development. The activities of the Levington Research Station were discussed.

Following each lecture, extensive discussions took place, involving questions both numerous and comprehensive. These were stimulating and rewarding sessions which were a good measure of the usefulness of the activities.

On 17 March 1987 a visit was made to the No.1 phosphoric acid pilot plant which is located at a site annexed from the main part of SRICI. The pilot plant was built in the 1960's. At present SRICI are preparing to revamp it and launch a new campaign of R&D in the production processes. A detailed inspection of the existing equipment took place. This was followed by a comprehensive examination and discussion of the design and flowsheet of the proposed revamped plant. The design was sound, though advice was given on modifications which would help avoid silica blockages in the fluorine recovery unit.

It was agreed that the revamped pilot plant would play an important part in the training of staff and operators of new industrial phosphoric acid plants as they come on-stream in China.

II. PHOSPHORIC ACID PRODUCTION TECHNOLOGY

Phosphoric acid, as a fertilizer intermediate, has been produced on an industrial scale for about 40 years. Traditionally the production process used was known as the dihydrate (DH) process, so called because of the nature of the by-product. However, since 1970 a process developed by Norsk Hydro Licensing, UK, has been employed in industrial phosphoric acid plants worldwide. This is known as the hemihydrate (HH) process (because of its slightly different by-product from the DH process), and since 1974 an extension of the HH process known as the hemidihydrate (HDH) process has been utilised in industrial production of phosphoric acid.

The main features of the HH and HDH processes are:

- The substantial energy saving in these production routes. Also, when by-product energy is available from other chemical plants, for example a sulphuric acid plant, instead of being used in the phosphoric acid process (as was the case traditionally) it can, when the HH and HDH process is employed, be used to generate an increased electricity capacity.

- The simplicity of operation of the HH process makes it ideal for a location where there is limited prior experience of chemical/fertilizer manufacture. Because it has fewer unit operations than other phosphoric acid processes, it also benefits from a lower capital cost.

- The HDH process achieves a phosphate (P_2O_5) recovery efficiency of 98.5-99% whereas traditional processes obtain around 94%. This is clearly beneficial for all locations and circumstances but becomes even more significant when the cost of the raw material (phosphate rock) is high due to mining, beneficiation or transport charges. It also saves on the consumption of the other main raw material - sulphuric acid.

- There is extensive industrial experience with the Norsk Hydro HH and HDH processes which has allowed the technology to be developed for a comprehensive range of raw materials and factory locations and conditions. (This cannot be offered by similar technologies).

  As a result, the application of the technology to China would not create new problems or circumstances which had not already been faced and overcome elsewhere. Moreover, a number of Chinese phosphate rocks have already been tested and found to be suitable for use in the HH and HDH processes.

Process descriptions are given below.

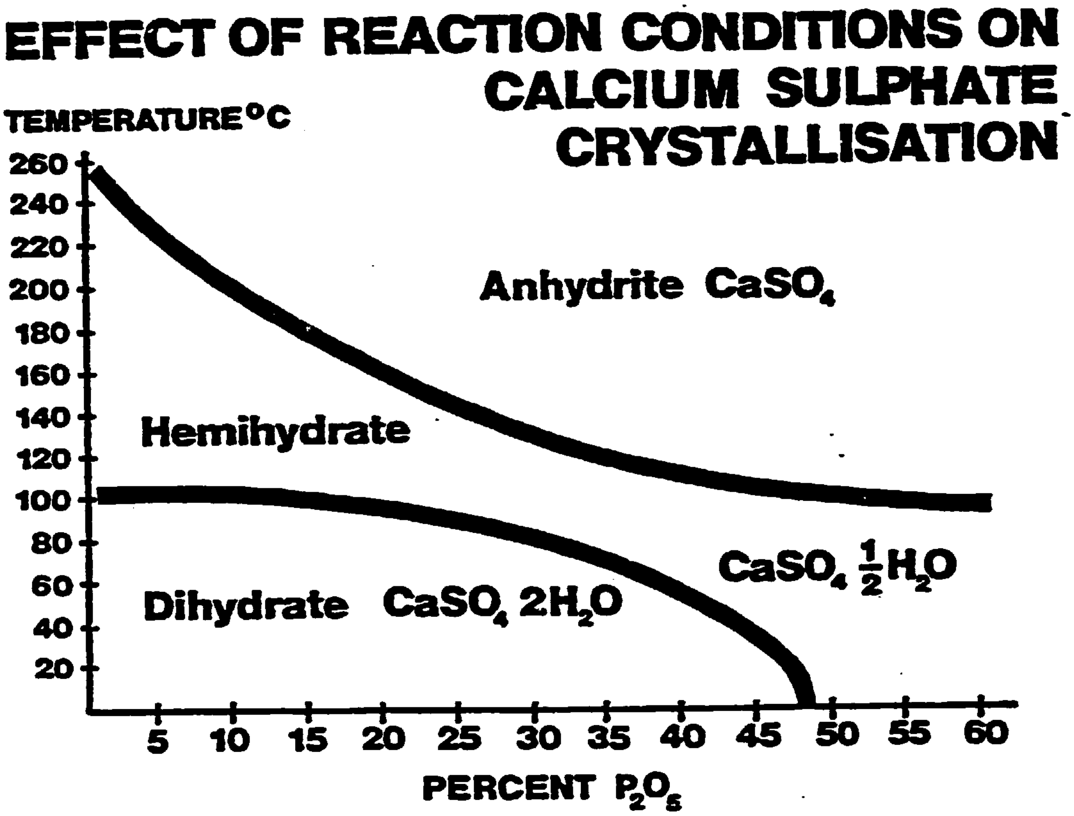
A. Process Description

The chemical reaction involved in phosphoric acid production can be expressed:

$$Ca_{10}(PO_4)_6F_2.CaCO_3 + 11H_2SO_4 + 11n\ H_2O \longrightarrow$$
$$6H_3PO_4 + 11CaSO_4nH_2O + 2HF + CO_2 + H_2O$$

the degree of hydration, n, being 0, ½ or 2 depending on whether the calcium sulphate is in the form of anhydrite, hemihydrate or dihydrate. This, in turn, is determined in accordance with the phase diagram shown in Figure 1.

Figure 1

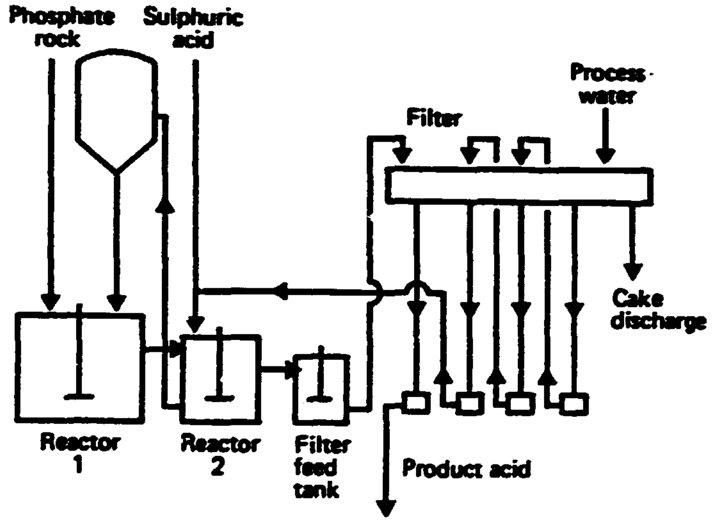


It can be seen that, to produce calcium sulphate in the dihydrate form, conventional processes operate typically at 80°C and 30% P_2O_5. Hemihydrate processes are controlled at 100°C and 40-50% P_2O_5, so that no separate concentration stage is required and significant reduction in energy consumption is achieved. Further, a number of unit operations necessary for a dihydrate process (e.g. intermediate acid storage, concentration, clarification) are avoided with the hemihydrate route.

Hemihydrate (HH) Process

The major operations involved in the HH process are shown in Figure 2. Reaction and filtration are the key process steps. The hemihydrate reaction proceeds in two distinct zones. It is therefore necessary to have at least two vessels or compartments. The preferred volumetric ratio is 2:1. Some filter feed buffer capacity between reactor 2 and the filter is desirable but not essential.

Figure 2



Hemihydrate Process

Phosphate rock is fed to reactor 1, sulphuric acid and dilute phosphoric acid ("return acid" from the filter) are fed to reactor 2. Slurry from reactor 2 is recycled to reactor 1, thus exposing the phosphate rock to sulphate ions under controlled chemical conditions. Heat is removed by air or vacuum cooling to maintain the reaction slurry temperature at 98-100°C.

Acid can be produced at between 40-52% P_2O_5 depending on downstream requirements.

The product acid and hemihydrate are separated by a horizontal vacuum filter with counter current wash stages. The product acid from the filter passes directly to storage. It does not require clarification or solids removal.

The HH process is simple with few unit operations. It is especially suited to mine site operations where phosphate rock is readily available at a relatively low cost.

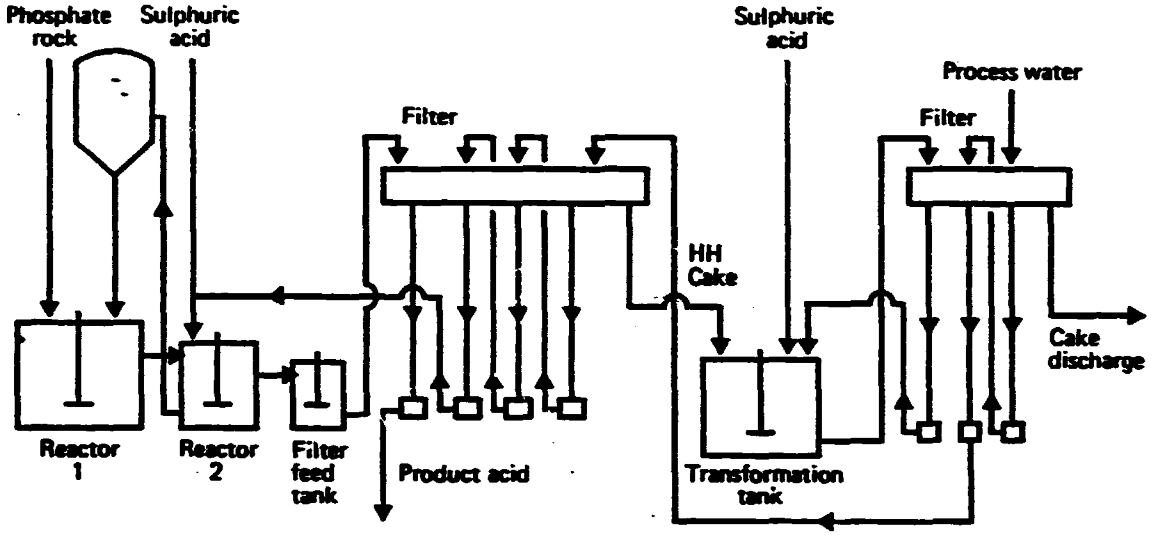
Hemidihydrate (HDH) Process

The HDH process is a development of the HH route whereby the P_2O_5 recovery efficiency is increased to around 98.5%.

In the HH process, as in the dihydrate process, the P_2O_5 recovery efficiency is affected by co-crystallisation of phosphate in the calcium sulphate crystal lattice. When phosphate rock costs are increased for example by the cost of transportation and handling, the lattice P_2O_5 loss from the phosphoric acid process can be reduced by using the HDH route. The addition of a transformation stage to the HH process, allows hemihydrate to be re-crystallised to dihydrate thus releasing lattice P_2O_5 into solution from which it can be recovered. This makes the HDH process particularly attractive for manufacturing locations remote from the source of phosphate rock.

A simplified flow diagram of the HDH process is shown in Figure 3 over page.

Figure 3



Hemidihydrate Process

The first stage of the process is almost identical to the HH process already described.

In the transformation stage the hemihydrate cake is discharged from the first stage filter into an agitated vessel. The operating conditions are controlled to ensure complete transformation of hemihydrate to dihydrate and to allow sufficient time for the dihydrate crystals to grow. The rate of transformation is increased by the addition of a small feed of sulphuric acid. Nearly all the lattice P_2O_5 co-precipitated with the hemihydrate is released into the liquid phase.

The dihydrate is then filtered and the cake washed with process or pond water. The filtrate containing the released P_2O_5 is returned to the hemihydrate reaction stage as the last wash on the hemihydrate filter.

B. Features

The main features of hemihydrate technology are energy saving, acid quality, simplicity of operation (for HH route), and high P_2O_5 recovery (for HDH route).

Energy Saving

Because the HH process can produce strong acid directly from the filter, it is not necessary to concentrate the product acid prior to its use in the downstream plants. Thus the evaporation step is avoided, therefore a saving of 1.5-2 tonnes steam per tonne P_2O_5 produced, can be achieved. When this steam has to be generated in a boiler, there is clearly a saving in fuel costs to be made. However when the steam is by-product from a sulphuric acid plant, the steam saving can be re-directed for use elsewhere in the fertilizer complex, or be used in electricity co-generation. When a condensing set turbo alternator can be used following a HH/HDH operation, the electricity generation is increased by 60% over that generated by a back pressure set required by dihydrate operation.

Another aspect of energy saving with the HH process is through a reduced rock grind requirement. The HH process will accept rocks of a coarser grind than a dihydrate process. This is illustrated by comparing the particle size distribution required by the different process routes. This is shown in Table 1.

Table 1

Comparison of Rock Grind Requirement for HH/HDH and DH Processes

BSS Sieve size (Mesh)	Aperture (Microns)	Particle Size Requirement % Through		72% BPL Khouribga Morocco as received % Through
		HH/HDH Plant	DH Plant	
10	1700	100	-	100
30	500	75	100	87
60	250	25	95	65
100	150	-	60	22
200	75	-	30	-

Acid Quality

During storage solid material will deposit in wet process phosphoric acid. The solid is a combination of calcium sulphate, and complexes of heavy metal ions such as fluosilicates of iron, aluminium and magnesium. A proportion of the calcium sulphate is in the form of fine particles which have passed through the filter cloth. This sludge needs to be removed because of the difficulties it creates in storage and transport vessels, in addition it can reduce the quality and concentration of the downstream product if it remains.

Clarification can be achieved by a number of methods, the most common being the use of sufficient storage capacity to hold the acid until post precipitation is complete and the sludge has settled and can be removed from the tanks. This process can be accelerated by the use of flocculating agents in the settling vessel. Solid removal can also take place with hydro-cylcones, solid bowl and nozzle centrifuges, Lamella settlers and filters.

The solids content of strong acid produced via the dihydrate route is higher than that from hemihydrate processes. This is because of the change in the solubilities of calcium and sulphate ions at different P_2O_5 concentrations. In dihydrate process acid at approximately 30% P_2O_5 ex filter, the solubility product of CaO and SO_4 at 30% P_2O_5 is approximately 0.8. In concentrated dihydrate process acid ex-vacuum evaporator at 50% P_2O_5, the solubility product of CaO and SO_4 is approximately 0.2.

The fourfold change in solubility causes the calcium sulphate to precipitate in the concentrated dihydrate acid. There is a similar tendency with fluosilicates and other complexes. Similar problems are not experienced with concentrated acid from hemihydrate processes. The solubility of calcium sulphate, etc. is the same in the hemihydrate acid but because it is produced at high strength directly in the reaction system all solids are in equilibrium with the acid phase, thus are filtered with the hemihydrate cake, and removed from the acid. As the hemihydrate acid ages and cools the equilibrium is not significantly disturbed therefore further precipitation is negligible.

An additional factor influencing solids precipitation is that the reaction in the hemihydrate mode causes a higher proportion of Al_2O_3 to be linked with the hemihydrate crystals than in the dihydrate process. Consequently hemihydrate acid contains less Al_2O_3 than dihydrate acid, thus reducing the proportion of Al_2O_3 in the acid available for the formation of complex salts.

A further point to note is that hemihydrate acid has a lower SO_4 content than dihydrate acid.

There are two benefits from the improved quality of the hemihydrate acid. Firstly, because of the relatively low solids content it is not normally necessary to clarify it before downstream use or transport, thus saving capital and operating costs, and the added P_2O_5 losses which occur with the increased handling operations can be avoided. In addition because the solids content of any acid lowers the analysis of the downstream product, the hemihydrate acid with its lower solids content improves the analysis of downstream product and makes it easier to achieve a required grade.

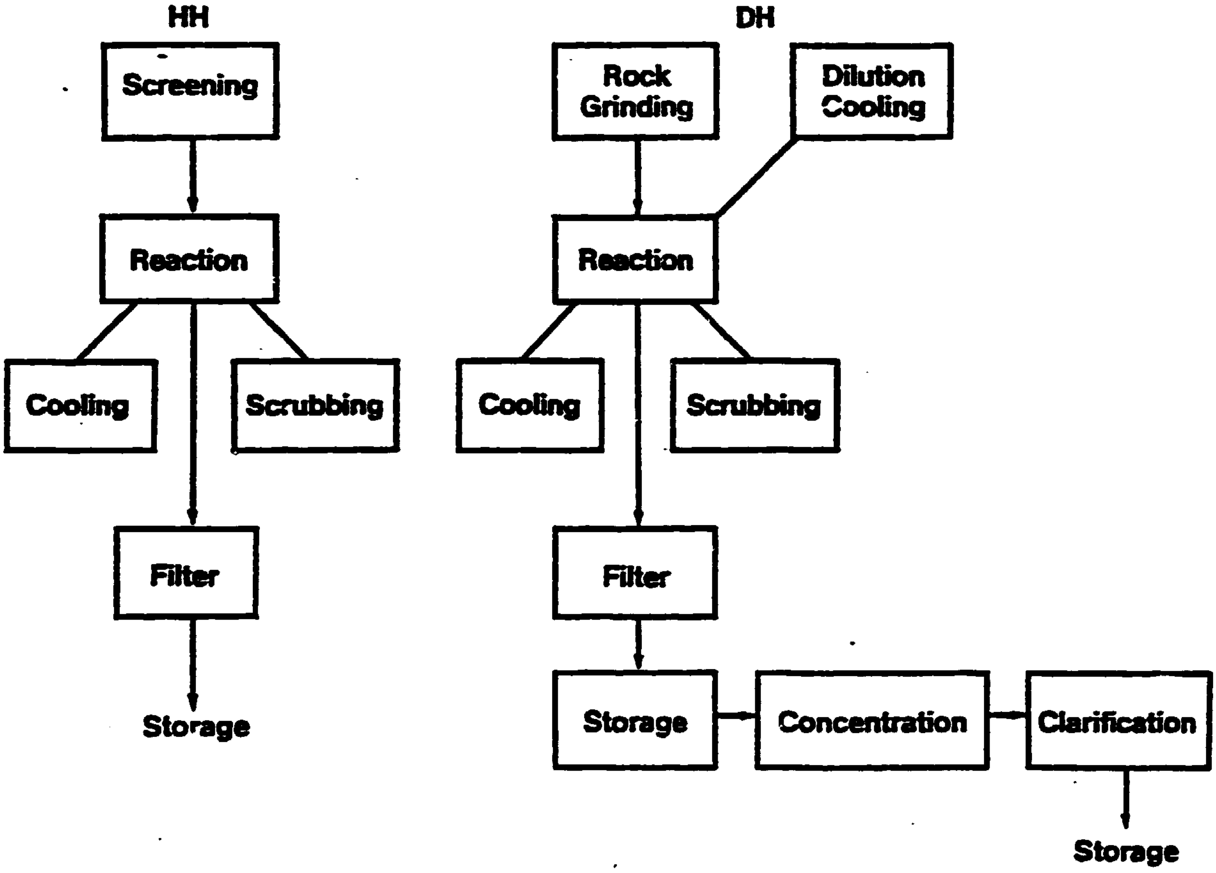
Furthermore the lower Al_2O_3 level in hemihydrate acid improves the chemical analysis of downstream products. Low aluminium improves citrate P_2O_5 solubility and increases nitrogen incorporation.

Simplicity of Operation

Figure 4 shows the difference between the number of unit operations required by the HH and dihydrate processes. As the HH process avoids, rock grinding, acid concentration, and clarification, the number of unit operations is substantially reduced thus leading to maintenance and capital cost savings.

Figure 4

Phosphoric Acid Production: Unit Operations



P_2O_5 Recovery Efficiency

The insoluble P_2O_5 loss from the HDH process when producing 42% P_2O_5 acid from 70 BPL Khouribga Morocco phosphate is given in Table 2.

Table 2

Insoluble P_2O_5 Loss from HDH Process

Item	% P_2O_5 in Dry Filter Cake
Unreacted Rock	0.05
Lattice	0.15

The P_2O_5 recovery efficiency based on these insoluble losses is over 99%. However, as with all phosphoric acid processes, there is additional water soluble loss from the filter. Typical results from a commercial HDH plant using mainly Khouribga Morocco phosphate shows water soluble losses of between 0.1-0.15% (of dry weight gypsum) which gives a total P_2O_5 recovery efficiency of between 98.0-98.5%.

C. Production Costs

The factors affecting production cost include raw materials consumptions, utility consumptions and the usage of other chemicals. All these factors, the most significant of which are phosphate rock, sulphuric acid and steam, are considered in more detail below.

Phosphate Rock

The consumption of phosphate rock is determined by the overall efficiency of the phosphoric acid production process. The single stage hemihydrate has a recovery similar to dihydrate, whilst that of the two-stage hemidihydrate is significantly higher. In both hemihydrate cases the losses associated with intermediate storage, evaporation and clarification are avoided.

Effective costs of phosphate rock are sensitive to freight charges. Thus the potential savings of the HDH route are of greatest interest when freight, mining or beneficiation costs are high.

Sulphuric Acid

The consumption of sulphuric acid per unit of phosphoric acid produced depends on the analysis of the phosphate rock and the overall P_2O_5 recovery. Thus comments made above in relation to rock consumption also apply here.

In addition, the sulphuric acid consumption is affected by the free SO_4 in the product. Since 40-50% P_2O_5 hemihydrate acid contains 2% SO_4 compared to 4-5% SO_4 in concentrated dihydrate acid, the usage of sulphuric acid is reduced.

Electricity

Power consumed in the hemihydrate process is almost identical to that consumed by the dihydrate route including concentration, and amounts to about 100 kWh/t P_2O_5. However the ability of the hemihydrate process to consume coarse phosphate rock avoids the usual grinding operation. This allows an important saving of electricity to be made amounting to about 30 kWh/t P_2O_5.

Steam

In the dihydrate route the concentration stage is a major consumer of steam. When producing 45% P_2O_5 acid from 28% P_2O_5 filter acid this amounts to nearly 2 tonnes steam/tonne P_2O_5. Apart from a small consumption for heating filter cake washing water, the hemihydrate process totally avoids the need for this steam, thus offering a major potential saving.

In the case where steam has to be generated deliberately by the combustion of hydrocarbon fuel, a saving representing the full value of the fuel is possible. Furthermore the operating and maintenance costs of the boiler can also be avoided.

Alternatively in the case where steam is available as a by-product, e.g. from an adjacent sulphuric acid plant, another use for the surplus steam can be considered. Of increasing interest is its utilisation for generation of electricity. Our information suggests that electricity generation can be increased by approximately 60% when using a condensing set turbo-alternator following a HH/HDH operation compared to a back-pressure set which is required with DH operation. The value of the electricity can range from the fuel cost avoided by the utility company to the full commercial price of power to the consumer. This will depend on local circumstances, however there is considerable current interest in co-generation projects.

Process Water

Considering the water balance for the hemihydrate route it is evident that less process water is called for (as filter cake washing water eventually being recycled to the reactor) because of the higher product strength.

This is even further reduced for the single stage process since less water of crystallisation is rejected with the hemihydrate filter cake.

A saving in process water can be an important consideration when it is in short supply.

Cooling Water

The cooling load of a hemihydrate unit is less by 20-40% than the equivalent dihydrate plant. This results from the lower heat of reaction and the greater natural losses by operating at a higher temperature. Consequently the load on a vacuum cooler condenser is reduced leading to lower cooling water consumption.

Avoiding the consumption of cooling water in the dihydrate concentration unit condenser permits a further substantial saving.

Defoamer

Because of the higher density and viscosity of a hemihydrate reaction slurry there is a tendency for gasification to occur. This is controlled to avoid reducing the reactor retention time and other operational difficulties by use of defoaming agents. The usage for hemihydrate, whilst not being excessive, is slightly higher than usual for the dihydrate route.

Chemicals

As explained earlier it is sometimes necessary with certain rocks to introduce a chemical additive to the filter system to avoid premature hydration of hemihydrate crystals.

The chemical is widely available and is of modest cost.

Operating Cost Tables

Table 3 illustrates the comparative operating costs for producing 45% P_2O_5 acid by the single stage hemihydrate (HH) process, the two-stage hemidihydrate (HDH) process and the dihydrate route. 70 BPL Khouribga Morocco rock is assumed. Table 3 refers to a European location where the rock cost includes freight charges.

It can be seen that the single stage hemihydrate process shows a cost advantage of over US $24/tonne P_2O_5. When using the two-stage HDH process, this increased to over US $36/tonne P_2O_5.

These figures have been calculated using a steam value equivalent to burning hydrocarbon fuel for its generation, namely US $11/tonne. For by-product steam from a sulphuric acid plant it is usual to allocate a value of about US $6/tonne.

Even at this reduced steam value the savings are attractive - over US $15/tonne P_2O_5 for the HH process and over US $27/tonne P_2O_5 for the HDH route.

Individual producers' costs are unlikely to coincide exactly with the examples given, but insertion of the specific figures will give a similar outcome.

Production of 50% P_2O_5 acid is possible with the HH process but at the penalty of reduced recovery of P_2O_5. As a result it is common for best economics to produce filter acid at 45% P_2O_5 and evaporate to the required higher concentration. In this case the production cost benefit quantified above will be retained.

No recovery penalty results in the production of 50% P_2O_5 acid by the HDH process. In this case further savings in steam consumption and cost benefits can be achieved.

Table 3

Typical Raw Material and Utility Costs in US Dollars per tonne P_2O_5 Producing 45% P_2O_5 Phosphoric Acid at a European Location From Morocco 70 BPL Rock

Item	Units	Price (US $)	Hemihydrate (HH)		Hemidihydrate (HDH)		Dihydrate (DH)	
			Consumption per tonne P_2O_5	Cost (US $)	Consumption per tonne P_2O_5	Cost (US $)	Consumption per tonne P_2O_5	Cost (US $)
Phosphate Rock	tonnes	50	3.36	168.00	3.20	160.00	3.36	168.00
Sulphuric Acid	tonnes	45	2.70	121.50	2.60	117.00	2.80	126.00
Steam	tonnes	11	0.18	1.98	0.18	1.98	1.90	20.90
Electricity	kWh	0.05	100	5.00	110	5.50	136	6.80
Process Water	m^3	0.02	6	0.12	6.5	0.13	7	0.14
Cooling Water	m^3	0.005	27	0.13	30	0.15	70	0.35
Defoamer	kg	1.50	1.0	1.50	1.0	1.50	0.7	1.05
Chemical	kg	0.80	1.0	0.80	1.0	0.80	-	-
TOTALS				299.03		287.06		323.24

Summary of Cost Savings

The savings shown in Table 3 are translated to annual savings for two typical production capacities.

Table 4

Location	Process	Saving (US$) per tonne P_2O_5	Annual Saving (million US$)	
			300 t P_2O_5/day	600 t P_2O_5/day
Europe	HH	24.21	2.29	4.58
	HDH	36.18	3.36	6.73

III. RESULTS

In general terms the activities resulted in the SRICI gaining a greater knowledge and appreciation of the industrial production techniques used in the manufacture of phosphoric acid. Prior knowledge of fertilizer and phosphoric acid processes had been gained by reading published papers and relevant journals, and by some visits to factories and research institutes in other countries. However, previous contact with the HH and HDH processes, SRICI explained, was sparse and superficial. This assignment was therefore timely and necessary.

This should be considered as a detailed introduction to, or foundation in, the technology. In order to maximise the effectiveness of the activities so far, it is important that they are followed by industrial experience of the technology. This can be gained in the first instance by visits to full-scale commercial plants and factories. From that, a number can be selected to be requested to give extended training in production methods, techniques, etc. Training programmes lasting from one to six months would probably be suitable.

Nevertheless, by the activities so far SRICI has already extended its knowledge of phosphoric acid manufacture. This has a number of implications. Firstly, it has extended the range of processes that SRICI can investigate and evaluate. As China is developing its phosphate and fertilizer industry, it is important that the full range of production routes is understood and can be considered for each new project. This will allow China to select the most efficient and appropriate route for the raw materials and conditions prevailing in each case. As from now, SRICI can begin to apply this new knowledge.

Furthermore, from the data provided on equipment design and selection, materials of construction and operating procedures SRICI can critically examine schemes and proposals for current and future projects in this field. Effectiveness in this role can be increased by the close and active involvement of SRICI in the planning, evaluation and implementation of new projects. An added role would be assistance in the training of operating personnel for new plants, utilising the pilot plant facilities.

IV. CONCLUSIONS

A successful start has been made in the strengthening of the SRICI in the field of phosphate and granular fertilizer production. In view of the increasing rate of progress and expansion of this industry in China, it is important that the momentum established in this project to date, is maintained and, if possible, increased.

There is a growing awareness that a text book knowledge of the technology, however thorough, is not sufficient to allow an effective part to be played in this rapidly expanding industry. It is recognised therefore that practical experience is essential if comprehensive evaluation of technologies available for new projects is to be possible. In addition, a knowledge of industrial techniques employed in fertilizer production worldwide is necessary to allow full technical support to be given to the phosphate and granular fertilizer industry in China.

A measure of the importance of industrial experience was shown in the discussion sessions at SRICI. These were intensive and lengthy and almost exclusively aimed at extending the appreciation of the practical application of the technology in full-scale plants.

As with all modern industries, fertilizer production is being progressed by companies and organisations throughout the world. SRICI is dedicated to play a part in that progress. In order to maximise effectiveness it is important to establish and maintain good and regular contacts with other members of the fertilizer industry internationally. This will ensure that SRICI keeps abreast of up-to-date developments which can be assimilated and, if appropriate, utilised in its work.

ANNEX 1

Senior SRICI staff attending general discussions:

Guo Xining - President
Xiao Xi Zhen - Vice Director (SRICI)
Secretary General (CECC)
Jiang Shu Qin - Secretary President Office
Ye Chun Lin - Secretary CFTC Project

Senior Staff attending general and technical discussions and lectures:

Shan Guang-Yu - Deputy Chief Engineer, Senior Engineer
Chen Ming Lei - Chief, Phosphates Division
Dai Yuan Fa - Deputy Chief Engineer
Chen Jing Yu - Vice Manager
Chai Xiaozia - Senior Engineer
Fan Jin Wen - Senior Engineer
Ye Chun Lin - Engineer
Zhang Lilin - Engineer
Zhang Guo Xin - Interpreter
Shen Qin Jia - Interpreter

There were approximately an additional 20 staff members attending the technical sessions.

PHOSPHATE ROCKS

$Ca_{10}(PO_4)_6X_2$ - General Formula

$Ca_{10}(PO_4)_6F_2$ - Fluorapatite

$Ca_{10}(PO_4)_6(OH)_2$ - Hydroxyapatite

$Ca_{10}(PO_4)_4Cl_2$ - Chlorapatite

X = Anion

SUBSTITUTED APATITE

$Ca_{10}(PO_4)_6{-}x(CO_3F)xF_2$ - Francolite

$Ca_{10}(PO_4)_6{-}x(CO_3F)x(OH_2)$ - Dahllite

CONSTITUENT ELEMENTS OF PHOSPATE ROCKS

PRIMARY	MAJOR	MINOR	TRACE	
>20%	UP TO 5%	UP TO 2%	PPM	
P	S	NA	BA	PB
CA	AL	K	U	ZN
	FE	CL	TI	MN
	F	SR	CR	V
	SI		AS	CU
	C		HG	MO
	MG			NI
			Rare Earths	

PHOSPHORIC ACID MANUFACTURE

MAIN REACTION

$$Ca_3(PO_4)_2 + 3H_2SO_4 + 3nH_2O \longrightarrow 2H_3PO_4 + 3CaSO_4 \cdot nH_2O$$

N = O, $^1/_2$ or 2 when Calcium Sulphate is in the form of Anhydrite, Hemihydrate or Dihydrate respectively.

SIDE REACTIONS

$$CaCO_3 + H_2SO_4 + nH_2O \longrightarrow CaSO_4nH_2O + CO_2 + H_2O$$

$$3CaF_2 + 3H_2SO_4 + SiO_2 \longrightarrow 3CaSO_4 + H_2SiF_6 + 2H_2O$$

$$H_2SiF_6 \longrightarrow SiF_4 + 2HF$$

$$3SiF_4 + 2H_2O \longrightarrow 2H_2SiF_6 + SiO_2$$

$$H_2SiF_6 + E_2O \longrightarrow E_2SiF_6 + H_2O \quad (E = Na \text{ or } K)$$

OVERALL REACTION

$$Ca_{10}(PO_4)_6F_2CaCO_3 + 11H_2SO_4 + 11nH_2O \longrightarrow 6H_3PO_4 + 11CaSO_4 \cdot nH_2O + 2HF + CO_2 + H_2O$$

