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ENTRAINED STATE HEATER FOR CHLORINATION OF ILMENITE

by

A. W. Nordin

Investigated by: Industrial Chemistry Section

Officer in Charge: R. E. Wilmshurst

P. A. Young. Director

THE AUSTRALIAN MINERAL DEVELOPMENT LABORATORIES
Adelaide South Australia

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SUMMARY

The success of the process for production of titania from ilmenite using a fluidised-bed chlorinator is dependent on the development of a system to supply sufficient heat during the chlorination reaction. This report records a study of the first three stages of a six point programme for the development of a system in which a stream of material discharged from a fluidised-bed chlorinator is heated in an entrained-bed heater and returned to the fluidised bed at a sufficiently high temperature to supply the heat required.

A literature survey was conducted to obtain information on the transport of solids between reactors, regenerators and heaters and to obtain details of various sealing devices to control the rate of transfer of solids from one system to another.

A pilot-scale model of the proposed fluidised-bed/entrained-state heater system was assembled to study the characteristics and effectiveness of selected types of sealing devices. A modified Dorrco type valve was found to be the most suitable with respect to sealing, capacity, reliability and construction.

A flow sheet for a production plant was prepared and sufficient design work completed to enable a preliminary cost estimate to be made.

The capital required for the production of 18,000 tons of titania per annum from ilmentie by the cyclic chlorination process is $A4 million. The operating costs for plants at Capel and Stradbroke Island have been estimated to be $A102 and $A106 per ton of titania. High depreciation (15%) and maintenance (10%) rates have been applied due to the severe operating conditions and these charges account for 50% of the operating cost. At the 1965 price of $A80 per ton for titania the process is not an economic proposition. Further development of the individual pieces of equipment could possibly reduce the estimated operating costs by $A10 per ton.

In view of the formidable technical difficulties still to be overcome, and the unfavourable economics of the process, no further work is recommended.

1. INTRODUCTION

Wilmshurst (1962) discussed some of the operating problems of the process including the difficulty of supplying heat to a fluidised-bed chlorinator. The Director, (Dr Coffer) suggested that it might be possible to develop a system in which a stream of material discharged from a fluidised-bed chlorinator was heated in an entrained-bed heater and returned to the fluidised bed at a temperature sufficiently above that of the chlorination bed to supply the heat required.

When endothermic reactions are carried out at high temperatures in fluidised-bed reactors, supply of heat to the bed may be difficult. For calcination reactions direct combustion of fuel with fluidising air is the usual method of supplying this heat. However, with some reactions, the combustion products from this direct firing would contaminate the gaseous products of the reaction and some other heating method is necessary. Heating the reactor from the outside is useful only for very small scale operations, so the use of an entrained-state heater for a fluidised-bed reactor has been developed.

An entrained-state heater is a tube furnace through which solids are carried by transport air, and in which fuel is burned directly in contact with the solids. By circulating some of the solids from the reactor through such a heater, superheating them, and returning them again, heat can be supplied to the fluid bed. A second fluid bed, directly fired, could be used as an alternative to the entrained heater.

The South Australian Government Department of Mines agreed to sponsor a project on the design, construction and testing of a unit of this kind. This programme arranged was:

1. To conduct a literature survey and to prepare a critical report on the various mechanisms proposed.
2. To select the most promising types of seals described and obtain information from experimental work of the characteristics of these devices.
3. To prepare an economic estimate to evaluate the potential of the process to the chlorination of ilmenite.
4. On the basis of 2 and 3 above, to design and construct a high temperature pilot unit.
5. To operate the high temperature unit to prove the feasibility of the system of the general case.
6. To operate the system for the chlorination of ilmenite.

This report completes the investigation of the first three stages of the programme.

2. MATERIAL EXAMINED

The material used in the tests was a beach sand rutile concentrate from Wyong Minerals Limited, NSW. This rutile was selcted because it was a convenient, closely sized material, and was hard enough to produce only a small amount of fines when recirculated through the system. A screen analysis of a sample of rutile is given in Table 1. The bulk density of the rutile was 2.45 g ml^{-1} and the true density was 4.2 g ml^{-1}.

3. EQUIPMENT

A sketch of the model of the proposed fluidised-bed/entrained-state system is shown in Figure 1. Essentially it consisted of a 12-inch diameter fluidised bed, a 4-inch diameter entrained-state "heater", a solids knockout vessel and a cyclone. Solids flowed down the standpipe from the fluidised bed to the valve whence they were entrained by the transport air and carried to the bottom of the entrained heater. Here the main portion of the entraining air mixed with the transport air and carried the solids up to the knockout vessel where the bulk of the solids was disentrained from the air stream. Very fine solids were collected in the cyclone. Gas cocks maintained a seal of solids in the return lines from the knockout vessel and cyclone to the fluidised bed to prevent air from blowing down these lines. The height of the fluidised bed could be varied from 6 inches to 4 feet. Suitable tappings (with air purges where necessary) were provided to measure the pressures at various parts of the system. Rotameters were used to measure the fluidising, transport and aeration air flow rates, and an orifice plate in the line from the Roots blower measured the flow rate of the main entraining air stream.

Sketches of seven of the nine valves tested are given in Figures 2 and 3. These mockups or models of the valves were constructed from pipe fittings. The levels taken up by the solids are shown in the drawings. In all cases these valves were attached to the bottom of the standpipe. The Esso Type A valve was modified after the original version failed to shut off effectively. The body of the valve and centre tube were lengthened by 2 inches so that the latter was 3½ inches below the transport air line.

For the U-bend valve, clear plastic tube was used as a constructional material. The USS Venturi valve was attached to the bottom of a separate ½-inch diameter standpipe, which extended 24 inches above and 34 inches below the distribution plate of the fluidised bed. The Dorrco valve was modified by the addition of a ½-inch length of ¾-inch diameter pipe to the body of the valve at the Tee end. Tests were conducted with one modified Dorrco valve at the bottom of the standpipe and another below the solids knockout vessel to replace the gas cock. In the latter case 3 feet of ¾-inch diameter "Perspex" standpipe was used between the knockout vessel and the valve.

For the final tests with the modified Dorrco valve, a 30^0 ¾-inch Y-piece which screwed to the bottom valve, replaced the T-piece and long sweep bend. Plastic tubing was used for transport air connections. This gave a more direct route to the entrained heater.

4. INVESTIGATION OF SEALING DEVICES

4.1 Experimental Procedures and Results

The operating procedure was designed to study the characteristics and effectiveness of the valves. The success of the unit would depend on the operation of these valves.

Suitable air flow rates and velocities were established in preliminary tests and maintained during the investigation. They were:

	Flow Rate
Main entraining air	192 $ft^3\ min^{-1}$
Transport air	34 $ft^3\ min^{-1}$
Total entraining air	226 $ft^3\ min^{-1}$
Velocity in entrained heater	43 $ft\ sec^{-1}$
Fluidising air	23 $ft^3\ min^{-1}$
Fluidising velocity	30 $ft\ min^{-1}$

Tests were conducted to determine the effect of the flow rate of the air used for aeration on the circulation rate of the solids in the system at various fluidised-bed heights. The weight of the solids circulating was determined by removing the return lines to the fluidised-bed unit and weighing the material collected for various times up to 10 minutes. The amount of solids collected from the cyclone only was approximately constant at 0.1 lb min^{-1} for aeration air flow rates of 1 to 10 litre min^{-1}.

The results of tests using a Dorrco valve in conjunction with steel and Perspex standpipes for fluidised-bed depths of 0.5 to 4 feet, are given in Tables 2 and 3, and are plotted in Figures 4 and 5. A series of tests were also conducted to test the ability of the Dorrco valve to seal effectively when the aeration air was shut off and also when both the aeration and the transport air were shut off. The results of these tests are given in Table 4. Tables 5 to 8 give the results of experiments to assess the suitability of other types of valves.

4.2 Discussion

4.2.1 Dorrco Valve

Figure 4 shows that for any particular fluidised-bed height the plot of aeration air flow rate against the solids circulation rate is best represented as an area between two limit curves, rather than a single definite curve. At flow rates greater than 4 or 5 litres per minute the circulation rate of solids tended to become constant. When the steel standpipe was used the value of this constant rate increased as the height of the fluidised bed was increased. The constant rate for a 4 feet fluidised bed was nearly twice that of a 3 feet bed and this was probably because the pressure at the top of the standpipe was greater than the pressure of the transport air at the valve. Hence fluidising air tended to go down the standpipe, through the valve, and into the entrained heater carrying solids with it. The flow of solids down the standpipe was much greater in this case than if it had been due to gravitational effects only.

The effect of using a different material for the standpipe can be seen by comparing Figures 4 and 5. The Perspex material apparently offered greater frictional resistance than the steel to the flow of solids down the standpipe, because the circulation rates were lower. The shape of the plots of Figures 4 and 5 were similar but the effect of increased fluidised-bed height was not confirmed.

The range of solids circulation rates obtained with the Dorrco valve, i.e., 0 to 20 pounds per minute made it suitable for use in the high temperature unit. The only doubt on this valve concerned its ability to shut off effectively when no aeration air was supplied to it but the transport air was left on. The results were confusing until the valve was modified.

4.2.2 Modified Dorrco Valve

This valve allowed only a trace of solids to circulate when no aeration air was supplied and transport air was kept on. Hence lengthening the valve had apparently had the desired effect of enabling the valve to shut off effectively. The maximum solids circulation rates obtained were similar to those obtained with the Dorrco valve and a Perspex standpipe, showing that this standpipe below the knockout vessel limited the flow of solids through the system.

The reduced transport air flow rate test showed that the velocity of the transport air could be reduced safely to about 33 feet per second in the 1 inch plastic line without seriously affecting the smooth circulation of the solids. At less than this velocity the solids tended to build up as slugs and the movement became erratic. The Y-piece on the Dorrco valve of the fluidised bed was a definite improvement, giving a much straighter line from the valve to the entrained heater, and eliminating several bends where abrasion had been severe.

The two extended runs showed that two Dorrco valves, one on the standpipe and the other below the knockout vessel, could be operated together to give a balanced circulation of solids. Once the system had settled down, alteration to the aeration air was necessary only every 2 hours. The extended runs also showed that the solids inlet bend in the bottom cone of the entrained heater could possibly be eliminated since the wear was not excessive during these tests. However the inlet should enter the cone at a fairly shallow angle to the vertical and an elbow with a hole at its corner might assist the air to entrain the solids before they abrade the opposite wall of the cone.

4.2.3 ICI Valve

The design of this valve was simple. An initial test revealed that the circulation rate was only 1 pound per minute and it was not investigated further. This value could be increased by moving the aeration air inlet further to the right (Figure 2 (b)).

The valve shut off effectively when no aeration air was supplied to it and the transport air was kept on.

4.2.4 Esso Type A Valve

The original valve failed to seal effectively. When no aeration air was supplied but the transport air was left on about $1\frac{1}{2}$ pounds per minute of solids circulated.

When the valve was modified (Section 3) only a negligible amount of solids circulated at no aeration air flow and the maximum rates were the same as before. The construction of this valve from high temperature ceramic materials would be difficult because the design is complicated.

4.2.5 Esso Type B Valve

This valve sealed more effectively than the Type A valve. A maximum circulation rate of 15 pounds per minute was obtained using a steel standpipe. The construction of this valve in ceramic high temperature materials would be difficult.

4.2.6 Plug Valve

The plug valve was the only mechanical valve tested. It performed fairly well and gave maximum solids circulation rates comparable with those of the Dorrco valve. It was limited by the flow of solids down the standpipe. In the fully open position it was liable to block suddenly and at all times the flow was erratic.

4.2.7 U-bend Valve

This valve required at least 3 litres per minute of aeration air irrespective of the fluidised-bed height, before any solids flow took place. For the first attempt on a 1 foot fluidised bed, the valve was packed too solidly for any solids flow to take place with up to 10 litres of air per minute. Only a large excess of aeration air emptied the outlet side of the valve and allowed it to refill with loosely packed solids. For fluidised-bed heights of less than 1 foot it was possible by supplying an excess of aeration air to stop the flow of solids by building up a large pressure in the bottom of the valve. The flow of solids throughout the tests was erratic and intermittent.

4.2.8 USS Venturi Valve

The Venturi valve was constructed from pipe fittings. The solids flow rates were low although the overflow standpipe used was $\frac{1}{2}$ inch in diameter. As long as there were some solids above the overflow tube, the solids circulation rates were not affected by the height of solids in the fluidised bed. The Venturi assembly showed signs of wear after 1 hour of testing. Electrostatic charging of the plastic tubing by the flowing solids had been observed previously and was greater during this test.

4.2.9 Abrasion

Serious abrasion of all curves and bends in the high velocity section between the valve and the entrained heater occurred during these tests. The plastic line was replaced six times. Its average life was 10 hours. A steel long-sweep bend endured nearly all the tests, but the steel elbow in the bottom of the entrained heater where the transport air and solids join the main entraining air abraded in 10-20 hours. In a high temperature refractory system this line must be straight and as short as possible to prevent such abrasion troubles. Elsewhere in the system abrasion did not appear to be serious.

The baffle of the knockout vessel was inspected and no sign of wear was observed. This design of the solids knockout vessel was therefore satisfactory for this system.

4.3 Conclusions

The Dorrco valve was the most suitable of those tested. Until the body of the valve was extended by $\frac{1}{2}$ inch the valve would not seal effectively. The modified Dorrco valve was therefore selected as the device to be used on the high temperature equipment.

The ICI and Esso valves sealed satisfactorily but their capacity was too low. Their construction from high temperature ceramic materials would be difficult.

The capacity of the plug valve was satisfactory but it was not sufficiently reliable for continuous operation. The U-bend valve behaved similarly. The operation of the Venturi valve was satisfactory but it would give a better performance at pressures above those anticipated in the pilot plant unit.

Abrasion of the bends and curves in the high velocity section of the model was very severe for both steel and plastic materials. The baffle of the solids knockout vessel showed no wear and the design of this unit was satisfactory.

5. COST ESTIMATE FOR THE ILMENITE CHLORINATION PROCESS

Previous cost estimates by Ketteridge and Nordin (1959) and Wilmshurst (1962), were modified to include the use of entrained heating for the fluidised-bed chlorinator. The recommendations of Ketteridge (1963) were used to determine the size of the chlorinator.

The size of the entrained heating equipment has now been determined, and an amended flow sheet, Figure 6, has been produced from that given by Wilmshurst.

It has been assumed that ilmenite will be processed at a rate of 100 tons per day. The titanium dioxide production was therefore 17,500 tons per year at Stradbroke Island and 18,900 tons per year at Capel, the tonnages being proportional to the grades of ilmenite.

Operating costs have been prepared for both Stradbroke Island and Capel plants using the respective ilmenites.

5.1 Capital Equipment Costs

The capital cost of the plant has been estimated by Method 3 in Aries and Newton (1955), using Lang factors. The purchased costs of the individual units were added to give the process equipment cost. This was then multiplied by 3.63 (Lang factor for solid-fluid process) to give the fixed capital cost. By adding the working capital (estimated to be 25% of annual product cost) the total capital was determined.

The unit costs were estimated from Aries and Newton. Factors of 4 and 6 were used to convert the purchased cost in mild steel to the cost in heat resistant stainless steel, and Inconel respectively.

Estimates of Winfield and Dryden (1962) were used to cost the fluidised-bed reactors. From a comparison of several standard items of equipment from the two sources an average factor of 1.32 was used to estimate the purchased cost of the item from the installed cost. Costs from both sources were brought up to date by the use of indices.

From Bauman (1962) the ratio of Australian equipment costs to USA costs was given as 0.86 and the exchange rate was given as 2.25 USA dollars to £A1 in 1961. Assuming the equipment cost ratio and the exchange rate were unchanged for 1965, the cost of equipment in \$A was given by the cost in USA dollars (\$) divided by 1.3.

5.1.1 Purchased Cost of Individual Items of Equipment

For location of the item, refer to the flow sheet (Figure 6).

1. Ilmenite Storage Hoppers

   Capacity of the two hoppers should be 1 week's supply, i.e. 350 tons or 5200 ft^3 each.
   Each hopper could be 15 ft square and 20 ft high with a square pyramid section 10 ft deep on the bottom.
   Allowing for ribbing of hoppers to take the high density ilmenite, purchase cost in 1965 = \$A20,000.

2. Ilmenite Elevator

   The elevator should be capable of handling at least 100 tons per day or 4¼ tons per hour continuously.
   The purchased cost of a spaced-bucket elevator (high speed centrifugal discharge) handling 14 tons per hour (100 lb/ft^3 material) was \$1800 in 1954 for a 100 ft lift. This elevator would handle about 20 tons per hour of ilmenite having a density of 150 lb per ft^3 and would operate intermittently for 5 hours per day.
   The estimated purchased cost in 1965 = \$A1800.

3. Ilmenite Weightometer and Belt Conveyor

   Ilmenite will be continuously weighed into the feed hopper from the elevator. The belt conveyor would be say 12 in. wide and 10 ft long. Purchased cost in 1965 = \$A800.
   From Riegel (1953) the estimated purchased cost of a weightometer for such a size belt conveyor was \$3000 in 1952.
   Purchased cost in 1965 = \$A3000.
   Total purchased cost in 1965 of conveyor and weightometer = \$A3800.

4. Ilmenite Feed Hopper

   The hopper should hold about 4 hours capacity, i.e. 16 tons of ilmenite. A hopper 6 ft in diameter and 13 ft deep including a 6 ft deep inverted cone would have this capacity.
   Purchased cost in 1954 was \$1700.
   Purchased cost in 1965 = \$A1800.

5. Ilmenite Screw Feeder

The capacity required is $4\frac{1}{4}$ tons per hour (9500 lb per hour), or 63 cu ft per hour. Considering ilmenite as a Class "e" material (Chemical Engineers' Handbook (1950), p 1345) a 12 inch diameter screw feeder running at about 12 rpm was required. Assuming a length of 10 ft and using steel, the purchase cost in 1954 was $420. For high temperature 310 alloy steel the cost would be $1680.
Purchased cost in 1965 = $A1800.

6. Fluidised-Bed Oxidiser

The size of this reactor was calculated by analogy with the chlorinator. Ketteridge (1963) estimated that a single stage fluidised-bed chlorinator treating 106.4 tons of oxidised ilmenite per day would have a capacity of 190 tons. Hence the average retention time in such a continuous reactor is 43 hours, but the batch retention time required to give a similar product was only 3 hours, i.e. ratio of about 14:1. Applying this ratio (which takes account of short circuiting in the continuous fluidised bed) to the oxidiser, the batch retention time for 95% oxidation is about $\frac{1}{2}$ hour, hence the average retention time must be about 7 hours in a single stage continuous unit.
The weight of the bed would be approximately $\frac{100}{24}$ x 7 or 29 tons.
For a fluidised-bed density of 17.2 tons per ft^3 (Ketteridge 1963) the bed volume is about 500 ft^3. An 11 ft diameter bed of height $5\frac{1}{4}$ ft would give this volume. The installed cost in 1965 of such a single stage fluidised bed reactor with brick lined steel shell would be $67,500.
Purchased cost of reactor in 1965 = $A40,000.
A cyclone should be added to this reactor. Assuming a gas velocity of 0.5 ft per second in the reactor, the flow rate of gas would be of the order of 3000 cfm.
The purchased cost in 1954 of a 3 ft diameter steel cyclone to handle this quantity of gas was $320. In heat resistant 310 alloy or refractory material with insulation lining the purchased cost in 1954 was $1280.
Purchased cost in 1965 = $1400.
Special oil injection burners for this reactor could cost an additional $A2000.
Total purchased cost in 1965 = $A44,000.

7. Rotary Airlock Feeder to Chlorinator

Costs of rotary airlock feeders were not available. However, from Winfield and Dryden (1962) the purchased cost in 1965 of a chamber chemical feeder handling 63 ft^3 per hour, was estimated to be $1770. This feeder is assumed to be constructed in mild steel. For high temperature application in a chlorine atmosphere the interior of the unit

would need to be ceramic coated, and the unit itself should be constructed from heat resistant 310 alloy.
Purchased cost in 1965 = $A8200.

8. Two Stage Fluidised-Bed Chlorinator
From Ketteridge (1963) a reactor for this application would have two stages arranged on the same horizontal plane, with the first stage 20 times the bed volume of the second. To give a suitable chlorine velocity through this reactor, a diameter of 19 feet would be required with a bed 4.8 ft deep. The installed cost of such a size single-stage fluidised bed reactor with a brick-lined steel shell in 1963 was $112,500. Although the required reactor was two stage, it was virtually only a single stage reactor with suitable partitioning and solids transfer devices. For this modification an addition of 20% was made to the cost.
Purchased cost in 1965 = $A80,000.
A cyclone for the effluent gases should be installed inside this reactor.
The total volume of gases at 1100°C is 6100 cfm.
The purchased cost in 1954 of a 4.5 ft diameter cyclone to handle this volume of gas was $550. Assuming this cost was for a steel cyclone then for a refractory unit the purchased price in 1965 would be $A2200.
Total purchased price in 1965 = $A84,000.

9. Rotary Airlock Feeder to Rutile Cooler
This cost should be the same as Item 7. Purchased cost in 1965 = $A8200.

10. Fluidised Bed Cooler
If this vessel is to be not only a solids cooler but also an air preheater then six stages would be necessary to preheat the air to 1000°C and to cool the solids to 180°C. Instead of a multi-stage reactor two other alternatives were considered:

    a. a baffled fluidised-bed reactor with a continuous temperature gradient down the vessel,

    b. a two-stage reactor with excess air in the bottom stage, and consequent rejection of much of this hot air and a top stage maintained at a higher temperature to give good preheating to the exit air.