MANUFACTURE OF PHOSPHORIC ACID

DIHYDRATE (DH) PROCESS : 30% P_2O_5 EX FILTER

80 °C

$CaSO_4 . 2 H_2O$ BY PRODUCT

HEMIHYDRATE (HH) PROCESS : 50% P_2O_5 EX FILTER

100 °C

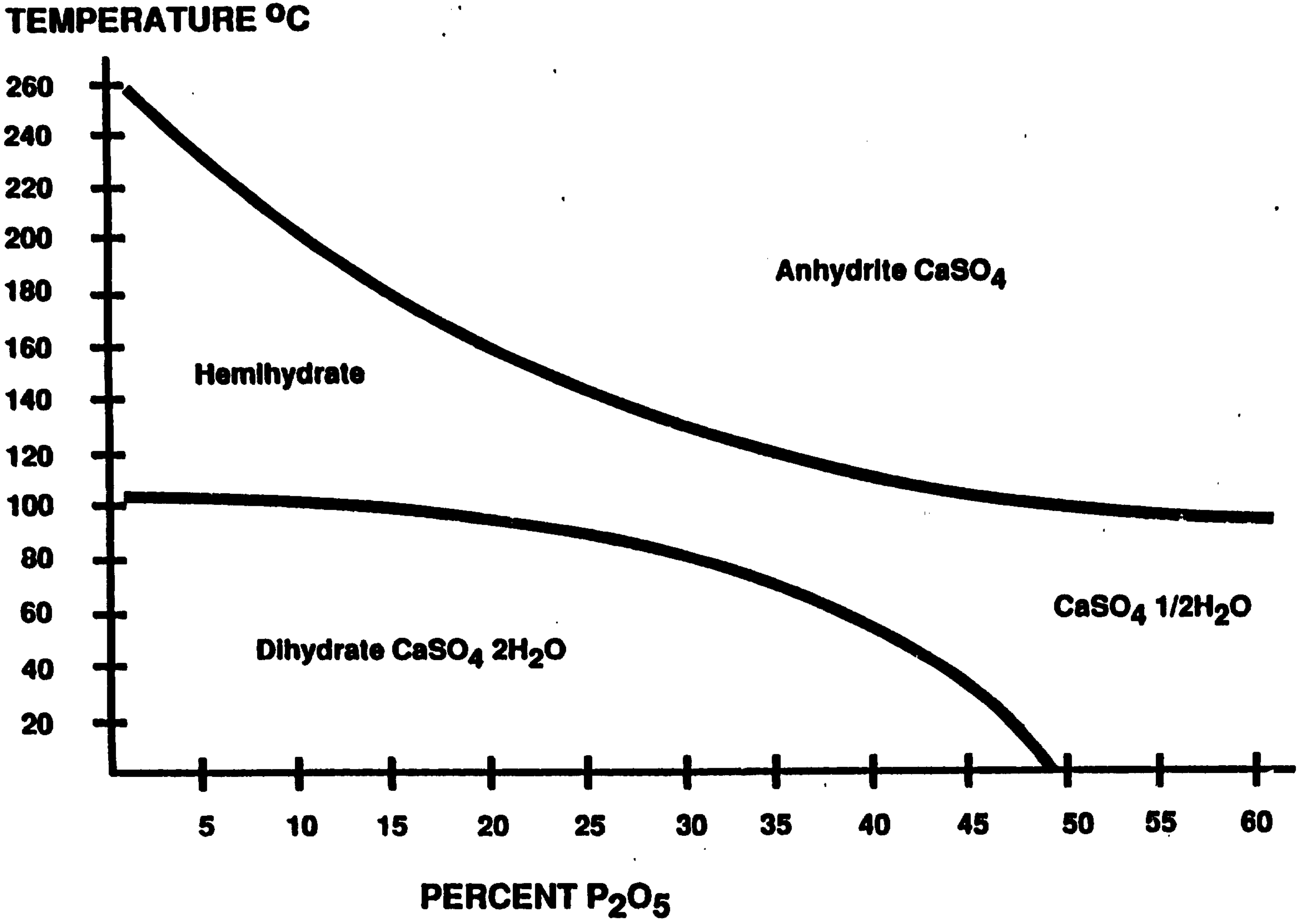
$CaSO_4 . 1/2 H_2O$ BY PRODUCT

HEMIDIHYDRATE (HDH) PROCESS : 50% P_2O_5 EX FILTER

100 °C

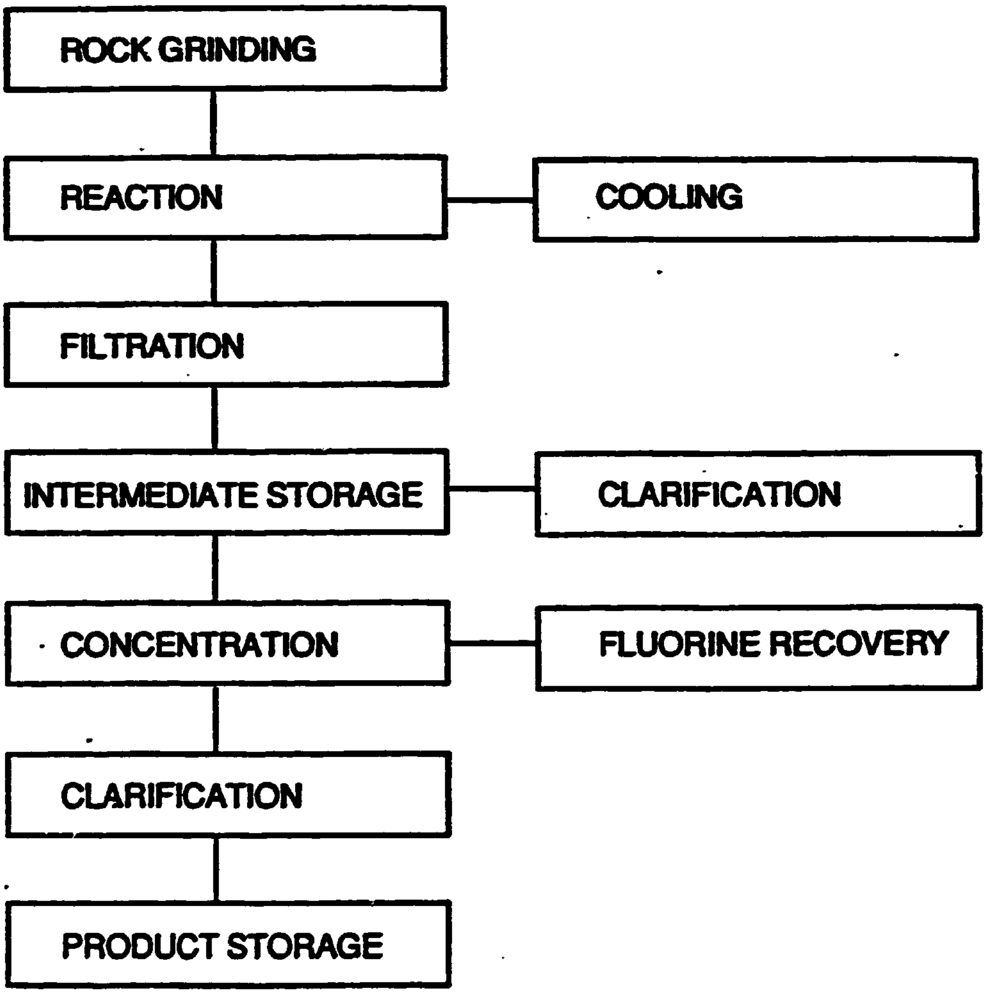
$CaSO_4 . 2H_2O$ BY PRODUCT

EFFECT OF REACTION CONDITIONS ON CALCIUM SULPHATE CRYSTALLISATION

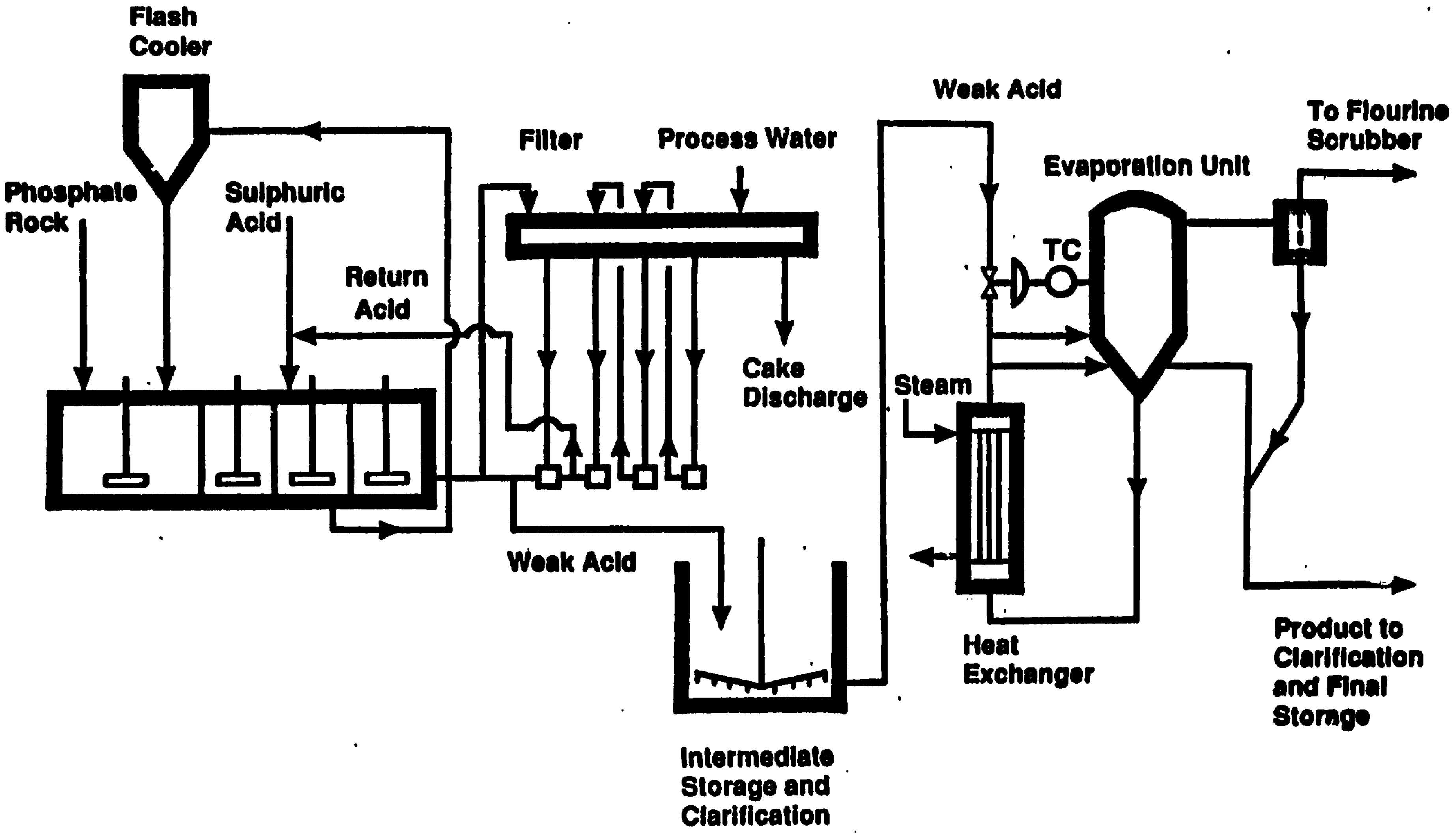


TYPICAL DIHYDRATE PROCESS

UNIT OPERATIONS

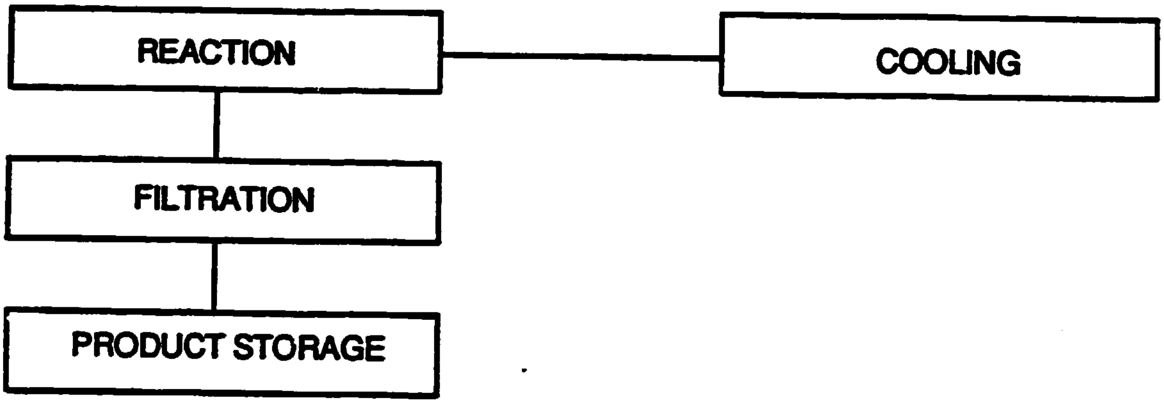


Dihydrate Process

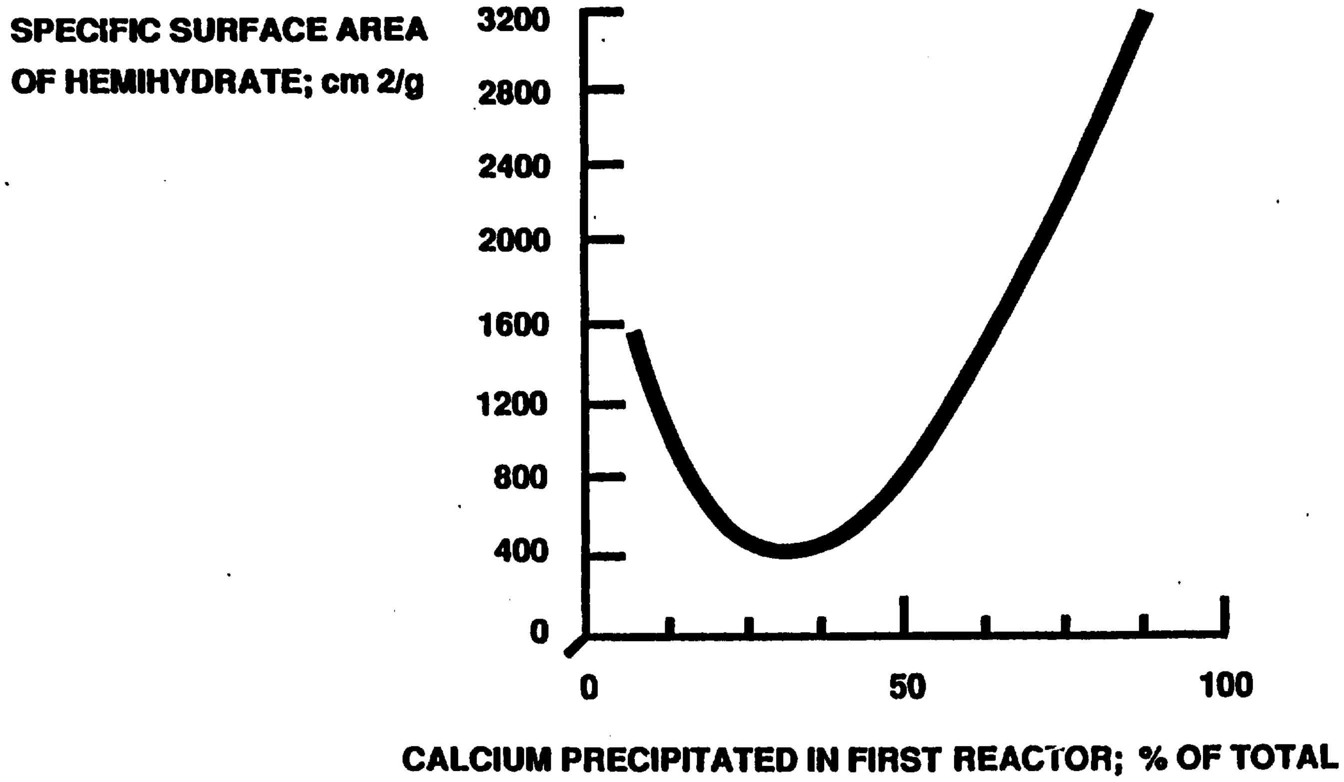


NHL HEMIHYDRATE (HH) PROCESS

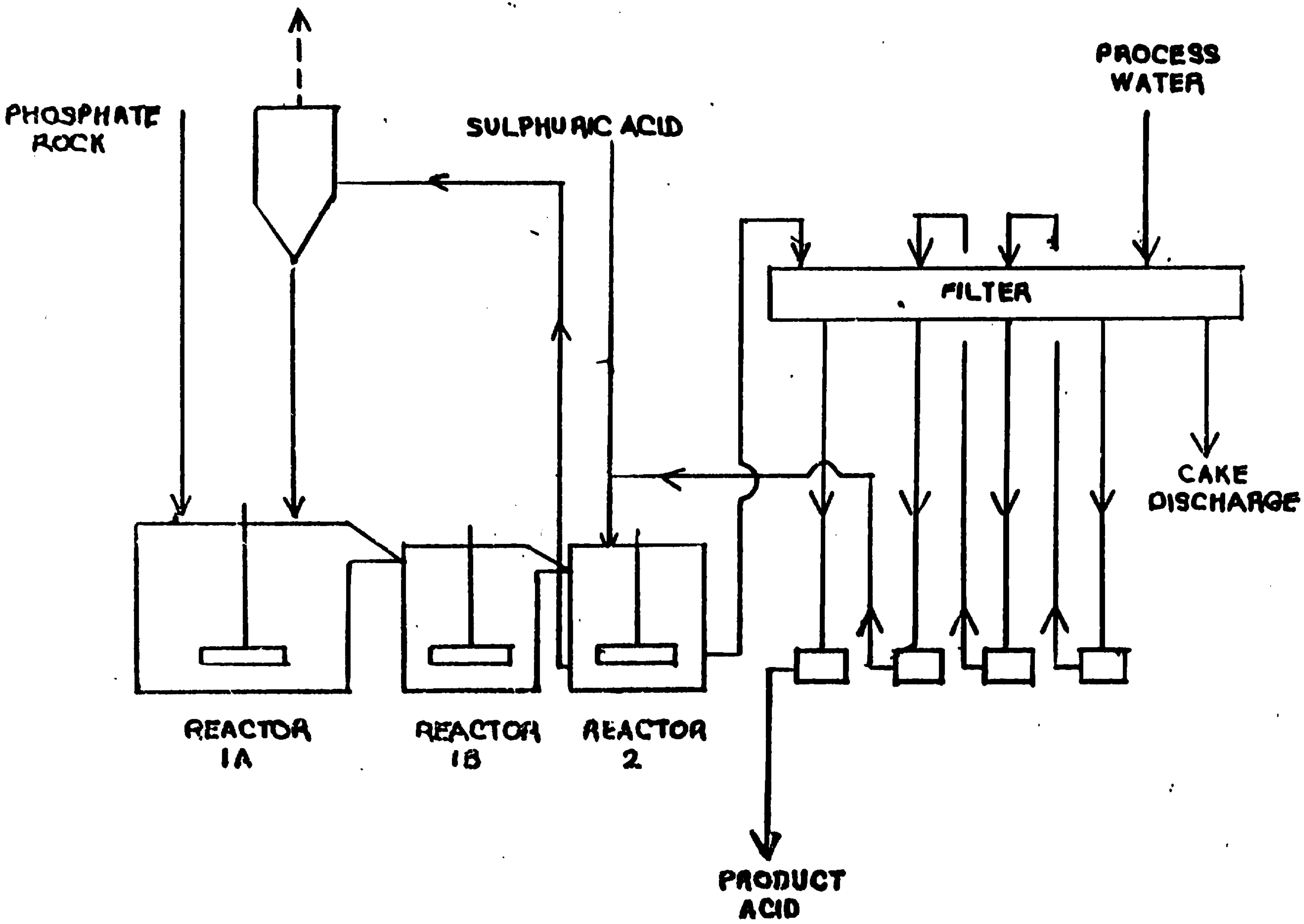
UNIT OPERATIONS



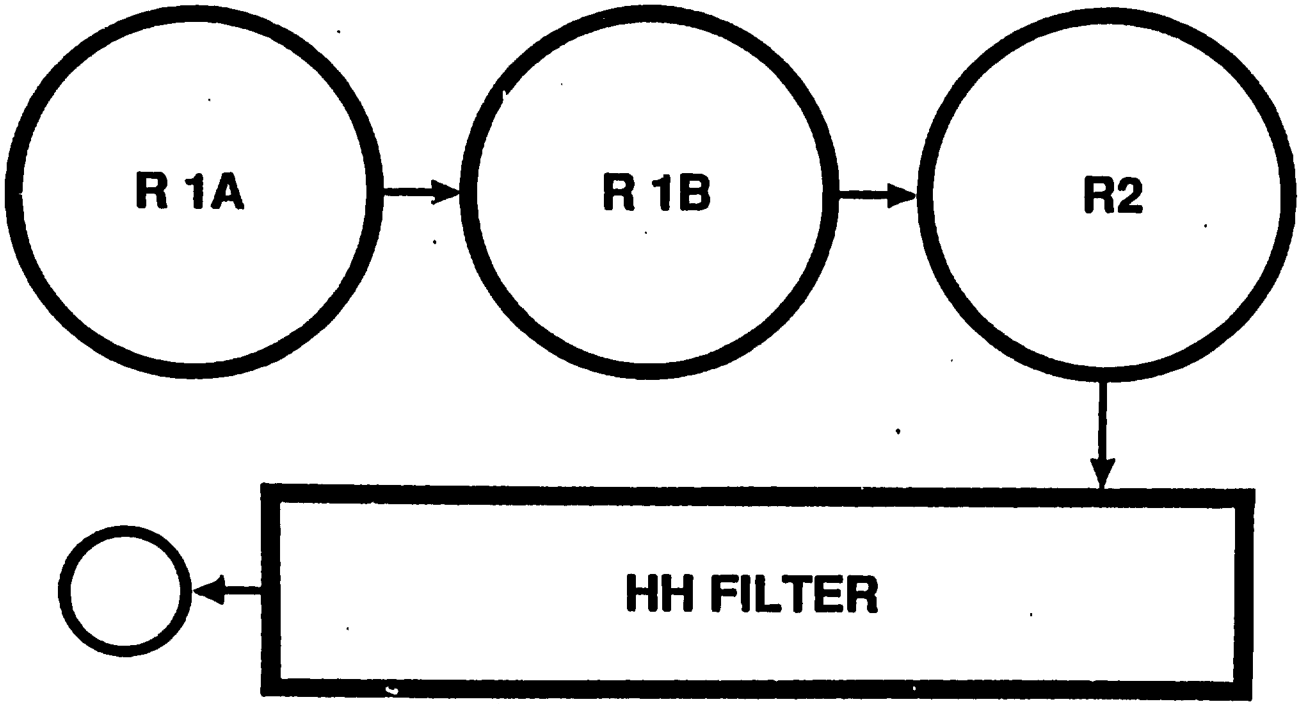
VARIATION OF SPECIFIC SURFACE AREA OF HEMIHYDRATE WITH PRECIPITATION RATIO



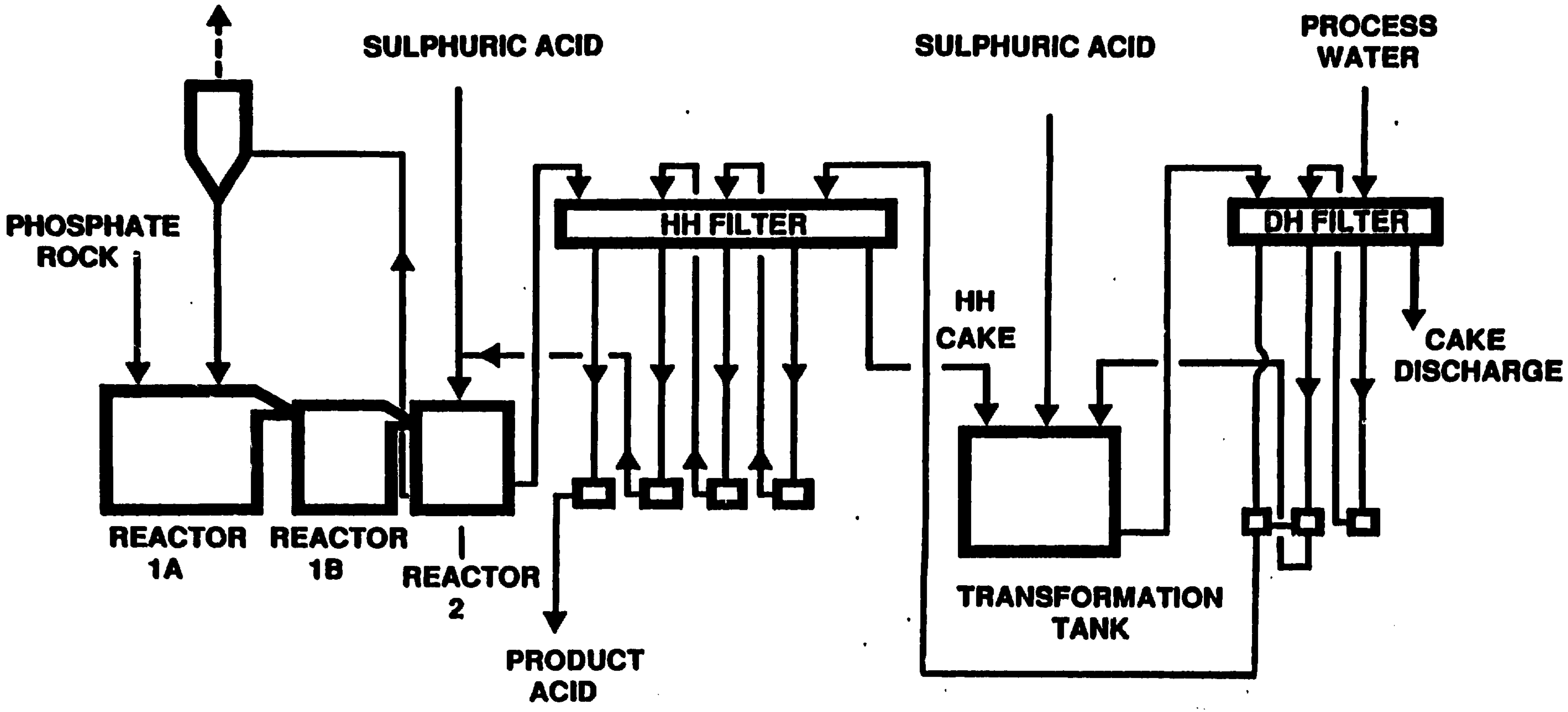
NORSK HYDRO LICENSING HEMIHYDRATE PROCESS



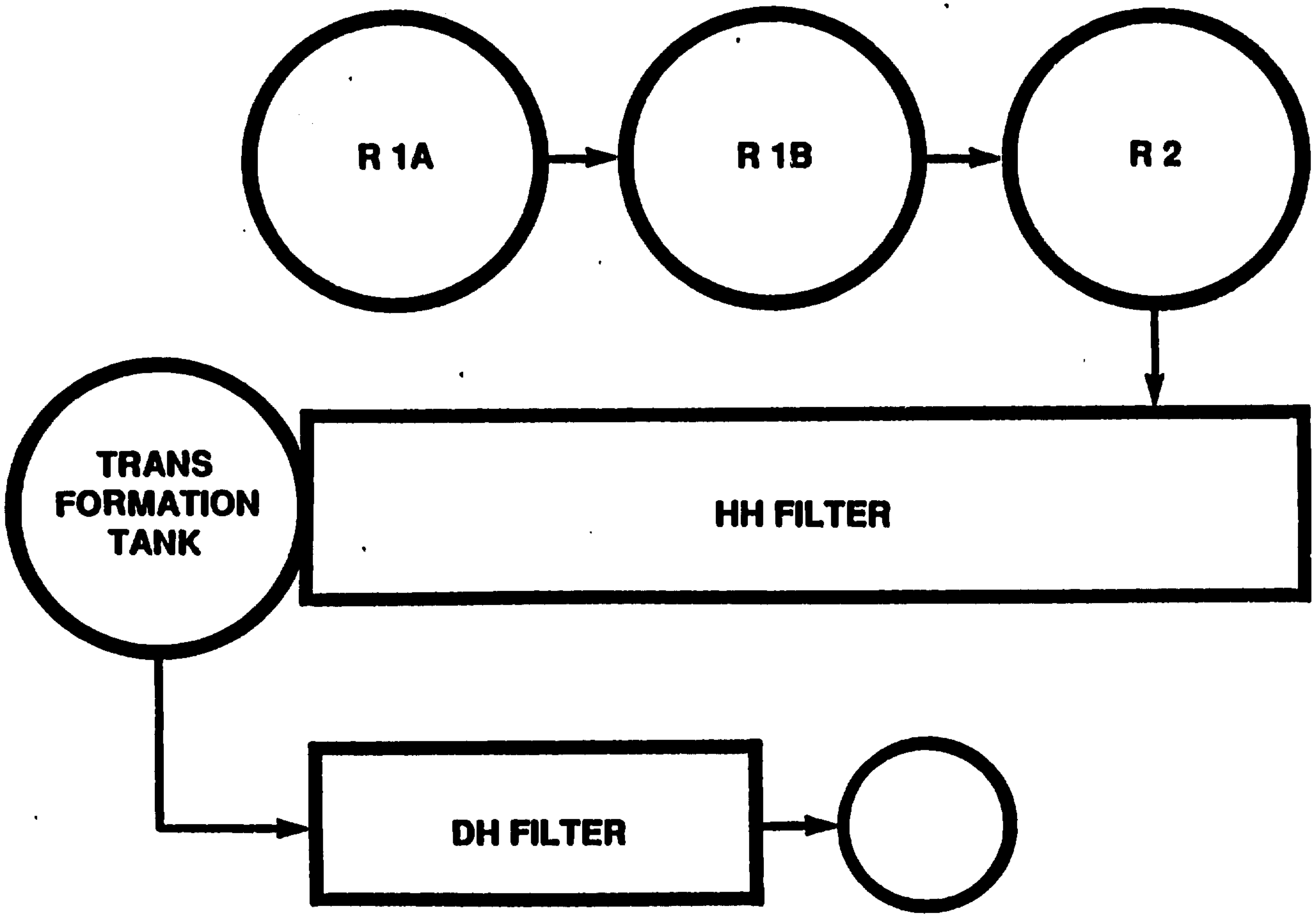
HEMIHYDRATE PLANT LAYOUT



NORSK HYDRO LICENSING HEMIDIHYDRATE PROCESS



HEMIDIHYDRATE PLANT LAYOUT



ROCK PHOSPATE PARTICLE SIZE REQUIREMENTS FOR HEMIHYDRATE AND DIHYDRATE PLANTS

BSS SIEVE SIZE (MESH)	APERTURE (MICRONS)	PARTICLE SIZE REQUIREMENT % THROUGH		SIZE DISTRIBUTION KHOURIBGA MOROCCO ROCK % THROUGH
		HH PLANT	DH PLANT	
10	1700	100		100
16	1000	-		98.3
30	500	75	100	92.7
60	250	25	95	71.3
100	150		60	28.2
200	75		30	9.6

HH PROCESS

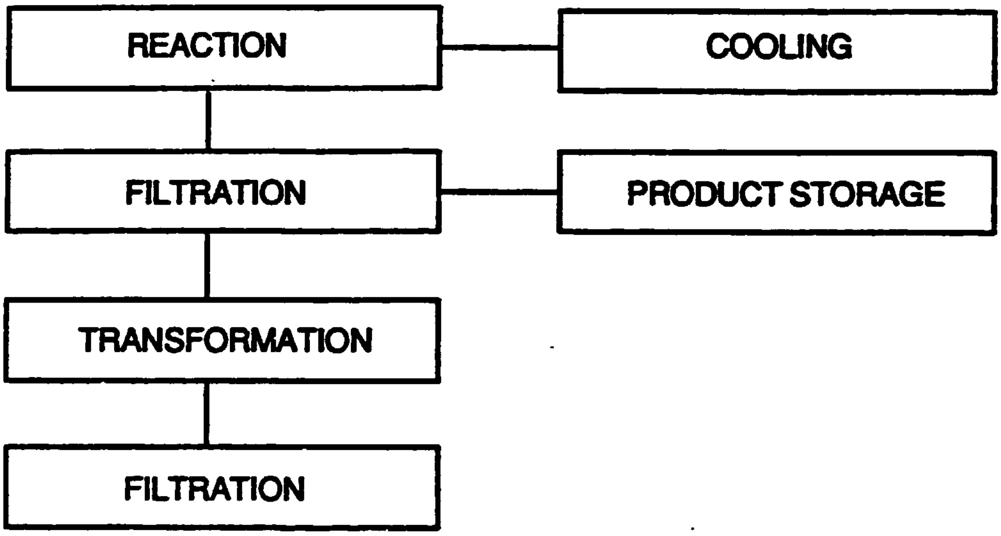
FEATURES	BENEFITS
Production of concentrated acid directly from the filter .	Energy savings and simplified plant operation.
Reduced rock grind requirement.	
improved acid quality.	

HH PROCESS

P_2O_5 recovery efficiency = 94%

NHL HEMIDIHYDRATE (HDH) PROCESS

UNIT OPERATIONS



HDH PROCESS

FEATURES

Very high P_2O_5 recovery efficiency.

Production of concentrated
acid directly from the filter.

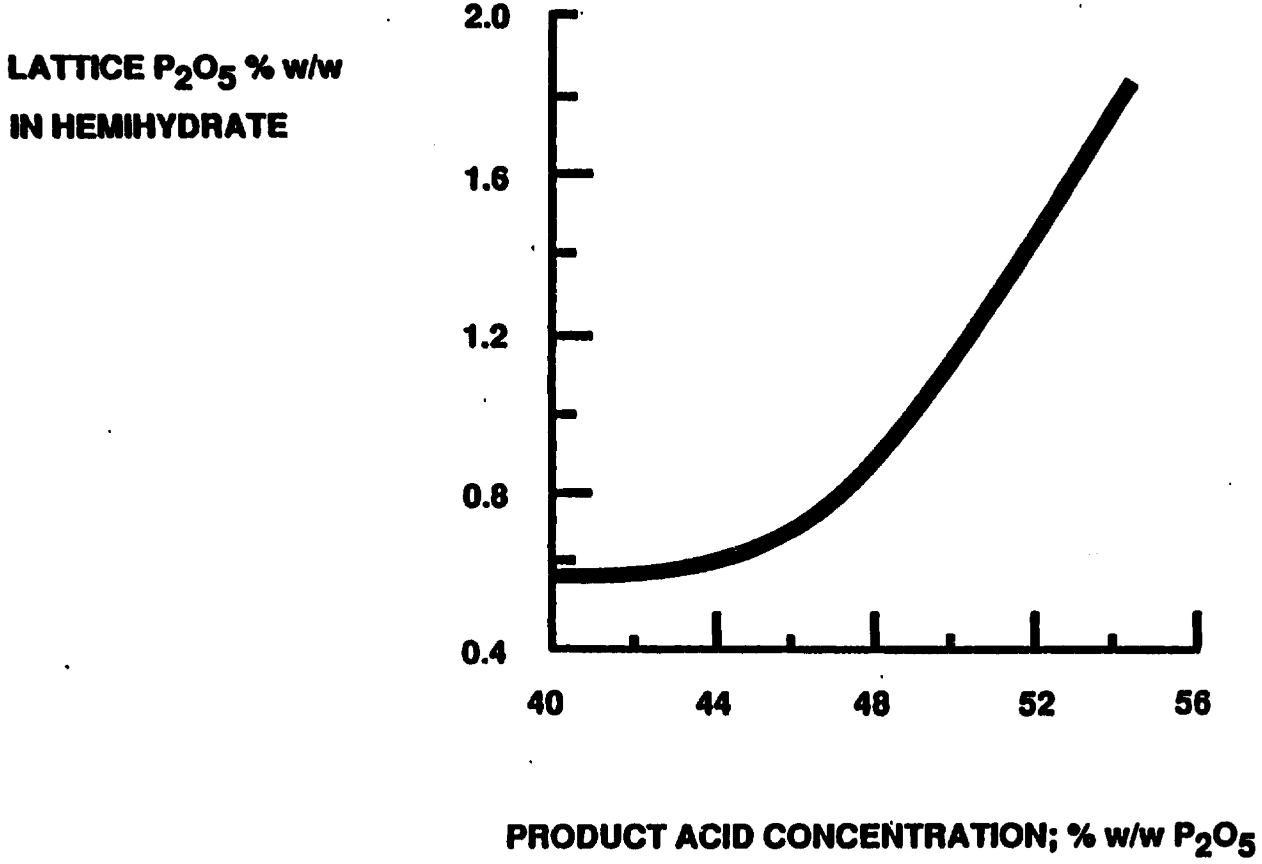
Reduced rock grind requirement.

Improved acid quality.

HDH PROCESS

FEATURES	BENEFITS
Very high P_2O_5 recovery efficiency.	Reduced raw material consumptions.
Production of concentrated acid directly from the filter.	Energy savings.
Reduced rock grind requirement.	Energy savings.
Improved acid quality.	Simplified plant operation.

VARIATION OF LATTICE P_2O_5 LOSS WITH PRODUCT ACID P_2O_5



RAW MATERIAL CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

	Consumption per tonne P_2O_5 Produced	
Item	HH Plant	DH Plant
Phosphate Rock, t	3.42	3.42
Sulphuric Acid, t	2.7	2.8

BASIS : Production of 42% P_2O_5 from 68 BPL Florida Rock

UTILITY CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

	Consumption per tonne P_2O_5 Produced	
Item	HH Plant	DH Plant
Steam, t	0.2	1.8
Electricity, kWh	100	140
Process Water, m^3	6	7
Cooling Water, m^3	30	65
Defoamer kg	1.5	1.0
Chemicals,kg	1.0	Nil
Labour	2+1/shft	3+1/shift

BASIS : Production of 42% P_2O_5 acid from 68 BPL Florida Rock

RAW MATERIAL AND UTILITY CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

	Consumption per tonne P_2O_5 Produced	
Item	HH Plant	DH Plant
Phosphate Rock, t	3.42	3.42
Sulphuric Acid, t	2.7	2.8
Steam, t	0.2	1.8
Electricty, kWh	100	140
Process Water, m^3	6	7
Cooling Water, m^3	30	65
Defoamer, kg	1.5	1.0
Chemicals, kg	1.0	Nil
Labour	2 + 1 / shift	3 + 1 /shift

BASIS: Production of 42% P_2O_5 Acid from 68 BPL Florida rock

TYPICAL PRODUCTION COSTS FOR 42% P_2O_5 ACID W. EUROPEAN LOCATION

ITEM		HH PLANT	DH PLANT
Phosphate Rock	\$60/t	205.2	205.2
Sulphuric Acid	\$45/t	121.5	126
Steam	\$6/t	1.2	10.8
Electricity	\$ 0.05/kWh	5	7
Process Water	\$0.02/m^3	0.12	0.14
Cooling Water	\$0.005m^3	0.15	0.33
Defoamer	\$1.5/kg	2.25	1.5
Chemicals	\$1.0/kg	1.0	-
		336.42	350.97

BASIS : 68 BPL FLORIDA ROCK

TYPICAL PRODUCTION COSTS FOR 42% P_2O_5 MINE SITE LOCATION

ITEM		HH PLANT	DH PLANT
Phosphate Rock	\$10/t	34.2	34.2
Sulphuric Acid	\$45/t	121.5	126
Steam	\$ 6/t	1.2	10.8
Electricity	\$0.05/kWh	5	7
Process Water	\$0.02/m^3	0.12	0.14
Cooling Water	\$0.005m^3	0.15	0.33
Defoamer	\$1.5/kg	2.25	1.5
Chemicals	\$1.0/kg	1.0	
		165.42	179.97

BASIS : 68 BPL Florida Rock

TYPICAL RAW MATERIAL CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

Consumption per tonne P_2O_5 produced

	HDH Plant	DH Plant
Phosphate rock	3.25 T	3.42 T
Sulphuric Acid (100%)	2.6 T	2.8 T

BASIS: Production of 42% P_2O_5 acid from 68% BPL Florida Phosphate (31.1% P_2O_5)

TYPICAL UTILITIES CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

	Consumption per tonnes P_2O_5 Produced	
ITEM	HDH PLANT	DH PLANT
Steam, t	0.2	1.8
Electricity, kWh	100	140
Process Water, m^3	4	7
Cooling Water, m^3	30	65
Labour	3 + 1/SHIFT	3 + 1/SHIFT
Defoamer , kg	1.5	1.0

BASIS : Production of 42% P_2O_5 acid from 68% BPL Florida Phosphate

TYPICAL RAW MATERIAL CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

	Consumption per tonne P_2O_5 produced	
	HDH PLANT	DH PLANT
Phosphate Rock	3.26 T	3.42 T
Sulphuric Acid (100%)	2.6 T	2.8T

BASIS : Production of 50% P_2O_5 acid from 68% BPL Florida Phosphate

TYPICAL UTILITIES CONSUMPTIONS FOR PHOSPHORIC ACID PRODUCTION

	Consumption per tonne P_2O_5 produced	
	HDH PLANT	DH PLANT
Steam	0.2 t	2.5 t
Electricity	110 kWh	140 kWh
Process Water	4 m^3	7 m^3
Cooling Water	30 m^3	77 m^3
Labour	3 + 1/shift	3 + 1/shift
Defoamer	1.5kg	1.0kg

BASIS : Production of 50% P_2O_5 acid from 68% BPL Florida Phosphate

TYPICAL PRODUCTION COSTS FOR 42% P_2O_5 PHOSPHORIC ACID

W. EUROPEAN LOCATION

ITEM		Cost $US per tonne P_2O_5 HDH PLANT	DH PLANT
Phosphate Rock	$60/t	195.6	205.2
Sulphuric Acid	$45/t	117.0	126
Steam	$ 6/t	1.2	10.8
Electricity	$0.05/kWh	5.5	7
Process Water	$0.02/m^3	0.1	0.1
Cooling Water	$0.005/m^3	0.2	0.33
Defoamer	$1.5/kg	2.3	1.5
		321.9	350.93

BASIS : From 68% BPL Florida Phosphate

TYPICAL PRODUCTION COSTS FOR 42% P_2O_5 PHOSPHORIC ACID

MINE SITE LOCATION

ITEM		Cost $US per tonne P_2O_5 HDH PLANT	Cost $US per tonne P_2O_5 DH PLANT
Phosphate Rock	$10/t	32.6	34.2
Sulphuric Acid	$45/t	117.0	126
Steam	$ 6/t	1.2	10.8
Electricity	$0.05/kWh	5.5	7
Process Water	$0.02/m^3	0.1	0.1
Cooling Water	$0.005m^3	0.2	0.33
Defoamer	$1.5/kg	2.3	1.5
		158.9	179.93

BASIS : From 68% BPL Florida Phosphate

TYPICAL PRODUCTION COSTS FOR 50% P_2O_5 PHOSPHORIC ACID

W. EUROPEAN LOCATION

Cost $US per tonne P_2O_5

ITEM		HDH PLANT	DH PLANT
Phosphate Rock	$60/t	195.6	205.2
Sulphuric Acid	$45/t	117.0	126
Steam	$ 6/t	1.2	15.0
Electricity	$0.05/kWh	5.5	7
Process Water	$0.02/m^3	0.1	0.1
Cooling Water	$0.005/m^3	0.2	0.4
Defoamer	$1.5/kg	2.3	1.5
		321.9	355.2

BASIS : Production of 50% P_2O_5 Acid from 68% BPL Florida Phosphate

ELECTRICITY GENERATION BY BACK PRESSURE AND CONDENSING TURBO ALTERNATORS

PHOSOPHORIC ACID PRODUCTION	TYPE OF PHOSPHORIC ACID PLANT	SULPHURIC ACID PRODUCTION	TYPE OF TURBO ALTERNATOR	OUTPUT
500 TPD P_2O_5	HEMIHYDRATE	1,350 TPD H_2SO_4	CONDENSING	13,000kW
500 TPD P_2O_5	DIHYDRATE	1,350 TPD H_2SO_4	BACK PRESSURE	7,500kW

ELECTRICITY COST CREDIT ASSOCIATED WITH HEMIHYDRATE AND DIHYDRATE PLANTS

PHOSPHORIC ACID PRODUCTION	TYPE OF PHOSPHORIC ACID PLANT	ELECTRICITY COGENERATION PER DAY	COST CREDIT	
			US$ PER DAY	US$ PER TONNE P_2O_5
500 TPD P_2O_5	HEMIHYDRATE	312,000 kWh	15,600	31.2
500 TPD P_2O_5	DIHYDRATE	180,000 kWh	9,000	18.0

COST CREDIT ASSUMES ELECTRICITY COST = US$0.05 PER kWh

NHL HEMIHYDRATE TECHNOLOGY: COMMERCIAL PLANTS

CLIENT	COUNTRY	PROCESS	START - UP	CAPACITY TPD P_2O_5	ROCK FEED
WINDMILL	HOLLAND	HH	1970	610	TOGO
RMHK TREPCA	YUGOSLAVIA	HDH	1974	160	JORDAN
A & W	U.K.	HDH	1980	500	MOROCCO
CSBP	AUSTRALIA	HDH	1981	500	68 FLORIDA
PIVOT	AUSTRALIA	HDH	1981	100	68 FLORIDA
HCI	CYPRUS	HH	1982	130	ALGERIA
WINDMILL*	HOLLAND	HH	1983	330	72 FLORIDA
ROYSTER*	USA	HH	1985	586	68 FLORIDA
BELLEDUNE*	CANADA	HH	1986	500	68 FLORIDA
SUPRA*	SWEDEN	HDH	1986	360	FLORIDA/ MOROCCO/ JORDAN
NFC	THAILAND	HDH	1987	810	JORDAN/ FLORIDA/ MOROCCO
QPL	AUSTRALIA	HH	1989	720	DUCHESS

* PLANT CONVERSION FROM DIHYDRATE UNIT

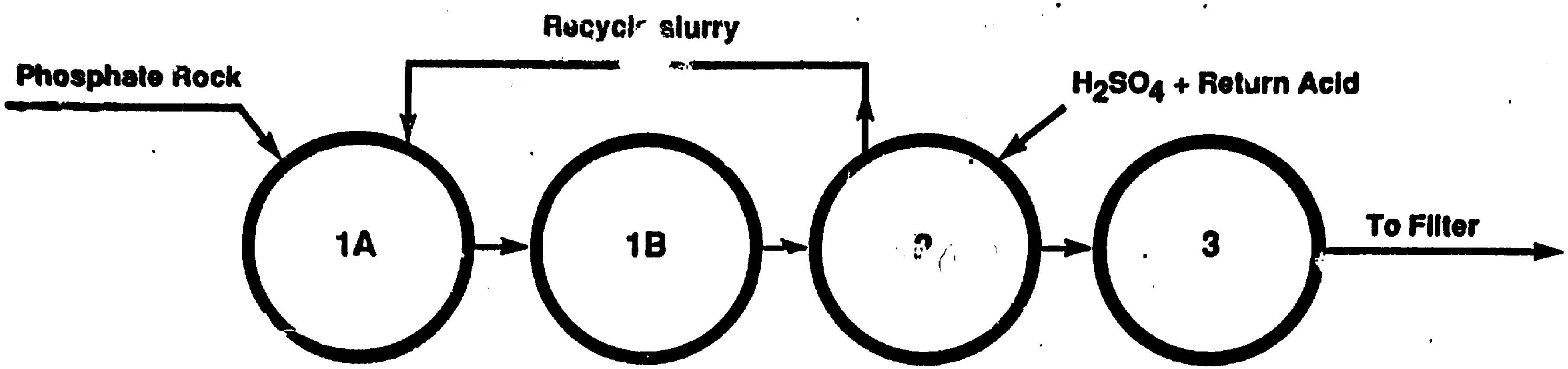
PHOSPHORIC ACID PLANT CONVERSIONS

Unit Operations:

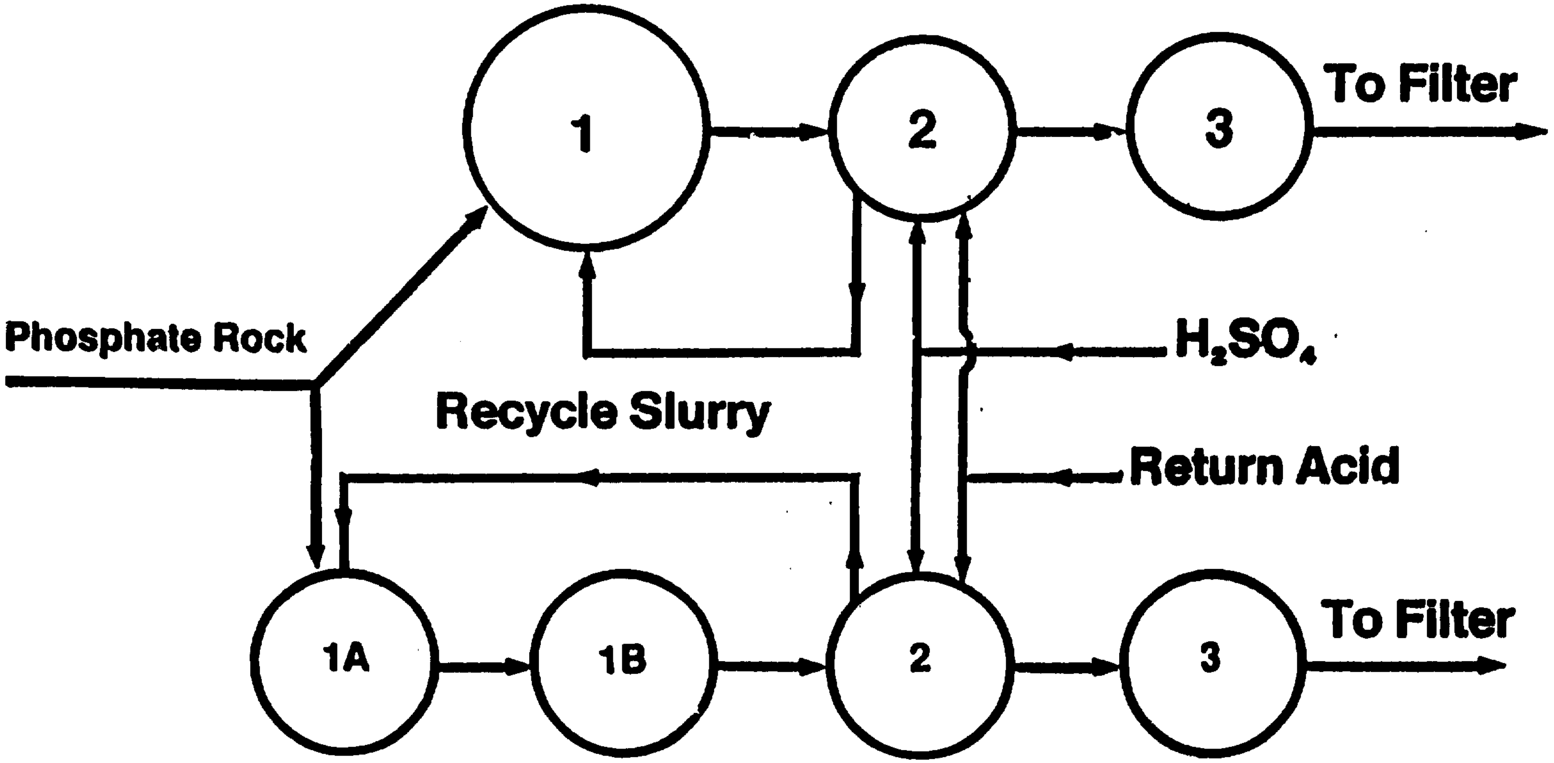
- Rock Feed
- Reaction + Agitation
- Cooling
- Gas Scrubbing
- Filtration
- Transformation

WINDMILL, HOLLAND

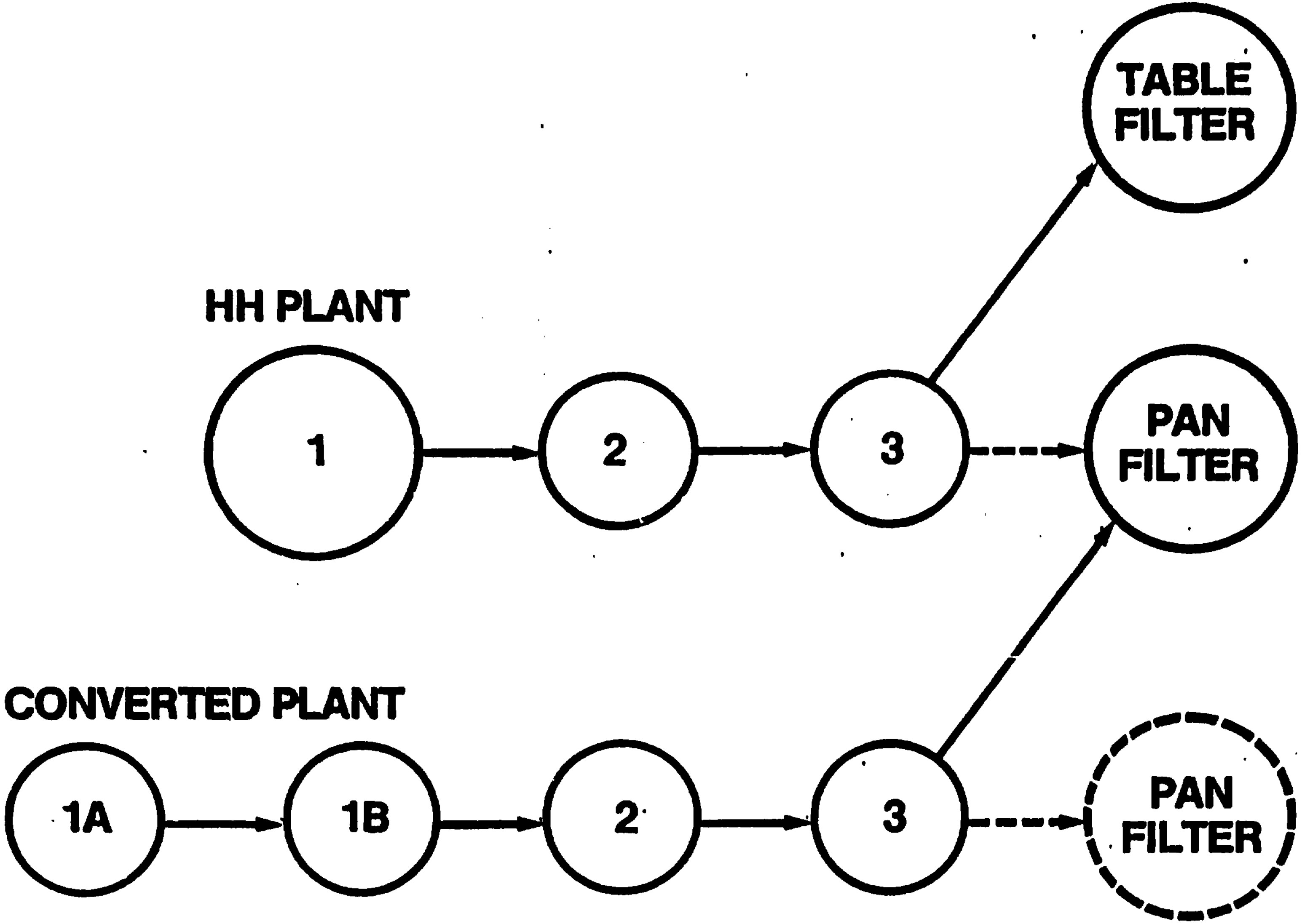
CONVERTED PLANT



WINDMILL, HOLLAND



WINDMILL, HOLLAND

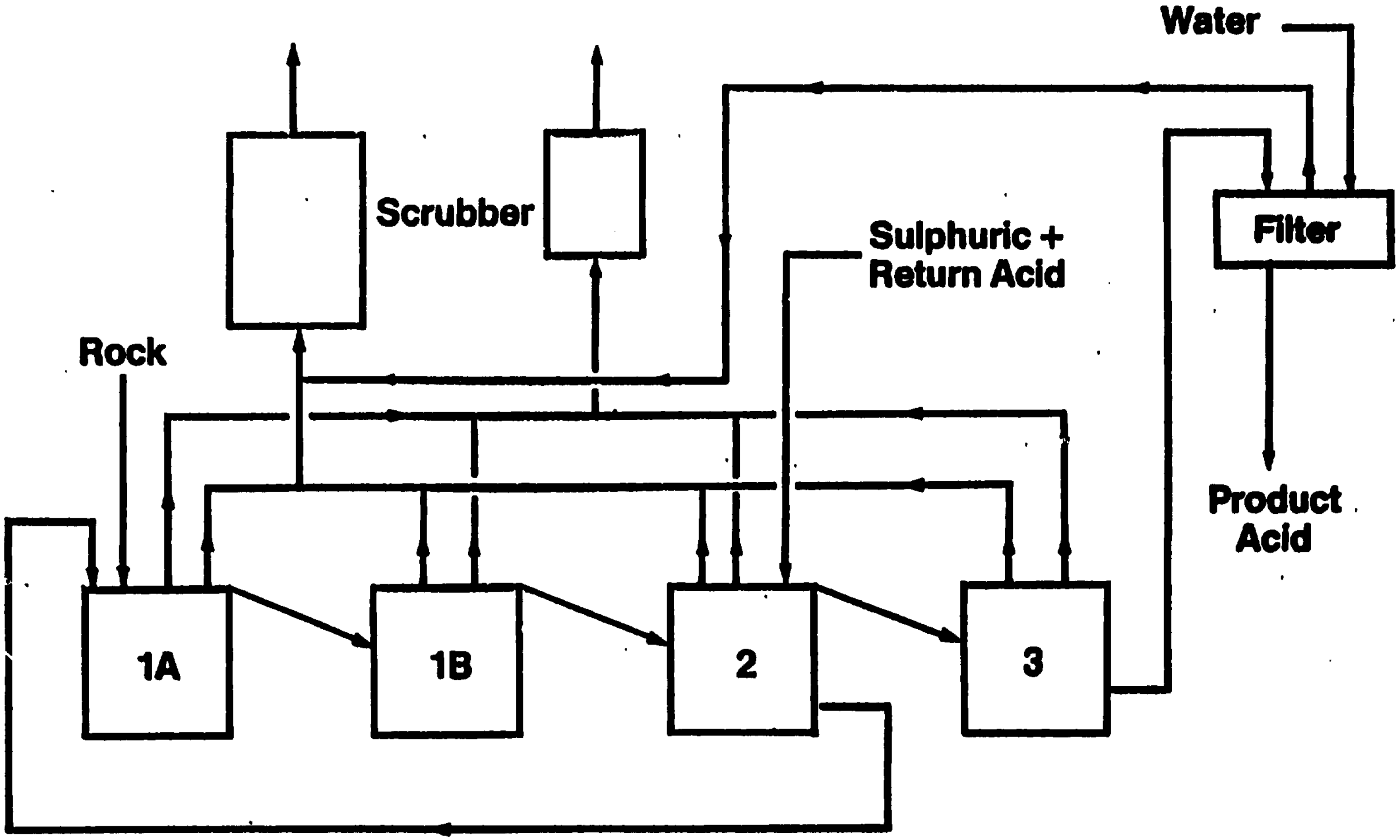


HH PLANT

CONVERTED PLANT

WINDMILL, HOLLAND

CONVERTED PLANT: SIMPLIFIED FLOWSHEET



ROYSTER CONVERSION

68 BPL FLORIDA ROCK

ITEM	% w/w
P_2O_5	30.0
CaO	44.0
F	3.4
Na_2O	0.5
Fe_2O_3	0.9
Al_2O_3	1.5
CO_2	3.0
SiO_2	8.5
H_2O	12.0

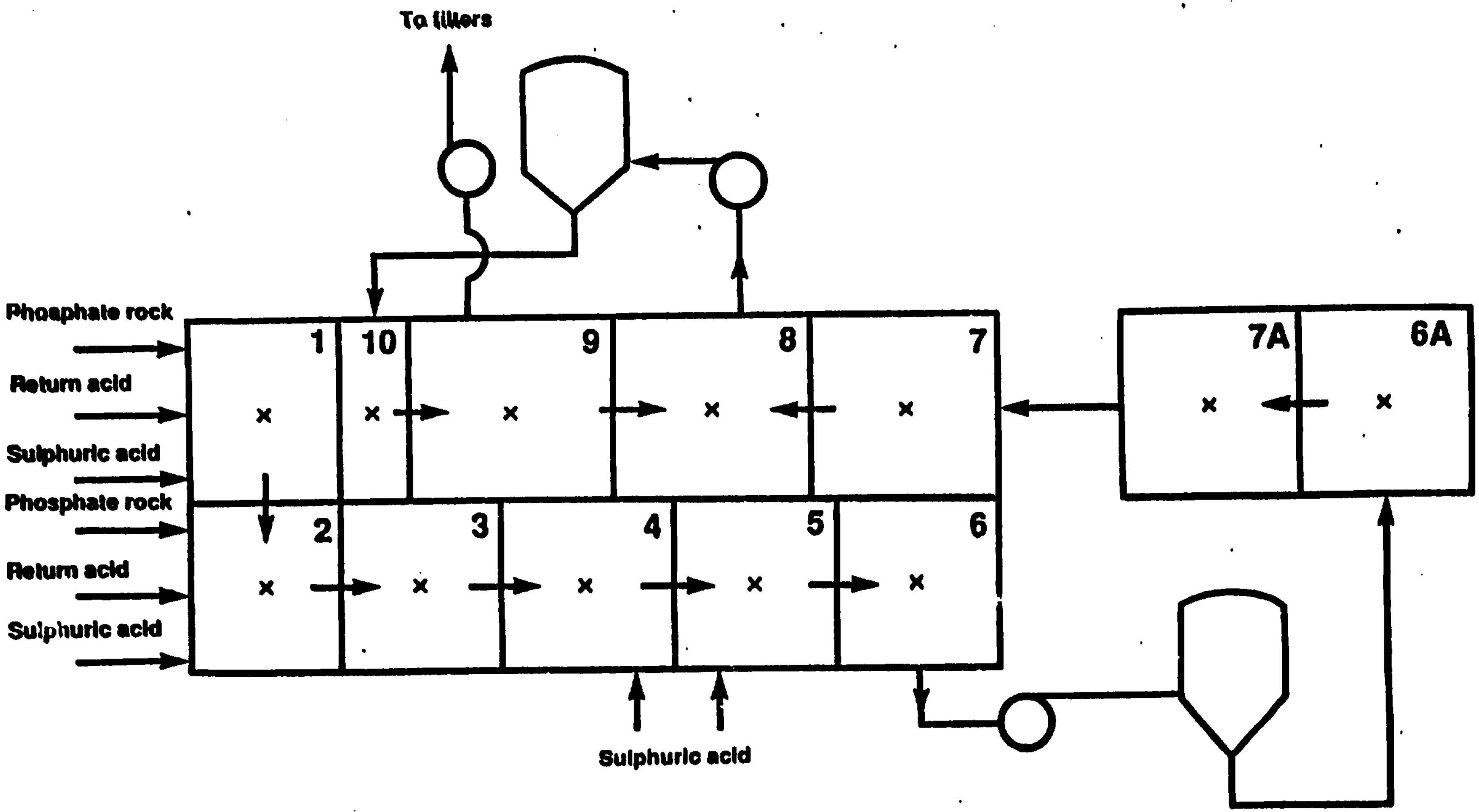
ROYSTER CONVERSION

68 BPL FLORIDA ROCK

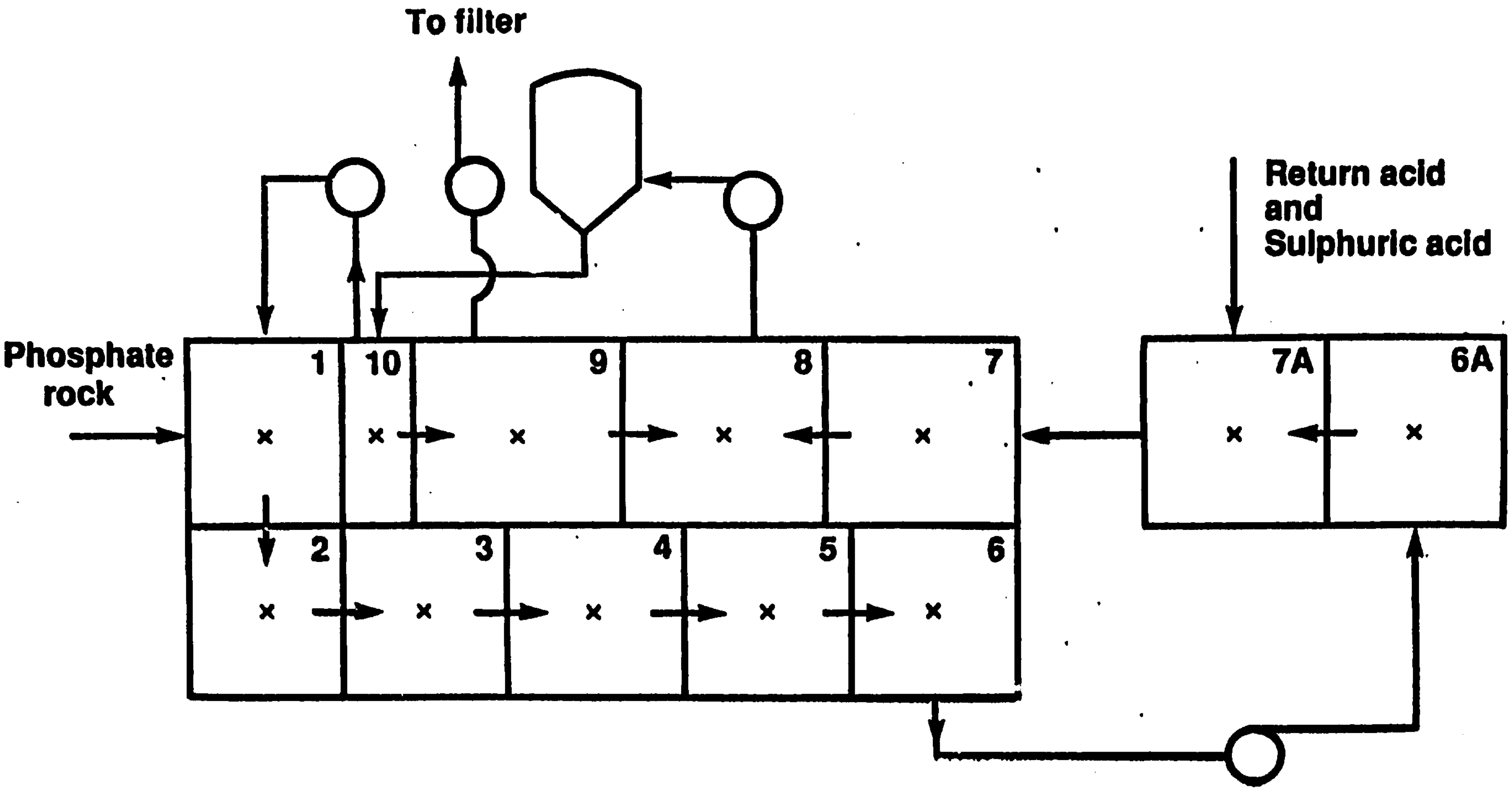
SIZE ANALYSIS :

BSS NO	Aperture,mm	% Through
10	1.7	97.9
16	1.0	82.1
30	0.5	32.6
60	0.25	10.7
100	0.15	3.0

Royster's Original Prayon Reaction System



Royster's Converted Plant: Hemihydrate Reaction System



ROYSTER CONVERSION

HEMIHYDRATE REACTION SYSTEM:

R1 = C1 - 6A = 512 m^3
R2 = C7, C7A, C8 = 290 m^3
FFT = C9 = 130 m^3

UNIT VOLUME = 1.3 m^3/tpd P_2O_5

ROYSTER CONVERSION

FILTERS

BIRD 24D = 108 m^2
(BIRD 24B = 57 m^2)

Achieved filtration rate = 5.6 tpd P_2O_5/m^2

ROYSTER CONVERSION

P_2O_5 LOSSES

		% CAKE
Water Soluble	P_2O_5	0.24
Insoluble	P_2O_5	1.4
TOTAL	P_2O_5	1.64

P_2O_5 Recovery Efficiency = 93.3%

BELLEDUNE CONVERSION

68 BPL FLORIDA ROCK

CHEMICAL ANALYSIS:

Item	% w/w
P_2O_5	32.0
CaO	45.4
SO_3	0.96
F	3.54
Cl	48 ppm
SiO_2	8.01
Na_2O	0.55
K_2O	0.11
MgO	0.43
Al_2O_3	1.55
Fe_2O_3	1.29
CO_2	3.56
Organic	0.36

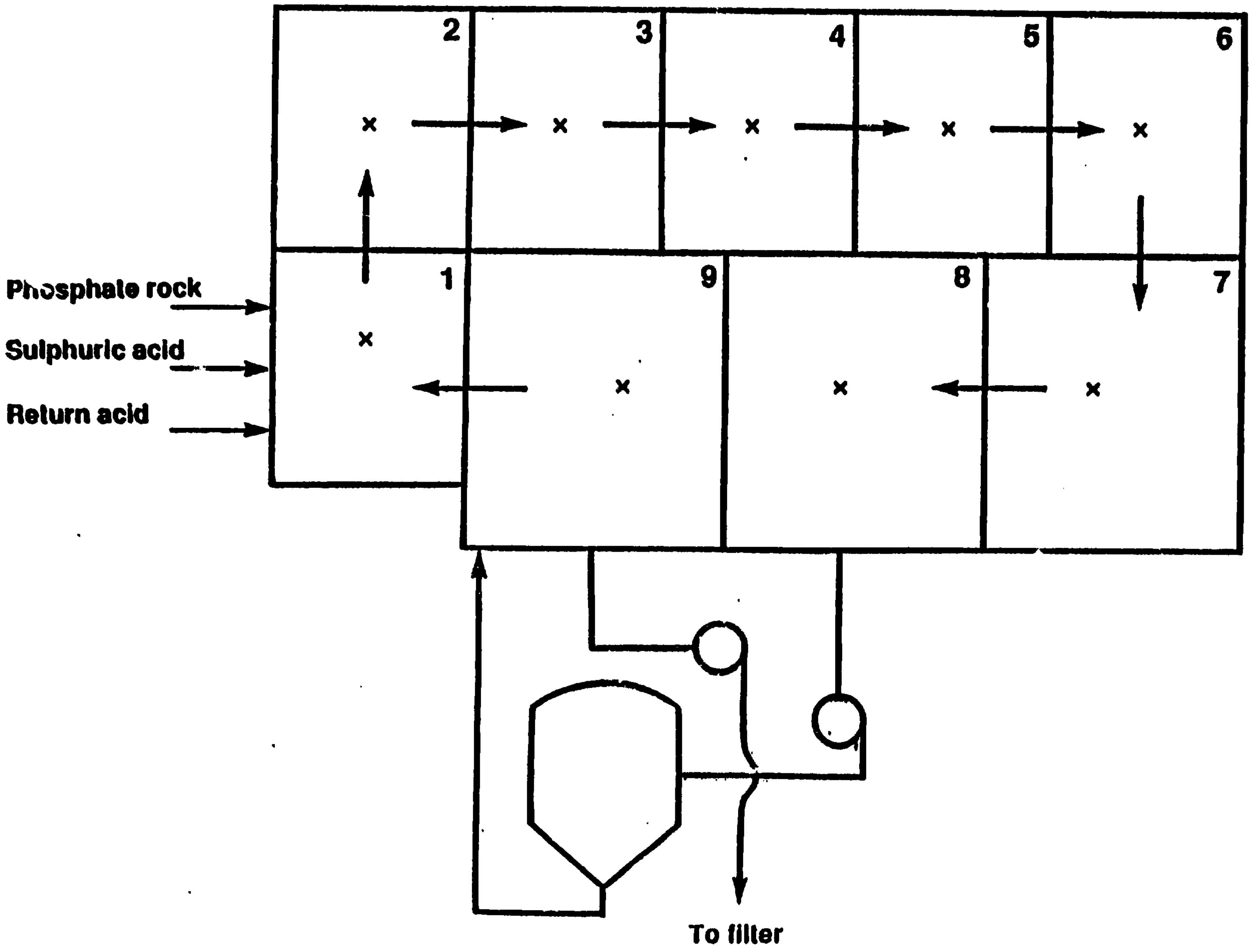
BELLEDUNE CONVERSION

68 BPL FLORIDA ROCK

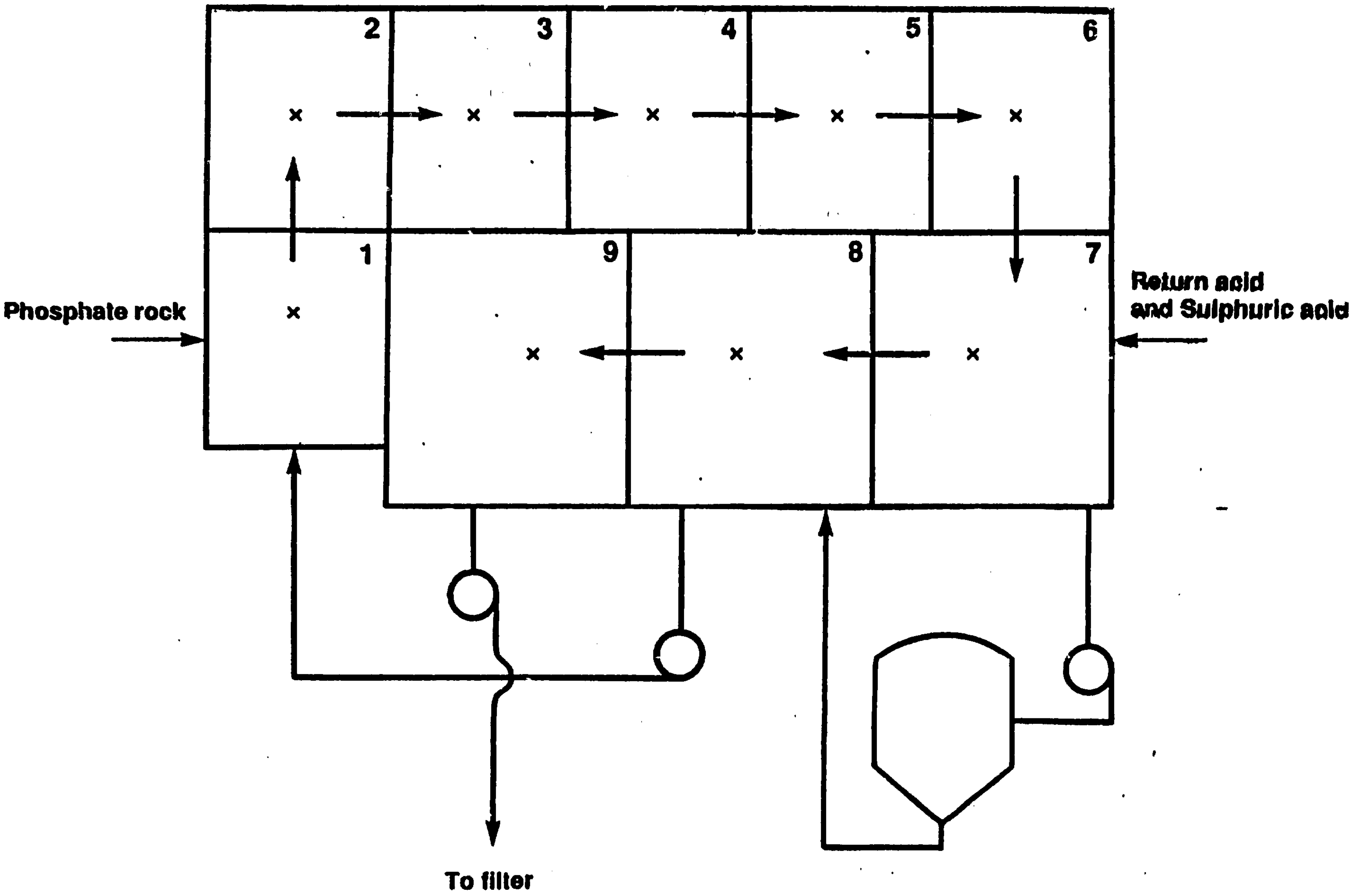
SIZE ANALYSIS :

BSS No	Aperture, mm	% Through
10	1.7	100
16	1.0	100
30	0.5	98.1
60	0.25	90.5
100	0.15	60.1

Belledune's Original Prayon Reaction System



Belledune's Converted Plant
Hemihydrate Reaction System



BELLEDUNE CONVERSION

HEMIHYDRATE REACTION SYSTEM:

R1 = C1 - 6 = 462 m^3
R2 = C7 + C8 = 232 m^3
FFT = C9 = 116 m^3

UNIT VOLUME = 1.26m^3/tpd P_2O_5

BELLEDUNE CONVERSION

FILTER = BIRD 24C = 84.5 m^2

ACHIEVED FILTRATION RATE = 6.5 tpd P_2O_5/m^2

BELLEDUNE CONVERSION

P_2O_5 LOSSES

		% CAKE
Water Soluble	P_2O_5	0.2
Insoluble	P_2O_5	1.5
		1.7

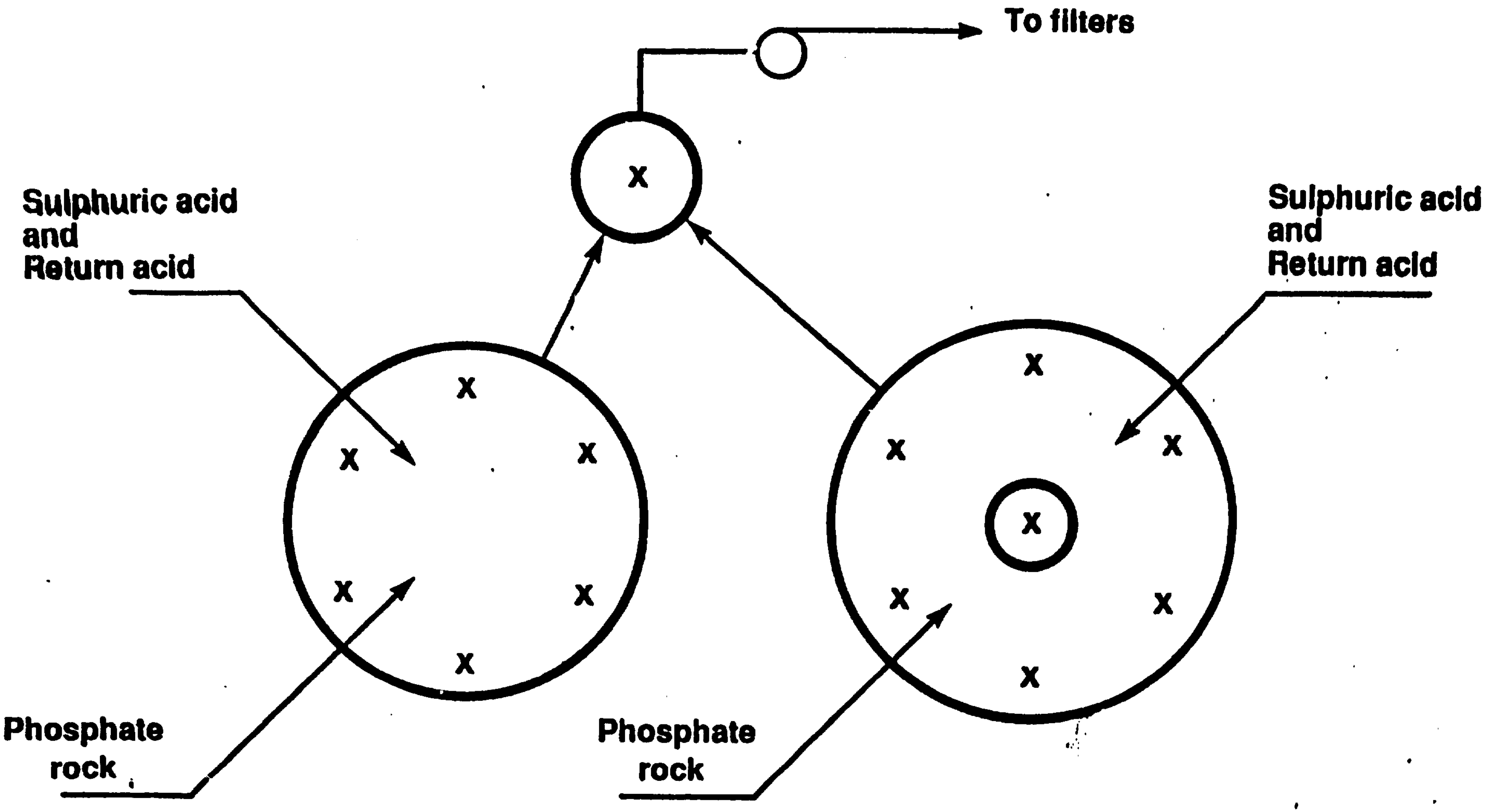
P_2O_5 Recovery Efficiency = 93.2 %

BELLEDUNE CONVERSION

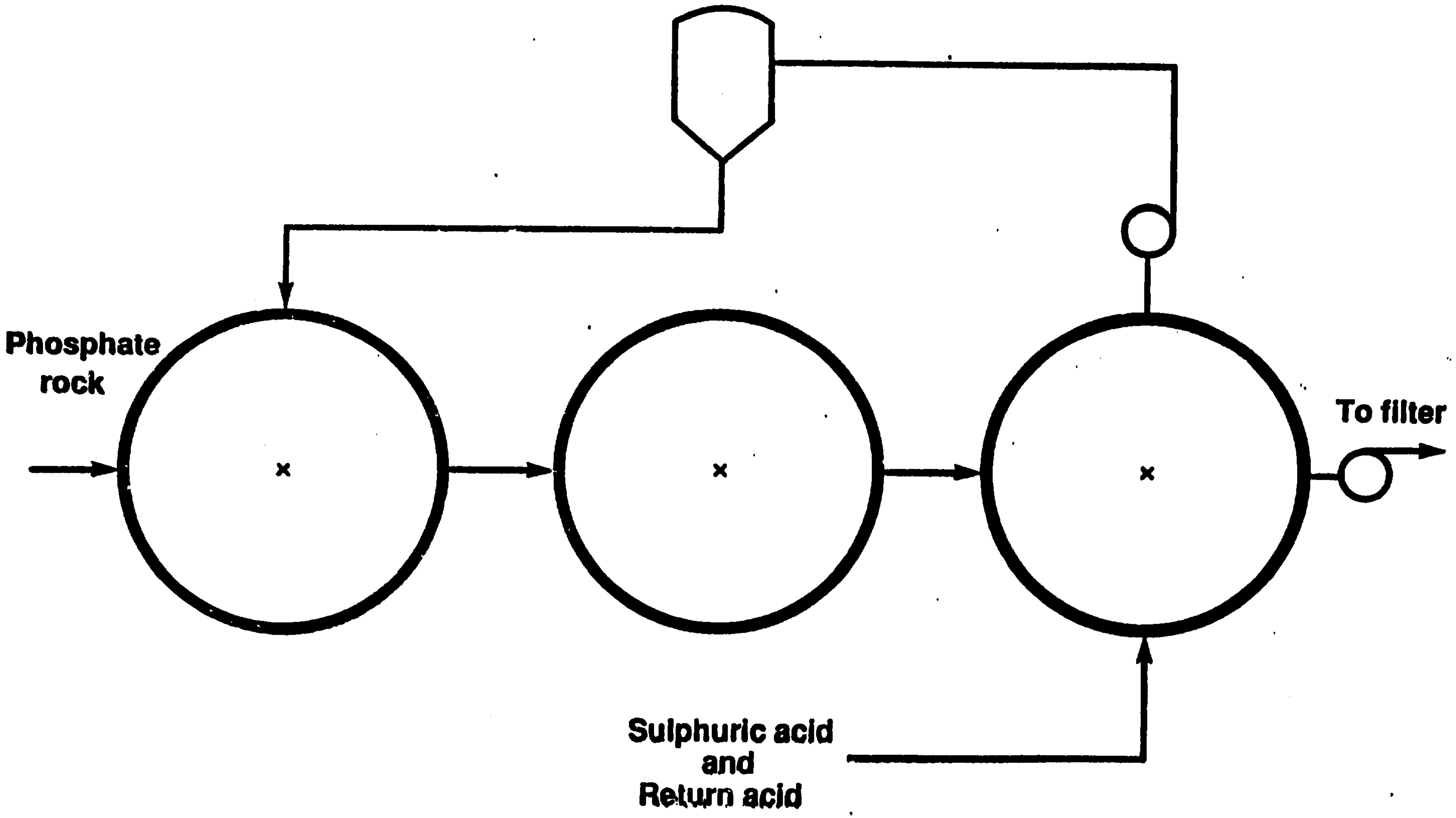
PRODUCT ACID ANALYSIS :

Item	% w/w
P_2O_5	41.1
SO_4	1.8
CaO	0.2
F	1.1
SiO_2	0.5
Al_2O_3	1.5
Fe_2O_3	1.7
MgO	0.6
Na_2O	0.05
K_2O	0.05

Supra's Original Dorr-Oliver Reaction System



Supra's Converted Plant: Hemihydrate Stage Reaction System



ANALYSIS OF 68% BPL FLORIDA PHOSPHATE

COMPONENT	% W/W
P_2O_5	31.6
CaO	45.8
SO_4	1.2
Fe_2O_3	1.18
Al_2O_3	1.4
MgO	0.45
Na_2O	0.55
K_2O	0.1
F	3.6
SiO_2	7.5

PROCESSING OF 68% BPL FLORIDA PHOSPHATE IN A HEMIDHYDRATE PLANT

P_2O_5 LOSSES	% W/W in	
	HEMIHYDRATE	GYPSUM
WATER SOLUBLE P_2O_5	5.74	0.198
LATTICE P_2O_5	0.984	0.192
UNREACTED ROCK P_2O_5	0.166	0.032
TOTAL P_2O_5	6.89	0.422

P_2O_5 RECOVERY EFFICIENCY BASES ON LOSSES IN GYPSUM CAKE

= 98.1%

PHOSPHORIC ACID ANALYSES FROM 68% BPL FLORIDA PHOSPHATE EX HEMIDIHYDRATE AND DIHYDRATE PLANTS

COMPONENT	HEMIDIHYDRATE ACID, % W/W	DIHYDRATE ACID, % W/W
P_2O_5	50.0	50.0
SO_4	2.0	4.8
Fe_2O_3	1.78	1.77
Al_2O_3	1.49	1.76
MgO	0.67	0.65
Na_2O	0.09	0.08
K_2O	0.1	0.08
F	0.7	1.3
SiO_2	0.1	0.1
SOLIDS	1.0	4.5

BY-PRODUCT $CaSO_4$ QUALITY
68 BPL FLORIDA ROCK

Component	HDH Process Gypsum % w/w	DH Process Gypsum % w/w	HH Process Hemihydrate % w/w
P_2O_5 (i. s.)	0.17	0.75	0.9
CaO	31.5	31.6	34.5
SO_4	52.5	52.6	54.5
Fe_2O_3	< 0.1	< 0.1	< 0.1
Al_2O_3	0.5	0.1	0.6
MgO	< 0.1	< 0.1	< 0.1
F	0.6	1.5	0.8
SiO_2	5.5	6.5	5.5

FLORIDA PHOSPHATE IN HDH PROCESS URANIUM DISTRIBUTION

	PHOSPHATE	PRODUCT ACID	GYPSUM
P_2O_5	31.6%	49.6%	0.422%
U	92 ppm	105 ppm	21 ppm

	HH RETURN ACID	HH CAKE	TRANSFORMATION LIQUOR
P_2O_5	40%	-	9%
U	107 ppm	93 ppm	237 ppm

TYPICAL DESIGN DATA
68 BPL FLORIDA ROCK -
HEMIHYDRATE PROCESS (HH)

PRODUCT STRENGTH	42% P2O5
REACTION VOLUME (Excluding filter feed tank)	1.5 m^3/tonne per day P2O5 produced
ROCK GRIND	100% < 1.7 mm 75% < 0.5 mm 25% < 0.25 mm
FILTER LOADING	6.3 tonnes per day P2O5 produced/m^2
POWER CONSUMPTION	100 kWh/tonne P2O5 produced
STEAM CONSUMPTION	0.2 tonnes/tonne P2O5 produced

THE ABOVE FIGURES ARE SUBJECT TO CHANGE FOR OTHER ROCK SOURCES, CONDITIONS, ETC.

72% BPL KHOURIBGA MOROCCO PHOSPHATE CHEMICAL ANALYSIS

COMPONENT	%W/W
P_2O_5	31.1
CaO	50.5
SO_3	1.83
F	3.7
Cl	0.024
SiO_2	2.88
Na_2O	0.90
K_2O	0.09
MgO	0.74
Al_2O_3	0.37
Fe_2O_3	0.23
SrO	0.14
TiO_2	0.04
CO_2	6.54
ORGANIC	0.07

72% BPL KHOURIBGA MOROCCO PHOSPHATE ACID ANALYSIS

COMPONENT	% W/W		
	HH ACIDS		DH ACID
P_2O_5	49.8	42.9	50.0
SO_4	2.0	2.0	4.5
Al_2O_3	0.1	0.1	0.45
Fe_2O_3	0.4	0.3	0.35
MgO	1.3	0.9	1.2
F	0.5	1.0	0.25
SiO_2	0.1	0.32	0.1
Cl	72 ppm	250 ppm	70 ppm
SOLIDS	1.0	1.0	4.5

FILTER SELECTION

CLIENT	FILTER TYPE	SUPPLIER	AREA M^2	DUTY
WINDMILL, HOLLAND	TABLE	UCB	70	HH
	TILTING PAN	EIMCO	63	HH
RMHK,TREPCA	TILTING PAN	EIMCO	50	HH
	BELT	NORDENGREN	15	DH
A & W	TILTING PAN	PRAYON	170	HH
	BELT	DELFILT	2 X 25	DH
CSBP	BELT	DELFILT	100	HH
	BELT	DELFILT	50	DH
PIVOT	BELT	EIMCO	45	HH
	BELT	EIMCO	24	DH
HCI	BELT	FILTERS PHILIPPE	45	HH
ROYSTER	TILTING PAN	BIRD	108	HH
			57	HH
BELLEDUNE	TILTING PAN	BIRD	88	HH
SUPRA	BELT	DELFILT	60	HH
	BELT	NORDENGREN	3 X 15	DH
NFC	BELT	DELFILT	2 X 135	HH
	BELT	DELFILT	100	DH

STAINLESS STEEL COMPOSITIONS

CAST

Name	C	Cr	Ni	Mo	Cu	Others
AISI 316	0.08	18.0 - 21.0	9.0 - 12.0	2.0 - 3.0	-	-
AISI 316L	0.03	17.0 - 21.0	9.0 - 13.0	2.0 - 3.0	-	-
AISI 317	0.08	18.0 - 21.0	9.0 - 13.0	3.0 - 4.0	-	-
40 V	0.08	25.0	5.0	2.5	2.5	-
HV 90	0.06	20.0	28.0	4.0 - 5.0	2.0 - 4.0	Nb
Uranus B 6 H	0.02	20.0	25.0	4.5	1.5	-
Durimet 20	0.07	18.0 - 22.0	27.5 - 30.5	2.0 - 3.0	3.0 - 4.0	-
Hastelloy C	0.15	15.5 - 17.5	51.0	16.0 - 18.0	-	V - 4 - 5

STAINLESS STEEL COMPOSITIONS

ROLLED

Name	C	Cr	Ni	Mo	Cu	Others
AISI 304	0.08	18.0 - 20.0	8.0 - 12.0	-	-	-
AISI 304L	0.03	18.0 - 20.0	8.0 - 12.0	-	-	-
AISI 316	0.08	16.0 - 18.0	10.0 - 14.0	2.0 - 3.0	-	-
AISI 316 Cb	0.08	16.0 - 18.0	10.0 - 14.0	2.0 - 3.0	-	Cb
AISI 316L	0.03	16.0 - 18.0	10.0 - 14.0	2.0 - 3.0	-	-
AISI 317	0.08	18.0 - 20.0	11.0 - 15.0	3.0 - 4.0	-	-
AISI 317L	0.03	18.0 - 20.0	11.0 - 15.0	3.0 - 4.0	-	-
HV 9	0.04	20.0 - 23.0	24.0 - 26.0	4.0	-	Nb
HV 9A	0.04	20.0 - 23.0	24.0 - 26.0	4.0 - 5.0	1.0-2.0	Nb
Uranus B6	0.02	20.0	25.0	4.5	1.5	-
Incoloy 825	0.03	21.5	41.8	3.0	1.8	Ti - 0.9
Hastelloy C	0.08	14.5 - 16.5	54.0	15.0 - 17.0	Co 2.5	V - 3.0 - 4.5
Hastelloy B	0.05	-	62.0	26.0 - 30.0	Co 2.5	V - 0.2 - 0.6

HDH PHOSPHORIC ACID PLANT

ON-STREAM FACTOR

85% : 310 OPERATING DAYS PER YEAR

TYPICAL DOWN TIME ALLOCATION

ANNUAL SHUTDOWN FOR MAINTENANCE = 14 DPY*
PLANNED WEEKLY MAINTENANCE, 8 HOURS = 16.5 DPY*
PLANNED WEEKLY WASHING, 7 HOURS = 14.5 DPY*
UNSCHEDULED STOPPAGES = 10 DPY*

* DPY = DAYS PER YEAR

FILTER SELECTION

TYPE	ADVANTAGES	DISADVANTAGES
BELT	HIGH SPEED - INCREASED FILTRATION RATE EASY ACCESS FOR MAINTENANCE GOOD CLOTH WASHING LONG CLOTH LIFE LOW ACID DILUTION RELATIVELY LOW CAPITAL COST	CLOTH TRACKING VACUUM LIMIT
TABLE	HIGH SPEED - INCREASE FILTRATION RATE EASY ACCESS FOR MAINTENANCE LOW ACID DILUTION GOOD DRAINAGE	CLOTH LIFE CLOTH CLEANING
TILTING PAN	WASH SEPARATELY	LIMITED SPEED NUMEROUS MOVING PARTS: HIGH MAINTENANCE POOR DRAINAGE HIGH ACID DILUTION RELATIVELY HIGH CAPITAL COST

EQUIPMENT SUPPLIERS

FILTERS : DELFILT, PANNEVIS, UCB, UCEGO

AGITATORS : PHILADELPHIA MIXERS, PROCHEM, LIGHTNIN

PUMPS : ASH, WARMAN, RHEINHUTTE, BUNGARTZ, WERNERT

MATERIALS OF CONSTRUCTION

REACTOR : CONCRETE, RUBBER LINED OR RLMS
CARBON BRICKS

VESSELS : RLMS

PIPEWORK : RLMS, POLYPROPYLENE, FRP

AGITATORS : RLMS + POLYPROPYLENE SHIELD
20 ALLOY E.G. 904L, HV9, SANRICO 28
FERRALIUM ALLOY 255

PUMPS : 20 ALLOY E.G. 904L, HV9, SANRICO 28
HIGH DENSITY POLYETHYLENE
FERRALIUM

CONTENTS

1. INTRODUCTION

Norsk Hydro Fertilizers (formerly Fisons) were pioneers in the field of granular fertilizer production in the UK during the 1930's and subsequently with the introduction of rotary drum granulation. Norsk Hydro's UK based process licensing department has designed and licensed over thirty successful granulation plants since its formation in 1965.

During the 1970's, technology developed by the company for the manufacture of Minifos - powder mono-ammonium phosphate - was adopted around the world. The Minifos process introduced the concept of pressure neutralisation of ammonia and phosphoric acid.

To meet the growing popularity of granular DAP these two concepts were merged to allow the production of DAP using a pressure neutraliser followed by a rotary drum granulation system. This process is described in some detail in this brochure.

By carrying out the first stage of ammoniation at pressure, the reactor can be safely operated at lower water contents. This significantly reduces the required fines recycle ratio in the granulation loop compared with conventional atmospheric reaction processes. Lower recycle means reduced equipment sizes hence reduced capital cost for the plant. Lower moisture content slurries also mean that less fuel is required for the drying stage so further reducing production costs.

By modifying the equipment layout slightly, the plant can be operated to produce a wide range of NPKs. Norsk Hydro Licensing (NHL) have licensed seven DAP or DAP based NPK granulation plants to client companies over the last decade.

2. PROCESS DESCRIPTION

This process description should be read in conjunction with the process flowsheet which is shown overleaf.

Liquid ammonia and phosphoric acid are fed to the pressure reactor. A small amount of sulphuric acid is usually required to control the product analysis and this is also fed to the reactor. The reactor is of the draught tube type. Ammonia is fed to the base of the central tube, phosphoric acid to the outer annulus. The resulting ammonium phosphate solution produced in the reactor is sprayed into the granulator. The pressure in the reactor is controlled at 1 kg/cm^{2}g by the release of reaction steam. Reaction steam is scrubbed with weak phosphoric acid in the ammonia scrubber to remove the majority of the ammonia carried over from the reactor. The ammonia scrubber is a co-current void tower with a high liquor spray density. The airstream drawn through the granulator also contains ammonia which is recovered in the ammonia scrubber. The exhaust from the ammonia scrubber is fed via a fan to the gas scrubber. Drier air containing dust and ammonia is also cleaned with weak phosphoric acid in the gas scrubber which operates counter-currently. The scrubber liquors are returned to the reactor.

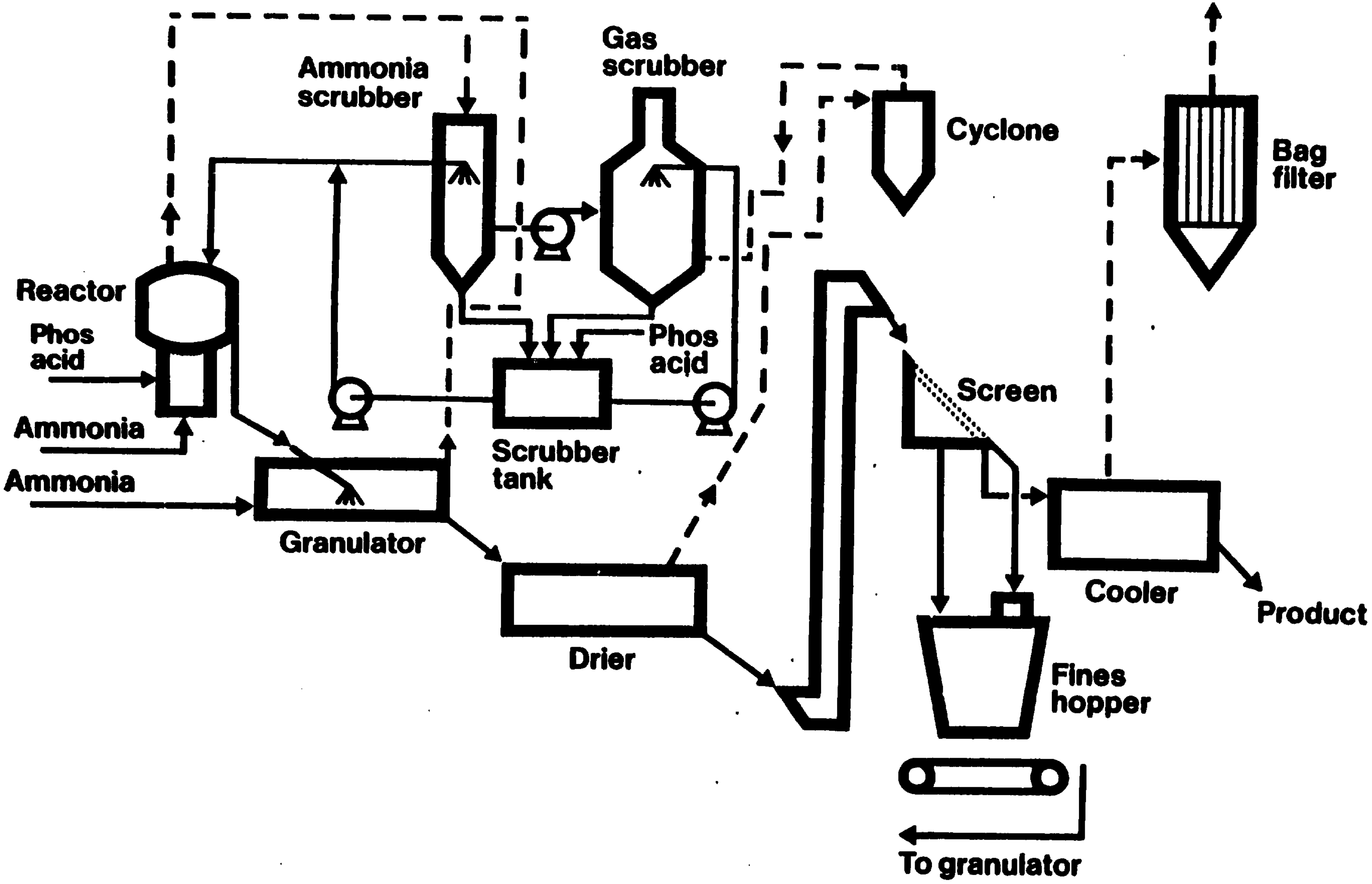
The ammonium phosphate solution is sprayed through a system of spray nozzles onto a bed of recirculating fines in the rotary granulator. Ammonia is added to achieve the required product analysis. Filler is added if necessary. The granulated material then falls directly into the rotary drier. Here the material is contacted with a hot airstream to reduce the moisture content of the solids for good storage properties. Drier air is heated by a direct fired gas or oil burner. From the drier the material is elevated to the screening section. These screens are double deck machines. Undersized material is separated on the lower deck for recycling to the granulator via the fines hopper which is located directly beneath the screens. Oversized material is separated on the upper deck and crushed before passing to the fines hopper.

The flowrate of product to storage can be regulated and excess product fed either to the crushers or direct to the fines hopper to maintain the recycle rate as required. Recycled material is metered back from the fines hopper to the granulator at a constant rate so that granulation is maintained at the optimum at all times.

The product stream is cooled in a simple fluidised bed cooler to a temperature satisfactory for product storage and bagging. The dust in the air stream from the cooler is recovered in a bag filter.

To provide a clean working environment dust extraction points are fitted at various transfer points. All the points are connected to a centralised bag filter where the dust is removed from the airstream.

Norsk Hydro Licensing DAP Process



3. TECHNICAL FEATURES

The Norsk Hydro Licensing process for the manufacture of DAP is similar to the conventional rotary drum granulation processes operating throughout the world. However, the NHL process has several features which provide significant advantages over competing processes.

3.1 Pressure Neutralisation

Conventional atmospheric reactors producing DAP operate at a temperature of 115-120°C. To be a true solution at this temperature the ammonium phosphate must be associated with approximately 20% water. At moistures less than this the reactor is operating with a slurry of suspended solids, which leads to pumping difficulties and problems of sedimentation and blockages. The NHL process operates at a pressure of 1 kg/cm^2 gauge so that the boiling point is elevated by about 20°C. Thus, the steep solubility curve of the water/ammonium phosphate-temperature system allows the significant advantage of a lower moisture content whilst maintaining the ammonium phosphate in a true solution. The amount of water fed from the reactor to the granulator is therefore minimised.

In process terms, this means operation with a recycle ratio of less than 4:1 compared to typically 6:1 for atmospheric reaction processes. The size of all equipment in the granulation loop is therefore some 30% smaller and capital cost is consequently reduced. As the plant throughput is smaller for the same product rate, operating costs are also lower.

In particular, fuel consumption is significantly lower than conventional processes, not only because of the lower recycle ratio, but also because granulation takes place at lower moistures.