The two-stage unit was selected. To achieve a compromise between obtaining hot air and cool product the temperature at the top and bottom stages were calculated. A temperature of 650°C in the top stage allowed outlet air to be discharged to the oxidisers at 650°C, and a temperature of 125°C in the bottom stage allowed discharge of the rutile at this fairly low temperature.

The throughput of air to the oxidiser and the bottom stage would be 2030 cfm at 650°C, and 5420 cfm at 125°C respectively. In an 11 ft diameter fluidised bed the gas velocity in the top stage was calculated to be 0.36 ft per second while that in the bottom stage was 0.95 ft per second which is a reasonable compromise to the recommended 0.5 ft per second. Using beds 4 ft deep in each stage the total gas retention time would be 15 seconds which should be sufficient for good heat transfer. The solids retention time will be several hours because of the large bed sizes. The installed cost of an 11 ft diameter two-stage fluidised-bed reactor with a brick lined steel shell in 1963 was $157,500. The bottom stage need not be brick lined, and say, 30% of the cost could be deducted, i.e. the installed cost in 1963 would be $110,250.
Purchased cost in 1965 = $A66,000.
A $2\frac{1}{2}$-ft cyclone capable of handling about 2030 cfm should be included. The purchased cost of a steel cyclone in 1954 was $230. The purchased cost of a heat resistant 310 alloy or refractory unit in 1954 was $920.
Purchased cost in 1965 = $A1000.
Total purchased cost in 1965 = $A68,000.

11. <u>Air Blower Supplying Cooler</u>
The purchased cost in 1954 of a rotary blower with a capacity of 4060 cfm at pressures from 10 to 15 psi, was $13,000.
Purchased cost in 1965 = $A13,000.

12. <u>Rotary Airlock from Cooler</u>
From Item 7 the purchased cost in 1963 of a mild steel rotary airlock was $1770. A mold steel airlock should be adequate because the temperature is only 125°C.
Purchased cost in 1965 = $A1400.

13. <u>Rutile Collection Hoppers</u>
These two hoppers should hold 1 week's production, i.e., 175 tons each. The density of the rutile product is about half that of the ilmenite feed, and the weight to be handled is also about half. Hence the same volume could be used for this application as for the ilmenite feed hoppers.
Total purchased cost in 1965 = $A20,000.

14. <u>Sealing and Feeding Device from Chlorinator to Entrained Heater</u>
A long standpipe with a Dorrco type gas operated valve may be used instead of a mechanical rotary airlock feeder.
Purchased cost in 1965 = $A1000.

15. Entrained Heater

Calculations based on heat balances showed that the entrained heater should be $3\frac{1}{2}$ ft in diameter and 25 ft high. This unit was considered to be similar to a fluidised-bed reactor.
A $3\frac{1}{2}$-ft diameter fluidised-bed reactor would be about 7 ft high so the entrained-heater unit is equivalent to about $3\frac{1}{2}$ fluidised beds with the bottoms, distributon plates and roofs removed. The installed cost in 1963 of a $3\frac{1}{2}$-ft diameter fluidised-bed reactor with brick lining was $21,600.
Then the installed cost of the equivalent of $3\frac{1}{2}$ fluidised bed units would be $76,000. After deducting say 60% for bottoms, distribution plates and roofs, and adding 50% for special inner lining of abrasion resistant refractory, the estimated purchased cost in 1965 = $A27,000.
Special oil burning equipment for the unit would cost $A6000.
Total purchased cost in 1965 = $A34,000.

16. Air Blower for Entrained Heater

This unit would require about 5000 cfm of air at room temperature against pressures of up to 10 psi. A centrifugal turbo compressor should be suitable.
The purchased cost in 1954 was $14,000.
Purchased cost in 1965 = $A14,000.

17. Solids Knockout Vessel

The purpose of this vessel is to remove from the gas stream the coarse solids from the entrained heater. It should be mounted directly above the entrained heater. The fine solids would be removed by cyclones. The unit would consist of a square sectioned baffled vessel with a right angled square pyramid on the bottom (to return the collected solids to the return line to the chlorinator). The calculated size of this vessel was 10-ft square by 16-ft high with the height of the pyramid another 16 ft.
An attempt has been made to cost the unit by analogy with cyclones. The size of the vessel was about equivalent to a cyclone between 10 and 11 ft in diameter having a capacity of 33,000 cfm.
The purchased cost in 1954 was estimated to be $2500.
Special abrasion resistent inner refractory lining with baffles pre-cast of a similar material would be required.
Total purchased cost in 1965 = $A20,000.

18. Cyclones for Collection of Fines from Entrained Heater

Three $5\frac{3}{4}$-ft diameter cyclones in parallel would handle the gas flow of 30,000 cfm. A single cyclone would have cost $820 in 1954.
The purchased cost of a refractory unit in 1965 would be $A5100.
Total purchased cost in 1965 = $A16,000.

19. Sealing and Feeding Device from Solids Collection Vessels to Chlorinator
As in Item 14 a Dorrco valve may be used. Purchased cost in 1965 = $1000.

20. Chlorine Reheater

The reheater would act as a high temperature heat exchanger, i. e. a refractory recuperator. One side would pass the hot exit gases from the cyclones of the entrained heating system while the other side would pass the gas to be heated, i. e. chlorine returning from the back reaction system to the chlorinator. The calculated temperature drop for the hot gas is from 1400 to about 1150°C and the calculated temperature rise of the chlorine from 440 to 1150°C.
The heat transfer area required was calculated to be over 4000 ft^2. The present cost of a recuperator similar to that described by Ketteridge and Nordin (1959) but with increase in the heat transfer area to 4000 ft^2 would be $A54,000. Using special high temperature refractory tubes, e.g. aluminous porcelain or mullite would increase the cost by say another 10%.
Purchased cost in 1965 = $A60,000.

21. Back Reactor
The back reactor unit described by Wilmshurst (1962) was a large chamber, 15 ft in diameter and 25 ft high into which the gas stream from the chlorinator and solid ferric oxide were introduced at the top. The solid would collect at the bottom of the chamber in a fluidised bed say 5 ft in diameter and the gases would pass upward through an annular disengagement zone to the gas cooler. The cost of this unit was estimated by anology with fluidised-bed reactors. In the back reactor both sensible and exothermic heat must be removed. Using the method of circulating ferric oxide as suggested by Wilmshurst (1962) the amount of heat to be removed and the weight of circulating ferric oxide can be calculated. For a production of 4 moles of TiO_2, the sensible heat in cooling chlorination gases from 1100-500°C = 134,000 cal and heat of reaction to be removed at 500°C =147,000 cal (Ketteridge and Wilmshurst (1963)).
The calculated amount of heat to be removed from the back reactor for 50 tons of TiO_2 per day, is 178 x 10^6 Btu per day. Heat taken out by 1200 cfm of chlorine at 500°C (56 tons/day) = 0.45 x 10^6 Btu per hour. Subtracting this quantity from the total heat to be removed, i. e. 7.42 x 10^6 Btu per hour, the heat to be taken out by the ferric oxide equals 6.97 x 10^6 Btu per hour. By balancing this heat against the sensible heat in "W" tons per day of ferric oxide heated from 100 to 500°C in the back reactor, W was found to be 519 tons.

This amount of ferric oxide must be circulated through the back reactor and ferric oxide cooler. The amount of chlorine used to raise this ferric oxide to the top of the back reactor must be calculated. Assuming 52-mesh ferric oxide with a density of 150 lb per cu ft, the choking velocity was calculated from Dallavalle's formula (Leva (1959)) to be a little over 10 ft per second. A solids loading of 1.0 appeared to be reasonable. At this loading the flow rate of chlorine was 432 cfm at 25°C (or 537 cfm at 100°C).

Then at 100°C in a 6-inch tube at gas velocity of about 45 ft per second, the solids velocities should be well above choking velocity.

The installed cost of the 5-ft fluidised-bed reactor with brick lined steel shell in 1965 would be $32,400 and the purchased cost in 1965 = $A20,000.

The installed cost of a main reactor chamber is estimated to be $109,000.

Purchased cost in 1965 = $A64,000.

Purchased cost in 1965 of complete reactor = $84,000.

A cyclone should be installed with this reactor. In addition to the circulating load of 167 tons of chlorine per day (3600 cfm at 500°C) there would also be fluidising gas (Cl_2) and chlorine conveying the recycle ferric oxide into the back reactor.

In a 5-ft diameter back reactor fluidised bed operated at 500°C, a quantity of about 1200 cfm would have a gas velocity of about 1 ft per second. Allowing another 1110 cfm at 500°C (535 cfm at 100°C) to convey the ferric oxide into the top of the back reactor through a 6-inch line at 100°C the gas velocity would be about 45 ft per second.

Thus total chlorine out of the back reactor would be

$$3600 + 1200 + 1110 = 5910 \text{ cfm at } 500^{\circ}\text{C}.$$

The diameter of the cyclone should be 4 ft to fit inside the back reactor. The purchased cost in 1954 for a mild steel cyclone of such a capacity = $550. Multiplying by a factor of 6 for cost of an Inconel cyclone = $3300. Then purchased cost in 1965 = $4230 or $A3264, say $A3400.

Thus the total purchased cost in 1965 = $A87,400 say, $A88,000.

22. Fluidised-Bed Gas Cooler

The purpose of this unit is to cool the exit gas from the back reactor to about 100°C for use in the fluidised bed ferric oxide cooler and for bleeding off 5% to the chlorine cleaning and recovery section of the plant. A large portion of this cooled gas is for use as a fluidising gas in the bottom of the back reactor and also to convey the ferric oxide to the top of the back reactor. The total volume of chlorine gas at 500°C was about 5910 cfm, or 2855 cfm at 100°C. At a gas velocity of about 1 ft per second the diameter of the cooler would need to be 8 ft. However, an 8 ft reactor will not give sufficient heat transfer area. For a 12-ft diameter reactor with a 6-ft deep bed the heat transfer area through the walls would be 228 ft^2, but the calculated total required transfer area is 414 ft^2. By use of say 20 water tubes, of 4-inch diameter across the bed, the equivalent of 190 ft^2 of heat transfer area could be gained. The gas velocity through the bed would now be only about 0.4 ft per second, hence the particle size would have to be kept to about minus 100 mesh for good fluidisation. The installed cost of a 12 ft diameter fluidised-bed reactor with an alloy-steel or a brick-lined steel shell in 1963 was $80,000 x 0.9 = $72,000 (Winfield and Dryden (1962)).

Because the chlorine temperature would be between 100 and 500°C, the reactor should be constructed of a metal such as Inconel for good heat transfer. The cost would be approximately 50% higher than that of high temperature alloy steel or refractory and insulation lined steel, i.e. cost would be $108,000.

The estimated purchased cost in 1965 = $\frac{\$108,000}{1.32}$ = $81,800 say, $A64,000.

This vessel must be jacketed to allow cooling water to keep the bed at 100°C. Add on about another 25% for jacketing the vessel, and adding the water tubes.

Purchased cost in 1965 = $A80,000.