The pressure reactor affords a degree of operating flexibility and is very stable in terms of process control, - the reactor operates with a residence time of around 30 minutes for the ammonium phosphate solution. Stabilising the operating conditions, - temperature, moisture content, and NH_3-P_2O_5 mole ratio, is crucial if production is to be consistently maximised and blockages avoided. The contents of the pressure reactor act as a buffer to smooth out process fluctuations such as changes in flow or composition of the reactants. This gives a major advantage over pipe reactor systems. Pipe reactors use the same principle - that of high temperature and pressure to produce a low water content slurry - but cannot offer as efficient or as stable a reaction as the pressure neutraliser.

The pressure neutraliser can be easily controlled so as to maintain slurry conditions independent of throughput. Operation over the range 50-100% is easily achievable without loss of efficiency.

Reaction steam is disengaged from the slurry in a controlled manner prior to spraying in the granulator. In this way the water and energy balance in the granulator is optimised and the moisture carrying burden of the granulator exhaust is reduced.

The pressure reaction system requires no pump to develop the pressure head necessary to distribute the solution through the spray nozzles in the granulator. This saves on both capital and operating costs for what can be a troublesome pumping duty.

3.2 Draught Tube Reactor

The pressure neutraliser has been in use for twenty years and as such is well proven. However, in the earlier pressure neutralisers, the use of a turbine type mechanical agitator and mechanical seals involved some maintenance effort.

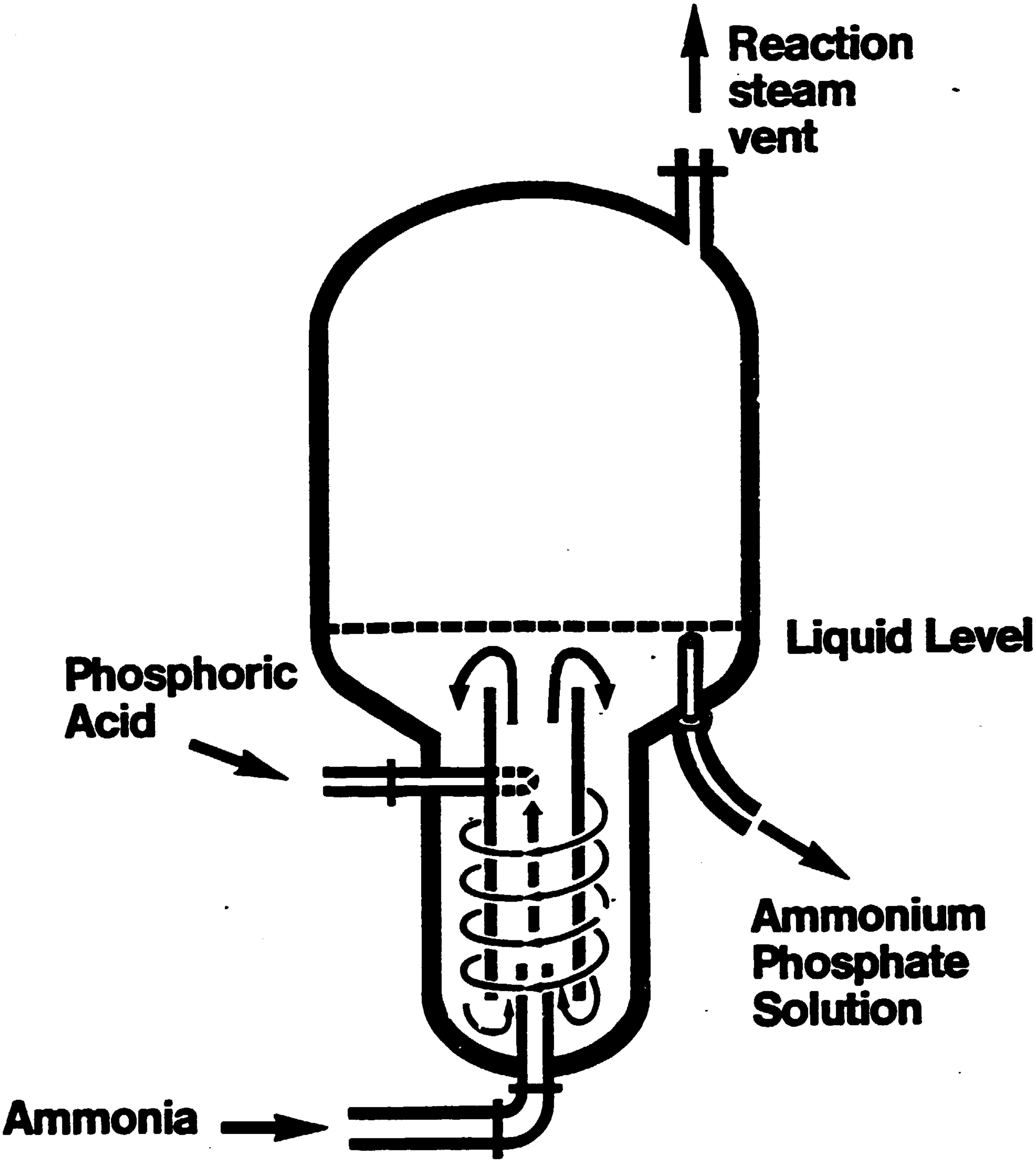
It was to eliminate this, and the power consumption associated with mechanical agitation, that the draught tube reaction system was developed. The objective of the reactor development was to exploit the energy of reaction to mix the contents of the reactor. The draught tube system used for this consists of two concentric tubes. The principle is illustrated on the diagram overleaf. The outer tube is closed at the bottom and forms the wall of the reactor. It is connected at the top to the wider cylindrical section which forms the disengagement space.

The inner tube is supported to form an annular channel. Phosphoric acid is fed tangentially into the annulus and ammonia is injected through a sparge pipe inserted into the base of the inner tube. The exothermic reaction occurs in the draught tube and causes a reduction in density which creates an upward movement of the reacting mass. The liquid rises up the central tube and descends through the annulus in a continuous circulating motion.

A high degree of internal mixing is achieved which ensures a very high reaction efficiency. The application of the draught tube reactor to commercial scale operation has proved to be very successful, with the first full scale plant having been in operation for over seven years.

3.3 Recycle Control

As stated previously, the operation of the pressure reactor system provides a major process advantage associated with the water balance over the granulator. This allows the NHL process to operate at substantially lower recycle ratios than conventional DAP processes. To ensure efficient operation the rate of return of recycle fines to the granulator is continuously metered and controlled. This is obtained by the use of a fines hopper fitted with an infinitely variable speed extractor and weighbelt. The hopper has a twenty minutes buffer storage capacity, although during normal operation the hopper is operated less than half full.


Reaction
steam
vent
Liquid Level
Phosphoric
Acid
Ammonium
Phosphate
Solution
Ammonia

In this way, unavoidable plant upsets and variations in granulation efficiency are effectively smoothed out before any major change in plant operation is required.

3.4 Dust Recovery and Bag Filters

Norsk Hydro Fertilizers has experience with both cyclones and bag filters for dust recovery duties on granulation plants. As it is necessary to use wet scrubbing to recover ammonia from the drier airstream, and the temperature and humidity can pose some difficulties for bag filters, cyclones are used on this duty. For the cooler and dedusting duties however bag filters are used.

NHF's operational experience with bag filters has enabled NHL to incorporate features to prevent any potential problems arising. By careful attention to the cloth cleaning mechanism, cloth material and start-up and shut-down procedures, the efficiency and simplicity of a bag filter system can be safely used.

The use of bag filters to remove all the dust from the cooler airstream allows it to be utilised as dilution air for the drier airstream. In this way, a further 20% reduction in fuel consumption can be made. This system is used successfully on NHF's own granulation plants in the UK.

4. PERFORMANCE DETAILS

In order to produce DAP of analysis 18:46:0 it may be necessary to adjust the formulation by the addition of a filler (usually sand) and a nitrogen bearing compound. This is most conveniently accomplished by using sulphuric acid which reacts with ammonia. The quantities of filler and sulphuric acid are small and depend on the source and quality of the phosphoric acid.

4.1 Raw Material Consumptions

Phosphoric acid, 52-54% P_2O_5 (as 100% P_2O_5)	: 461 kg/tonne
Ammonia (as 100% NH_3)	: 221 kg/tonne
Sulphuric acid, 96-98% (as 100% H_2SO_4)	: 25 kg/tonne
Filler	: 60 kg/tonne.

4.2 Utility Consumptions

Steam, 4 kg/cm^{2}g	: 20 kg/tonne
Fuel oil, Net CV 9,500 Kcals/kg	: 3 kg/tonne
Power	: 35 kWh/tonne
Process water	: 0.35 m^3/tonne

4.3 Product Quality

Nitrogen content	: 18%
Total P_2O_5 content	: 46%
Moisture	: 1.0% or 1.5% (to client's specification)
Size range, 2-4 mms	: 95%
Bulk density	: 1.0 t/m^3
Hardness, 2-3.35 mms	: 6 kg/granule

4.4 Typical Operating Conditions

Pressure Neutraliser

Pressure	: 1.0 kg/cm^{2}g
Temperature	: 143-147°C
Water content	: 10-14%
Mole ratio	: 1.45-1.5/1

Granulator

Temperature : 90°C
Water content : 2%
Mole ratio : 1.75-1.85/1
Recycle ratio : 4/1

Drier

Air inlet temperature : 250°C
Air outlet temperature : 90°C
Solids outlet temperature : 85°C

Scrubbers

Liquor temperature : 80-85°C
Mole ratio : 0.4-0.5/1
P_2O_5 content : 41-46%

5. CAPITAL COSTS

Capital cost can vary tremendously depending on the engineering contractor involved, the source of the equipment supply etc. However, with a Western European contractor a typical battery limits cost for a 1,000 tpd DAP plant in 1986 would be:-

$$\$14.5 \times 10^6.$$

This figure is exclusive of the building and off-sites.

6. REFERENCE LIST

AMMONIUM PHOSPHATE SLURRY GRANULATION

Company	Location	Capacity	Date	Remarks
CNO	Oostende, Belgium	190,000 tpa	1962	Pressure Reactor
ANIC	Gela, Sicily	120,000 tpa	1967	Atmospheric Reactor
Industrias Luchsinger Madorin SA	Rio Grande, Brazil	450,000 tpa	1975	Pressure Reactor
Hemijska Industrija Veles	Titov Veles, Yugoslavia	200,000 tpa	1979	Pressure Reactor
Hemijska Industrija Zorka Sabac	Sabac, Yugoslavia	155,000 tpa	1980	Pressure Reactor
Hellenic Chemical Industries	Vassiliko, Cyprus	160,000 tpa	1982	Pressure Reactor
Industrija Nafta	Kutina, Yugoslavia	472,000 tpa	1983	Pressure Reactor
Hindustan Lever	Haldia, India	150,000 tpa	1986	Pressure Reactor

NORSK HYDRO FERTILIZERS PROCESS FOR AMMONIUM PHOSPHATE BASED GRANULAR NPK

PROCESS DESCRIPTION

This process description for a granular DAP/NPK plant refers to the enclosed sketch.

Ammonia and phosphoric acid are fed, under flow control, to the draught tube reactor. The resulting ammonium phosphate solution can be fed to either the granulator or the Minifos tower. The pressure in the reactor is automatically controlled at 1 kg/cm^2g for DAP based grades. The reaction between ammonia and phosphoric acid is highly exothermic, and this heat is removed by evaporating a controlled amount of water. The resulting steam is scrubbed with a phosphoric acid solution in the ammonia scrubber to remove the majority of the ammonia carried over from the reactor. The ammonia scrubber is a co-current void tower vessel with a high liquor spray density. The air stream drawn through the granulator also contains ammonia which is scrubbed out in the ammonia scrubber. The exhaust from this vessel is fed via the ammonia scrubber fan to the gas scrubber. Here a dilute solution of phosphoric acid is used to remove any residual ammonia and the small quantity of ammonia and dust contained in the drier exhaust gases. This scrubber is operated in a counter-current manner. The scrubbing liquor is recirculated via an integral sump and pump. A constant blowdown of liquor to the ammonia scrubber is metered by means of level control. An identical recirculation system is used for the ammonia scrubber, with the blowdown being fed to the reactor. The ammonium phosphate solution is sprayed through a system of spray nozzles onto a bed of recirculating fines in the rotary granulator.

For the production of most NPK grades, the reactor can be operated to produce powder MAP, Minifos. The $NH_3:P_2O_5$ mole ratio is 1:1 compared to 1.4:1 for DAP production. The reactor pressure for Minifos production is 2.1 kg/cm^{2}g, which reduces the moisture content of the boiling ammonium phosphate solution to around 10%. The solution is fed to a spray tower through a specially designed steam injection nozzle. The tower operates on a natural draught principle, with a low air velocity to minimise the entrainment of dust in the air stream. The ammonium phosphate is solidified within a couple of metres after discharge from the nozzle, and the powder is cooled sufficiently for ease of handling at the base of the tower, where it is recovered with a twin bladed scraper. It can then be fed either directly to one of the raw material hoppers for immediate use in the granulation plant, or to intermediate storage for later use. The air from the tower and the steam produced in the reactor are scrubbed with water in the spray tower chimney. The blowdown from this scrubber is fed to the reactor as temperature control water. Therefore, the Minifos process can be operated with no liquid effluent.

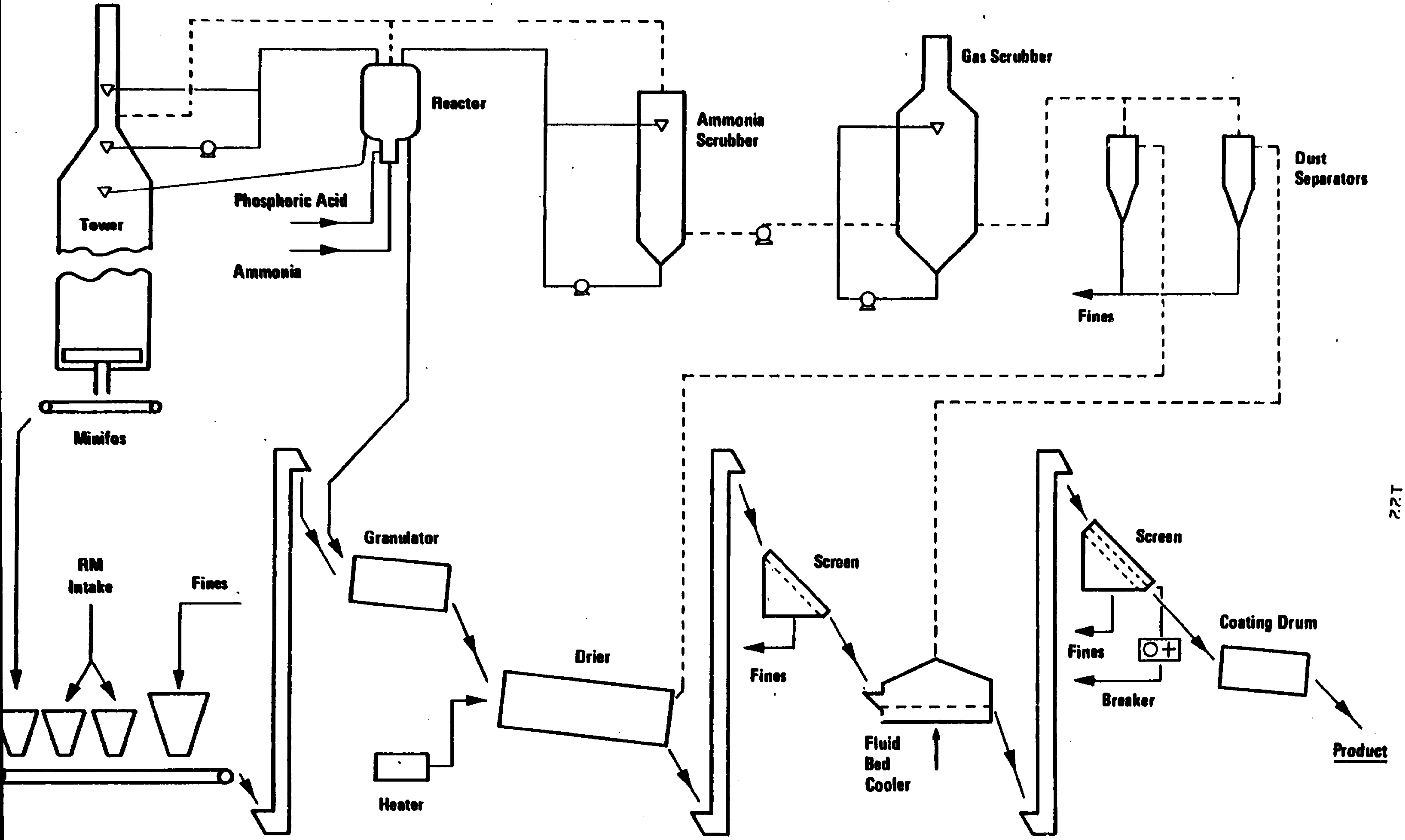
The other raw materials, usually urea, potash, filler and Minifos are recovered by mechanical shovel from storage and fed via the intake system to the raw materials hoppers. The intake system has a magnet to remove metallic objects, and a grizzly to remove large lumps from the feed. These lumps are crushed and returned to the intake elevator boot. The raw materials are accurately metered by extractor weighbelts onto the collector conveyor where the recycled fines are also added. These are elevated to the granulator where small quantities of ammonia, sulphuric acid and steam are used to achieve the optimum conditions. The granulated material falls directly down a chute into the rotary drier. The material is contacted co-currently with a hot air stream to reduce the moisture content of the granules to a value satisfactory for storage of the product. The air is heated by means of a direct fired gas or oil burner.

Any large lumps of build-up are removed by a trommel fitted to the end of the drier and broken up by the ex-drier lump breaker. The material is then elevated to the hot screens. The flow is equally divided over the screens which are double deck machines. On the upper cloth large oversize particles (greater than 20 mm) are removed and fed via a conveyor directly to the pulverisers. This flow is small representing only 1 to 2% of the plant throughput. On the lower deck the cloth is split into two parts. The first section has a small mesh cloth which removes the reject fines size particles. The second section has a large mesh cloth which removes the smaller sized product. This can be either incorporated with the product stream, or diverted by means of a flap fitted beneath the screen into the fines bin. The product/oversize stream from the screens is fed to a fluid bed cooler. There the solids temperature is reduced to a satisfactory value for product storage/bagging and oversize crushing. The material leaving the cooler is elevated to the cold screens.

The flow is equally divided over the screens which are double deck machines. On the upper cloth the oversize is removed, crushed in the pulverisers and recovered in the fines bin. On the lower cloth the product is separated from any residual fines. The flowrate of product to storage can be regulated and excess product fed to the pulverisers to maintain the recycle fines flow as required.

For the majority of NPK grades, especially ones containing urea, coating of the product with oil and dust is essential for good product storage properties. This is applied in a rotary drum. The oil and dust flows are automatically ratioed to the product flowrate as measured on the weighbelt.

The airstream from the drier contains dust which is recovered directly to the solids section of the process by cyclones. This stream is then scrubbed in the gas scrubber before discharging to atmosphere. The scrubbing liquor blowdown is fed back to granulator when Minifos is being made in the liquids section of the process.


Tower
Reactor
Phosphoric Acid
Ammonia
Ammonia Scrubber
Gas Scrubber
Dust Separators
Fines
Minifos
RM Intake
Fines
Granulator
Heater
Drier
Screen
Fines
Fluid Bed Cooler
Screen
Fines
Breaker
Coating Drum
Product

The dust in the air stream from the fluid bed cooler is recovered in a bag filter. Air is extracted from several points in the process where material is being transferred between items of equipment. This air has a considerable dust content, which is also recovered in a bag filter. The air streams leaving the bag filters are sufficiently free of dust that they can be discharged directly to atmosphere without further scrubbing.

TECHNICAL FEATURES

Norsk Hydro Fertilizers process for the manufacture of NPK fertilizer is similar to the conventional rotary drum granulation processes operating throughout the world. However, Norsk Hydro Fertilizers process has several features which provide significant advantages over competing processes.

Pressure Neutralisation

The well established technique of neutralizing phosphoric acid with ammonia under pressure was first developed by Fisons, now operating as Norsk Hydro Fertilizers Limited, in the 1960's. It has been employed on a number of granulation plants where the ammonium phosphate solution from the pressure reactor is fed directly to the granulator for the manufacture of NP compounds such as DAP, and NPK compounds. Minifos, Norsk Hydro Fertilizers powder MAP, has been used throughout the world as a high quality raw material for the manufacture of granular NPK.

Norsk Hydro Fertilizers pressure reactor is capable of working at both 1 kg/cm^2g pressure for the production of DAP based grades and at 2.1 kg/cm^2g pressure for the production of MAP powder. Operation at 2.1 kg/cm^2g elevates the boiling point of the reaction mixture to around 170°C thus enabling the moisture to be reduced to 10% whilst still maintaining the ammonium phosphate in solution. The reduction in pressure through the spray nozzle from 2.1 kg/cm^2g to atmospheric is used to flash off water and solidify the MAP to a fine powder.

The size range of the powder is controllable by the addition of steam to the spray line. Product moisture at the base of the tower is 7% which enables easy handling.

Conventional atmospheric reactors producing DAP operate at a temperature of 115-120°C. To be a true solution at this temperature the ammonium phosphate must be associated with approximately 20% water. At moistures less than this the reactor is operating in the slurry regime which leads to problems of sedimentation and blockages. Norsk Hydro Fertilizers process operates at a pressure of 1 kg/cm^2 gauge so that the boiling point of the reaction mixture is elevated by some 20°C. Thus, the steep solubility curve of the water/ammonium phosphate - temperature system allows Norsk Hydro Fertilizers process the major advantage of operating at lower moistures whilst maintaining the ammonium phosphate in the solution regime. The amount of water fed from the reactor to the granulator is therefore minimised. In terms of process operation this means that Norsk Hydro Fertilizers process operates with a lower fines recycle ratio; for example, 4:1 compared to 6:1 for atmospheric reaction processes manufacturing DAP. This leads to a reduction in the size of all the equipment related to the total throughput of the plant and consequently capital costs are reduced. As the plant throughput is smaller for the same output rate, operating costs are also lower.

The pressure reaction system requires no pump to develop the pressure head necessary to distribute the solution through the spray nozzles in the granulator. This saves on both capital and operating costs for what remains an arduous pumping duty, despite recent developments in design and materials of construction.

By carrying out the reaction in an enclosed pressure vessel there is no possibility for the spillage and overflow from the reactor characteristic of the atmospheric process. This saves time and effort in plant cleaning and improves raw material efficiency. The reactor is positioned above the granulator thus allowing greater access to the plant at ground floor level.

Draught Tube Reactor

Norsk Hydro Fertilizers have developed a reactor which requires no external agitation to promote efficient reaction. Maintenance problems associated with agitators and mechanical seals are thus eliminated and power consumption for the plant is reduced. The principle of operation is to provide a high degree of mixing by means of a high internal recirculation of solution. The phosphoric acid feed is added to the outer annulus and the ammonia injected at the base of the central tube. The reaction takes place inside the central tube and the steam generated leads to an apparent reduction in density. The difference in hydrostatic head between the inner tube and outer annulus results in a high liquid recirculation rate.

This gives a reaction efficiency of 100% and the ammonia loss from the reactor is merely due to the vapour pressure of ammonia over ammonium phosphate solution. The lower ammonia loss considerably reduces the scrubbing duty for the ammonia recovery in the gas scrubbers for DAP based grades. For MAP production the ammonia loss from the reactor in the reaction steam is so low that water scrubbing in the tower chimney is quite adequate. In fact, as the steam is at a pressure of 2.1 kg and is practically clean, it can be used to vaporise the incoming liquid ammonia feed.

The major advantage of the Norsk Hydro Fertilizers pressure reactor over other reactor systems is its flexibility and stability of process control and operation. The draught tube reactor operates with a residence time of around thirty minutes for the ammonium phosphate solution. Maintaining the ammonium phosphate solution at the required temperature, moisture content, and $NH_3:P_2O_5$ mole ratio is crucial to maximise the plant's production rate. The contents of the pressure reactor act as a buffer to smooth out process fluctuations such as changes in flow or composition of the reactants, which particularly occur in the scrubbing section. This gives a major advantage over pipe reactor systems. A pipe reactor is designed for a specific set of conditions and any deviation from these significantly increases the probability of blockages in the pipe.

The solids content of the phosphoric acid is another potential cause of blockages. This is not the case for the Norsk Hydro Fertilizers process, which has several plants which are operating with acid containing exceptionally high solids content.

Pipe reactors are prone to blockage when operating on pure ammonium phosphate solution. This condition has been alleviated by the addition of a significant quantity of sulphuric acid in the pipe which obviously reduces the flexibility of the process in terms of product NP analysis that can be obtained.
Norsk Hydro Fertilizers process does not have this process requirement for sulphuric acid addition.

Norsk Hydro Fertilizers pressure reaction process is well proven over a number of years, and our records indicate an average process availability well in excess of 90%. A large turn down ratio is another advantage of the draught tube reactor. Operation over the range 50-100% rate is achievable without loss of efficiency.

Norsk Hydro Fertilizers have designed plants using a range of materials of construction for the reactor, from 316 stainless steel to 20 grade alloys (Incalloy, UB6, HV9 etc). The choice is dependent on the analysis of the phosphoric acid to be used. Reactor life is in excess of ten years.

To summarise, the major advantages of the combined powder MAP/slurry DAP draught tube reactor system are:-

- Greater flexibility of production rate and product analysis.

- Greater plant availability. The Minifos powder MAP section is operated independently of the granulation section, therefore production can continue when the granulation plant is shut down.

- Stability of operation as process variations are smoothed out by buffer capacity in the reactor.

- High ammonia efficiency, thus no acid scrubbing is required for MAP production allowing steam to be utilised for ammonia vaporisation.

- Proven reliability; thirty one Minifos plants have been designed by Norsk Hydro Fertilizers since the mid 60's.

Gas Scrubbing

The scrubbing system is based on the use of void towers for ammonia and dust scrubbing; a system Norsk Hydro Fertilizers have employed on their own plants for over thirty years. Gaseous exhaust streams from the granulator and reactor contain ammonia which is first scrubbed in the ammonia scrubber and finally in the gas scrubber where any residual ammonia is recovered. Particulate matter from the drier air stream is also removed in the gas scrubber.

A dilute phosphoric acid scrubber liquor is employed in order that any variation in the quantity of ammonia lost from the granulator or reactor can easily be recovered without resulting in the crystallisation of ammonium phosphate in the scrubbing liquor with its attendant problems of sedimentation and pipeline blockages. A high scrubbing efficiency is achieved and since the liquor is eventually fed to the reactor or granulator there is a minimal loss of raw material from the plant. There is no liquid effluent.

All phosphoric acid contains a certain percentage of fluorine as hydrogen fluoride (HF). A proportion of this is evolved in the ammonia and gas scrubbers. If very low fluorine emission standards are essential - less than 10 mg/m^3 F - then a further stage is required in the gas scrubber. This stage consists of an extra section of void tower with a packed section at the top, and utilises water as the scrubbing medium. The method of operation can be either once through, using sea water which is discharged directly back into the sea, or if the regulations do not allow this, recirculating, discharging a small flow of concentrated liquid effluent. This is usually neutralised before disposal.

Incorporating the effluent back into the process is not feasible as the absorbed fluorine would be re-released. The inclusion and/or design and operation of this item in the process is entirely dependent on the local legal regulations concerning the discharge of liquid and gaseous effluents.

Rotary Drum Granulation

Norsk Hydro Fertilizers were pioneers in the field of granular fertilizer production in the 1930's and subsequently with the introduction of rotary drum granulation. We now operate seven granulation plants at four sites in the UK, six of these have rotary drum granulators and three have the facility for ammonium phosphate solution addition. Additionally, we have licensed and designed a further twenty seven plants throughout the world, twenty three of which are operational, with the remaining four at the design or construction stage. All these plants are based on the rotary drum principle, and seven have the facility of ammonium phosphate solution addition directly to the granulator. The current design for a rotary granulator has evolved from over forty years' operating experience. A particularly important point for NPK grades is the positioning and method of addition of the liquid feeds - ammonium phosphate, ammonia, steam, and if required, sulphuric acid. The granular material in the drum is quite moist and readily adheres to the shell of the drum. This build-up is not allowed to accumulate and in Norsk Hydro Fertilizers process is continually removed by a system of flexible rubber panels fitted to the inside of the shell. This system is far superior to other methods of drum cleaning scrapers which are prone to build-up themselves, and external hammers which are environmentally undesirable in terms of noise in a working area.

Drying

There has been considerable research by Norsk Hydro Fertilizers into the mechanisms and techniques of fertilizer drying. The drier represents the largest item of equipment in the process and normally determines the size of building required to house the plant.

The design of this unit is therefore optimised in terms of size, solids drying profile, number and positioning of internal flights, and hammer placement. The air flow, solids and air temperature profiles are closely and, more importantly, individually controllable to achieve the required product moisture content. This is particularly advantageous for NPK fertilizers which are temperature sensitive materials.

The air heater is normally a direct fired burner using either gas or oil as the fuel.

Cooling

Product and oversize cooling is required for the majority of NPK grades. This is because fertilizer containing urea and potash is sticky when hot. Therefore, hot crushing of this material would quickly lead to pulveriser build-up, and thus increase the frequency of plant shutdowns for cleaning. Norsk Hydro Fertilizers recommend a fluid bed cooler as this type of machine has the distinct advantages of lower capital and maintenance costs, and less material breakdown due to attrition. It also allows improved plant layout. As an alternative, Norsk Hydro Fertilizers are prepared to offer a rotary drum cooling design, if this is preferred by the client.

As a method of energy conservation the re-use of the cleaned cooler exhaust air as dilution air in the drier air heater offers potential savings in excess of 20% of the fuel consumption for urea based NPK grades.

Screens

The screens employed on Norsk Hydro Fertilizers granulation plants are of the inclined vibrated screen cloth type. These have a higher capacity per unit area of screen cloth than other types of screens, thus minimising the amount of space required in the plant layout.

They have a very high screening efficiency because of the flexibility given by the mechanisms for on-line adjustment - screen angle and cloth vibration. The mechanism of granulation, producing a surplus of product size material, coupled with the ability to closely control the quantity of small sized product rejected into the fines stream means that rounded granules with a fairly close size range will be available from the screening section.

Recycle Control

As stated previously, the operation of the pressure reactor system provides a major process advantage associated with the water balance over the granulator. This together with the liquid phase controlled granulation mechanism for ammonium phosphate solution based grades allows the Norsk Hydro Fetilizers process to operate at substantially lower recycle ratios than conventional AP slurry processes. To ensure efficient operation under this regime, the rate of return of recycled fines to the granulator is continuously metered at a fixed value. This is obtained by the use of a fines hopper fitted with an infinitely variable speed extractor and weighbelt. The hopper has a twenty minutes buffer storage capacity, although during normal operation the hopper is operated less than half full. In this way, unavoidable plant upsets and variations in granulation efficiency are effectively smoothed out before any major change in plant operation is required.

Dedusting System

On a modern fertilizer plant it is essential to have an efficient and reliable dust extraction system. However, Norsk Hydro Fertilizers design goes beyond this and minimises the size of dust extraction system required by careful attention to plant layout, minimising the number of areas of dust generation at material transfer points, selection of equipment such as screens which are totally enclosed to prevent dust emission. The dust extraction system operates independently of the process and has its own fan and dust recovery bag filter.

PERFORMANCE DETAILS

Raw Materials

The combination of the various raw materials available permit the widest range of analyses possible to be produced on a Norsk Hydro Fertilizers NPK granulation plant. The plant can be designed to operate on any combination of the following:

Nitrogen sources : Urea, ammonium nitrate, ammonium sulphate
P_2O_5 sources : Mono ammonium phosphate (usually powder)
Di ammonium phosphate (solution)
Single superphosphate
Triple superphosphate
Phosphate rock
K_2O sources : Potassium chloride, potassium sulphate
Filler : Locally available material (usually sand)

Urea and ammonium nitrate are not chemically compatible, therefore either one or the other is chosen as the primary nitrogen source. The optimum choice for a new fertilizer site is usually urea, MAP, DAP and potassium chloride. These allow production of the highest possible chemical analyses, with lower concentrations being formulated with the use of filler. Typically ceiling analyses for MAP and DAP based fertilizers are as follows for the most common NPK ratios.

DAP based		MAP based	
DAP	18:46:0	MAP	11:56:0
	19:19:19		18:18:18
	28:28:0		27:27:0
	14:35:14		9:27:18
	10:30:20		

As can be seen, slightly higher analyses are possible for DAP based fertilizers. However, the penalty for this is one of plant output rate. For a unit capable of manufacturing 1000 tpd of DAP, 1100-1200 tpd of DAP slurry based granular NPK are possible.

The use of powder MAP enables this output to be increased to 1600 tpd of NPK fertilizer. Therefore a plant as shown in the flowsheet where both DAP slurry and MAP powder are available provides the best solution in terms of output rate, achievable analysis, and operational flexibility.

Typical raw materials specifications are:

Phosphoric acid - merchant grade

Total P_2O_5 : 52-54%
H_2SO_4 : 2-4%
Solids : 1-4%
Temperature : Ambient
Pressure : 6 kg/cm^{2}g (at grade)

Ammonia

NH_3 : 99.5% minimum
H_2O : 0.5% maximum
Oil : 10 ppm maximum
Temperature : -33 or 0°C dependent on storage
Pressure : 7 kg/cm^2 (at grade)

Sulphuric acid

H_2SO_4 : 96-98%
Temperature : Ambient
Pressure : 6 kg/cm^{2}g (at grade)

Potash

K_2O : 60% minimum
H_2O : 0.5% maximum
Condition : Free flowing powder 99% $<$ 2 mm size

Urea

N : 46% minimum
H_2O : 0.5% maximum
Condition : Solid Prills 99% $<$ 2 mm size

Filler

Type : Sand or equivalent free flowing inert material
H_2O : 5% maximum
Size : 99%< 1.7 mm

Raw Material Efficiency

The following raw material efficiencies are achieved, based on measurable losses from the plant.

Nitrogen	99%
P_2O_5	99.9%
K_2O	99.9%

Utilities

The following utilities are required:

Steam

Low pressure
Condition : dry saturated
Pressure : 4 kg/cm^{2}g
Quantity : 50 kg/tonne of product
Medium pressure
Condition : dry saturated
Pressure : 8 kg/cm^{2}g
Quantity : 50 kg/tonne of product

Electric Power

Voltage : 440 V
Frequency : 50 Hz
Phase : 3
Usage : 25-40 kWh/tonne of product

Process Water

Suspended solids : nil
Chloride : 50 ppm maximum
Pressure : 7 kg/cm^{2}g (at grade)
Temperature : Ambient
Quantity : 0.35 m^3/tonne of product

Air heater fuel - alternatives

Fuel Oil

Type : medium/heavy fuel oil
Calorific value : 10,000 kcals/kg minimum
Temperature)
Pressure) : to suit burner
Quantity : 3-18 kg/tonne of product

Gas

Type : natural gas
Calorific value : 10,000 kcals/Nm^3 typically
Quantity : 3-18 Nm^3/tonne of product

Process air

Condition : clean, filtered
Pressure : 7 kg/cm^2 g
Temperature : Ambient

Instrument air

Condition : clean, filtered and oil free
Pressure : 7 kg/cm^2 g
Temperature : Ambient

NORSK HYDRO FERTILIZERS

REFERENCE LIST

SLURRY GRANULATION PROCESS

COMPANY	COUNTRY	CAPACITY	DATE	ROUTE
Fisons Fertilizers	England	190,000 tpa	1960	MAP + AP grades
Fison-UCB	Belgium	150,000 tpa	1962	AN based
Fisons Fertilizers	England	210,000 tpa	1966	AN/AP grades
ANIC	Italy	120,000 tpa	1967	DAP + AP grades
Asur	Spain	150,000 tpa x 2	1969	AN based/GAN
ILM	Brazil	450,000 tpa	1975	DAP/Urea based
Shahpur	Iran	275,000 tpa	1974	DAP
Zletovo	Yugoslavia	200,000 tpa	1979	DAP/Urea based
Zorka	Yugoslavia	155,000 tpa	1980	AN Based
State	Iraq	300,000 tpa x 2	1982	TSP
HMC	Cyprus	160,000 tpa	1982	DAP + AS/AP grades
Kutina	Yugoslavia	472,000 tpa	1983	DAP/AP grades/Urea based

LOW RECYCLE GRANULATION PROCESS

COMPANY	LOCATION	CAPACITY	DATE	ROUTE
Fisons Fertilizers	England	100,000 tpax7	1959	AN based
Fisons Fertilizers	England	100,000 tpax2	1962	AN based/GAN
Donau Chemie	Austria	75,000 tpa	1964	PK grades
Windmill	Holland	220,000 tpa	1968	AN based
Donau Chemie	Austria	115,000 tpa	1969	PK grades
Grassland	Ireland	35,000 tpa	1969	AS based
Fertiberia	Spain	120,000 tpa	1970	Urea-MAP based
Fosfato Thomas	Uruguay	60,000 tpa	1972	Urea-Superphos based
Quimbrasil	Brazil	150,000 tpa	1974	Urea-Superphos based
Elekiroz	Brazil	70,000 tpa	1975	Urea-Superphos based
Manah	Brazil	150,000 tpa	1976	Urea-Superphos based
Quimur	Uruguay	200,000 tpa	1976	Urea-Superphos based
Fusa	Brazil	210,000 tpa	1977	Urea-Superphos based
Duslo	Czechoslovakia	350,000 tpa	1977	CAN
Elekiroz Do Nordeste	Brazil	70,000 tpa	1977	Urea-Superphos based
Egsas	Turkey	310,000 tpa	1978	Urea-MAP based
Tekfen	Turkey	330,000 tpa	1981	Urea-MAP based
State	Iraq	250,000 tpa	1982	Urea-MAP based
Polimex	Poland	405,000 tpax2	1983	Urea-MAP based

CONTENTS

1. INTRODUCTION

Norsk Hydro Fertilizers (formerly Fisons) were pioneers in the field of granular fertilizer production in the UK during the 1930's and subsequently with the introduction of rotary drum granulation.

In common with most other major European nations the UK fertilizer industry developed utilising ammonium nitrate as the major nitrogenous intermediate in the manufacture of compound fertilizers. However for many parts of the world urea has become the principle source of fertilizer nitrogen. The erection of large ammonia and urea factory complexes, frequently close to the source of the feedstocks, has created a situation whereby this material has offered considerable attraction as an easily handleable, safe and cheap form of nitrogen for straight application. Agriculture in emergent areas of the world is based on the use of urea as the principle nutrient nitrogen source. As a result the manufacturing capacity for this material is now several times higher than that for ammonium nitrate. Urea is therefore an important material both as a straight fertilizer and increasingly so in combination with other intermediates for the manufacture of complete balanced fertilizers.

In the 1960's Fisons started a programme of work which enhanced the understanding and quantification of the urea-superphosphate (USP) system. In this work, the effect of temperature on urea hydrolysis and loss of water soluble P_2O_5, with recommendations for the operation of the drying stage and product storage conditions were clearly stated. The stickiness of the USP system in the granulation and drying stages was already known at this time, as was the importance of free acid neutralisation in minimising this effect.

Subsequently, the main effort of development and exploitation has been geared to the urea-ammonium phosphate (UAP) system, since fertilizers based on this form of phosphate have a very high nutrient content and allow the maximum saving in costs of storage, bagging, distribution and application.

Since its formation in 1965 Norsk Hydro's UK based process licensing department has designed and licensed over thirty successful granulation plants of which twelve have utilised urea as the principle nitrogen source.

2. PROCESS DESCRIPTION

This process description should be read in conjunction with the process flowsheet which is shown overleaf.

Solid raw materials together with fines are metered to the rotary granulator where ammonia, sulphuric acid and steam can be added, as appropriate, to achieve optimum conditions.

The granulated material falls directly down a chute into the rotary drier. Here the material is contacted with a hot air-stream to reduce the moisture content of the solids for good storage properties. Drier air is heated by a direct fired gas or oil burner.

From the drier the material is elevated to the screening section. These screens are single deck machines where undersized material is separated out for recycling to the granulator via the fines hopper.

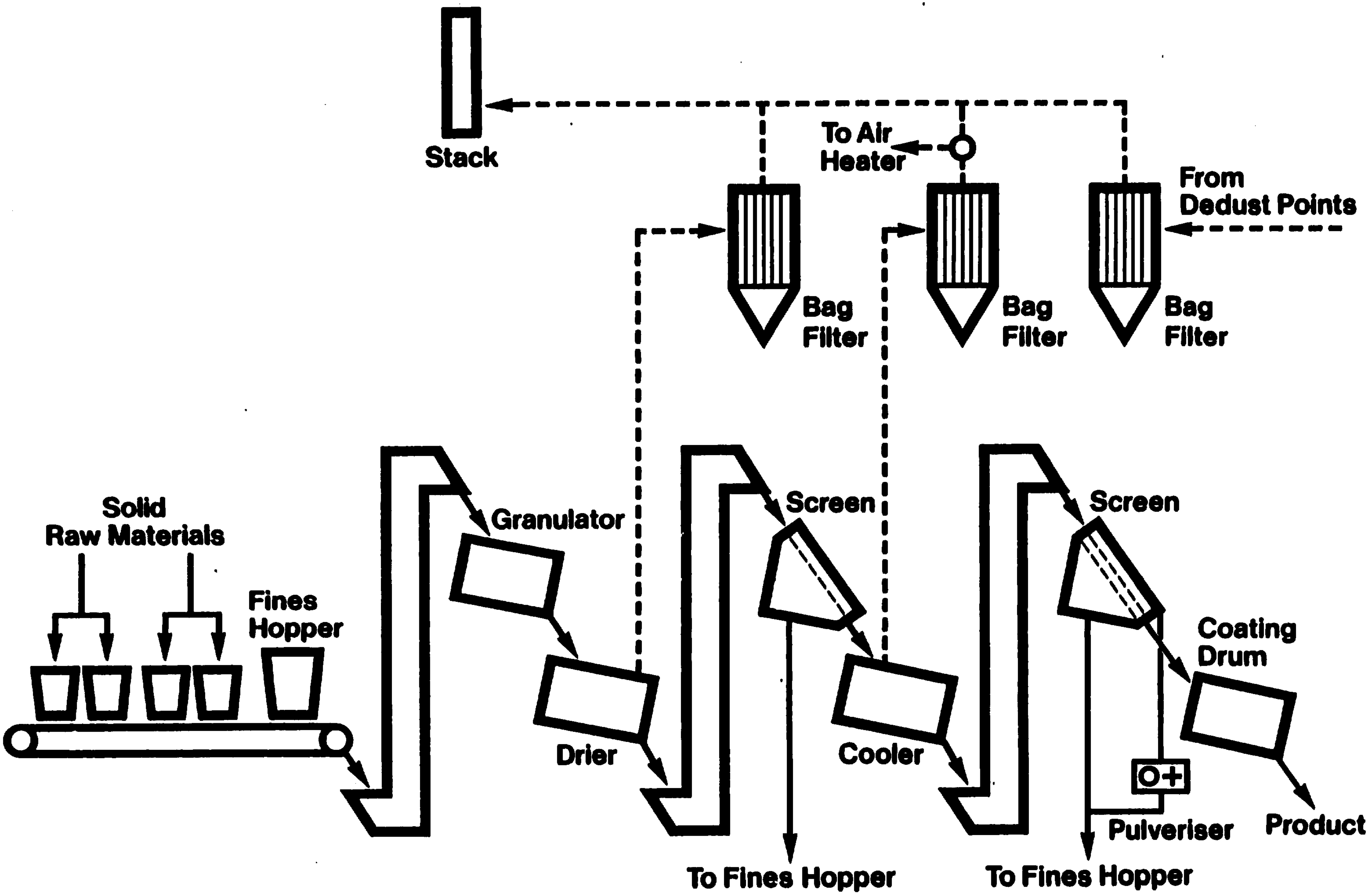
The combined product and oversize stream from the screens passes to the cooler. This can either be a counter-current rotary drum or a fluidised bed unit. The solids temperature is reduced to a satisfactory value for product storage/bagging and trouble-free oversize crushing.

From the cooler the material is elevated to a second screening section. These screens are double deck machines. On the upper cloth the oversized material is removed and crushed before passing to the fines hopper which is located directly beneath the screens. On the lower cloth the product is separated from any residual fines which also pass to the fines hopper. Recycled material from the fines hopper is metered to the granulator at a constant rate.

The product from the screens is coated with dust and oil in a small rotary drum to ensure good storage properties before passing to store.

The airstreams from both the drier and cooler contain dust which is recovered by bag filters. Air is also extracted from several transfer points to provide a clean working environment. The dust is also recovered from this airstream in a centralised bag filter.

Norsk Hydro Licensing Urea Based Granulation Process



3. TECHNICAL FEATURES

The Norsk Hydro Licensing process for the manufacture of urea based NPK fertilizer is similar to the conventional rotary drum granulation processes operating throughout the world. However, the NHL process has several features which provide significant advantages over competing processes.

3.1 Solids based Granulation

The NHL process utilises all the raw materials in the solid form. Powder MAP can either be imported or manufactured on site using the NHL 'Minifos' process. The 'Minifos' process is a simple, low capital cost operation which does not require an ammonia scrubbing step. No additional operators over those necessary for the granulation plant are required.

The use of all solid feeds means that the recycle ratio, and consequently the overall size of the plant, is purely dependent on the efficiency of the granulation rather than being determined by a water balance as with a slurry based process. In practice this means a recycle ratio of between 1.5 and 2/1 compared with at least 4/1 for the slurry route. This low recycle ratio means all the equipment in the granulation loop is small resulting in very low capital and operating costs.

Solids based granulation is a very simple operation; the reaction step ('Minifos' production) is carried out independently from the granulation step and no ammonia recovery step is required for either the reaction or granulation stages. The result is a high on-stream time and an operation that can be accomplished easily by unskilled operators.

3.2 Recycle Control

The production rate and therefore the return of off-specification material from the screens is dependent upon the efficiency of the granulation. For this reason, and because the recycle rate is so low, it is necessary to continuously meter and control the rate of return of recycle fines to the granulator. This is obtained by the use of a fines hopper with an infinitely variable speed extractor and weighbelt. The hopper has a twenty minutes buffer storage capacity to smooth out natural variations in granulation efficiency.

3.3 Raw Material and Product Flexibility

The process can handle almost all the commonly used fertilizer raw materials in any reasonable combination. The only real exception to this is ammonium nitrate which cannot be used in combination with urea.

The following points should be noted however:-

a) The addition of potash to the system has the effect of reducing the melting point of the mixture necessitating lower temperatures to be used for drying and oversize to be cooled before crushing to prevent pulveriser build-up.

b) The use of superphosphates as the only source of P_2O_5 is possible provided proper control is exercised. Fine pH control is necessary to prevent the free acidity in the superphosphate leading to stickiness and rapid over-granulation. The products produced have a relatively low water solubility caused by urea hydrolysis.

c) Granulation becomes more difficult as the percentage of urea in the formulation increases. NHL have however successfully produced grades with up to 50% urea in the formulation.

d) Excessive amounts of filler also depress granulation although NHL have successfully produced grades with as much as 25% filler in the formulation.

e) For the production of grades based on urea and MAP, heat and ammoniation are necessary to promote good granulation. This is achieved by the addition of small quantities of sulphuric acid and ammonia.

NHL have successfully produced a very wide range of urea based grades with N contents ranging from 5-27%, P_2O_5 from 0-34% and K_2O from 0-30%. The following product nutrient ratios have been produced:-

NP	NPK
1-2.5-0	1-4-5
1-2-0	1-3-2
1-1-0	1-2-3
2-1-0	1-2-1
	1-1-1
	2-1-2
	2-1-1

3.4 Cooling

Compound fertilizers containing both urea and potash are sticky when hot and therefore crushing of this material directly after the drying operation would quickly lead to pulveriser build-up and thus increase the frequency of plant shut-downs for cleaning. For this reason NHL designs cater for oversize as well as product cooling.

3.5 Dust Recovery and Bag Filters

Bag filters are used to recover dust from all the airstreams within the plant thus eliminating the need for wet scrubbing.

The Norsk Hydro group has wide experience with the utilisation of bag filters both on their own production plants as well as on licenced plants. Bag filters have been successfully employed to recover dust from drier, cooler and plant dedusting airstreams.

This operational experience has enabled NHL to incorporate design features to prevent any potential problems arising. By careful attention to the cloth cleaning mechanism, cloth material and start-up and shut-down procedures, the efficiency and simplicity of a bag filter system can be safely used.

The use of bag filters to remove all the dust from the cooler airstream allows it to be utilised as dilution air for the drier airstream. In this way, a further 20% reduction in fuel consumption can be made. This system is used successfully on NHF's own granulation plants in the UK.

4. PERFORMANCE DETAILS

Naturally, performance figures and operating conditions will vary depending on the grades to be produced and the raw materials that are available. The figures given below are typical and are examples of 1-1-0 and 1-1-1 ceiling analysis grades.

4.1 Raw Material Consumptions

		27-27-0	18-18-18
Urea, kg/tonne	:	411	272
Powder MAP, kg/tonne	:	475	314
Potash, kg/tonne	:	-	300
Filler, kg/tonne	:	20	27
Sulphuric acid, kg/tonne	:	40	40
Ammonia, kg/tonne	:	31	21
Coating dust, kg/tonne	:	15	15
Coating oil, kg/tonne	:	4	4

4.2 Utility Consumptions

Steam (3 kg/cm^2g), kg/tonne	:	15	15
Fuel oil (net CV 9480 Kcal/kg) kg/tonne	:	2.5-3.0	2-2.5
Power, kWh/tonne	:	20	20
Process water, m^3/tonne	:	Nil	Nil

4.3 Product Quality

Nitrogen content, %	:	27.0	18.0
Total P_2O_5 content, %	:	27.0	18.0
K_2O content, %	:	Nil	18.0
Moisture, %	:	0.7	0.7
Size range, 1.5-4 mms, %	:	85	85
Bulk density, t/m^3	:	0.9-0.95	0.9-0.95
Hardness, kg/granule	:	2.5-3.0	2.5-3.0

4.4 Typical Operating Conditions

		27-27-0	18-18-18
Granulator			
Temperature	:	75-80	75-80
Water content	:	1.4	1.2
pH (10% solution)	:	6.3	5.9
Recycle ratio	:	2/1	2/1
Drier			
Inlet air temperature	:	120	110
Outlet air temperature	:	85	85
Solids outlet temperature	:	80	80

5. CAPITAL COSTS

Capital costs can vary tremendously depending on the engineering contractor involved, the source of the equipment supply etc. However, with a Western European contractor a typical battery limits cost for a 1,000 tpd NPK plant in 1986 would be:-

$$\$11 \times 10^6.$$

This figure is exclusive of the building and off-sites.

6. REFERENCE LIST

UREA BASED COMPOUND FERTILIZER GRANULATION

COMPANY	LOCATION	CAPACITY	GRADES	DATE	FORMULATIONS
Fertilantes de Iberia SA	Huelva, Spain	120,000 tpa	8-24-16	1970	Urea/MAP/KCl/ Filler
			9-27-18		Urea/MAP/KCl/ Filler
			9-18-27		Urea/MAP/KCl/ Filler
			12-24-12		Urea/MAP/KCl/ Filler
			15-15-15		Urea/MAP/KCl/ Filler
			18-18-18		Urea/MAP/KCl
			21-10-10		Urea/MAP/K_2SO_4
Fosfato Thomas SA	Montevideo, Uruguay	60,000 tpa	15-15-15	1972	Urea/SSP/DAP/ Phos Rock/KCl
Industrias Quimica Brasileira	Ponta Grossa, Brazil	150,000 tpa	18-9-18	1974	Urea/SSP/MAP/ KCl
Productos Qimicas Elekiroz SA	Izvarzea, Brazil	70,000 tpa	12-6-12	1975	Urea/AS/SSP/ KCl
Compania Quimica Uruguaya SA	Montevideo, Uruguay	200,000 tpa	15-15-15	1976	Urea/SSP/DAP/ KCl
			13-31-0		Urea/SSP/DAP
Productos Qimicas Elekiroz do Nordeste	Recife, Brazil	70,000 tpa	5-20-25	1977	Urea/MAP/ Phos Rock/KCl
			12-6-24		Urea/AS/DAP/ KCl
			14-8-11		Urea/MAP/ Phos Rock/KCl
			20-10-20		Urea/DAP/ Phos Rock/KCl
Ege Gubre Sanayii SA	Foca, Turkey	310,000 tpa	20-20-0	1978	Urea/MAP/ Filler
			15-15-15		Urea/MAP/ KCl/Filler

COMPANY	LOCATION	CAPACITY	GRADES	DATE	FORMULATIONS
Hemijska Industrija Veles	Titov Veles, Yugoslavia	200,000 tpa	8-16-24	1980	Urea/MAP/KCl/Filler
			8-24-16		Urea/MAP/KCl/Filler
			10-20-30		Urea/MAP/KCl/Filler
			10-30-20		Urea/MAP/KCl
			14-14-14		Urea/MAP/KCl/Filler
			15-15-12		Urea/MAP/K_2SO_4/Filler
			15-15-15		Urea/MAP/KCl/Filler
			16-16-16		Urea/MAP/KCl/Filler
			16-32-0		Urea/MAP/Filler
			25-0-0		Urea/AS/Filler
Toros Fertilizer & Chemical Industries	Ceyhan, Turkey	330,000 tpa	14-23-14	1981	Urea/AS/MAP/KCl
			15-15-15		Urea/MAP/KCl/Filler
			20-20-0		Urea/MAP/Filler
			23-23-0		Urea/MAP/Filler
			24-24-0		Urea/MAP/Filler
			26-13-0		Urea/MAP/Filler
Industrija Nafta	Kutina, Yugoslavia	472,000 tpa	18-18-18	1983	Urea/MAP/KCl
State Organisation of Minerals	Alkaim, Iraq	250,000 tpa	18-18-18	1983	Urea/MAP/KCl
			27-27-0		Urea/MAP
Polimex-Cekop	Police, Poland	405,000 x 2 tpa	17-17-17	1988	Urea/MAP/KCl/Filler

"DISPOSAL OF "GYPSUM" FROM THE HEMIHYDRATE PROCESSES"

by

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1. INTRODUCTION

The hemihydrate route for producing phosphoric acid is becoming increasingly used commercially as a result of its technical and economic benefits. Two variants of the Norsk Hydro process are available, (a) the single-stage HH process discharging calcium sulphate hemihydrate, and (b) the two-stage HDH process which produces the dihydrate form.

Disposal of hemihydrate is a new requirement in the phosphoric acid industry and there may be unnecessary concern that it creates a serious problem. This paper concentrates on disposal of hemihydrate, although reference is also made to disposal of dihydrate from the HDH process.

Descriptions of the two process variants are given with a review of the advantages. Economic data is presented to illustrate the potential savings.

Methods employed for the disposal of hemihydrate and dihydrate from Norsk Hydro licensed plants are listed. More detail is given for the three single-stage hemihydrate plants operating in Europe and North America. An account of some of the operational difficulties overcome is included.

It is concluded that provided the properties of hemihydrate are properly understood and the correct provisions made, disposal of calcium sulphate from hemihydrate processes is no more troublesome than dihydrate. The important benefits of adopting hemihydrate technology are not diminished by "gypsum" disposal considerations.

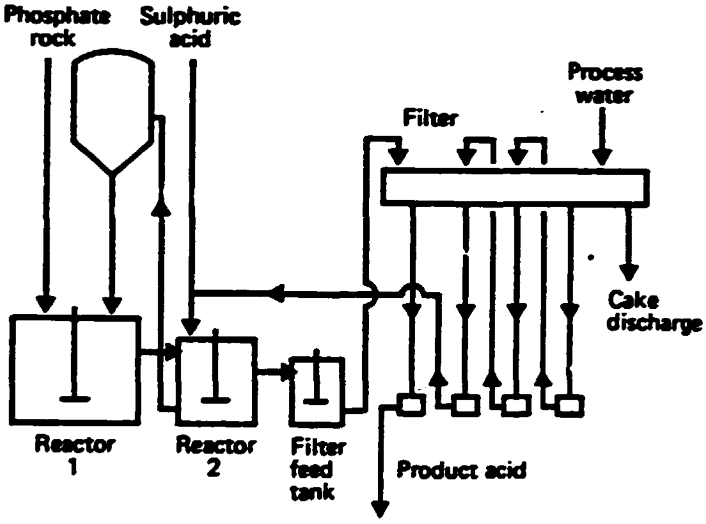
2. NORSK HYDRO HEMIHYDRATE PROCESSES

Two variations of the hemihydrate process have been developed by Norsk Hydro (formerly Fisons Fertilizers) in the United Kingdom. A description of the two processes is given below followed by a review of the main advantages and economic factors.

2.1 Process Description

The major operations involved in the single-stage hemihydrate (HH) process are shown in Figure 1. Reaction and filtration are the key process steps. The hemihydrate reaction proceeds in two distinct zones with calcium sulphate being produced and discharged in the hemihydrate.

Figure 1



Hemihydrate Process

Phosphate rock is fed to reactor 1, sulphuric acid and dilute phosphoric acid from the filter are fed to reactor 2. Slurry from reactor 2 is recycled to reactor 1, thus exposing the phosphate rock to sulphate ions under controlled chemical conditions. Heat is removed by air or vacuum cooling to maintain the reaction slurry temperature at 98-100°C.

Acid can be produced at between 40-52% P_2O_5 depending on downstream requirements.

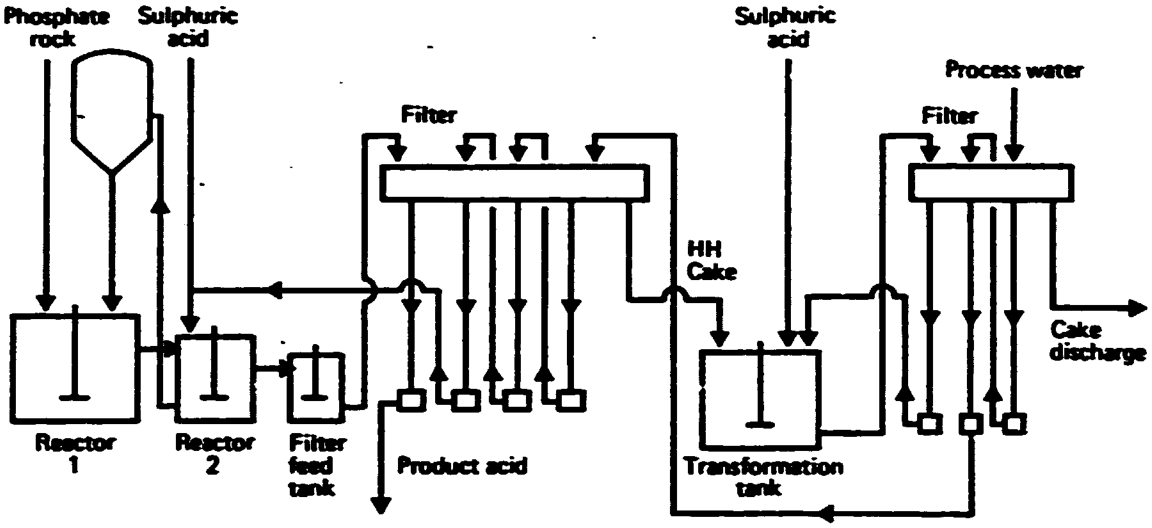
The product acid and hemihydrate are separated by a horizontal vacuum filter with counter current wash stages. The product acid from the filter passes directly to storage. It does not require clarification or solids removal.

The HH process is simple with few unit operations. It is especially suited to mine site operations where phosphate rock is readily available at a relatively low cost.

The two-stage hemidihydrate (HDH) process is a development of the HH route by the addition of a transformation stage allowing hemihydrate to be re-crystallised to dihydrate thus releasing lattice P_2O_5 into solution from which it can be recovered. The P_2O_5 recovery efficiency is increased to around 98.5%, making the HDH process particularly attractive for manufacturing locations remote from the source of phosphate rock. Calcium sulphate is discharged in the dihydrate form.

A simplified flow diagram of the HDH process is shown in Figure 2.

Figure 2



Hemidihydrate Process

The first stage of the process is almost identical to the HH process already described.

In the transformation stage the hemihydrate cake is discharged from the first stage filter into an agitated vessel. The operating conditions are controlled to ensure complete transformation of hemihydrate to dihydrate and to allow sufficient time for the dihydrate crystals to grow. The rate of transformation is increased by the addition of a small feed of sulphuric acid. Nearly all the lattice P_2O_5 co-precipitated with the hemihydrate is released into the liquid phase.

The dihydrate is then filtered and the cake washed with process or pond water. The filtrate containing the released P_2O_5 is returned to the hemihydrate reaction stage as the last wash on the hemihydrate filter.

2.2 Advantages of the Processes

The three main advantages of the hemihydrate processes are energy savings, product acid quality and simplicity of operation.

Strong phosphoric acid is produced directly from the filter of the hemihydrate processes, thereby avoiding the concentration step necessary with the old dihydrate route. Between 1½ and 2 tonnes of steam per tonne P_2O_5 can be saved resulting in fuel savings or the opportunity to generate increased supplies of electricity. This translates into an important economic benefit.

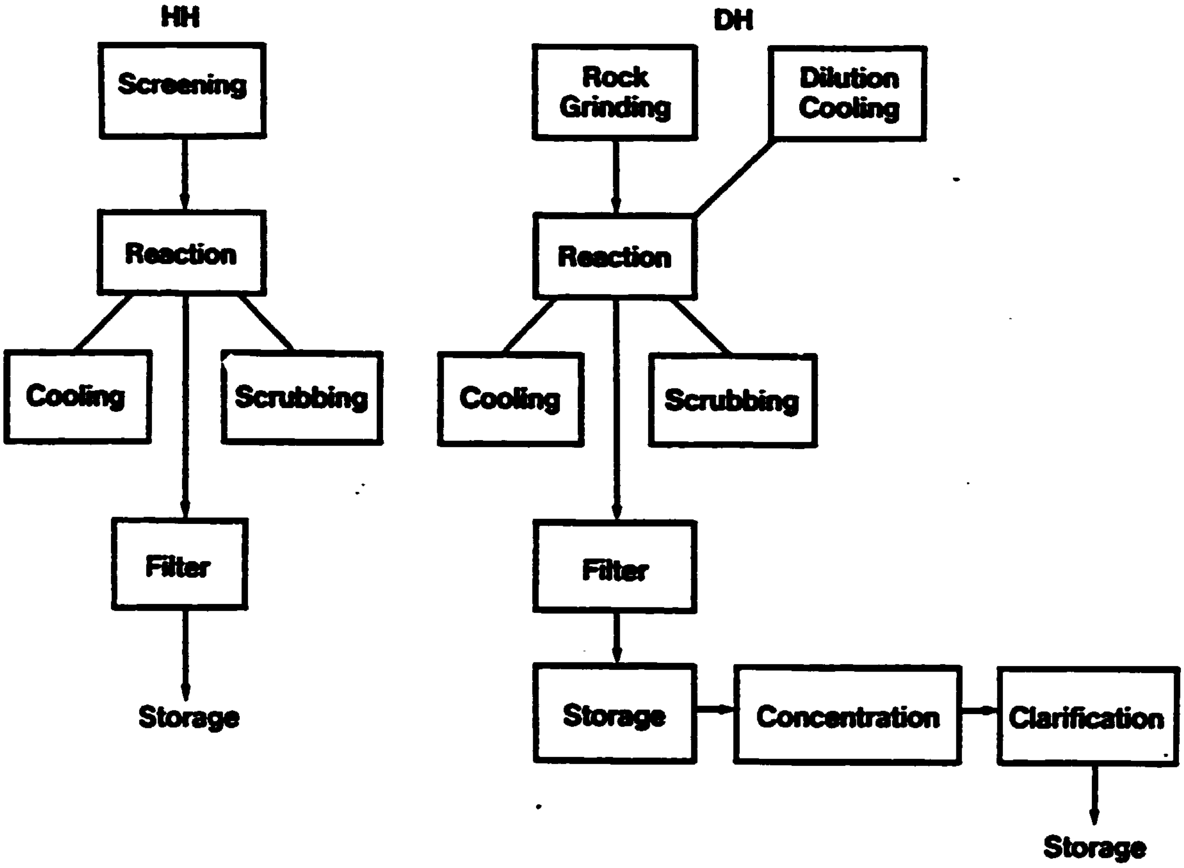
Operating conditions within the hemihydrate reaction system allows a relatively coarse grade of phosphate rock to be processed, sometimes completely avoiding the need for grinding. This results in a valuable power saving and reduction of operating costs.

Hemihydrate acid has a lower aluminium content than dihydrate acid and it exhibits reduced post precipitation. The free sulphate level is only 2% compared with 4-5% in dihydrate acid evaporated to 40-50% P_2O_5. These factors make hemihydrate acid easier and cheaper to handle and enable downstream products to be produced in grade more easily even with low grade rock.

The number of unit operations required in the hemihydrate route is reduced compared with dihydrate. This is illustrated in Figure 3. The avoidance of rock grinding, acid concentration, intermediate storage and clarification reduces capital cost and leads to maintenance cost savings.

Figure 3

Phosphoric Acid Production: Unit Operations



2.3 Economics of the Processes

Table 1 illustrates the operating costs of the single-stage HH process compared with dihydrate. A rock-mine location is assumed. A saving of over US $24 per tonne P_2O_5 is indicated.

Table 1

Typical Raw Material and Utility Costs in US Dollars per tonne P_2O_5, producing 45% P_2O_5 acid from Florida 68% BPL Rock at a Rock-Mine Location

Item	Units	Price (US $)	Hemihydrate		Dihydrate	
			Consumption per tonne P_2O_5	Cost	Consumption per tonne P_2O_5	Cost
Phosphate Rock	tonnes	20.00	3.41	68.20	3.41	68.20
Sulphuric Acid	tonnes	45.00	2.70	121.50	2.80	126.00
Steam	tonnes	11.00	0.18	1.98	1.90	20.90
Electricity	kWh	0.05	100.00	5.00	136.00	6.80
Process Water	m^3	0.02	6.00	0.12	7.00	0.14
Cooling Water	m^3	0.005	27.00	0.14	70.00	0.35
Defoamer	kg	1.50	1.00	1.50	0.70	1.05
Chemical	kg	0.80	1.00	0.80	0.00	0.00
TOTALS				199.24		223.44
SAVING				24.20		

The higher recovery achieved with the HDH process makes it more suitable for a location remote from the source of phosphate. Table 2 shows the operating cost for a Western European or Far Eastern location. In this case the HDH route shows an advantage of over US $35 per tonne P_2O_5.

Production of 45% acid from Florida 68 BPL rock is used in each case. Obviously the calculations can be reproduced for different sets of circumstances, but the savings usually remain significant.

Table 2

Typical Raw Material and Utility Costs in US Dollars per tonne P_2O_5, producing 45% P_2O_5 acid from Florida 68% BPL Rock at a Western European or Far Eastern Location

Item	Units	Price (US $)	Hemidihydrate		Dihydrate	
			Consumption per tonne P_2O_5	Cost	Consumption per tonne P_2O_5	Cost
Phosphate Rock	tonnes	50.00	3.26	163.00	3.41	170.50
Sulphuric Acid	tonnes	45.00	2.60	117.00	2.80	126.00
Steam	tonnes	11.00	0.18	1.98	1.90	20.90
Electricity	kWh	0.05	110.00	5.50	136.00	6.80
Process Water	m^3	0.02	6.50	0.13	7.00	0.14
Cooling Water	m^3	0.005	30.00	0.15	70.00	0.35
Defoamer	kg	1.50	1.00	1.50	0.70	1.05
Chemical	kg	0.80	1.00	0.80	0.00	0.00
TOTALS				290.06		325.74
SAVING				35.68		

2.4 Commercial Plants

Whilst the economic savings illustrated above are impressive in themselves, it is important for the technology to be practical and operable. Since 1970, Norsk Hydro hemihydrate technology has been in continuous commercial operation. The plants licensed and their start-up date is shown in Table 3. At the time of writing, ten plants have been put into production using HH or HDH technology.

Table 3

Norsk Hydro Hemihydrate Technology: Commercial Plants

Client	Country	Start-up	Process
Windmill	Holland	1970	HH
RMHK Trepca	Yugoslavia	1974	HDH
A & W	UK	1980	HDH
CSBP	Australia	1981	HDH
Pivot	Australia	1981	HDH
HCI	Cyprus	1982	HH
Windmill*	Holland	1983	HH
Royster*	USA	1985	HH
Belledune*	Canada	1986	HH
Supra*	Sweden	1986	HDH
NFC$^+$	Thailand	1987	HDH

* Plant conversion from dihydrate unit

\+ Letter of Intent

3. CALCIUM SULPHATE HEMIHYDRATE

3.1 Rocks Tested and Used

Many different phosphate rocks from all parts of the world have been tested in the laboratory units and pilot plants at the Levington Research Station, United Kingdom. The more important rocks having been subject to testing in the hemihydrate mode are listed in Table 4.

Table 4

Norsk Hydro Hemihydrate Technology - Phosphates Tested in Laboratory and Pilot Plant

Phosphate Rock	
Florida - various grades	Vernal, USA
Khouribga - various grades	Wooley Valley, USA
Calcined North Carolina	Phalaborwa
Uncalcined North Carolina	Duchess
Langebaan	Gafsa
Sahara	Calcined Idaho
Araxa, Brazil	Togo
Jordan	Kola
Nauru	China - various grades
Syrian	Nauru
Taiba	Youssoufia

A shorter list of the more commonly available commercial rocks have been utilised in the full-scale plants in operation details of which are given in Table 5.

Table 5

Norsk Hydro Hemihydrate Technology: Commercial Plants

Client	Country	Capacity tpd P_2O_5	Rock Feed
Windmill	Holland	610	Togo
RMHK Trepca	Yugoslavia	160	Jordan
A & W	UK	500	Morocco
CSBP	Australia	500	68 Florida
Pivot	Australia	100	68 Florida
HCI	Cyprus	130	Algeria
Windmill*	Holland	330	72 Florida
Royster*	USA	586	66 Florida
Belledune*	Canada	500	68 Florida
Supra*	Sweden	360	Florida/Morocco/ Jordan
NFC	Thailand	810	Florida/Morocco/ Jordan

* Plant conversion from dihydrate unit

3.2 Chemical Analyses of Phosphate Rocks and Hemihydrates

Chemical analyses for the more important constituents of phosphate rock with the equivalent analyses for the hemihydrate produced are given for several important rocks in Table 6. Data are obtained from full-scale plants and the pilot plants at Levington and are relevant to the production of 48% P_2O_5 acid.

Table 6

Chemical Analyses of Phosphate Rock and Hemihydrate

Component %	Florida 68		Togo		Morocco 72		Algeria		Jordan 72	
	Rock	Hemihydrate	Rock	Hemihydrate	Rock	Hemihydrate	Rock	Hemihydrate	Rock	Hemihydrate
P_2O_5 Total	30.6	2.06	35.6	1.9	31.4	1.68	30.6	1.54	33.7	1.36
CaO	45.8	35.6	50.3	36.6	51.1	38.1	51.2	37.8	52.1	38.5
SO_3	1.08	47.1	0.22	50.4	1.83	51.1	2.63	52.0	0.9	51.2
F	3.3	0.88	3.81	0.78	3.65	0.78	3.72	0.48	3.7	0.89
Al_2O_3	1.16	0.63	1.08	0.68	0.37	0.23	0.46	0.14	0.29	0.16
Fe_2O_3	1.39	0.1	0.57	0.02	0.23	0.03	0.38	0.05	0.21	0.03
MgO	0.59	0.02	0.12	0.002	0.75	0.05	1.11	0.01	0.28	0.03
Cd (ppm)	20	7	57	21	16	6	25	9	10	4
SiO_2	8.76	5.09	4.5	2.24	2.91	0.79	1.97	0.33	3.03	1.13
H_2O Combined	2.38	5.29	0.95	5.29	1.9	5.68	1.72	5.85	1.53	5.76

All analyses are given on a dry basis

In the hemihydrate process the composition of the hemihydrate is dependent not only on the rock analysis but also on the concentration of the phosphoric acid produced.

Unlike the dihydrate process, which operates at acid concentrations between 26-30% P_2O_5, the hemihydrate process is much more flexible, and acid concentrations between 40-50% are quite permissible.

As the acid concentration increases the insoluble P_2O_5 content of the hemihydrate also increasese and changes are more rapid above 45% P_2O_5. Aluminium concentrations also increase with acid concentration and at acid concentrations above 45% P_2O_5 a higher proportion of the aluminium originally fed in the rock is discharged with the hemihydrate compared to calcium sulphate discharged with the dihydrate process.

Impurities such as Fe_2O_3, MgO, Cd and SiO_2 are mainly controlled by rock composition and are similar for both processes.

Fluorine levels are generally lower in the hemihydrate process as more is evolved during the reaction stage arising from the use of a higher temperature and acid concentration. If the fluorine scrubbing liquors are disposed with the calcium sulphate effluent then the overall discharge will be similar.

3.3 Physical Characteristics of the Crystals

Crystals from the hemihydrate process can be produced in a variety of shapes in the same way as in the dihydrate process.

In the dihydrate process the basic crystal shape is the form of flat rhombic plates whereas the hemihydrate process produces crystals which are basically hexagonal needles.

Modification of these crystal shapes occurs in both processes due to the effect of impurities present in the rock. Aluminium and fluorine are two of these impurities which are particularly important. Other impurities, such as the alkali metals and silica, which can affect fluorine concentrations, also have influence.

Hemihydrate crystals can be in the form of single hexagonal needles, needles with different degrees of twinning, through to loose agglomerates and finally to very compact agglomerates. The compact agglomerates are spherical in overall shape.

The crystals are characterised by the use of an air permeability technique which provides a measurement of specific surface area, porosity and permeability.

Typical ranges of these values according to the Levington method of assessment are:

Specific Surface Area (SSA)	400 - 2000
Porosity	0.4 - 0.7
Permeability	10 - 60

For good filterability a high value of permeability is needed. Permeability is affected by specific surface area and, in particular, porosity. Ideally, a high permeability should be accompanied by a low SSA and a medium value for porosity. High permeabilities caused by high porosities are not ideal due to the high moisture contents of the filter cake and a reduction in washing efficiency during filtration. It is also important during disposal of the hemihydrate to a gypsum stack that adequate de-watering can occur before the onset of conversion to dihydrate. A high porosity is a disadvantage from this aspect.

Photographs taken on an electron microscope are given in Figures 4 to 8 and show typical crystals of hemihydrate produced from the five rocks given in Table 6. Data obtained from the air permeability measurement are also given.

3.4 Dihydrate Crystals from HDH Process

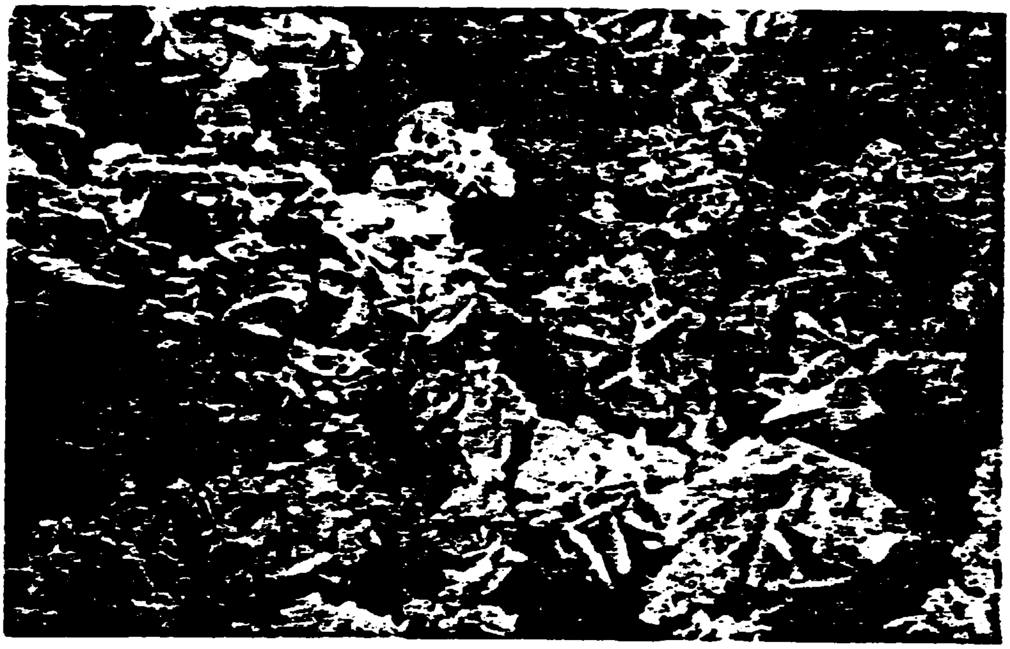
In the transformation stage of the HDH process, crystals of calcium sulphate hemihydrate dissolve and re-crystallise as dihydrate. The shape of the dihydrate crystals produced depends on the impurities present in the transformation liquor, which in turn are controlled by the conditions of the hemihydrate reaction stage and the composition of the rock feed.

Most rocks produce dihydrate from the transformation stage in the form of agglomerates which have good drainage properties.

Figure 4

Hemihydrate from Florida 68 BPL Rock

SSA - 1388; Porosity - 0.636; Permeability - 28

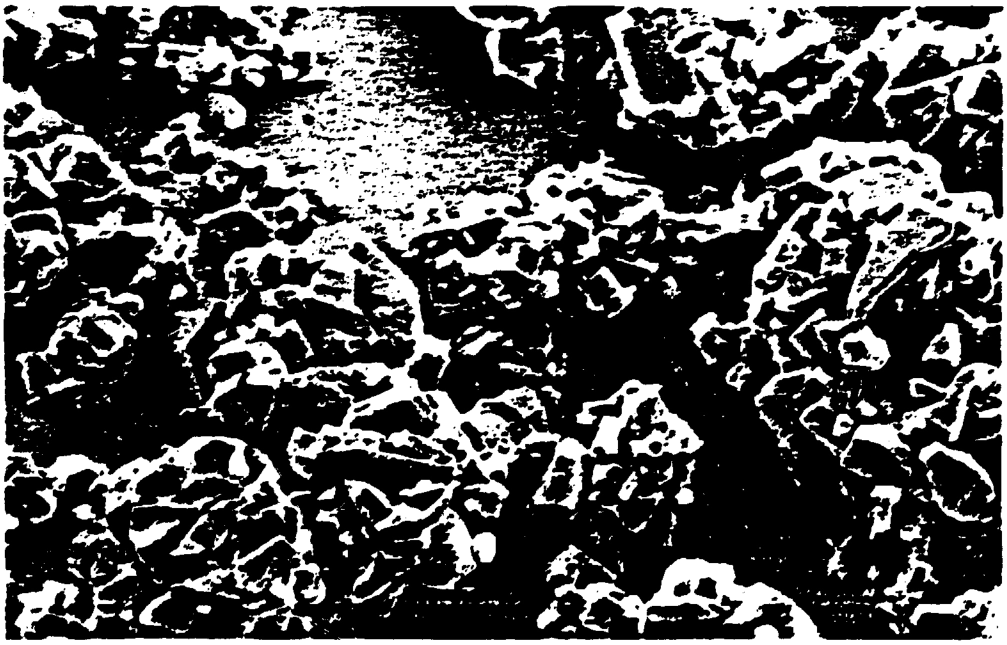


Magnification x 500

Figure 5

Hemihydrate from Togo Rock

SSA - 1263; Porosity - 0.61; Permeability - 25



Magnification x 500

Figure 6

Hemihydrate from Morocco 72 BPL Rock

SSA - 682; Porosity - 0.514; Permeability - 32

Magnification x 500

Figure 7

Hemihydrate from Algerian Rock

SSA - 1174; Porosity - 0.54; Permeability - 14.5

Magnification x 500

Figure 8

Hemihydrate from Jordan 72 BPL Rock

SSA - 1940; Porosity - 0.71; Permeability - 30

Magnification x 500

Typical ranges obtained from the air permeability measurement are as follows:

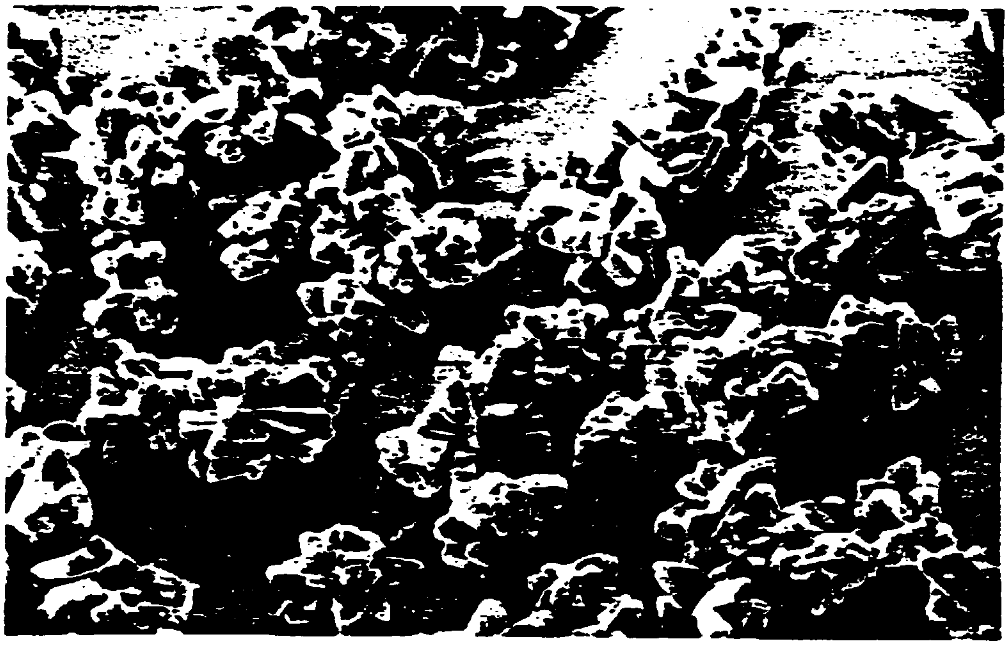
Specific Surface Area (SSA)	400 - 2000
Porosity	0.45 - 0.55
Permeability	20 - 60

Figure 9 shows dihydrate crystals from the HDH process when processing Florida rock.

Figure 9

Dihydrate from HDH Process, Florida Rock

SSA - 1040; Porosity - 0.5; Permeability - 17



Magnification x 500

4. DISPOSAL OF CALCIUM SULPHATE FROM HEMIHYDRATE PLANTS

The chemistry of calcium sulphate hemihydrate differs from dihydrate and has an impact on the disposal method. This section considers the stability of hemihydrate and gives brief details of the main methods of disposal.

4.1 Chemistry

Calcium sulphate hemihydrate is stable at higher P_2O_5 concentrations and temperatures (e.g. 40% P_2O_5, 100°C) whereas dihydrate is the stable form at lower concentrations and temperatures. Thus, whilst calcium sulphate crystallises as hemihydrate in the reaction system of the HH and HDH processes, dihydrate is the stable form elsewhere in the system, for example, at the weaker end of the filtration circuit. This phenomenon is exploited in the HDH process, but must be suppressed in the hemihydrate filtration and disposal system. Norsk Hydro has developed the use of a hydration inhibitor which is injected into the filtration system and has the ability of slowing down the transformation for sufficient time to complete the filtration cycle.

The same approach is relevant to the disposal of hemihydrate cake in slurry form and the inhibitor is successfully added to the slurry pumping system. This delays the hydration process for sufficient time to avoid solids build up in the pumps and piping and also avoids the formation of minute dihydrate crystals at the disposal site.

Calcium sulphate from the HDH process is in the dihydrate form and is therefore stable in the disposal system exactly as from the dihydrate route. No special precautions are required. However, the crystal form of the dihydrate could affect any disposal system in which solids are accumulated.

4.2 Disposal Methods

Calcium sulphate hemihydrate from Norsk Hydro licensed plants is disposed of in a variety of ways. These are briefly described below, more details of three sites being given in Section 5.

(a) To the Sea or River

Hemihydrate slurry is simply slurried with water and pumped to a nearby river or sea where it dissolves and disperses.

(b) To "Gypsum Stack"

Hemihydrate slurry is pumped as a slurry to the disposal area where the solids are deposited and the liquid phase recycled to the phosphoric acid plant. The solids accumulate and the level is progressively built up to create a stack or hill of material.

This is similar in principle to a lagoon.

Additionally, dihydrate from the HDH process is also disposed of as a moist solid. Cake from the dihydrate filter is discharged to lorries which transport the material for disposal at a remote land-fill site.

Table 7

Methods of Disposal from Norsk Hydro Licensed Plants

Client	Process	Method of Disposal
Windmill (2 Plants)	HH	Slurry to River
RMHK Trepca	HDH	Slurry to Lagoon
A & W	HDH	Slurry to Sea
CSBP	HDH	Slurry to Lagoon
Pivot	HDH	Solid
HCI	HH	Slurry to Sea
Royster	HH	Slurry to "Stack"
Belledune	HH	Slurry to Sea
Supra	HDH	Slurry to "Island"
NFC	HDH	Slurry to "Stack"

5. DISPOSAL TECHNIQUES

More detailed descriptions of the hemihydrate disposal methods are given in this Section.

Particular reference is made to operational aspects which have required resolution.

5.1 Disposal to River/Sea

This technique will be illustrated by reference to the Windmill operation, with some comparative data from Belledune.

Windmill operates two hemihydrate units each with its separate but similar system.

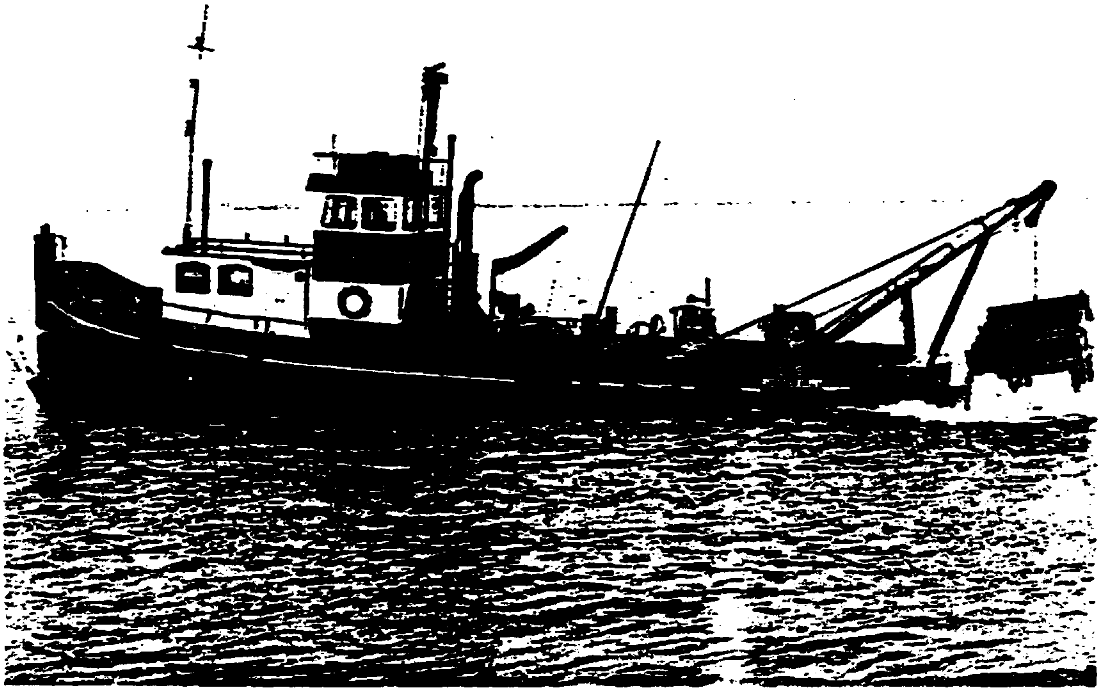
Hemihydrate cake is discharged from the filter and flushed with river water to an agitated slurrying tank. Metered quantities of the hydration inhibitor are added to the slurrying tank. The slurry is then pumped continuously through a polyethylene pipeline over land to the river. The discharge is below the water level at all states of the tide, angled downwards by 20 degrees and at right angles to the flow of the river.

The river adjacent to the Windmill plant is a major shipping route as well as being close to a densely populated area. Consequently the Authorities pay close attention to the Windmill discharge.

Deposition of only some of the heaviest solids (SiO_2) is detected in the vicinity of the slurry discharge point. To avoid accumulation, the deposit is disturbed twice per week by a plough-like device towed by a small ship. See Figure 10.

Figure 10

Ship with Plough at Windmill



There has never been any difficulty with scaling or plugging of the pipelines. This is ensured by flushing the lines with fresh river water after any plant shutdown and before stopping the hemihydrate pumping system. The addition of the hydration inhibitor is considered essential.

The Belledune plant is close to the Gulf of St Lawrence - in effect the North Atlantic. A similar set-up is employed but the discharge is above the water level. Although operating experience is limited at the time of writing, there has been only a small amount of build up at the discharge point which is not troublesome although being monitored. There has been no plugging of the pipeline.

Mechanical details of the systems are given in Table 8 below.