In 1954 the cost of a 3-ft diameter mild-steel cyclone to treat 2855 cfm of chlorine gas at 100°C would probably be $310. Mild steel should be suitable for dry chlorine.

Purchased cost in 1965 = $A400.

Thus total purchased cost in 1965 = $A80,400 say, $A82,000.

23. <u>Blower for Chlorine Leaving Back Reactor</u>

The blower is required to deliver 5910 cfm of chlorine gas at 500°C against a pressure of about 10 psi. The purchased cost of a mild steel centrifugal turbo blower of this capacity and pressure rating was $16,000 in 1954. Constructed from Inconel the unit would cost $96,000.

Purchased cost in 1965 = $A96,000.

24. <u>Rotary Airlock from Gas Cooler</u>

This unit may be smaller than the one described in Item 7 and the purchased cost could be reduced to say, $1000. For handling dry chlorine, mild steel could be used.

Purchased cost in 1965 = $A800.

25. Chlorine Bleed and Purifying System

In a cyclic system 5% of the circulating gas stream should be bled off (to keep down the build up) of such gases as nitrogen, oxygen, oxides of carbon and hydrogen chloride. The chlorine in this bleed can be recovered. The following items would be required:

a. Compressor. A Nash Hytor type cast iron unit capable of compressing 115 cfm at room temperature to about 100 psi would be suitable. To cool the chlorine gas to room temperature a heat transfer area of about 140 ft^2 would be required. For a water jacketed heat exchanger, the 1965 purchased cost of the mild steel unit would be $A2000.
No accurate cost data was available on the Nash Hytor compressor, but a cost was estimated for a similar unit working as a vacuum pump. The purchased cost in 1954 of such a unit was $2900. A cast iron construction should be adequate, but a higher pressure unit may cost say, 50% more. Purchased cost in 1965 = $A4400.
Purchased cost for cooler and compressor in 1965 = $A6400.

b. Condenser. The complete unit would consist of a shell and tube condenser and a refrigeration system capable of cooling to minus 50°F. The calculated heat transfer area of the condenser was 108 ft^2. A condenser should be available for $A1400. A plant to remove 177,000 Btu per hour, requiring 15 tons of refrigeration was estimated to cost $A30,000.
Purchased cost of condenser and refrigeration plant in 1965 = $A32,000.

c. Chlorine Vaporizer. The vaporizer for injecting the separated liquid chlorine into the main gas stream again could be similar to the condenser in part "b" but with hot water as the heating medium. The calculated heat transfer area is 170 ft^2.
Purchased cost in 1965 = $A1600

Purchased cost of complete chlorine bleed and purifying system in 1965 = $A40,000.

26. Rotary Airlock to Ferric Oxide Cooler

The airlock would be required to handle 24 tons per hour of ferric oxide at 500°C in a chlorine atmosphere. At 150 lb per ft^3 of ferric oxide the required throughput would be 358 ft^3 per hour. The capacity of the airlock feeder described in Item 7 would probably be 60 ft^3 per hour when operating at about 1 rpm. A similar sized unit could be used rotating at 6 rpm.
Purchased cost of a unit made of Inconel would be $A8200.

27. Fluidised Bed Ferric Oxide Cooler, Chlorine Heater
The circulating ferric oxide (519 tons/day) and the ferric oxide product (50 tons/day) would be cooled from 500°C to 440°C by the chlorine gas sent back to the chlorinator. The chlorine gas would be heated from 100°C to 440°C. If the gas velocity was assumed to be 1 ft per second for 3320 cfm at 440°C then the diameter of the cooler would need to be just over 8 ft. The depth of bed was taken as 4 ft.
Purchased cost of a fluidised bed reactor with a brick lined steel shell, in 1965 = $30,000.
The cost of a $3\frac{1}{2}$ ft diameter cyclone to handle 3320 cfm of chlorine must be included also.
The purchased cost of a cyclone of this capacity in mild steel in 1954 was $340.
Purchased cost of an Inconel cyclone in 1965 = $A2000.
Total purchased cost in 1965 = $A32,000.

28. Rotary Airlock to Air Cooled Ferric Oxide Cooler
This unit has a similar duty to Item 26 except that the ferric oxide is slightly cooler, and one side is exposed to air.
Purchased cost in 1965 = $A8200.

29. Fluidised Bed Air Cooled Ferric Oxide Cooler
This unit would cool the ferric oxide from 440°C to 100°C for recirculation (519 tons/day) and then discharge it as by-product (50 tons/day). Air would be used as the cooling medium and this would enter at 25°C and leave at 100°C. The volume of air at 25°C would be 43,000 cfm or 53,500 cfm at 100°C. A 20 ft diameter unit with a bed 5 ft deep was chosen giving a gas velocity of about 2.8 ft per second. The installed cost of a 20 ft diameter fluidised bed in either alloy steel or in brick lined mild steel in 1965 would be $117,000 (Winfield and Dryden (1962)). Since this unit need not be brick lined deduct say 60% from this cost.
Purchased cost in 1965 = $A28,000.
The cost of 4 cyclones of $6\frac{1}{2}$ ft diameter in parallel to handle 53,500 cfm must be included.
The purchased cost of a $6\frac{1}{2}$ ft cyclone in 1954 was $1000.
Total purchased cost of cooler and 4 cyclones in 1965 = $A32,000.

30. Rotary Airlock Discharge from Ferric Oxide Cooler
The function of this unit is similar to that of Item 26 except that chlorine is not present. Hence a mild steel unit should suffice.
Purchased cost in 1965 = $A1400.

31. Air Blower for Ferric Oxide Cooler

The capacity of the air blower has been calculated to be 43,000 cfm against a pressure of 3 to 4 psi.
The purchased cost of a centrifugal turbo blower in 1954 was \$53,000.
Purchased cost in 1965 = \$A54,000.

32. Dry Grinding Mill

The recycle ferric oxide must be ground dry at 100°C. Probably the most suitable mill for this application would be a 6 ft diameter by 12 ft rod mill operating in open circuit to give a product with a minimum of fines. For a capacity of 520 tons per day the purchased cost of a suitable ball mill in 1954 was \$58,000.
The purchased cost in 1965 = \$A58,000.

33. Rotary Airlock from Grinding Mill

A unit similar to Items 26 and 28 would be suitable.
Purchased cost in 1965 = \$A8200.

34. Ferric Oxide Collection Hopper

Assuming that the bulk density of the ferric oxide is about the same as the ilmenite, i.e. 150 lb per ft^3, then 1 week's production i.e, 350 tons should be held in a hopper similar to Item 1.
Purchased cost in 1965 = \$A10,000.

35. Blower for Chlorine Returning to Chlorinator

The blower would be required to handle 167 tons per day or 3320 cfm at 440°C against a pressure of 10 psi. The purchased cost of a steel centrifugal turbo blower of this capacity and pressure rating was \$11,500 in 1954. Constructed from Inconel the purchased cost would be \$69,000.
Purchased cost in 1965 = \$A68,000.

36. Total Capital Investment

The purchased costs of the individual items of equipment as summarised in Table 9 amount to \$A1,008,800. Multiplying this figure by Lang's factor of 3.63 gives an estimated total fixed capital of \$A3,660,000. For working capital, 3 months operating expenses was allowed. The manufacturing cost has been calculated to be \$A106 per ton.
Estimated working capital = \$A464,000.
Estimated total capital investment = \$A4,124,000.

5.2 Operating Costs

For estimating the operating costs per ton of rutile product it was assumed that Capel ilmenite contained 54% TiO_2 and that the Stradbroke Island concentrate contained 50% TiO_2. The operating costs were assessed on the basis of treating 100 tons of either material per day at their respective locations.

5.2.1 Raw Materials

Ilmenite, oxygen and chlorine were considered under this heading. As the oxygen content in the gas streams was low, little would be lost. However, the oxidation of ilmenite was assumed to be only 95% complete and additional oxygen must be added to the back reactor to maintain the ferric chloride; oxygen ratio in the stoicheiometric proportions.

The deficiency represented 0.15 tons or 4000 ft^3 per day for Capel ilmenite containing 26.5% FeO and 17% Fe_2O_3. For the Stradbroke Island ilmenite containing 31.5% FeO and 14.5% Fe_2O_3 the deficiency was 0.18 tons or 4800 ft^3 of oxygen per day at standard conditions.

To compensate also for the mechanical loss of chlorine, a make up of 1% or 1.67 tons per day of the chlorinating gas has been assumed for Capel ilmenite.

For Stradbroke Island ilmenite an additional loss could occur due to fixation, as this material may contain up to 1.6% of manganous oxide and 0.2% Cr_2O_3. The extra chlorine required was estimated at 1.28 tons per day and the total make up chlorine for Stradbroke Island ilmenite would be 2.95 tons per day.

The estimated material costs at Capel and Stradbroke Island are given in Tables 10 and 11.

5.2.2 Labour

The estimated cost of labour at Capel and Stradbroke Island is $A3.2 and $A3.4 per ton TiO_2 (Wilmshurst (1962)) at the adjusted production rates.

5.2.3 Fuel

The fuel oil required for the oxidiser is 0.45 tons per day The heat from the exothermic reaction and the sensible heat in the preheated fluidising air should be almost sufficient to heat the ilmenite to 1000°C. The entrained heater would require 17.1 tons per day of fuel oil. Hence the total fuel requirement is 17.55 tons per day.

At $A25.4 per ton of oil (Wilmshurst (1962)) the fuel cost at Capel is $A8.2 per ton TiO_2. Assuming the same price, the fuel cost at Stradbroke Island is $A8.8 per ton TiO_2.

5.2.4 Power

The installed horsepower for each item is summarised in Table 12. Assuming a load factor and efficiency of 80% and a cost of 1.5 pence per unit, the power costs at Capel and Stradbroke Island are $A10.8 and $A11.8 per ton TiO_2 respectively.

5.2.5 Depreciation and Maintenance

These costs have been taken as 15% and 10% per annum respectively of the fixed capital costs which have been assumed to be the same for each plant.

The annual charges are $A916,000 and the depreciation and maintenance cost per ton TiO_2 is $A48.4 at Capel and $A52.4 at Stradbroke Island.

5.2.6 Miscellaneous

Miscellaneous costs cover interest, rates and taxes, stores, etc., and have been assumed to be 5% per annum of total capital cost, or $A11.0 and $A11.8 per ton TiO_2 at Capel and Stradbroke Island respectively.

The operating costs are summarised in Table 13.

No credits have been assigned to the ferric oxide or to the waste heat (Wilmshurst (1962)).

5.3 Discussion

5.3.1 Capital Costs

This estimate of capital costs is much higher than all previous estimates. The 1963 cost of the 6 major items considered by Ketteridge and Nordin is only 80% of the cost of the corresponding units in the present report. The flow sheet incorporating Wilmshurst's modified form of back reactor system and the entrained heater and its ancilliary equipment has been drawn out and the extra items of equipment and the larger size of the units have added considerably to the capital costs. The use in the present report of a recent article by Winfield and Dryden (1962) gave more accurate costs than were available previously. Hence the current estimates for the fluidised bed reactors are higher than those previously reported.