Table 8

Mechanical Details of Windmill and Belledune Hemihydrate Disposal Systems

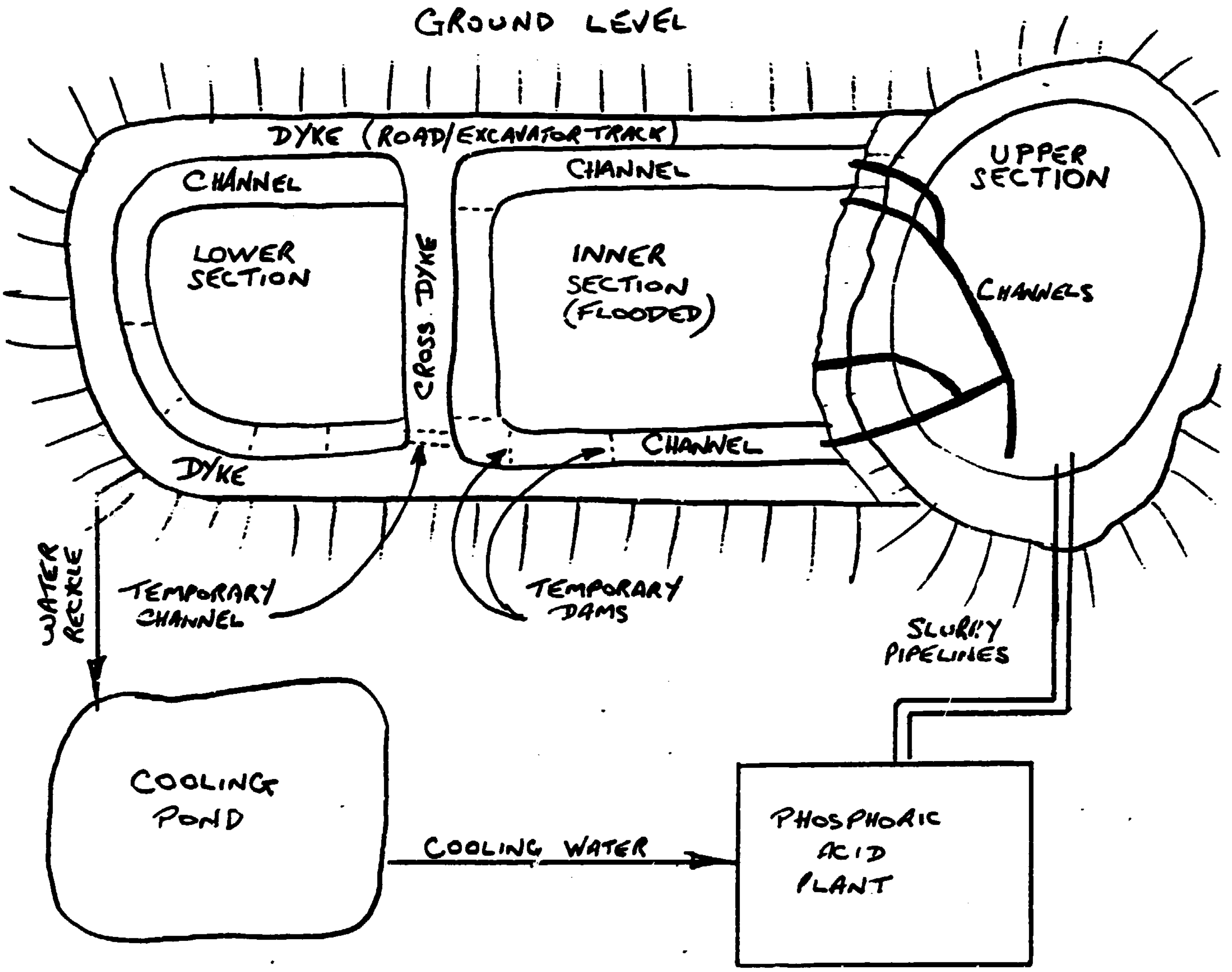
Item	Windmill (2 Systems)	Belledune
Length of pipeline, (m)	1,000	900
Diameter of pipeline, (mm)	200	560
Projection into river/sea, (m)	5	3
Discharge above/below water level, (m)	0.3-3 below	Above
Material of pipeline	Polyethylene	HDPP
Pumping stages	One	One
Velocity, m/s	1.8	0.8
Slurry solids content, (%)	20 - 40	5

5.2 Disposal to "Gypsum Stack"

The plant discharging hemihydrate to a stack is located at Mulberry, Florida, USA, and operated by the Royster Company. Each gypsum stack is unique and its management is specialised, details being beyond the scope of this paper. However, a short description of the Royster system is given below and illustrated by a sketch plan in Figure 11 and photograph in Figure 12.

Figure 11

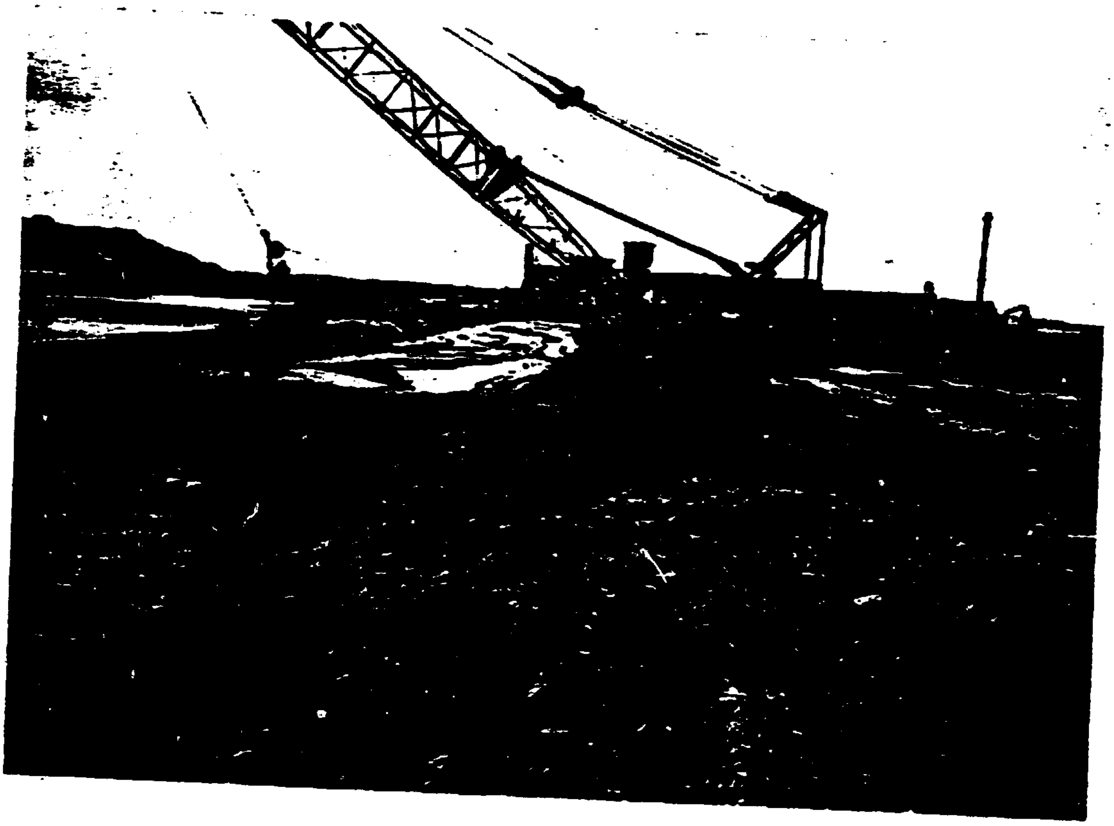
ROYSTER "GYPSUM"STACK - PLAN VIEW



The stack, located immediately adjacent to the phosphate factory and the water cooling pond, is of an elongated shape with a higher section at one end occupying about 25% of the total area. The upper and lower sections are surrounded by gypsum banks, or dykes, which contain the liquid covering a majority of the surface area. The lower section is divided into two by a further gypsum dyke.

Figure 12

Excavation of the Hemihydrate at Royster



The method of operation is for slurry to be fed to the higher section from which it flows down channels to the lower section. Initially a channel is excavated near the edge of the lower section, parallel to each dyke, eventually leading to the centre. Slurry is first fed to one channel down which it slowly flows. Solid calcium sulphate deposits in the channel gradually filling it, whilst clear liquor flows to and collects in the central area, eventually draining to the cooling pond. The stack serves the dual purpose of decanting and cooling (by evaporation) of the liquid phase.

When the first channel is full of solid, the flow is diverted to the other and solids removed by drag line to build up the dyke (which also acts as an excavator track and access road). The process is repeated for each channel alternately until the dyke has been built up to sufficient height to create a rim. The slurry is then fed to the central area until it is full, then the whole process is repeated. In this way the stack gradually grows, with steeply sloped sides.

Hemihydrate is pumped as a slurry using re-cycled pond water through one of two 700 polyethylene pipelines with diameters of approximately 250 mm up the wall of the stack to the upper zone. There is one pumping station at the phosphoric acid plant with a booster part-way up the stack.

In the initial stages of operation two problems were encountered. Firstly, there was a build-up of scale on the inner walls of the pipeline, and secondly, the slurry was very slow to de-water on the stack.

Scale build-up in the pipe was removed by physically flexing the pipe and dislodging the hard solid. This was quick and effective but tended to block the intermediate pump with large lumps. Eventually scaling was largely avoided by the remedy employed to overcome the de-watering problem.

The slowness of the slurry to de-water on the stack caused the channels to fill with a high water content thixotropic mass. When excavated to create a higher dyke and road surface the load-bearing characteristics were unacceptable. Conditions deteriorated to the point where there was a risk of closing the plant down.

Close investigation indicated that calcium sulphate hemihydrate was hydrating within the residence time of the pipe and re-precipitating as tiny dihydrate crystals, much smaller than experienced from the original dihydrate plant. The fine crystals obviously were slower to sediment from the slurry and retained a high percentage of liquid phade between the crystals.

The cause of the rapid hydration was the use of a less expensive, but inferior, inhibitor, compared with that found effective elsewhere. Correction to the alternative material caused an immediate improvement and the de-watering characteristics resembled those of gypsum from the original plant.

Calcium sulphate now arrives at the stack in the hemihydrate form, in large crystals grown in the reaction system to provide rapid filtration. Hydration on the stack is delayed for a period long enough to achieve de-watering of the large hemihydrate crystals and conversion to dihydrate occurs slowly after the excavation stage has been completed.

Use of the preferred inhibitor has largely eliminated any scaling problems in the piping. However, the usual practice is to operate on one line with the other on wash, alternating weekly.

6. CONCLUSION

The HH process forms large crystals of calcium sulphate hemihydrate which, whilst stable in the reaction system, tend to convert to dihydrate in the disposal system. Use of a hydration inhibitor delays conversion sufficiently to allow trouble-free disposal.

Five commercial plants have successfully demonstrated the feasibility for disposal of hemihydrate, the first having been in operation for over 15 years.

Dihydrate from the HDH process is stable and similar to that from the old dihydrate route for disposal.

It is concluded that disposal of calcium sulphate from Norsk Hydro hemihydrate plants can be achieved without difficulty and is comparable with the conventional process.

ANNEX 4

HEAT AND MASS BALANCES PROVIDED IN LECTURES

HH HEAT AND MASS BALANCE FOR 68FLORIDA

CONFIDENTIAL

% DILUTION OF HH FILTER = 1.0

MATERIAL IN			HEAT IN	
MATERIAL	% W/W	KG/HOUR	DEG C	MJ/HOUR
		SULPHURIC ACID		
S03	80	45050		
H20(C)	17.99	10136		
H20(F)	1.99	1126		
OTHERS	0	0		
TOTAL		56312	25	-2589
		PHOSPHATE ROCK		
P205	31	22163		
CAO	45	32172		
S03	.99	714		
SI02	8.49	6076		
F	3.69	2645		
CL	.01	8		
NA20	.49	357		
K20	.09	71		
MGO	.39	285		
FE203	1.49	1072		
AL203	1.29	929		
C02	3.49	2502		
H20(C)	.99	714		
H20(F)	1.49	1072		
OTHERS	2.55	1829		
CORRECTION	-1.57	-1116		
TOTAL		71493	40	1043
		REACTOR VENTILATION AIR		
AIR	99.01	8660		
H20	.98	86		
TOTAL		8746	25	168
		HEAT OF REACTION		
				47847
		AGITATOR POWER		
				1044
		FILTER CAKE WASH WATER		
H20		57552	55	7229
		AIR TO VACUUM SYSTEM		
AIR	99.04	1139		
H20	.95	11		
TOTAL		1150	25	22
		FILTER DILUTION WATER		
H20		1153	30	24

MATERIAL OUT			HEAT OUT	
MATERIAL	% W/W	KG/HOUR	DEG C	MJ/HOUR
		FLASH COOLER GASES		
P205	0	0		
SI02	4.02	501		
F	6.37	793		
CL	.02	3		
C02	4.01	500		
H20	88.22	10977		
OTHERS	.02	3		
CORRECTION	-2.7	-335		
TOTAL		12442	95	29327
		REACTOR VENTILATION GASES		
SI02	1.69	208		
F	2.15	264		
C02	16.35	2001		
H20	9.9	1212		
AIR	70.76	8660		
OTHERS	.03	4		
CORRECTION	-.92	-112		
TOTAL		12237	70	3541
		REACTOR HEAT LOSS		
				792
		PRODUCT ACID		
P205	41.99	20833		
CAO	.13	69		
S03	1.53	763		
SI02	.47	238		
F	1.16	580		
CL	0	3		
NA20	.08	44		
K20	.03	19		
MGO	.49	248		
FE203	1.84	917		
AL203	1.26	625		
H20(C+F)	48.06	23844		
OTHERS	3.35	1666		
CORRECTION	-.5	-246		
TOTAL		49603	90	7576
		HEMIHYDRATE CAKE(DRY BASIS)		
P205	.97	886		
CAO	35.31	32102		
S03	49.49	45001		
SI02	5.64	5128		
F	1.1	1006		
NA20	.34	312		
K20	.05	51		
MGO	.04	37		
FE203	.16	154		
AL203	.33	304		
H20(C)	6.8	6191		
OTHERS	.18	169		
CORRECTION	-.47	-424		
TOTAL		90914	67	3248
		ACID IN HEMIHYDRATE CAKE		
P205	1.59	443		
H20	98.4	27275		
TOTAL		27718	67	4932
		FILTER VACUUM HEAT LOSS		
AIR	36.36	1139		
H20	63.63	1993		
TOTAL		3132	60	5030
		FILTER SENSIBLE HEAT LOSS		
				270
		FILTER RADIATION HEAT LOSS		
				171
		FILTER EVAPORATION HEAT LOSS		
H20		360		901

TOTAL IN 196406 54788

TOTAL OUT 196406 54788

HDH HEAT AND MASS BALANCE 155
FOR
68BPL FL [redacted] CONFIDENTIAL

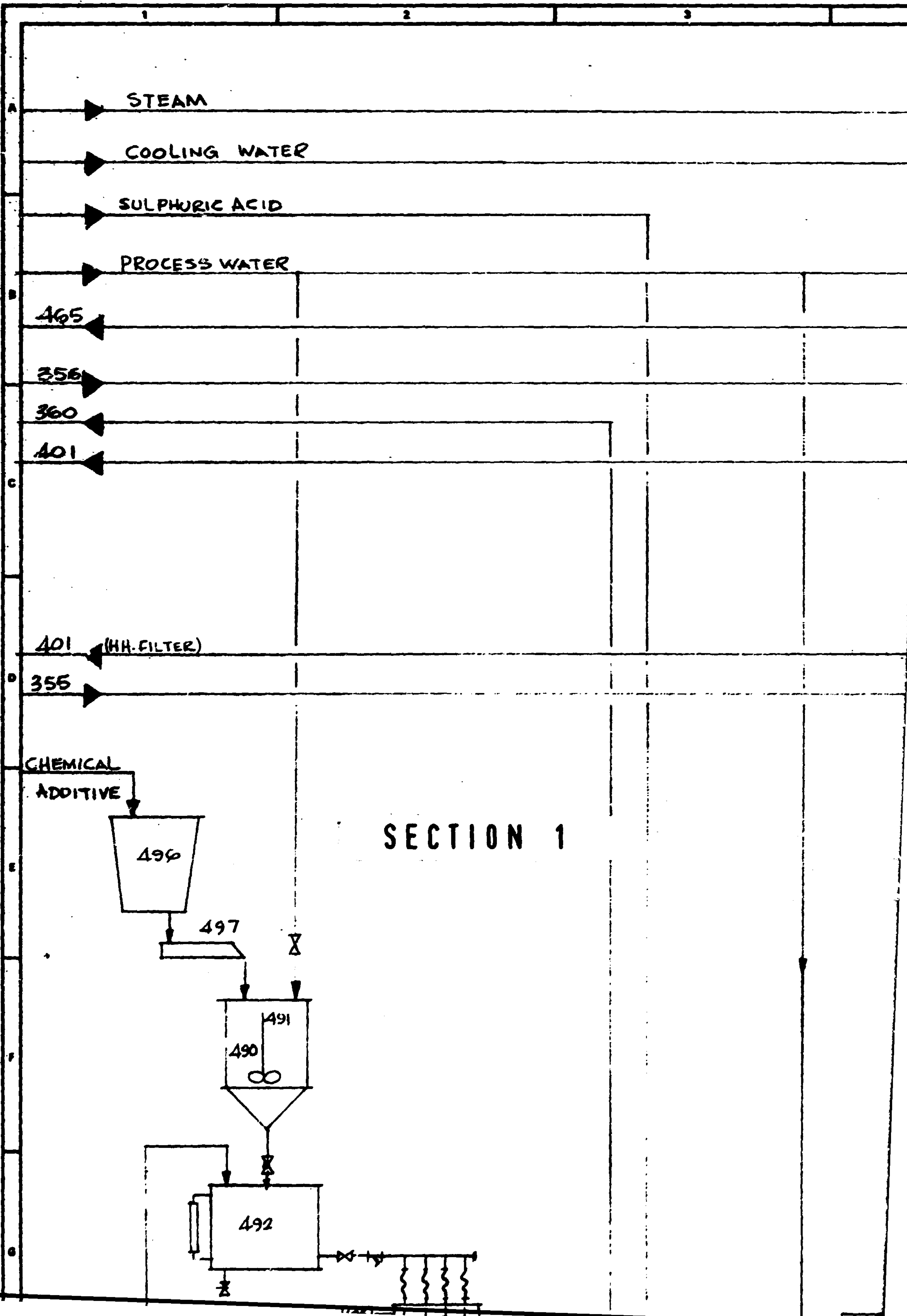
% DILUTION OF HH FILTER = 1

```
        MATERIAL  IN           HEAT  IN
MATERIAL  % W/W  KG/HOUR  DEG C  MJ/HOUR
              SULPHURIC ACID
SO3        80     72243
H2O(C)     17.99  16254
H2O(F)     2.01   1806
OTHERS     0      0
TOTAL             90303    25      -4151


              PHOSPHATE ROCK
P2O5       30.88  38071
CAO        45.54  56130
SO3        .98    1220
SIO2       8.41   10371
 F         3.46   4270
 CL        .01    17
NA2O       .39    488
K2O        .09    122
MGO        .49    610
FE2O3      1.38   1708
AL2O3      1.28   1586
CO2        3.76   4636
H2O(C)     1.97   2440
H2O(F)     .99    1232
OTHERS     1.74   2150
CORRECTION-1.47   -1802
TOTAL             123249   40      1768

         REACTOR VENTILATION AIR
AIR        99.01  40716
H2O        .989998  407
TOTAL             41123    30      994

           AIR TO VACUUM SYSTEM
AIR        99.06  1898
H2O        .940002  18
TOTAL             1916     30      46

          FILTER DILUTION WATER
H2O               2076     30      43

             HEAT OF REACTION
                                   80385

              AGITATOR POWER
                                   2880

              SULPHURIC ACID
SO3        80     7820
H2O(C)     17.99  1759
H2O(F)     2.01   195

OTHERS     0      0
TOTAL             9774     25      -450

         HEAT OF REACTION/TRANS.
                                   27304

              AGITATOR POWER
                                   1440

          FILTER CAKE WASH WATER
H2O               146496   40      9199

           AIR TO VACUUM SYSTEM
AIR        99.04  2479
H2O        .959999  24
TOTAL             2503     30      60
                  ADDITIVE
SIO2       90     0
H2O        0      0
OTHERS     0      0
TOTAL             0        40      0

TOTAL IN 417440   119518
```

```
        MATERIAL  OUT          HEAT  OUT
MATERIAL  % W/W  KG/HOUR  DEG C  MJ/HOUR
            FLASH COOLER GASES
P2O5       0      0
SIO2       4.2    809
F          6.66   1281
CL         .03    7
CO2        4.82   927
H2O        87.07  16733
OTHERS     0      0
CORRECTION-2.82   -540
TOTAL             19217    95      43197

         REACTOR VENTILATION GASES
SIO2       .66    337
F          .84    427
CO2        7.31   3709
H2O        11.24  5700
AIR        80.29  40716
OTHERS     0      2
CORRECTION-.35    -180
TOTAL             50711    70      16366

            SENSIBLE HEAT LOSS
SENSIBLE                           450
RADIANT                            285
TOTAL                              735

            REACTOR HEAT LOSS
                                   1131


            FILTER VACUUM LOSS
AIR        36.36  1898
H2O        63.64  3322
TOTAL             5220     60      8384

         FILTER EVAPORATION LOSS
H2O               600              1501

         FILTER CLOTH SPRAY LOSS
H2O               4788             11947

               PRODUCT ACID
P2O5       42     37500
CAO        .19    178
SO3        1.66   1491
SIO2       .04    44
 F         .89    803
 CL        0      8
NA2O       .03    35
K2O        .01    17
MGO        .64    580
FE2O3      1.85   1660
AL2O3      .64    580
H2O(C+F)   52.33  46731


OTHERS     -.01   1
CORRECTION-.39    -341
TOTAL             89285    90      13638

               COOLING LOSS
H2O               3950             10310

            REACTOR HEAT LOSS
                                   157

        DIHYDRATE CAKE(DRY BASIS)
P2O5       .11    219
CAO        30.19  55951
SO3        42.91  79500
SIO2       4.95   9180
 F         .94    1758
NA2O       .24    452
K2O        .05    104
MGO        .01    29
FE2O3      .02    47
AL2O3      .54    1005
H2O(C)     19.3   35775
OTHERS     1.07   1991
CORRECTION-.4     -741
TOTAL             185270   45      4260

          ACID IN DIHYDRATE CAKE
P2O5       .64    352
SO3        .53    293
H2O        98.52  53955
OTHERS     .3     165
TOTAL             54765    45      4586

            FILTER VACUUM LOSS
AIR        68.21  2479
H2O        31.79  1155
TOTAL             3634     54      2954

          RADIANT AND SENSIBLE
                                   352

TOTAL OUT    417440   119518
```

ANNEX 5

P&I DIAGRAMS PROVIDED IN LECTURES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A
B
C
D
E
F
G
STEAM
COOLING WATER
SULPHURIC ACID
PROCESS WATER
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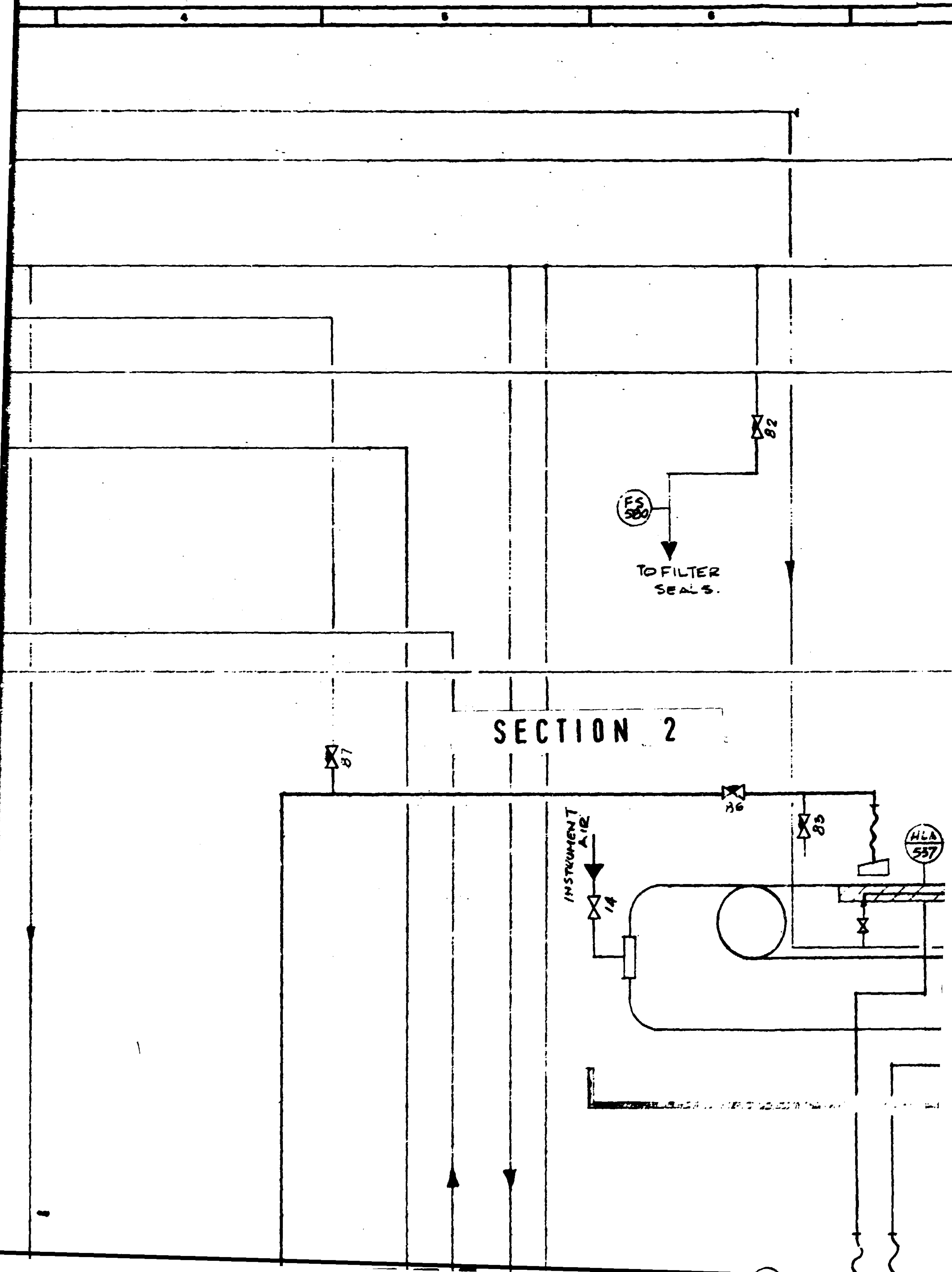
356
360
401
401 (HH. FILTER)
355
CHEMICAL ADDITIVE
SECTION 1
496
497
491
490
492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5
6
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FS 580
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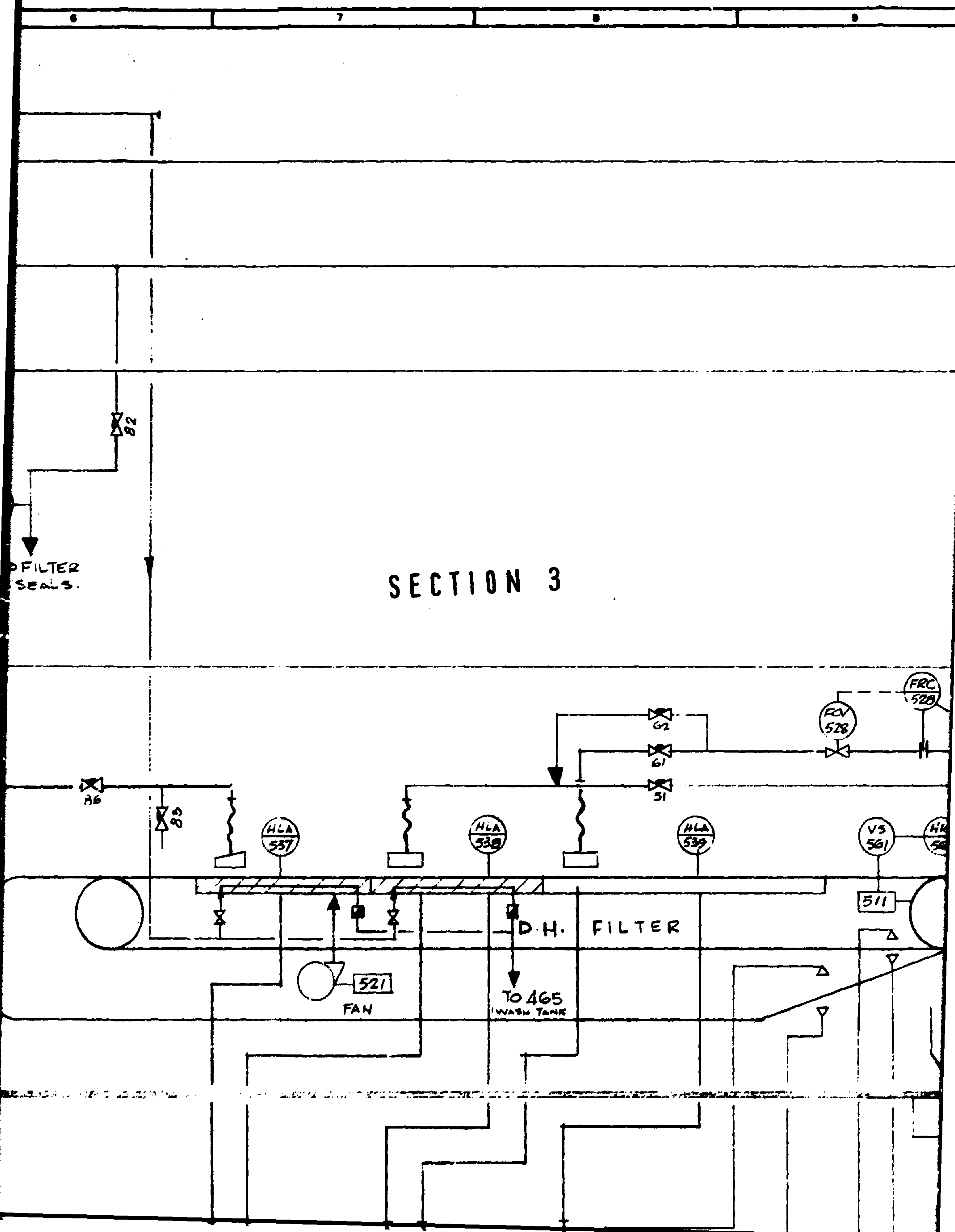
SECTION 2
87
86
83
INSTRUMENT AIR
14
HLA 537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7
8
9
82
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SEALS.
SECTION 3
FRC
528
FCV
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HLA
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HLA
538
HLA
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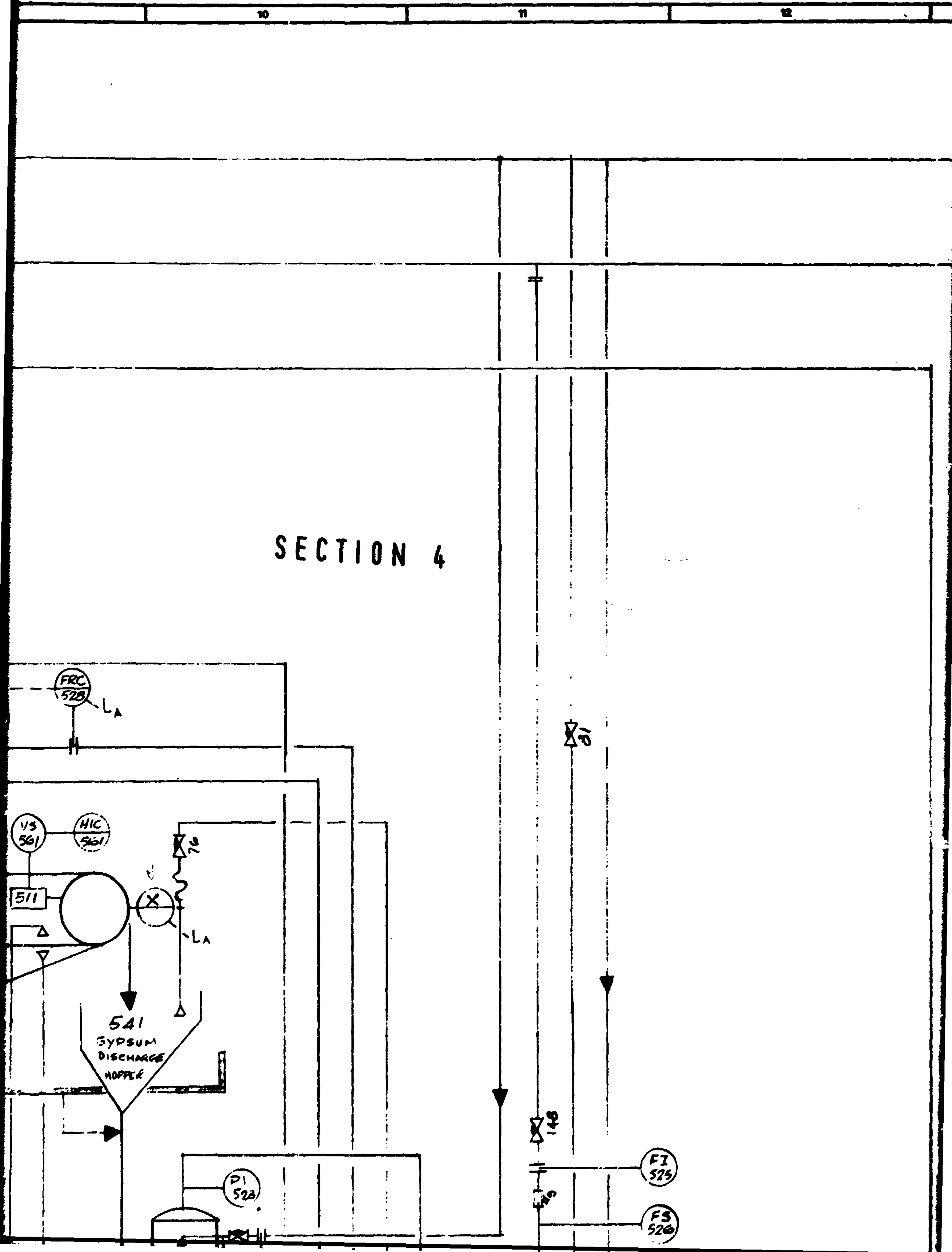
VS
561
511
D.H. FILTER
521
FAN
TO 465
(WASH TANK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11
12
SECTION 4
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VS 561
HIC 561
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LA
541
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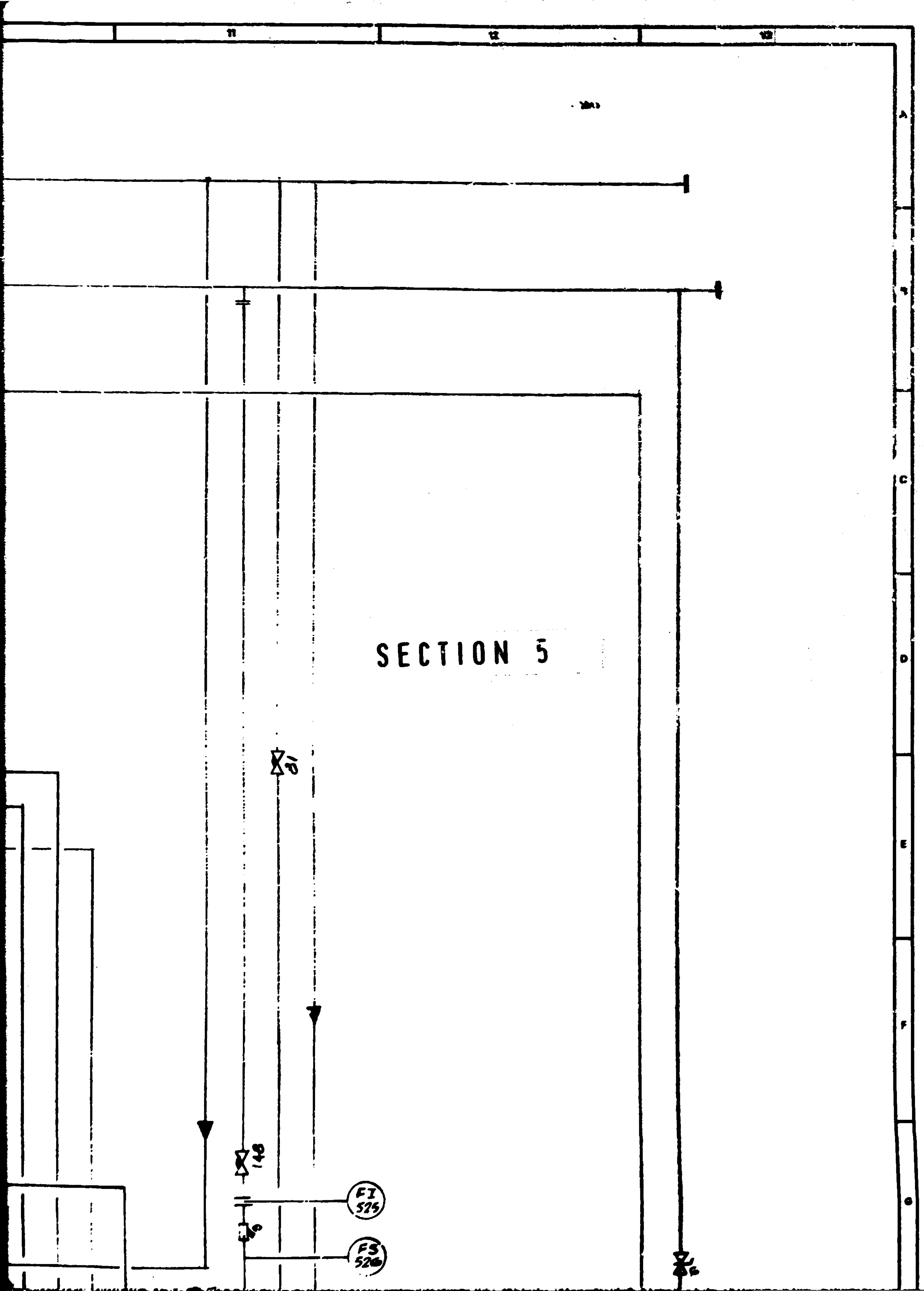
PI 523
FI 525
FS 526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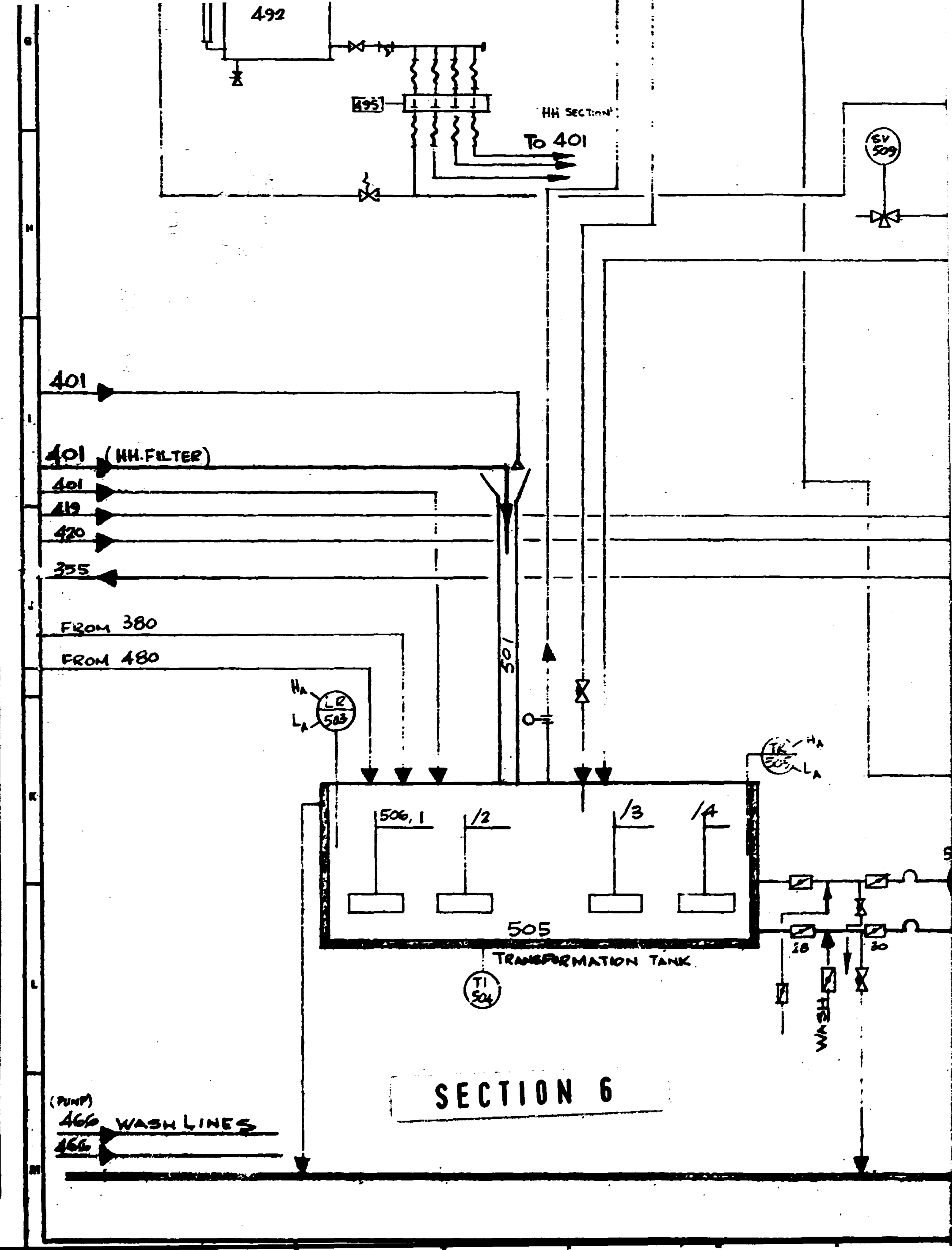
12
13
A
B
C
D
E
F
G
SECTION 5
10
148
FI
525
FS
526


492
495
HH SECTION
To 401
SV 509
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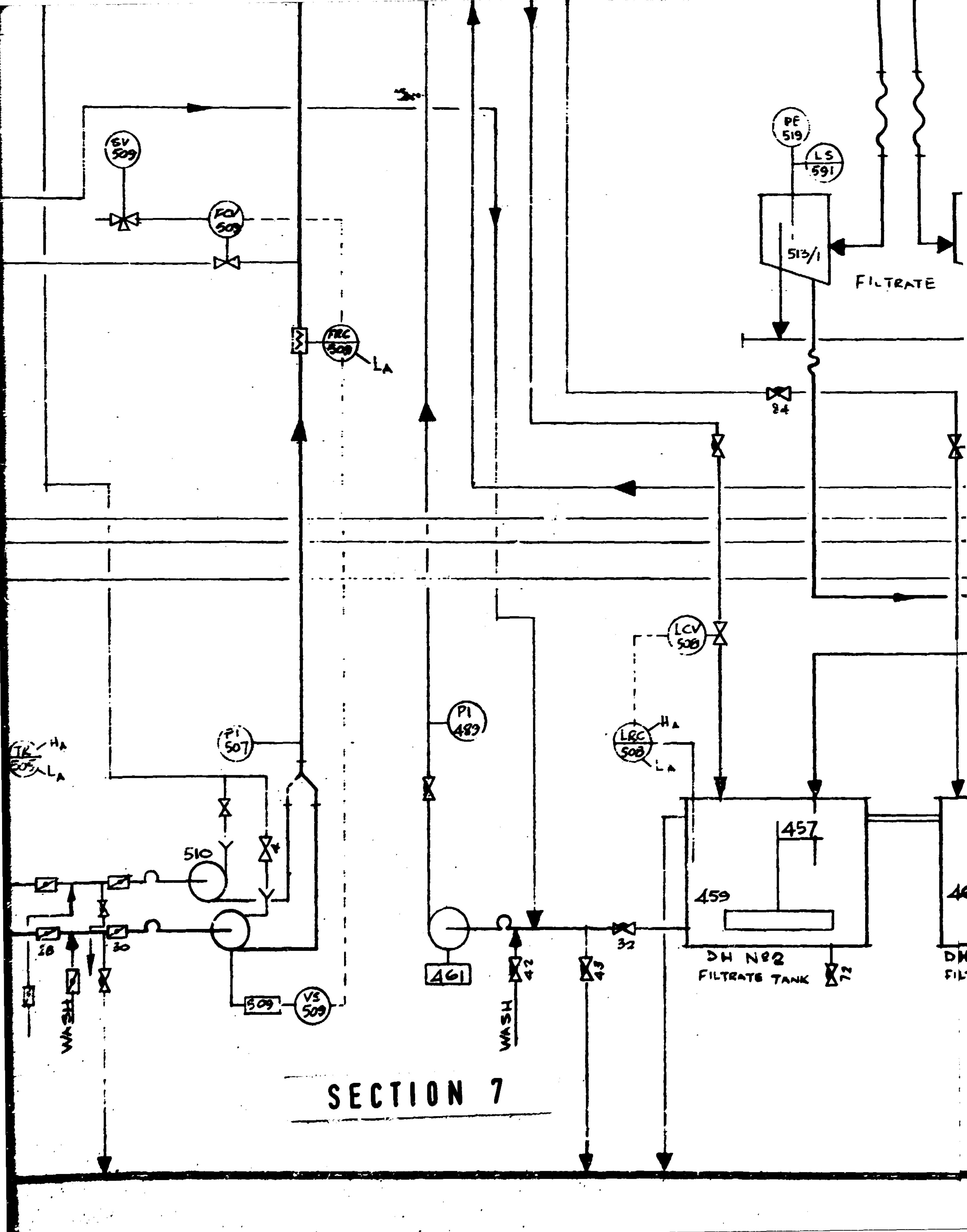
401 (HH.FILTER)
401
419
420
355
FROM 380
FROM 480
HA
LR 503
LA
501
TK 505
HA
LA
506, 1
/2
/3
/4
505
TRANSFORMATION TANK
TI 504
28
30
WASH
SECTION 6
(PUMP)
466 WASH LINES
466
G
H
I
J
K
L
M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WASH
509
VS 509
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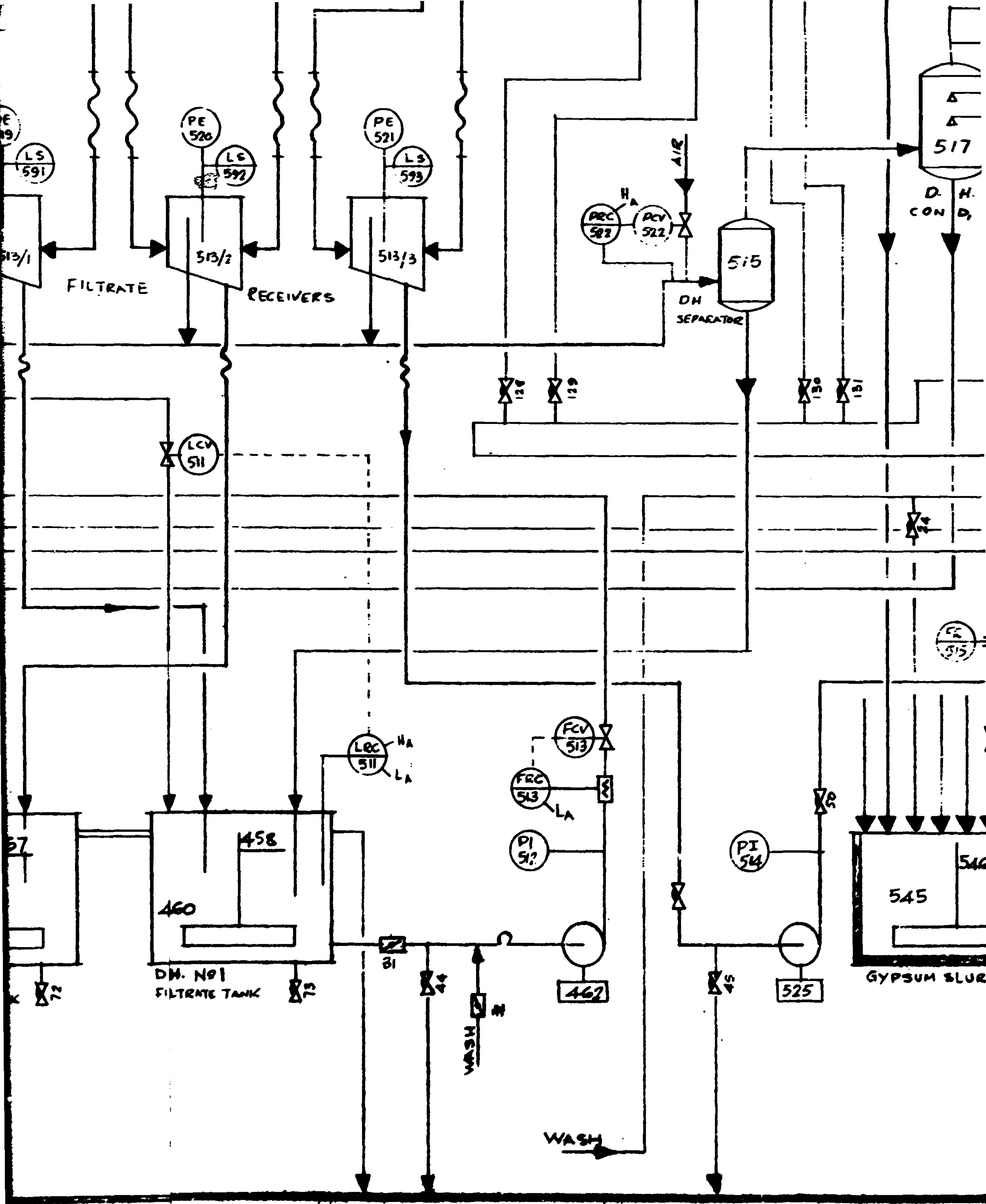
459
DH No2
FILTRATE TANK
72

SECTION 7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LS 591
513/1
FILTRATE RECEIVERS
PE 520
LS 592
513/2
PE 521
LS 593
513/3
AIR
HA
PRC 522
PCV 522
515
DH SEPARATOR
517
D. H. CON D,
128
129
130
131
LCV 511
LRC 511
HA
LA
FCV 513
FRC 513
LA
PI 512
PI 514
FC 515
57
458
460
DH. No 1 FILTRATE TANK
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73
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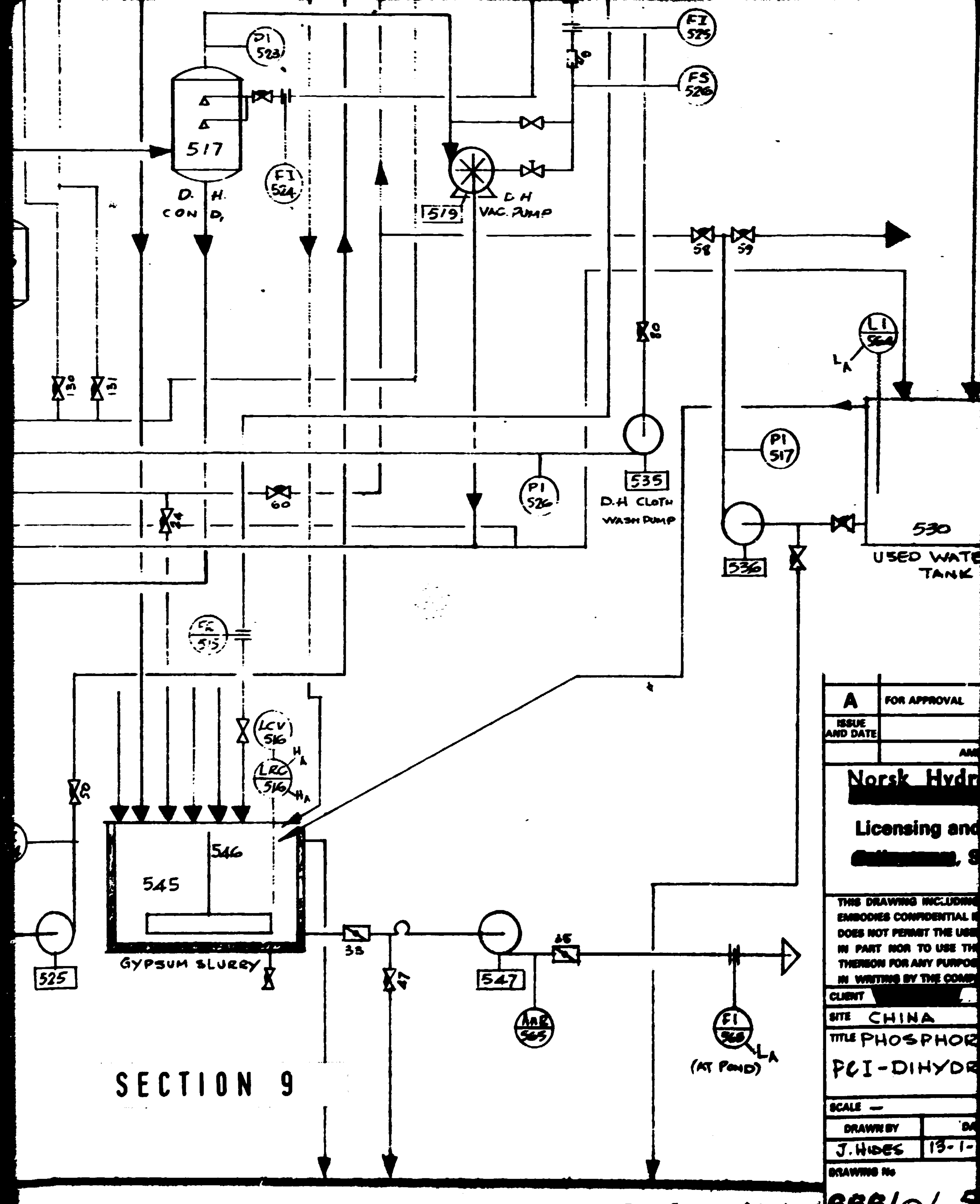
44
45
WASH
WASH
462
525
545
546
GYPSUM SLUR

SECTION 8



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TITLE PHOSPHOR
PCI-DIHYDR

SCALE —

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J. HIDES	13-1-

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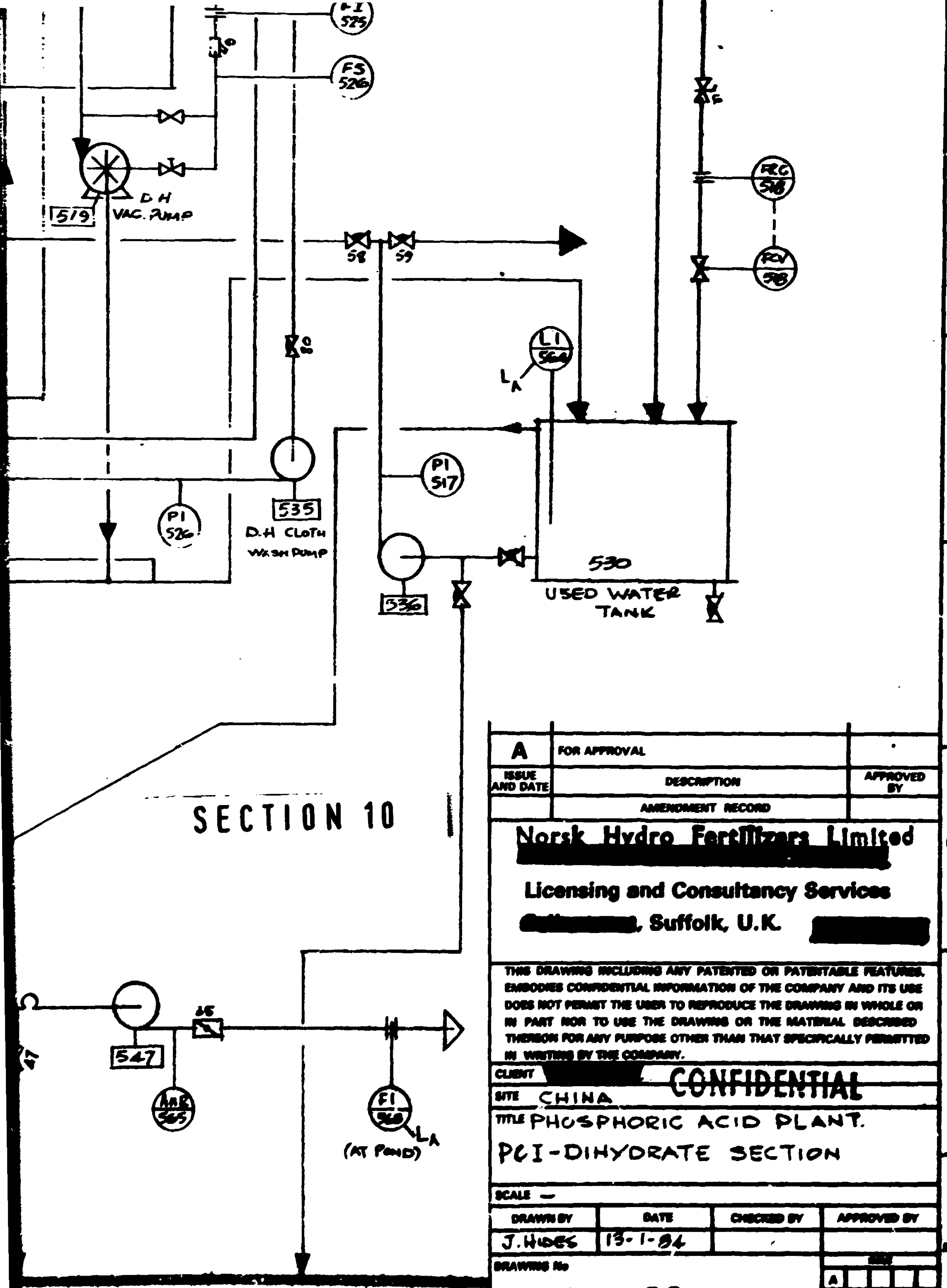

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ISSUE AND DATE	DESCRIPTION	APPROVED BY
	AMENDMENT RECORD	

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TITLE PHOSPHORIC ACID PLANT.
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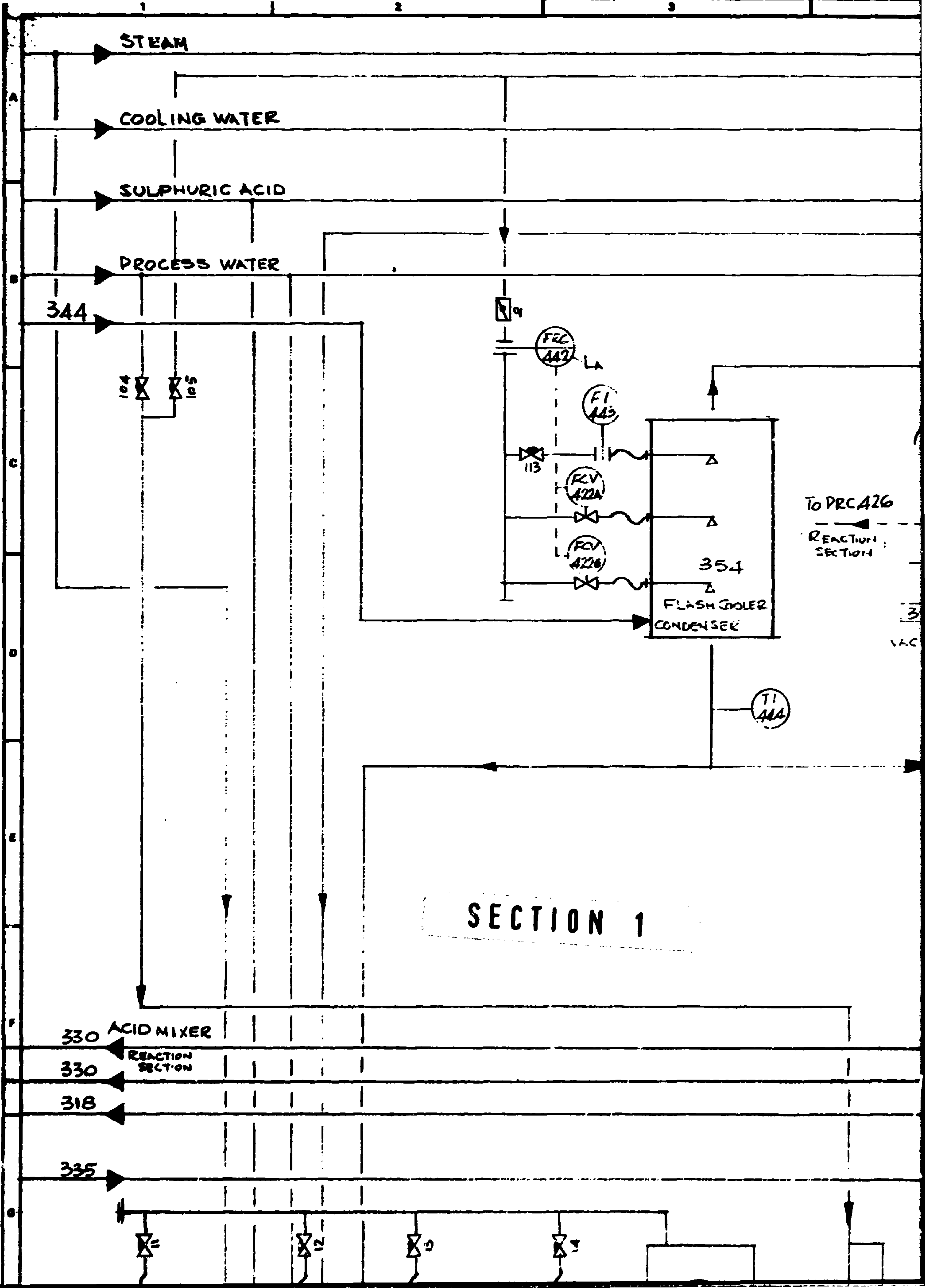
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2
3
A
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C
D
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STEAM
COOLING WATER
SULPHURIC ACID
PROCESS WATER
344
104
105
9
FRC 442
LA
FI 443
113
FCV 422A
FCV 422B
354
FLASH COOLER CONDENSER
To PRC 426
REACTION SECTION
TI 444
SECTION 1
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REACTION SECTION
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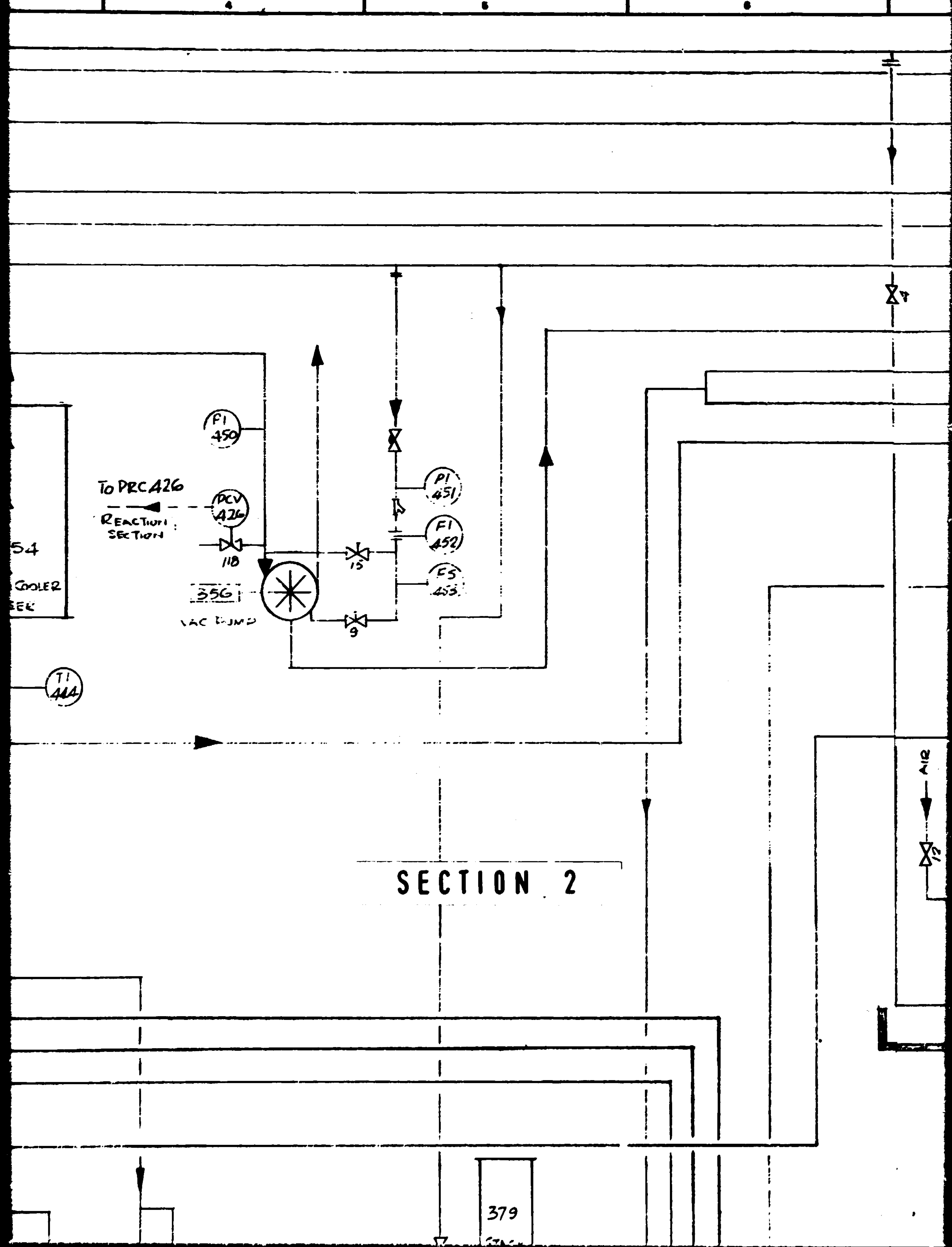
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5
6
7
FI 450
To PRC 426
PCV 426
Reaction Section
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Cooler
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Vac Pump
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PI 451
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FS 453
TI 444
AIR
17
SECTION 2
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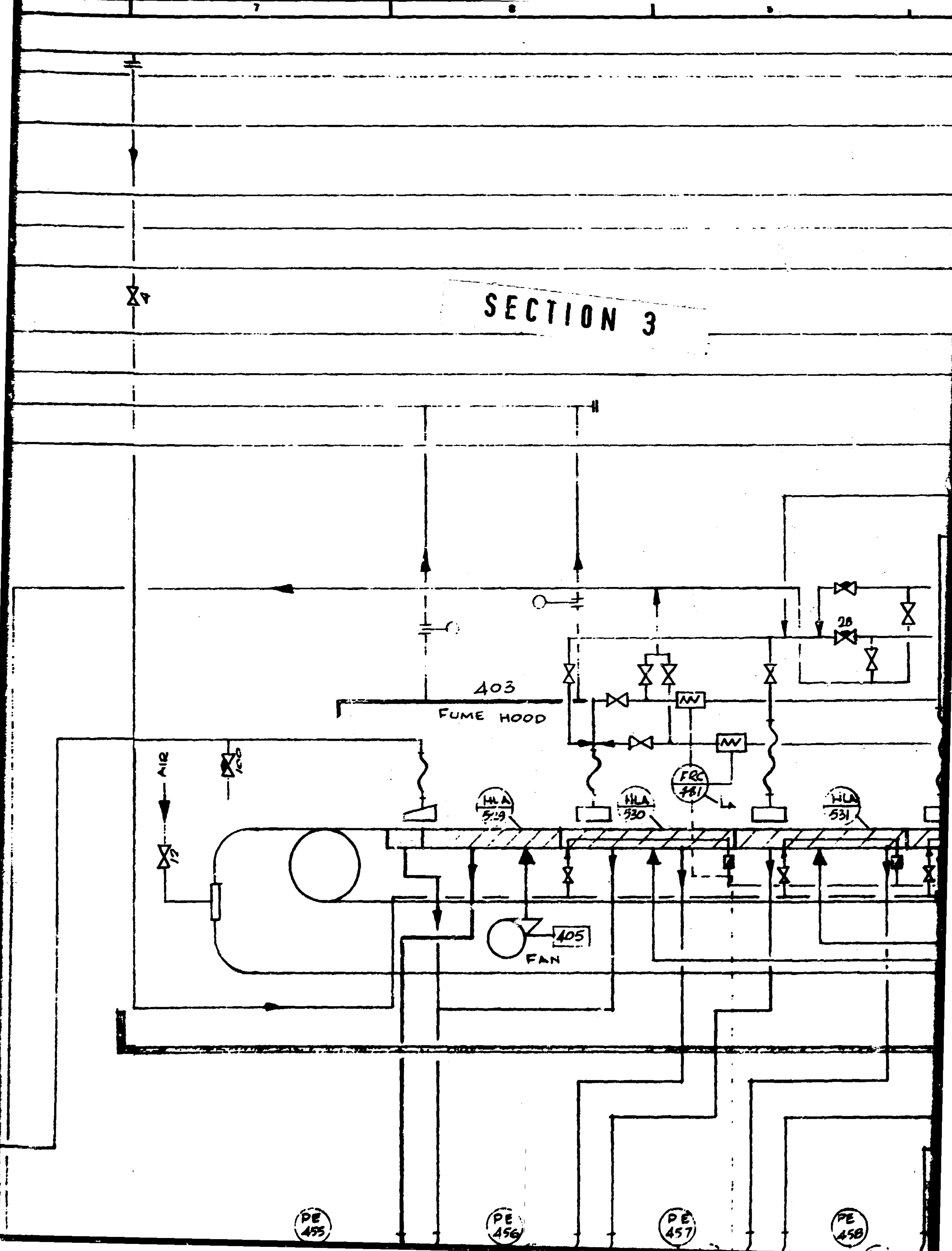
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8
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SECTION 3
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AIR
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HLA 530
HLA 531
FRC 481
LA
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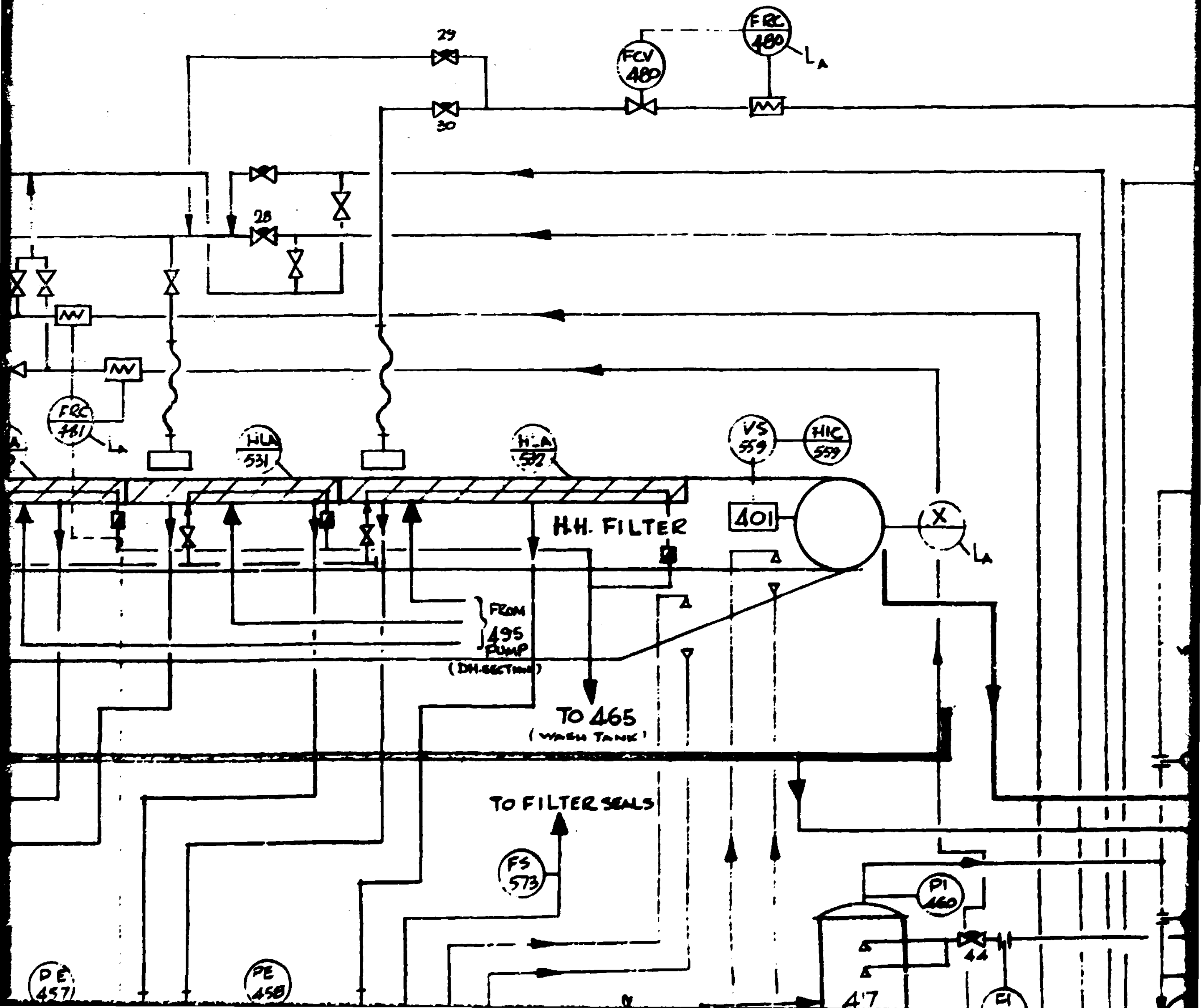
FAN
PE 455
PE 456
PE 457
PE 458

SECTION 4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30
FCV 480
FRC 480
LA
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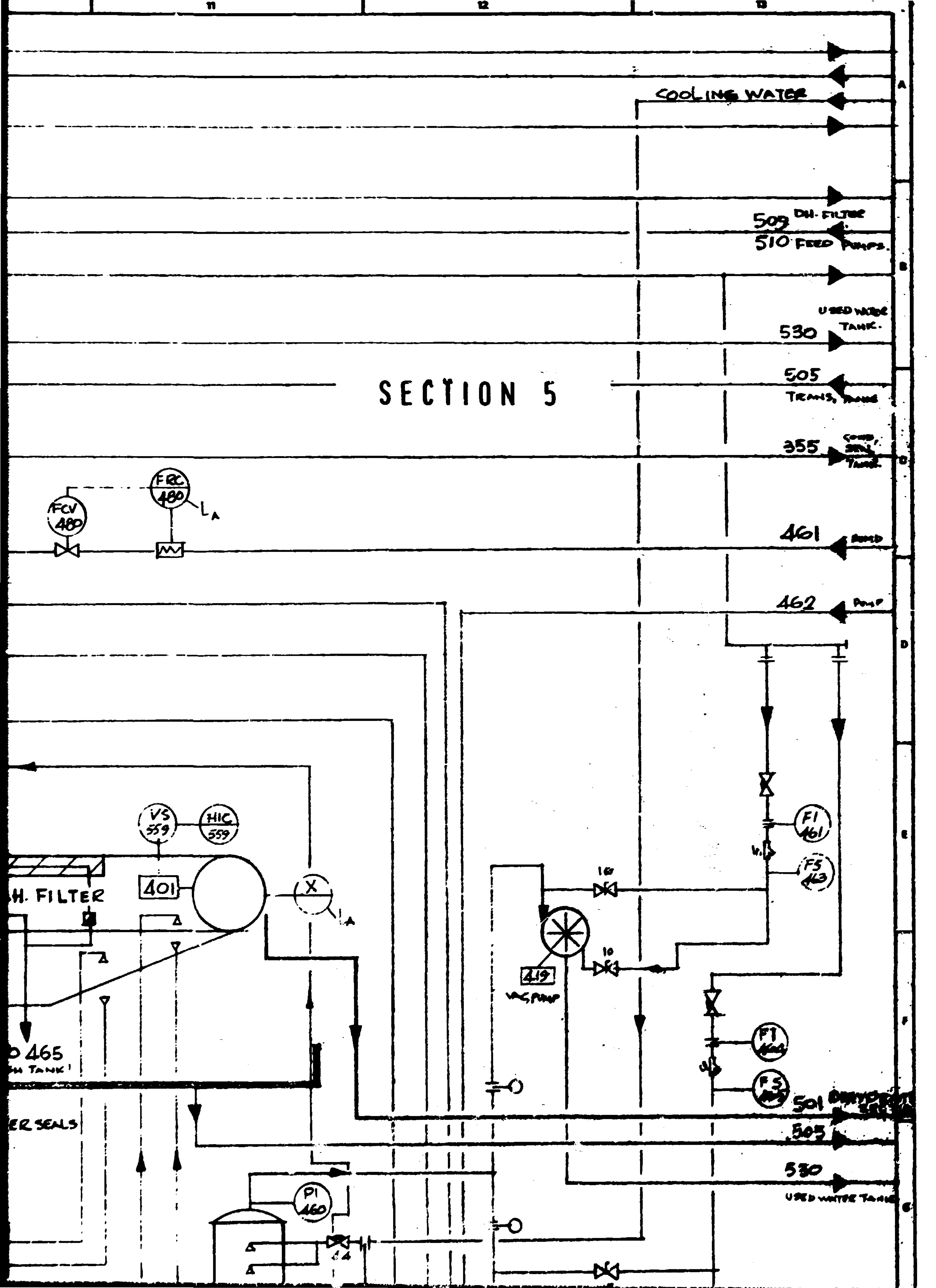
FRC 481
LA
HLA 531
HLA 532
VS 559
HIC 559
401
X
LA
H.H. FILTER
FROM 495 PUMP (DH SECTION)
TO 465 (WASH TANK)
TO FILTER SEALS
FS 573
PI 460
44
47
PE 457
PE 458
FI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12
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A
B
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D
E
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510 FEED PUMPS.
USED WATER TANK.
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SECTION 5
505
TRANS, TANK
355
COND, SEAL TANK.
FRC 480
FCV 480
LA
461
PUMP
462
PUMP
VS 559
HIC 559
401
H. FILTER
X
LA
FI 461
FS 463
10
10
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FS 465
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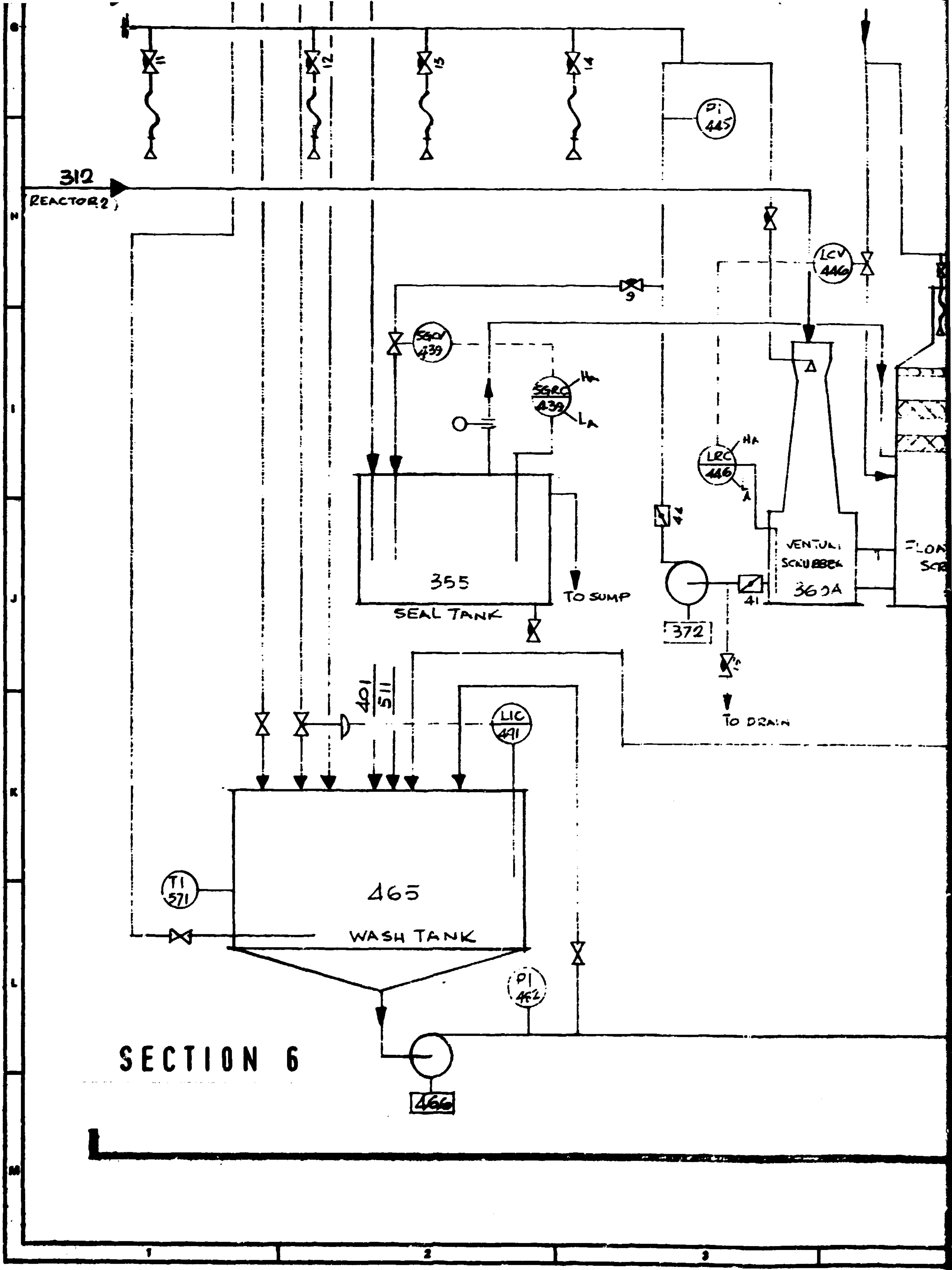
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SGRC 439
HA
LA
LRC 446
HA
LA
44
VENTURI SCRUBBER
369A
FLOA
SCR
355
SEAL TANK
TO SUMP
372
41
TO DRAIN
401
511
LIC 491
TI 571
465
WASH TANK
PI 462
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SECTION 6
G
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I
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K
L
M
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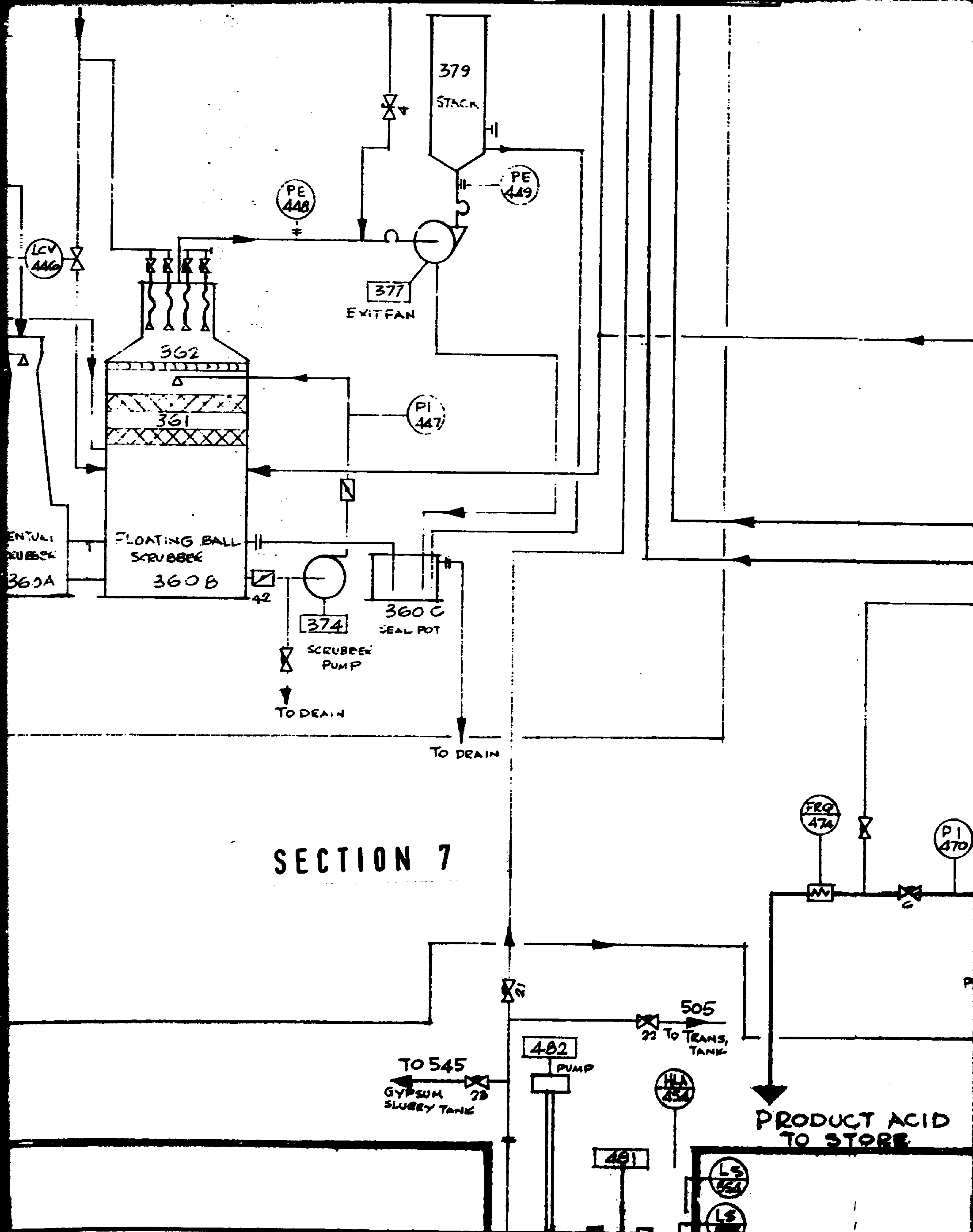
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LCV 446
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361
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ENTURI
SCRUBBER
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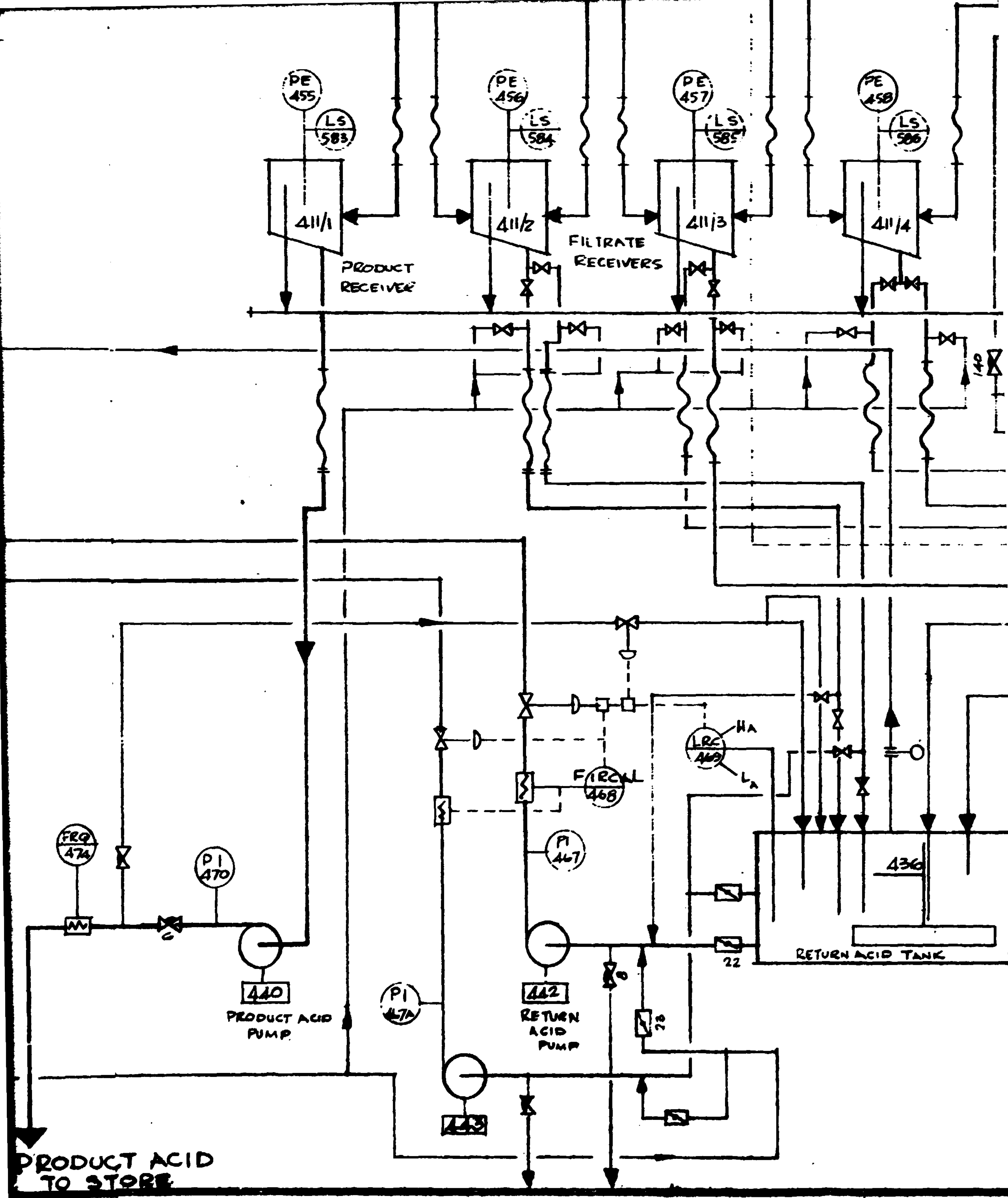
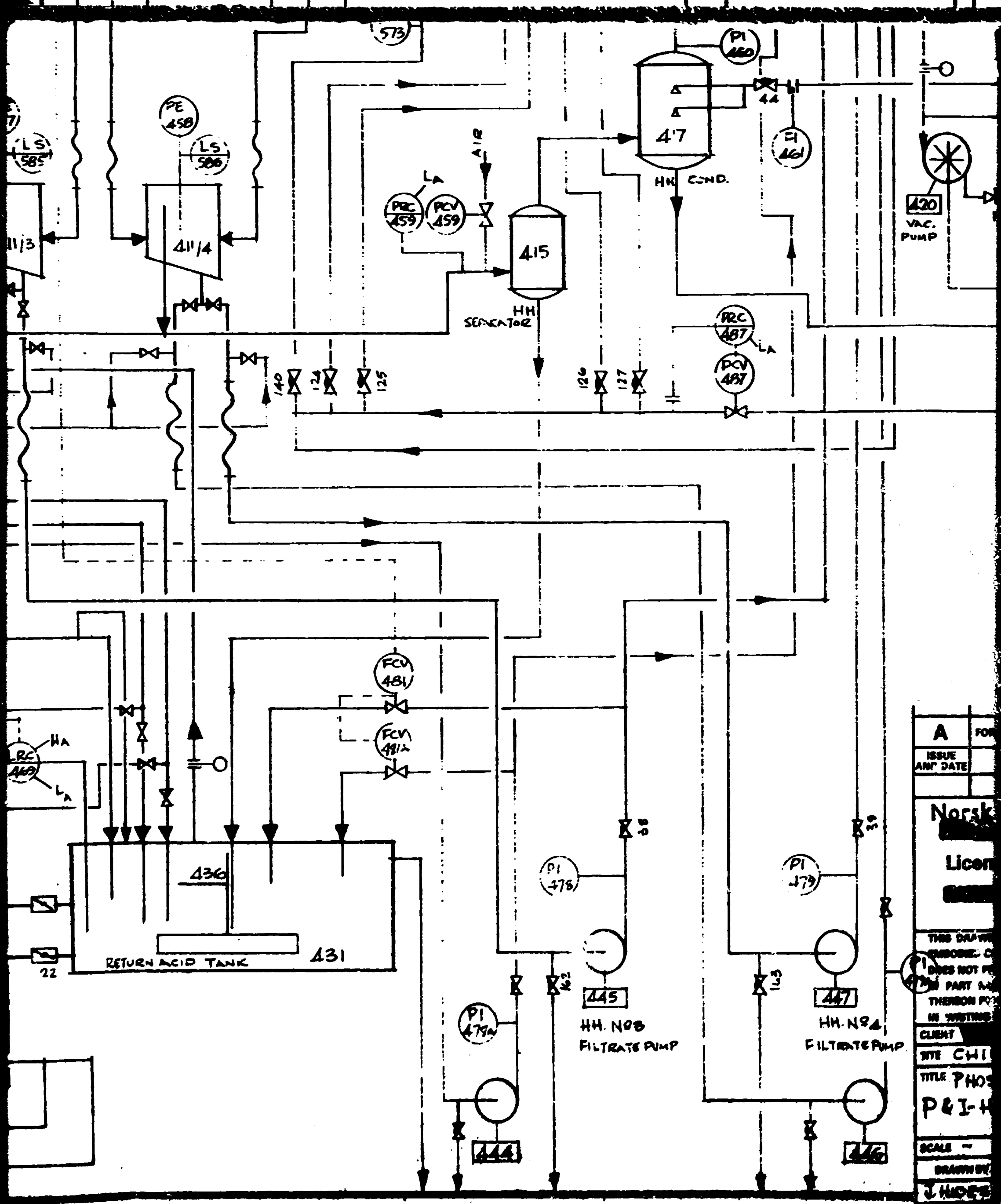
FLOATING BALL
SCRUBBER
360 B
42
374
SCRUBBER
PUMP
360 C
SEAL POT
TO DRAIN
TO DRAIN
SECTION 7
FRQ 474
PI 470
6
21
505
22 To TRANS, TANK
482
PUMP
TO 545
GYPSUM SLURRY TANK
23
HLA 434
PRODUCT ACID TO STORE
481
LS 564
LS