Certain items, e.g. feeders and blowers, should be constructed from heat resistant stainless steel or Inconel and this resulted in the cost of such items being higher than the previous estimate.

5.3.2 Operating Costs

The estimated operating costs for plants located at Capel and Stradbroke Island are $A102 and $A106 per ton of titania respectively. At the present price of rutile, $A80 per ton[a] the process is not an economic proposition.

The major items in the operating cost are depreciation and maintenance. A risk would be taken if these costs were reduced because the operating conditions are expected to be severe. The availability of cheap chlorine on the site would reduce the operating costs slightly at Stradbroke Island but would have little effect at Capel. Power consumption has been estimated to be higher than in the previous estimate, due to the use of more and larger blowers. The miscellaneous charges at 5% of the total capital are high. However, Wilmshurst used 10% of the total capital for these costs.

The small credits for waste heat and ferric oxide allowed by Wilmshurst have not been deducted as it is doubtful if these could be obtained.

(a) Australian Mineral Industry Quarterly Review, December, 1963.

A price of $A70-80[a] per ton for rutile may be close to the economic maximum for making titania pigment via the chloride process, and for the proposed process to be economical the markets may be restricted to the traditional outlets, e.g. metal and welding rod manufacture.

A rutile price of $A100 per ton would yield a return of 6-8% on the total capital investment. If other processes to convert ilmenite to rutile undercut these prices then the proposed process would not be economical. Whether the rutile product would command a higher price than the less pure natural rutile is not known.

5.3.3 Possible Cost Reductions

It should be noted that little process optimisation has been carried out on the suggested flow sheet. A reasonable flow sheet has been costed to give the order of capital and operating costs likely to be incurred if a plant were built to this design. By eliminating certain items of equipment the capital cost, and hence the depreciation and maintenance charge, could be reduced considerably, but the operating cost would not be affected greatly.

Elimination of the cooling of the chlorine and ferric oxide from the back reactor and the associated major Items 22, 27, 29 and 31, and other various rotary airlocks, would save capital of about $A800,000. On the debit side a much larger quantity of ferric oxide would be needed to allow the circulating ferric oxide to air cool slowly in heaps on the grounds. Also materials handling equipment would be required to collect the ferric oxide for recirculation and elevation to the top of the back reactor. The cost of these extra storage facilities and handling equipment may not amount to more than $A200,000. A net saving of the order of $A600,000 could be made at the expense of a larger area of land and possibly higher labour costs. The total operating costs would be reduced by about $A6 per ton of titania by this alteration.

Elimination of the product cooler, Item 10, and allowing the product to air cool on the ground, would save capital, but this would contaminate the product and make difficult the handling of the hot (1100°C) product. Covered concrete aprons could be used to store the product while it is allowed to cool. This would save capital of about $A240,000 but the cooling storage facilities and materials handling equipment could cost about $A80,000 so that the net saving would be $A160,000 or $A2 per ton of titania. However, the oxidiser, Item 6, would use more fuel as the air is not preheated. This additional fuel oil cost may be of the order of $A0.2 per ton of titania.

One other possible saving is the recovery of the heat in the gases from the oxidiser and chlorine reheater in a waste heat boiler. By using this heat for power generation it may be possible to reduce the power costs by up to $A6 per ton of titania.

The labour figure used in the estimate was rather low. Since maintenance has been listed separately, labour should include operating personnel only. For 50 tons per day production, the plant may require 0.8 man hours per ton per step (Aries and Newton (1955)). For five main steps this gives the labour requirement as 4.0 man hours per ton or 200 man hours per day, i.e. about 8 men continuously on shift. The operating labour should consist

(a) Australian Mineral Industry Quarterly Review, June, 1965.

of a superintendent at say $A100 per week, 4 leading hands at say $A60 per week and 32 operators at say $A40 per week, giving a total of $A1620 per week. Allowing 50% extra for long service, annual and sick leave, public holidays, overtime, etc, the total becomes $A2430 per week. The labour costs at Capel and Stradbroke Island would then be $A6.4 and $A7.0 per ton of titania. The increase is about $A3 per ton of titania over the figures used in the estimate.

The total saving from the above considerations could be of the order of $A760,000 in capital excluding the cost of boilers and power generating equipment. A saving of about $A11 per ton of titania may be possible.

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APPENDIX A

LITERATURE SURVEY

Entrained Heater for Fluidised Beds

1. INTRODUCTION

As the first step towards the investigation of such a system for heating fluidised beds, a literature survey was conducted. The results of the survey will be discussed under the following three headings:

1. Various fluidised bed catalytic and non-catalytic processes used mainly in the petroleum refining industry. These all involve the transport of solids between reactors, regenerators and heaters, all of which are either entrained state or fluidised beds.
2. Details of the various sealing devices used to control the rate of solids transfer from one vessel to another in such systems. The operating temperature (up to 1200^{0}C) precludes the use of most mechanical type valves.
3. Correlations of theory and practice in the transport of solids by gases. Such information is necessary for design purposes.

The literature survey included various text books and also Chemical Abstracts (1947 to the first half of 1962 inclusive). Chemical Titles (second half of 1962 and first half of 1963 inclusive) were used to bring the survey up to date.

2. ENTRAINED STATE AND FLUIDISED BED PROCESSES INVOLVING TRANSPORT OF SOLIDS

Turner[1] discusses various types of fluid catalytic crackers used in petroleum refining. Details are given of pressure control of a conventional fluid catalytic cracker consisting of a reactor and regenerator side by side.

Sittig[2] outlines the evolution of fluid cracking units from the wartime models through to the Model IV unit which incorporates U-bend catalyst transport lines between the reactor and regenerator.

In the UOP, Orthoflow and Model IV catalytic cracking processes[3] the flow of catalyst is controlled by slide valves, special plug valves and U-bends respectively.

The Lurgi-Ruhrgas sand cracker[4, 5, 6] does not use a catalyst, but inert coarse sand is used as a heat exchange material. The sand is heated and cleaned in an entrained state reactor by combustion of contaminating carbon and then dropped into a fluid-bed reactor where it supplies the heat of reaction for the cracking of oil vapours to olefins. Blanding[7] describes a very similar process.

Murphree et al[8] discuss the relative merits of "upflow" or entrained state unit, and "downflow" or fluid bed cracking processes. The main advantages of the fluid bed unit are the much smaller dust collection system and the ease of control of solids hold-up. Hence the capital cost of the fluid-bed process should be less than the entrained-state system.

Resen[9] describes in detail the Model IV fluid catalytic cracking unit which operates at a velocity of $2\frac{1}{2}$ feet per second compared with $1\frac{1}{2}$ in earlier models. Reference is made to the U-bend lines connecting the reactor and regenerator, and to the two stage dust collection cyclones built inside these vessels. Wickham[10] briefly discusses the Orthoflow catalytic cracking process.

3. DETAILS OF SEALING DEVICES FOR SOLIDS TRANSFER CONTROL

Turner[1] discusses various types of valves for controlling the removal of solids from fluid beds. Both mechanical, e.g. slide and plug valves, and non-mechanical, e.g. U-bend and ICI valves are discussed. Sittig[2] mentions briefly such devices when discussing the use of solid feeders. A detailed study by Olive[11] mentions the above types and screw and rotary vane feeders. Schnacky[12] claims that his shredder-seal feeder combines the features of the rotary vane feeder and the choke screw conveyor.

One method[13] of circulating solids from one high temperature fluid-bed reactor to another and back again without contamination of the respective gases involves varying the pressure of one reactor first above and then below the fixed pressure of the second reactor. The only apparent disadvantages of this process are the variation in temperature and bed depth within the reactors and the selection of suitable inert purge gases for any particular system.

The ICI valve[14] and the Dorr Company valve[15,16] are non-mechanical flow control devices which utilise the natural angle of repose of the unfluidised solids to halt the flow of solids. When a small amount of aeration gas is supplied to the valve, the angle is lowered and flow of solids can be regulated.

Other designs of non-mechanical valves are the special transfer wells of Esso Research and Engineering Co.[17] A small amount of aeration gas supplied to solids in the well enables the solids to flow down one tube and up an adjacent tube.

Other methods are the USS Venturi system[18] which can be used to transfer solids from a low pressure to a higher pressure fluidised bed, and the Esso Research and Engineering Co. U-bend system[19], which operates on a similar principle to the Esso transfer wells.

The following US patents which were noted during the survey are not so readily applicable as the previous methods. Two patents,[20,21] refer to dense phase conveying of solids by gases, while another[22] gives details of a hollow stem plug valve to control the flow of solids between two fluidised beds. Two more[23,24] use a differential in bed density caused by different gas velocities in the two beds, and show how solids can be transported from one fluidised bed to another. Finally, one patent[25] uses a second slide valve in the solids standpipe to reduce valve erosion, and another [26] gives details of the use of steam and air as aeration gases in the standpipe itself of a catalytic cracking regenerator unit.

4. TRANSPORT OF SOLIDS BY GASES – CORRELATIONS, THEORY AND PRACTICAL DESIGNS

A number of papers reported experimental work on the transport of solids by gases and compared the results with theoretical data. Farbar[27] describes the use of flow nozzles and Venturi tubes[28] to meter gas-solid mixtures in horizontal and vertical flow. In both devices the gravimetric gas flow must be constant for the various derived relations to be correct. Leva[29] summarises a number of papers[30, 31, 32, 33] on the pressure drop for the pneumatic conveying of solids. Wilhelm and Valentine[34] showed that by properly adjusting solids and gas flow rates, the whole fluidised state spectrum from the dense-phase bed through the dispersed phase into pneumatic transport could be produced. Zenz[35, 36] presents a fluidised-state phase diagram for the above entire range of superficial gas velocities. Correlations to determine saturation carrying capacity for pneumatic conveying of solids are also given by Zenz[36] together with velocities necessary to prevent saltation and chocking of solids in horizontal and vertical tubes respectively. Recent articles[37, 38, 39] give additional correlations for the determination of pressure drops in pneumatic conveying of solids.

Practical designs of pneumatic solids conveyors based partly on theory and partly on an empirical approach are given by Michell[40], Hudson[41] and Fischer[42]. A patent[43] shows how a vessel to "knock-out" solids from the gas stream of a pneumatic lift is designed to eliminate right angle bends in the lead-in line, and to reduce maintenance costs. Several articles by Fischer[44, 45] and Reta[46] discuss pneumatic conveying systems for the application of particles of plastic in petrochemical plants.

5. CONCLUSIONS

5.1 Advantages and Disadvantages of Entrained State Heater

Compared with a fluidised-bed heater, the entrained-state heater has certain advantages and disadvantages. The greatest advantage is the high gas velocities (up to 100 feet per second) involved. The high solids velocities are especially important when the temperature employed is close to the sintering temperature of the solids. Another advantage of the entrained-state heater is that it elevates the solids to a suitable height for gravitation into the fluidised bed reactor. It occupies less floor space than a fluidised-bed heater but is more elevated which involves more costly steelwork. Due to the higher gas velocities direct firing may be more difficult for the entrained-state heater than for a fluidised-bed unit. The entrained-state heater can be bottom fed for sintering solids.

The entrained-state heater has few disadvantages. The high velocity solids will cause considerable wear on the walls and bends of the unit, and the preliminary collectors and cyclones must be large enough to collect the entire solids flow. A low velocity fluidised bed heater requires little dust collection equipment. The method of circulating solids from a fluidised bed reactor to a heater and back again by controlling the pressure of one unit alternately above and below the other can only be used if the heater is a fluidised bed.

Summarising, the entrained state heater appears to be the more suitable unit for this application, particularly since it may be required to operate at temperatures approaching the solids sintering temperature.

5.2 Selection of Sealing Devices for Solids Transfer Control

As a result of this survey, sealing devices have been selected for control of solids transfer for the entrained-state heater. In all but one case non-mechanical valves have been selected, as they are more simply constructed in high temperature refractory materials than mechanical valves.

Both the ICI and Dorr Company valves are simple in design and should be the first choice for construction in refractory materials. The Esso Research and Engineering Co. non-mechanical transfer wells may shut off more efficiently but are more intricate in design. Both the USS Venturi system and the Esso Research and Engineering Co. U-bend system were tested but both are considered to be inferior to the above valve system. The one non-mechanical valve to be tested is a plug valve similar to the Dorr Company cone valve and should operate at higher temperatures than other mechanical valves but is unlikely to be suitable for use above 900-1000°C.

5.3 Correlations, Theory and Practical Design in Pneumatic Transport Systems

Although a number of papers have been published giving correlations for the transport of solids by gases, in most cases these correlations hold only for the particular system being tested. The theories of different authors do not agree, especially regarding the pressure drops likely to be encountered in such systems, nor do they all fit the experimental results (of the various studies which have been made). The most useful correlations are the saturation carrying capacity of gases and the saltation and chocking gas velocities in horizontal and vertical tubes respectively. Probably pressure drops are best predicted from results of model work or from small scale direct measurements. Several authors presented practical methods based partly on theory and partly on empirical relations for the design of systems for conveying solids by gases. However, most agreed that designs of such systems still rely to a considerable extent on experience and are still largely an art rather than an exact science.

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TABLES 1 TO 13

FIGURES 1 TO 6

TABLE 1: SCREEN ANALYSIS OF RUTILE

Mesh BSS	Weight %
+ 52	0.5
- 52 + 72	29.95
- 72 + 100	61.5
- 100 + 150	7.9
- 150 + 200	0.1
- 200	0.05
	100.0

TABLE 2: SOLIDS CIRCULATION RATES
Expressed as pounds per minute

Aeration Air litres min^{-1}	Bed Heights 0.5 ft	1 ft	2 ft	3 ft	4 ft
	Dorrco Valve, Steel Standpipe				
0.1	0.4	0.4	0.4	0.4	0.6
1	0.6-3	2.5	4	4-7	7
2	4-7	7-10	12	11-13	16
3	7-12	12-16	13-17	14-19	35
4	12-17	17	12-19	18-22	36
5	15	16	18	20	nd(a)
6	15	16	18	20	35
8	15	16	18	20	36
10	15	16	18	20	35
	Dorrco Valve, Perspex Standpipe				
1	1.5	nd	nd	2.5	-
2	3	nd	nd	5	-
3	5	8	nd	10	-
4	12	14	13	10	-
5	13	14	14	10	-
6	13	14	14	10	-
8	13	14	14	10	-
10	13	14	14	10	-

(a) nd - Not determined.

TABLE 3: SOLIDS CIRCULATION RATES
Expressed as Pounds per minute

Aeration Air litres min^{-1}	Bed Heights		
	0.5 ft	1 ft	2 ft
	Modified Dorrco Valve, Perspex Standpipe		
0.1	trace	trace	trace
1	1	1	1-2
2	4-7	3-4	5-6
3	7-9	9-10	5-9
4	11-12	9-11	6-7
6	11-12	12	7-10
8	12-13	11-12	7-14
10	13	12-13	7-13

Note: In the experiments from which the above results were obtained, the solids circulation rate was insufficient to seal the perspex standpipe. It was necessary to use excess aeration air to prevent the solids from being blown down the aeration air inlet line.

TABLE 4: SOLIDS CIRCULATION RATES

Valve Position	Fluidised Bed Height ft	Circulation Rate lb min^{-1}
	Dorrco Valve, No Aeration Air	
Horizontal	1	0.03-0.30
	2	0.03-0.30
Valve body lengthened by $^1/_{16}$ inch.	1	0.01
	2	0.02-0.70
As above but slightly inclined upwards.	0.5	trace
	1	trace
	2	trace
	Dorrco Valve, No Aeration or Transport Air	
	0.5	trace
	1	trace
	2	trace

TABLE 5: SOLIDS CIRCULATION RATES
Expressed as pounds per minute

Aeration Air litres min^{-1}	Bed Height ft	Valve Types, Steel Standpipe ICI	Esso A	Modified A	Esso B
0.1	0.5	0.02	1.7	0.1	Nil
1	0.5	0.4	1.9	nd[a]	2
2	0.5	0.4	2.1	0.1	4
3	0.5	0.8	2.5	nd	5-8
4	0.5	0.8	2.9	0.1	7-10
6	0.5	nd	nd	nd	13
10	0.5	0.6	2.9	3.6	12-18
10	1	nd	nd	1-4	14
10	2	0.9	6.5	4.5	15
Nil	2	nd	1.8	0.1	Nil

(a) nd - Not determined.

TABLE 6: SOLIDS CIRCULATION RATES
Expressed as pounds per minute
Plug Valve

Valve Position	Bed Heights 0.5 ft	1 ft	2 ft
Closed	Nil	trace	trace
¼ open	0.1	0.5-1	0.7-1.3
½ open	2-3	2-3	4-7
¾ open	10-13	10-12	12-13
Fully open	16	16-18	19-21

TABLE 7: SOLIDS CIRCULATION RATES
Expressed as pounds per minute
U-Bend Valve

Aeration Air litres min^{-1}	Bed Height		
	0.5 ft	1 ft	2 ft
0.1	Nil	Nil	Nil
1	Nil	Nil	Nil
2	Nil	Nil	Nil
3	0.1-0.6	0.06-0.3	0.5
4	1	1-2	1-2
5	4-5	3-7	6-8
6	9-12	11-17	17
7	4-7	6	nd[a]
8	5-8	6	8-9
10	5-6	6-8	9-10
10	Nil	Nil	nd

(a) nd - Not determined.

TABLE 8: SOLIDS CIRCULATION RATES
Venturi Valve

Fluidised Bed Height		Circulation Rate
Unexpanded in.	Expanded in.	lb min^{-1}
26	29.9	3
25	28.8	3
24	27.6	3-4
23	26.5	3
22	25.3	0.4-2.5

TABLE 9: SUMMARY OF PURCHASED COST OF INDIVIDUAL ITEMS OF EQUIPMENT

Item No.	Name of Item of Equipment	Estimated Purchase Cost $A
1	Ilmenite storage hoppers	20,000
2	Elmenite elevator	1,800
3	Ilmenite weightometer and belt conveyor	3,600
4	Ilmenite feed hopper	1,800
5	Ilmenite screw feeder	1,800
6	Fluidised bed oxidiser	44,000
7	Rotary airlock feeder to chlorinator	8,200
8	Two-stage fluidised-bed chlorinator	84,000
9	Rotary airlock feeder to rutile cooler	8,200
10	Rutile product fluidised bed cooler	68,000
11	Air blower supplying rutile cooler	13,000
12	Rotary airlock from rutile cooler	1,400
13	Rutile collection hoppers	20,000
14	Sealing and feeding device from chlorinator to entrained heater	1,000
15	Entrained heater	34,000
16	Air blower for entrained heater	14,000
17	Solids knockout vessel above entrained heater	20,000
18	Cyclones for collection of fines from entrained heater	16,000
19	Sealing and feeding device from solids collection vessel to chlorinator	1,000
20	Chlorine reheater	60,000
21	Back reactor	88,000
22	Fluidised bed gas cooler	82,000
23	Blower for chlorine leaving back reactor	96,000
24	Rotary airlock from gas cooler	800
25	Chlorine Bleed and Purifying System	40,000
26	Rotary airlock to ferric oxide cooler	8,200

(contd.)

TABLE 9: CONTINUED

Item No.	Name of Item of Equipment	Estimated Purchase Cost $A
27	Fluidised bed ferric oxide cooler, chlorine heater	32,000
28	Rotary airlock to air cooled ferric oxide cooler	8,200
29	Fluidised bed air cooled ferric oxide cooler	32,000
30	Rotary airlock out of air cooled ferric oxide cooler	1,400
31	Air blower for air cooled ferric oxide cooler	54,000
32	Dry grinding mill	58,000
33	Rotary airlock from grinding mill	8,200
34	Ferric oxide collection hopper	10,000
35	Blower for chlorine returning to chlorinator	68,000
		Total $A1,008,800

TABLE 10: RAW MATERIAL COSTS
Capel

Material	Quantity/day	Price	Cost per Ton TiO_2
Ilmenite	1000 tons	\$A8/ton[a]	\$A14.8
Oxygen	4000 ft^3	\$A6.88/1000 ft^3[b]	\$A 0.6
Chlorine	1.67 tons	\$A158.6/ton[b]	\$A 5.0
		Total	\$A20.4

(a) Australian Mineral Industry Quarterly Review, December, 1963.
(b) Wilmshurst (1962).

TABLE 11: RAW MATERIAL COSTS
Stradbroke Island

Material	Quantity/day	Price	Cost per Ton TiO_2
Ilmenite	100 tons	\$A2.50/ton[a]	\$A 5.0
Oxygen	4800 cu ft	\$A6.88/1000 cu ft	\$A 0.6
Chlorine	2.95 tons	\$A207.2/ton[a]	\$A 12.2
		Total	\$A17.8

(a) Wilmshurst (1962).