PE 455
LS 583
411/1
PE 456
LS 584
411/2
PE 457
LS 585
411/3
PE 458
LS 586
411/4
PRODUCT RECEIVER
FILTRATE RECEIVERS
140
LRC 469
HA
LA
FIRC 468
L
PI 467
FRQ 474
PI 470
440
PRODUCT ACID PUMP
PI 467A
442
RETURN ACID PUMP
443
436
RETURN ACID TANK
22
10
20
PRODUCT ACID TO STORE

SECTION 8


573
PI 460
PE 458
LS 585
LS 586
411/3
411/4
AIR
LA
PRC 459
PCV 459
415
HH SEPARATOR
417
HH COND.
44
PI 461
420
VAC. PUMP
PRC 487
LA
PCV 487
140
124
125
126
127
FCV 481
FCV 481/2
HA
LRC 469
LA
436
431
RETURN ACID TANK
22
PI 478
PI 479
PI 479A
445
HH. NO3 FILTRATE PUMP
447
HH. NO4 FILTRATE PUMP
444
446
A
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SCALE
DRAWN BY
J. HODGS
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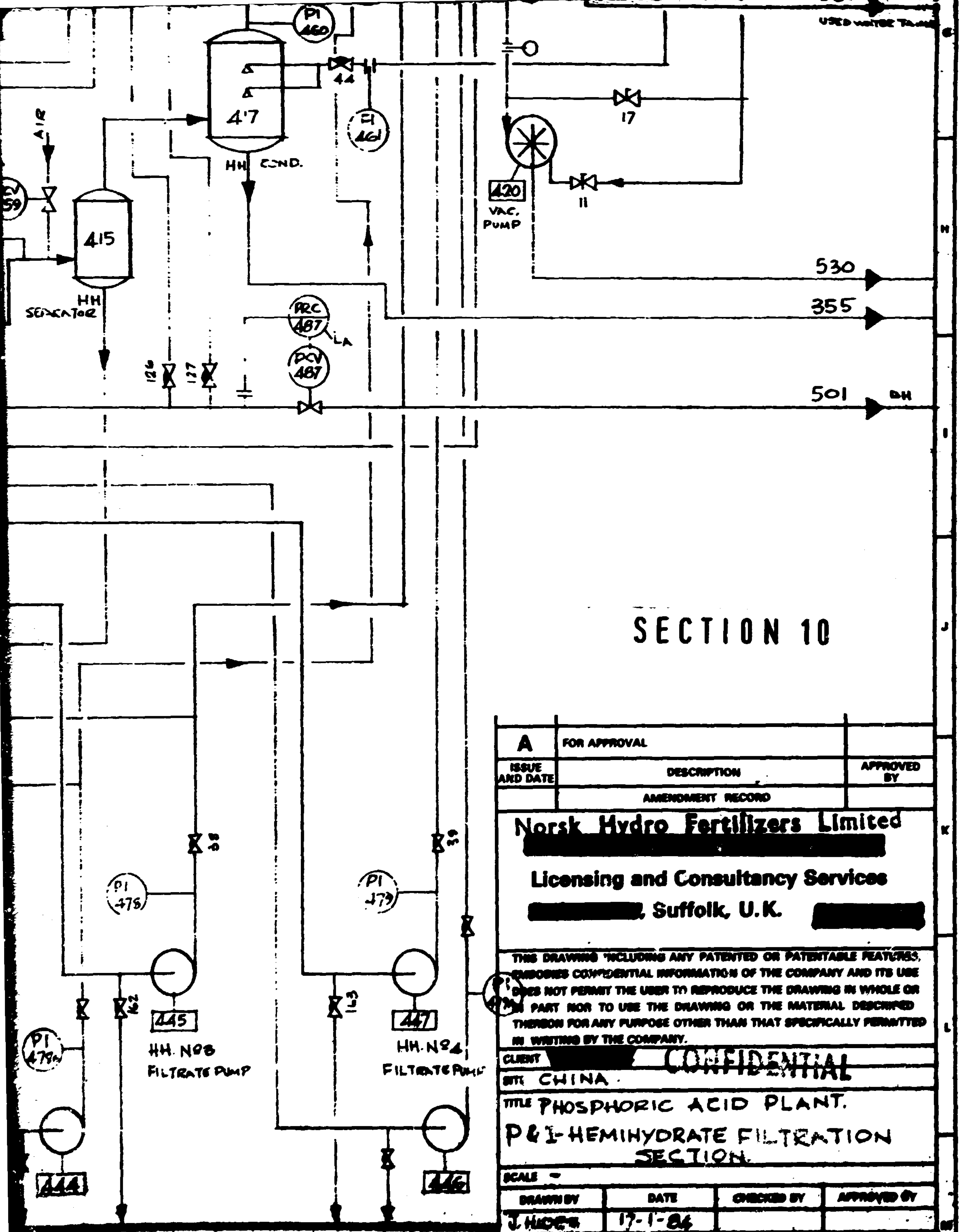
686/



SECTION 10

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[redacted]
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TITLE PHOSPHORIC ACID PLANT.
P&I-HEMIHYDRATE FILTRATION SECTION.
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J. HIDES	17-1-84		

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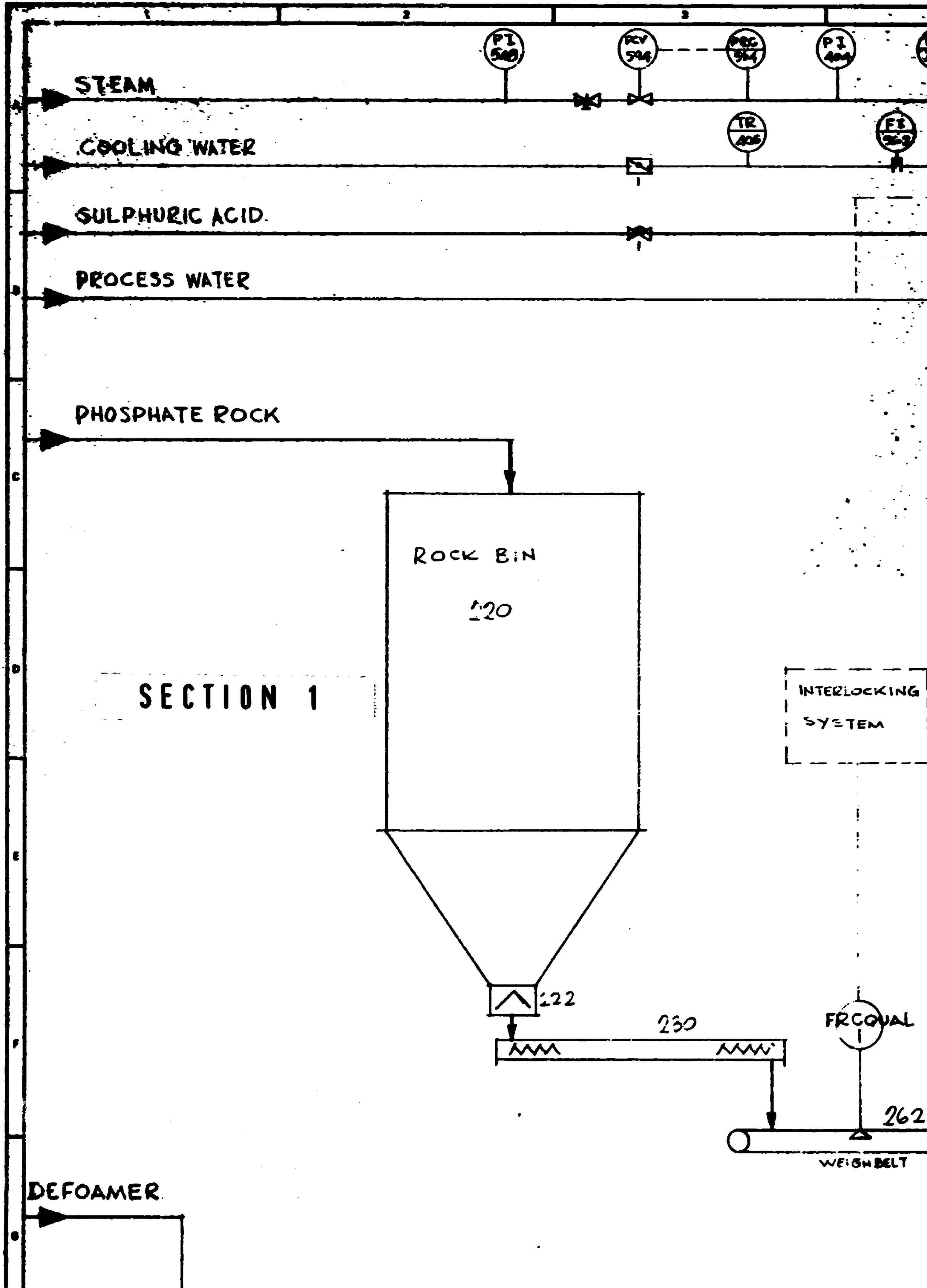
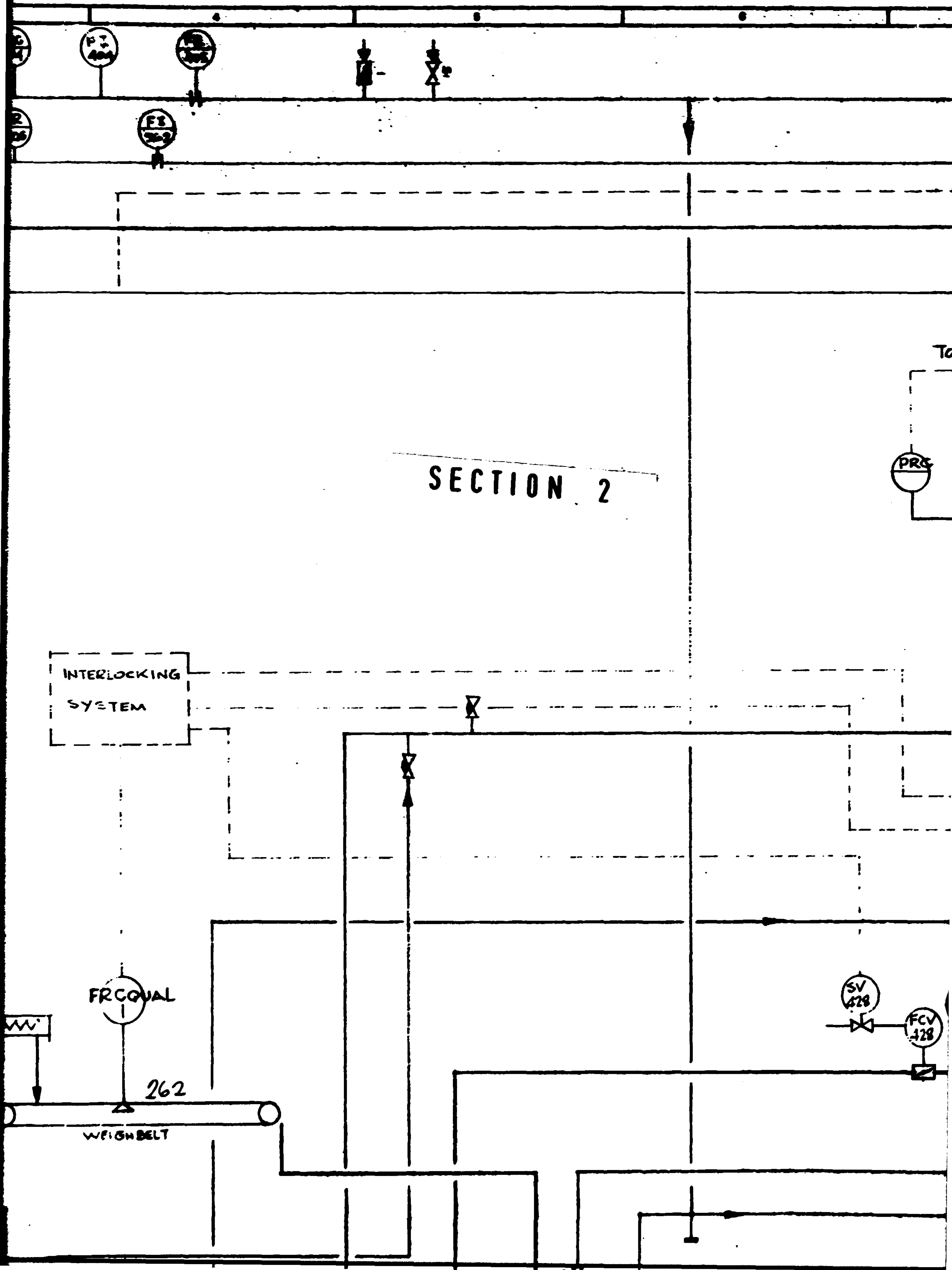
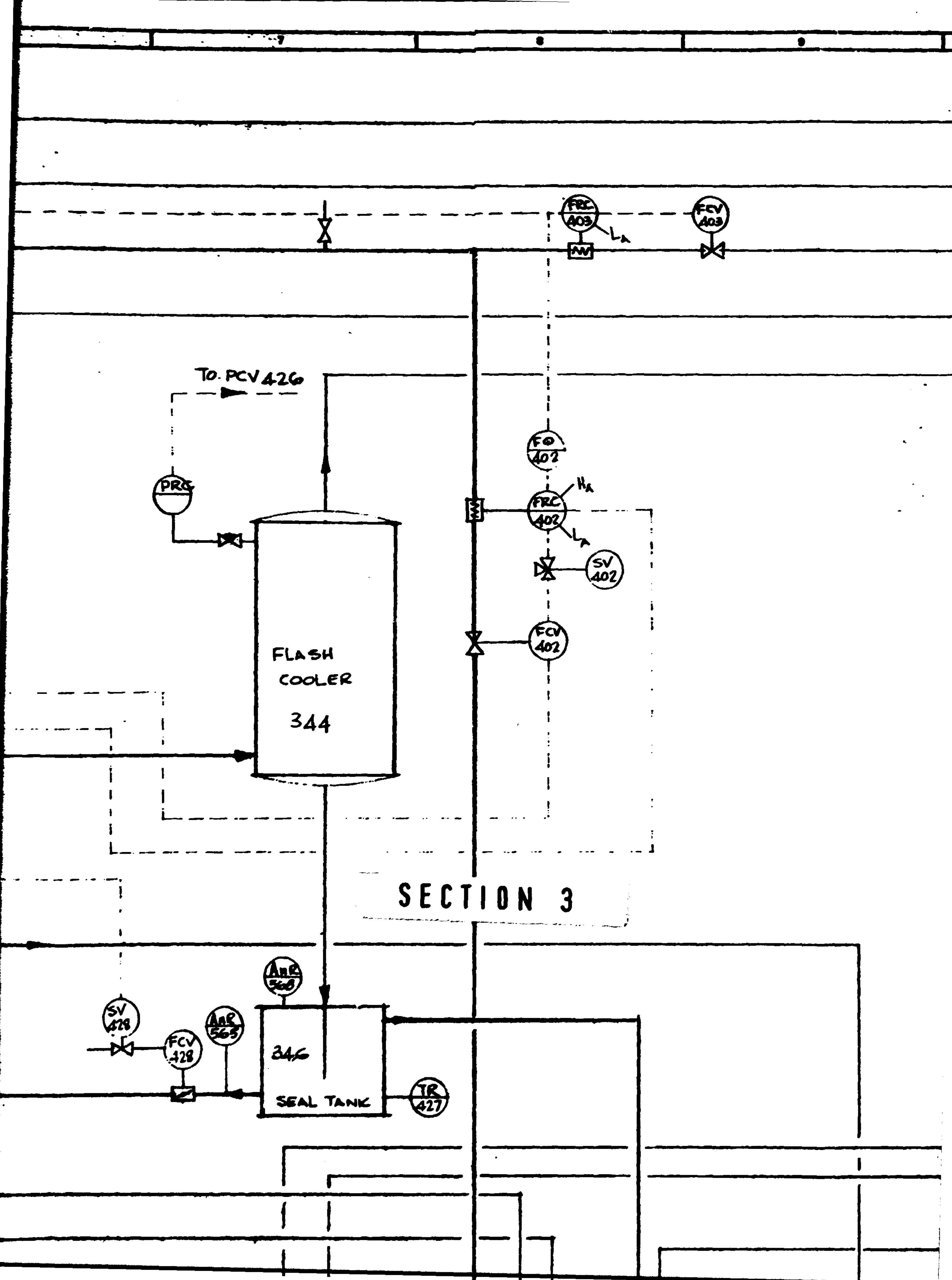
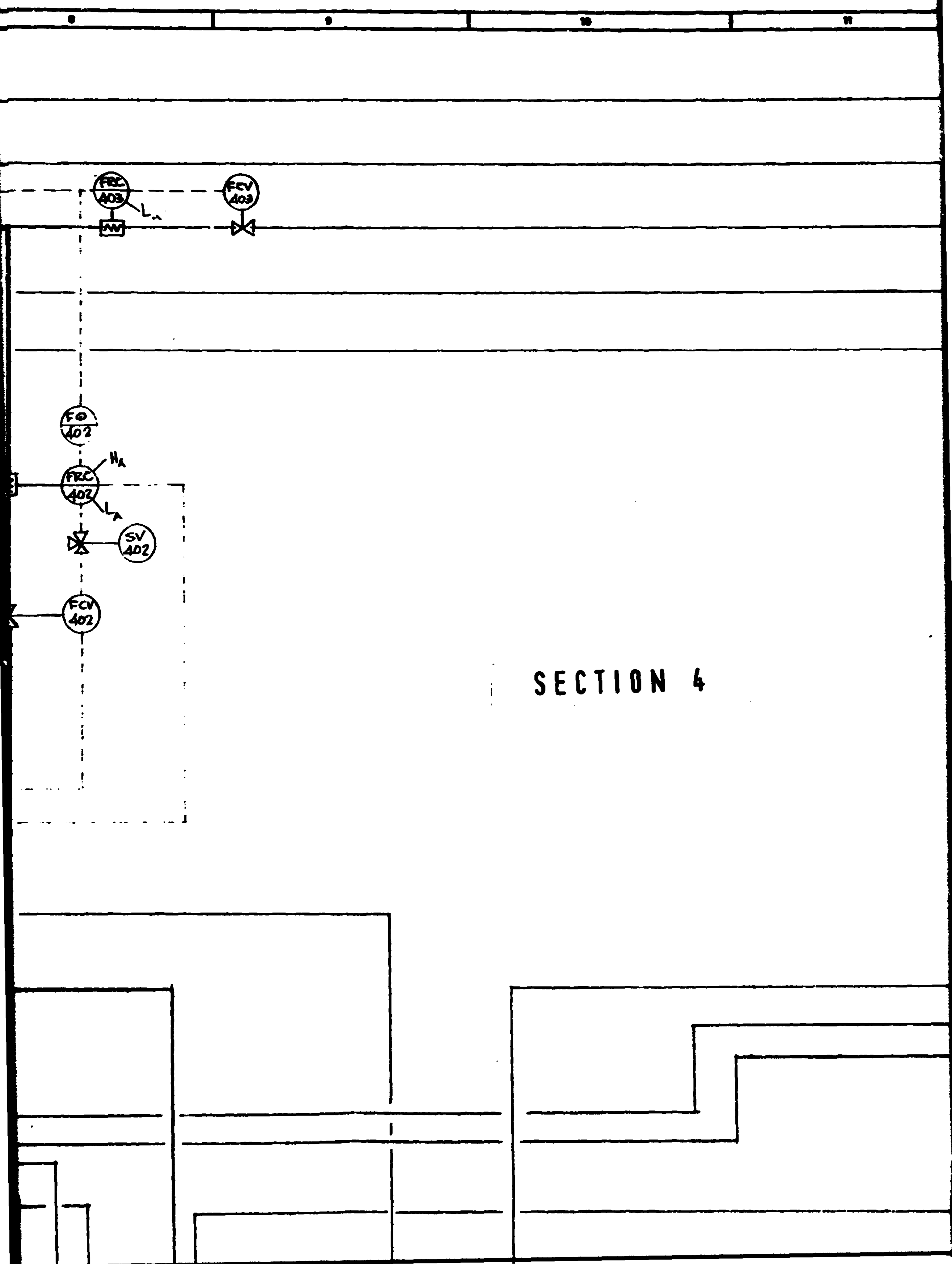
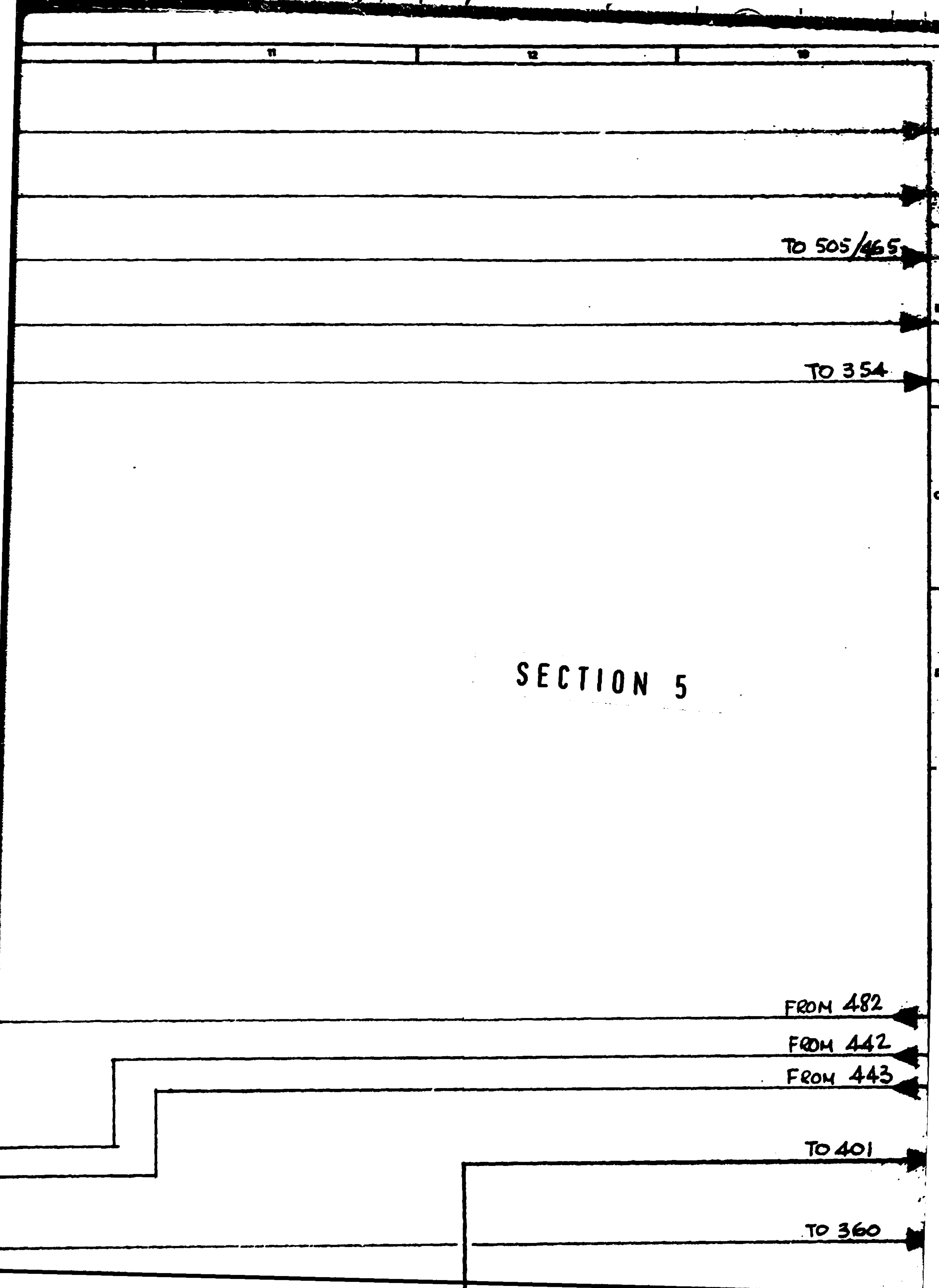
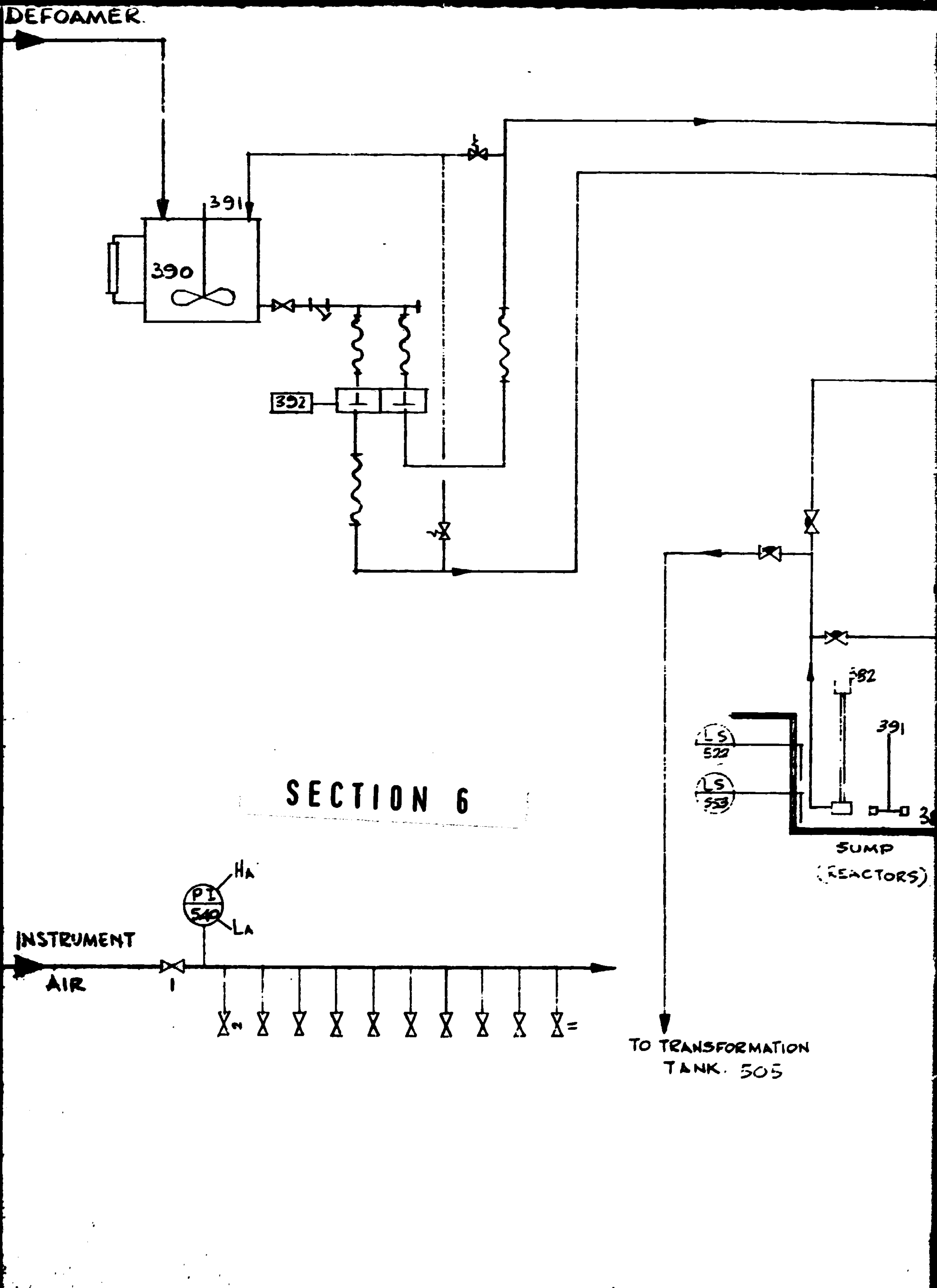

1
2
3
STEAM
PI 508
PCV 594
PRC 594
PI 404
COOLING WATER
TR 406
FI 362
SULPHURIC ACID
PROCESS WATER
PHOSPHATE ROCK
ROCK BIN
220
SECTION 1
INTERLOCKING SYSTEM
222
230
FRCQUAL
262
WEIGHBELT
DEFOAMER
A
B
C
D
E
F
G

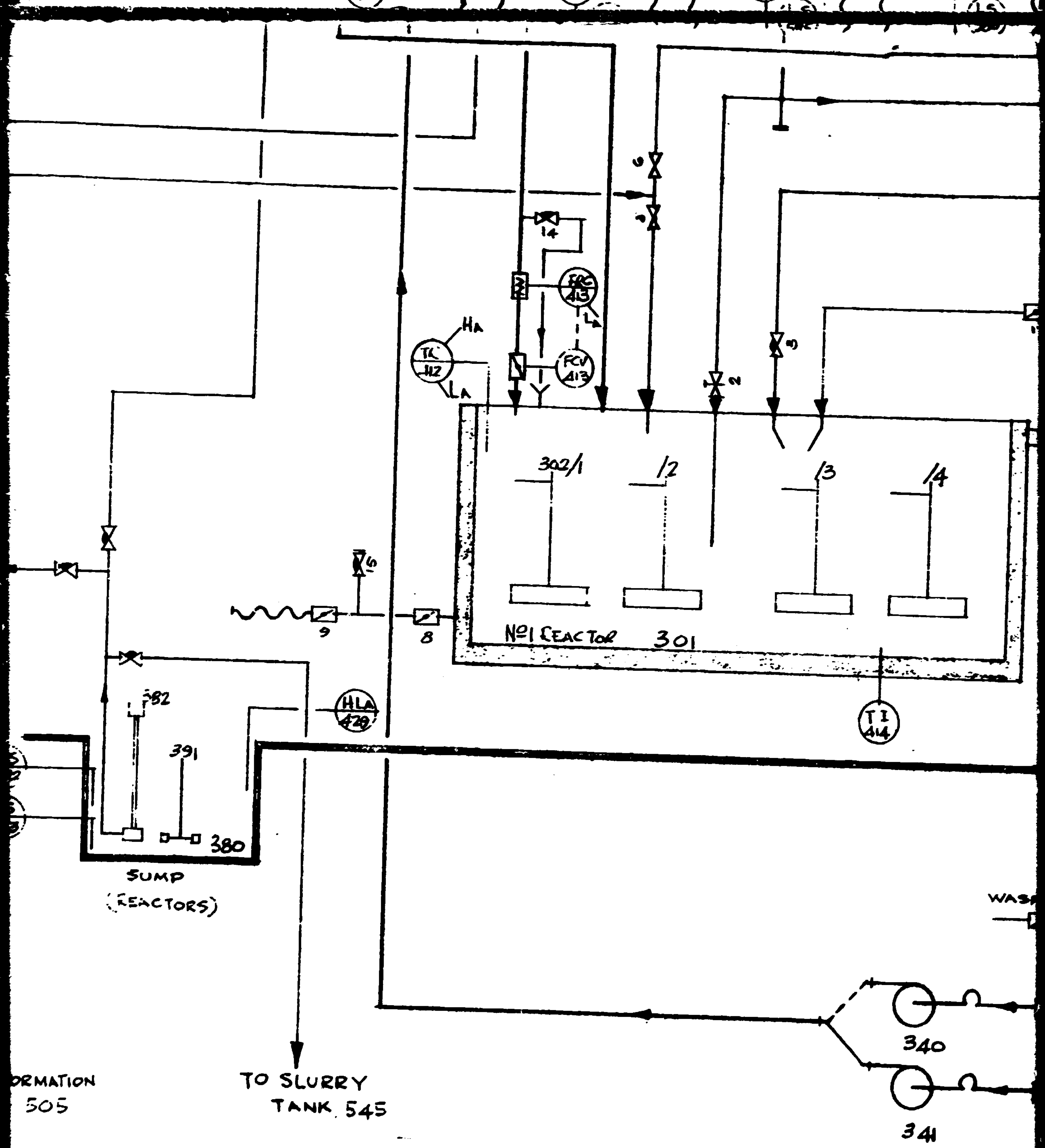

4
5
6
FT 404
FE 362
To
PRC
SECTION 2
INTERLOCKING SYSTEM
FRCQUAL
262
WEIGHBELT
SV 428
FCV 428


7
8
9
FRC 403
FCV 403
LA
TO PCV 426
PRC
FO 402
HA
FRC 402
LA
SV 402
FCV 402
FLASH COOLER 344
SECTION 3
AnR 568
SV 428
AnR 565
FCV 428
346
SEAL TANK
TR 427


8
9
10
11
FRC 403
L
FCV 403
FQ 402
H
FRC 402
L
SV 402
FCV 402
SECTION 4

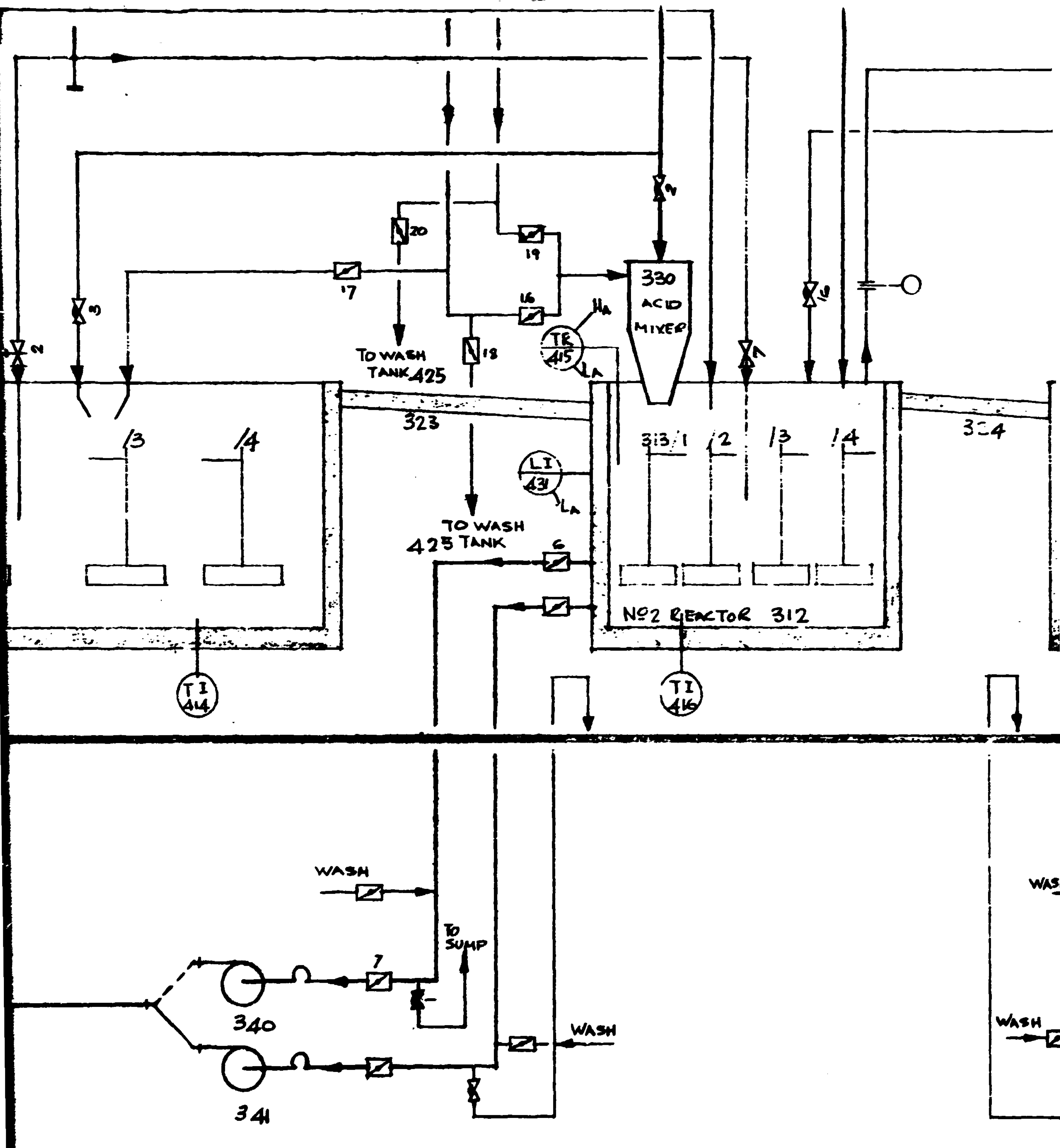

11
12
13
TO 505/465
TO 354
SECTION 5
FROM 482
FROM 442
FROM 443
TO 401
TO 360


DEFOAMER
391
390
392
SECTION 6
PI
540
HA
LA
INSTRUMENT
AIR
I
382
391
LS
522
LS
533
SUMP
(REACTORS)
TO TRANSFORMATION
TANK. 505

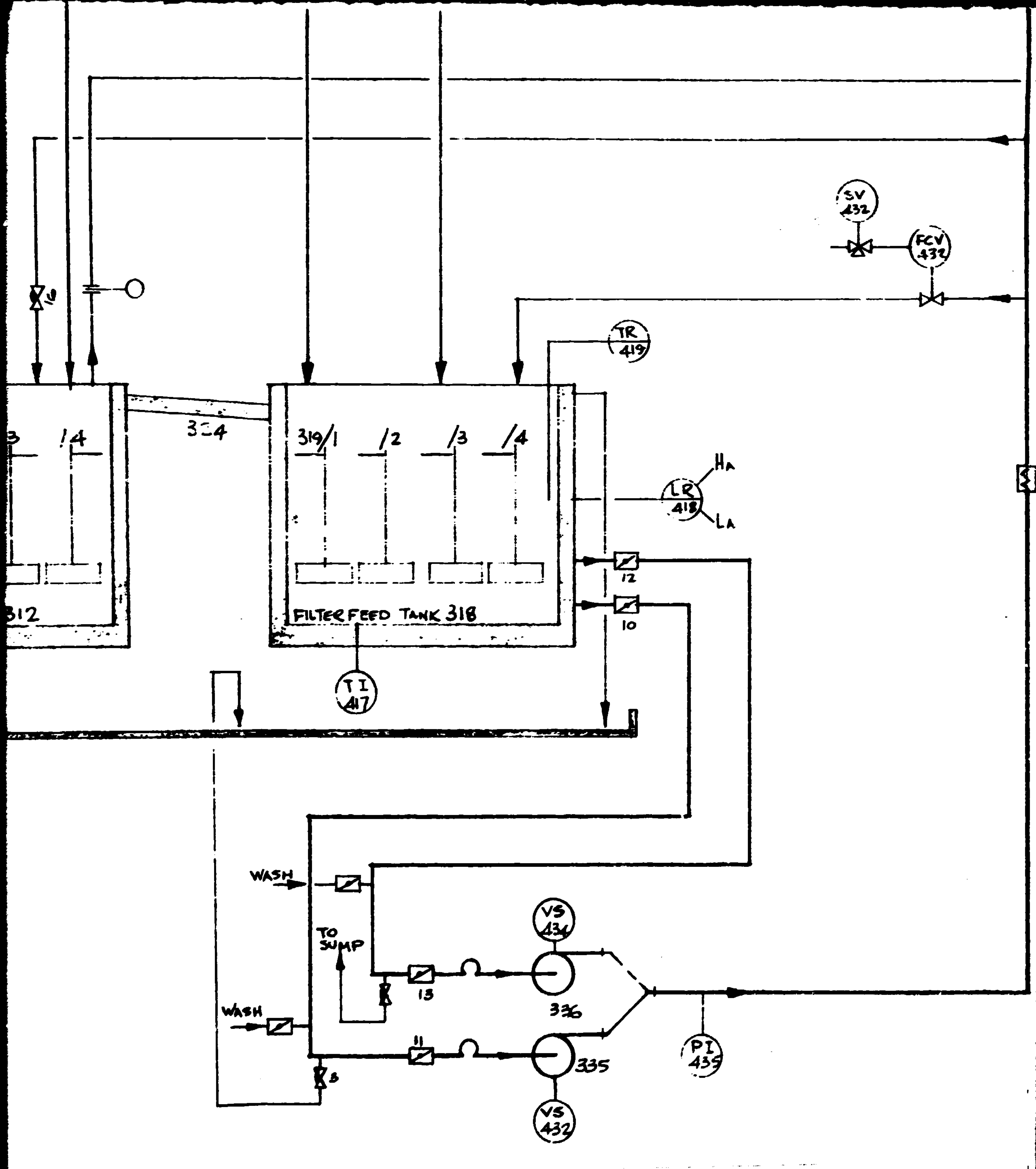


14
FRC 413
HA
TC 412
LA
FCV 413
LA
302/1
/2
/3
/4
9
8
№1 REACTOR 301
382
391
380
HLA 420
TI 414
SUMP
(REACTORS)
WAS
340
341
ORMATION
505
TO SLURRY
TANK 545

SECTION 7

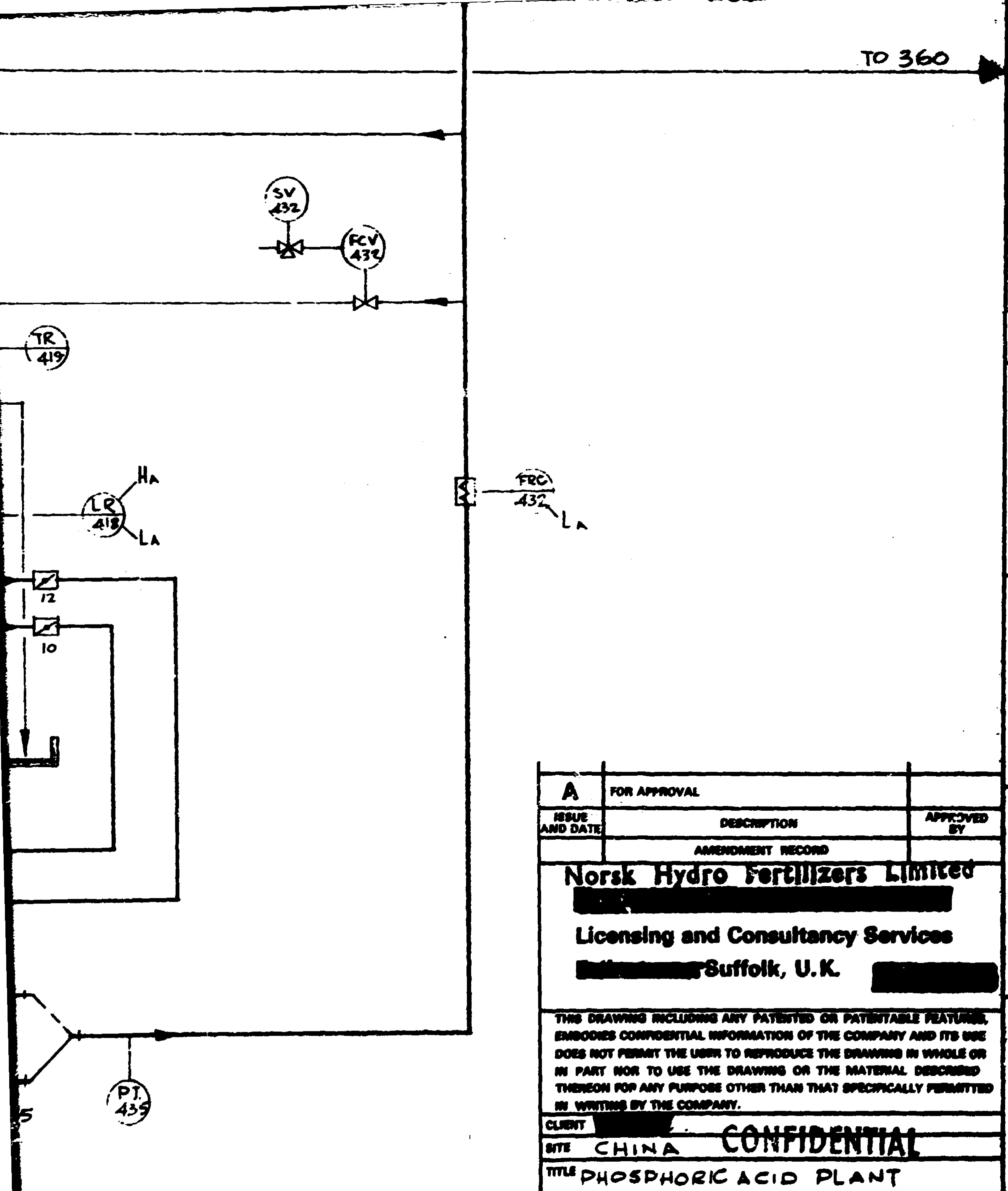

20
19
17
16
18
330 ACID MIXER
TE 415
HA
LA
TO WASH TANK 425
323
/3
/4
313/1
/2
/3
/4
3_4
LI 431
LA
TO WASH 425 TANK
6
No2 REACTOR 312
TI 414
TI 416
WASH
TO SUMP
7
340
341
WASH
WAS
WASH

SECTION 8



SECTION 9

Norsk Hydro Fertilizers Lin



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SITE CHINA CONFIDENTIAL

TITLE PHOSPHORIC ACID PLANT

P&I - REACTION SECTION

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J HIDES	10-1-84		

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