TABLE 12: INSTALLED HORSEPOWER OF ITEMS OF EQUIPMENT

Item	Estimated Installed Horsepower
Ilmenite elevator	4
Ilmenite weightometer and belt conveyor	1
Ilmenite screw feeder	2
Rotary airlock feeder to chlorinator	2
Rotary airlock feeder to rutile cooler	2
Air blower supplying rutile cooler	440
Rotary airlock from rutile cooler	2
Air blower for entrained heater	285
Blower for chlorine leaving back reactor	370
Rotary airlock from gas cooler	2
Chlorine bleed and purifying system	120
Rotary airlock to ferric oxide cooler	2
Rotary airlock to air cooled ferric oxide cooler	2
Rotary airlock out of air cooled ferric oxide cooler	2
Air blower for air cooled ferric oxide cooler	920
Dry grinding mill	150
Rotary airlock from grinding mill	2
Blower for chlorine returning to chlorinator	200
Total	2508

TABLE 13: COST OF PRODUCT TITANIA PER TON

Item	Capel $A	Stradbroke Island $A
Raw materials	20.4	17.8
Labour	3.2	3.4
Fuel	8.2	8.8
Power	10.8	11.8
Depreciation and maintenance	48.4	52.4
Miscellaneous	11.0	11.8
Total	102.0	106.0

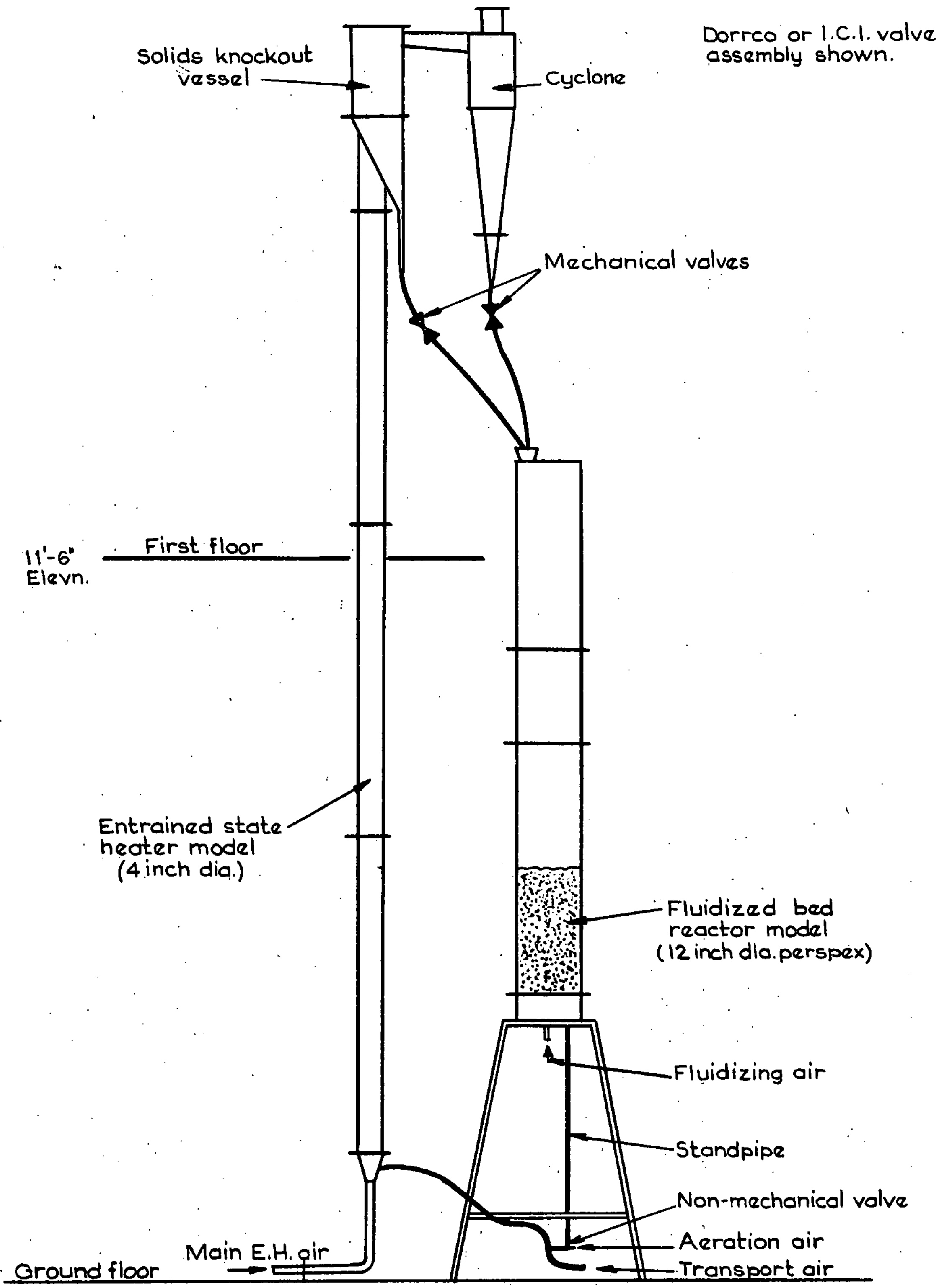


FIG. 1: MODEL OF SYSTEM FOR ENTRAINED HEATING OF FLUIDIZED BED REACTORS

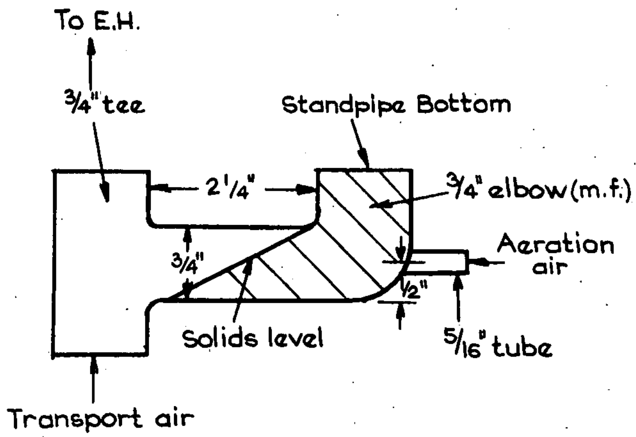


Fig. 2a: Dorrco Valve

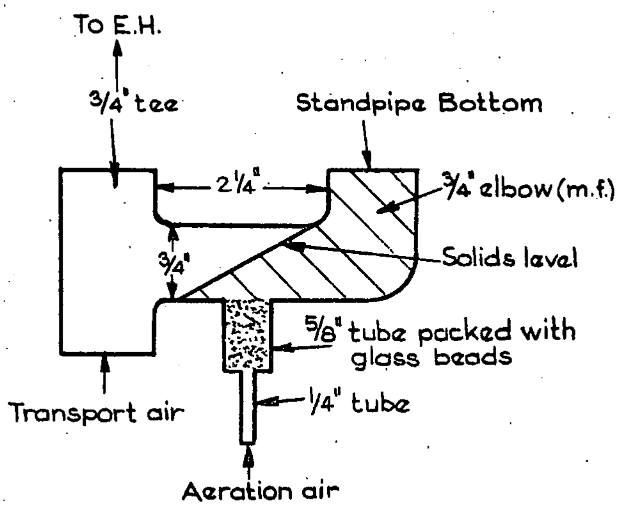


Fig. 2b: ICI Valve

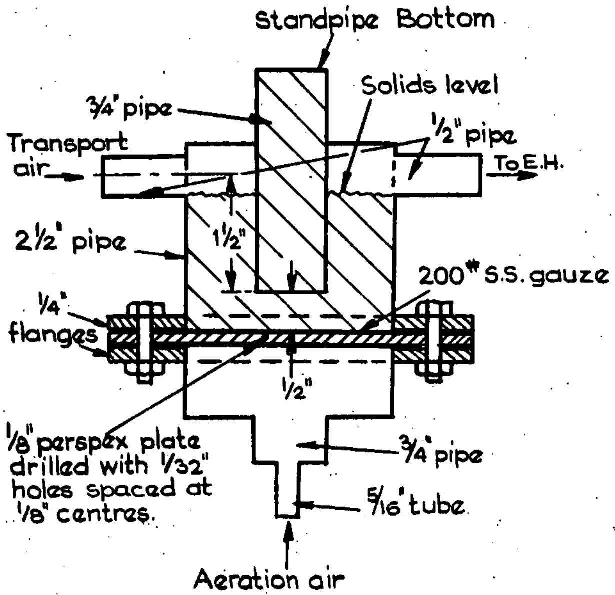


Fig. 2c: Esso Type "A" Valve
Original version

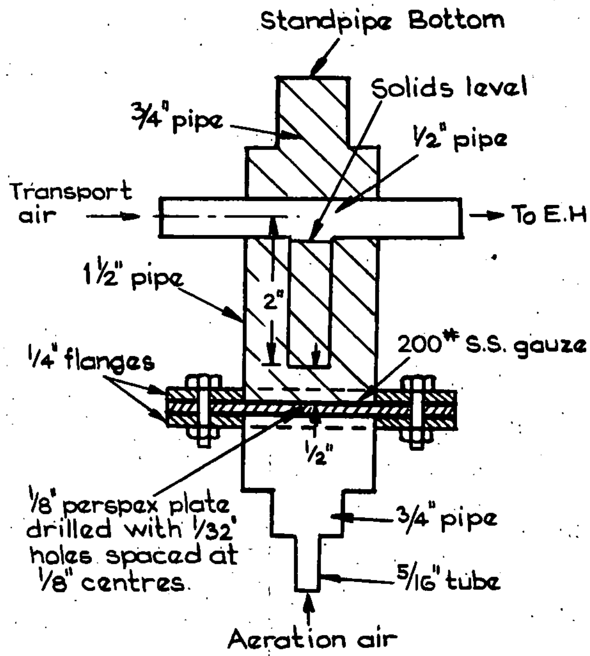


Fig. 2d: Esso Type "B" Valve

FIG. 2: DETAILS OF FIRST FOUR VALVES TESTED IN MODEL
Sections taken through centre of each valve

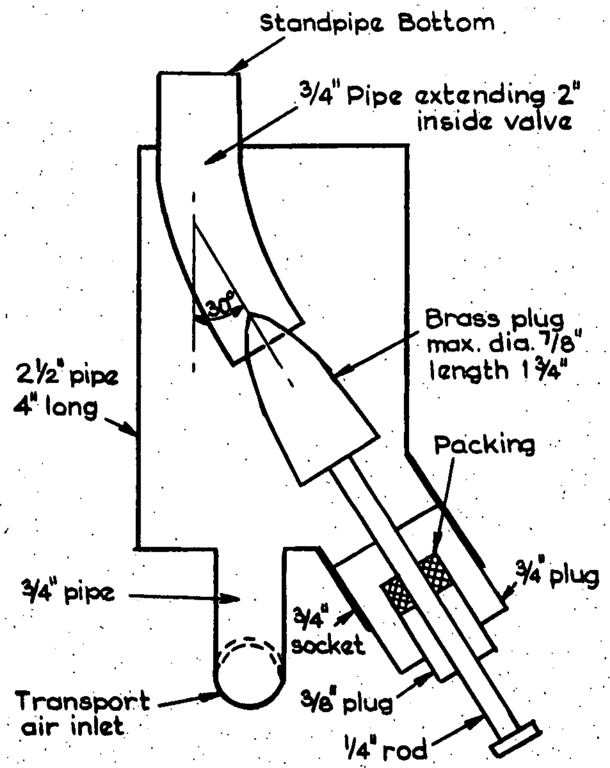


Fig. 3a: Plug Valve
Section through centre of valve

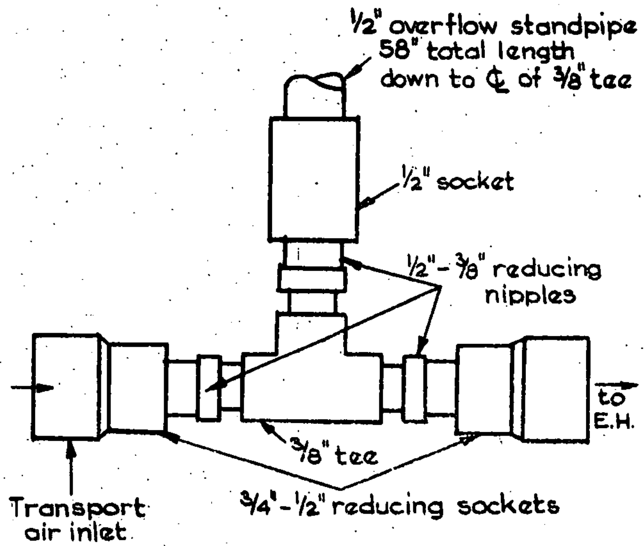


Fig. 3c: USS Venturi Valve

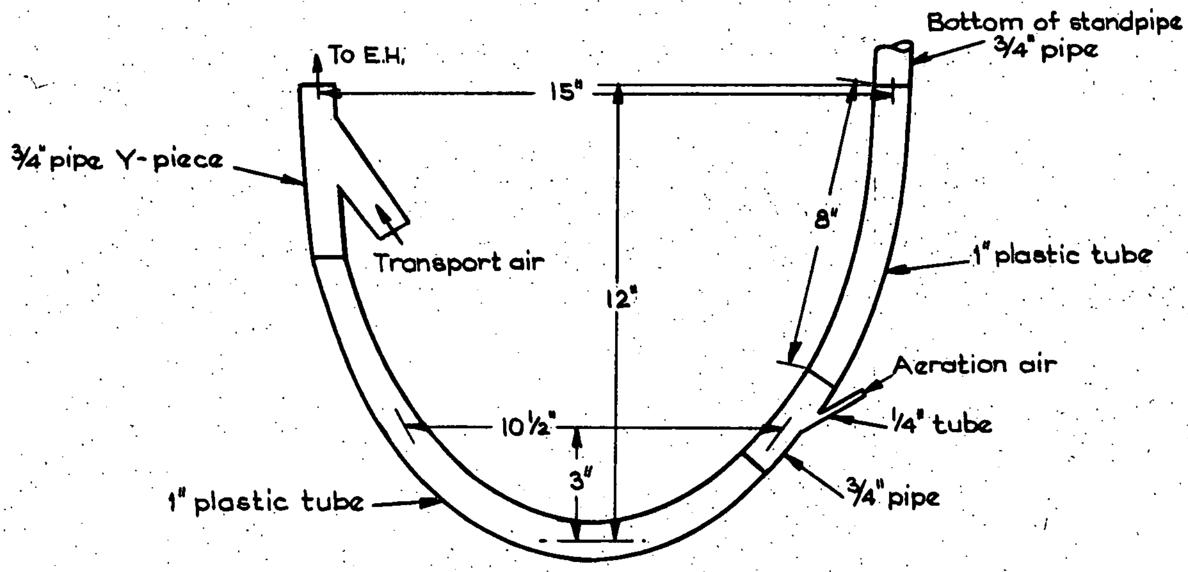


Fig. 3b: U-Bend Valve

FIG. 3: DETAILS OF LAST THREE VALVES TESTED IN MODEL

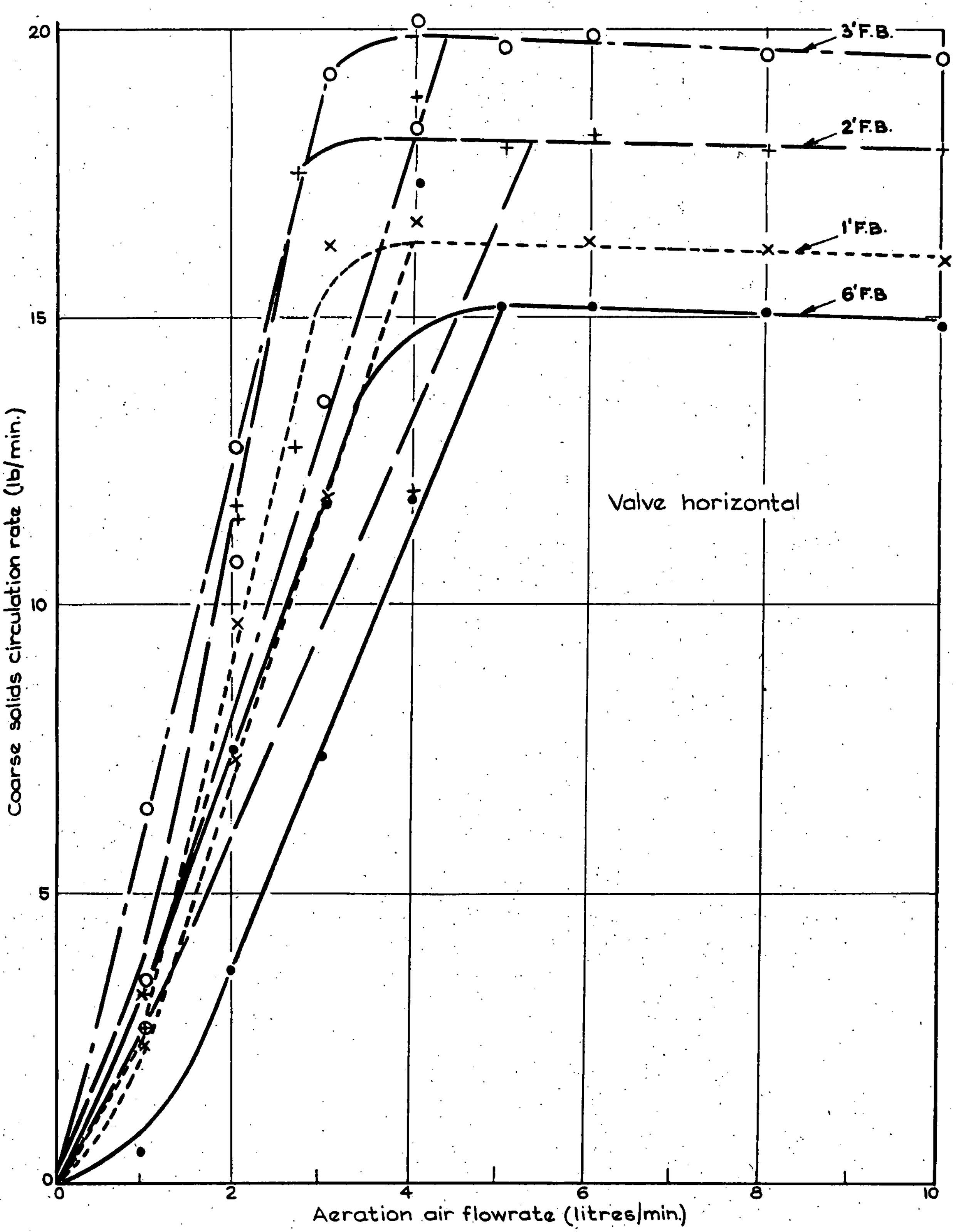


FIG. 4: SOLIDS CIRCULATION RATES
Dorrco Valve - Steel Standpipe

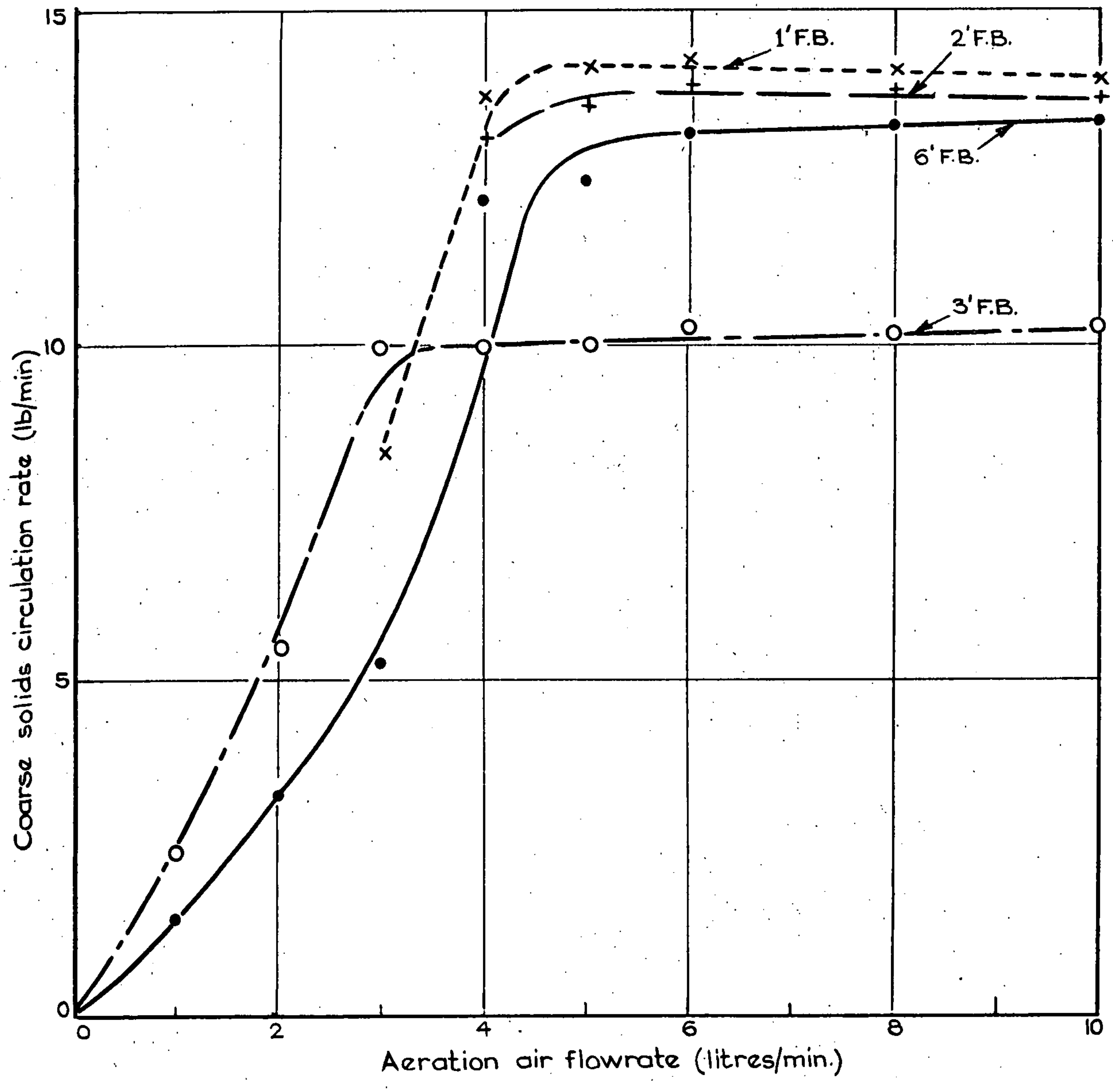
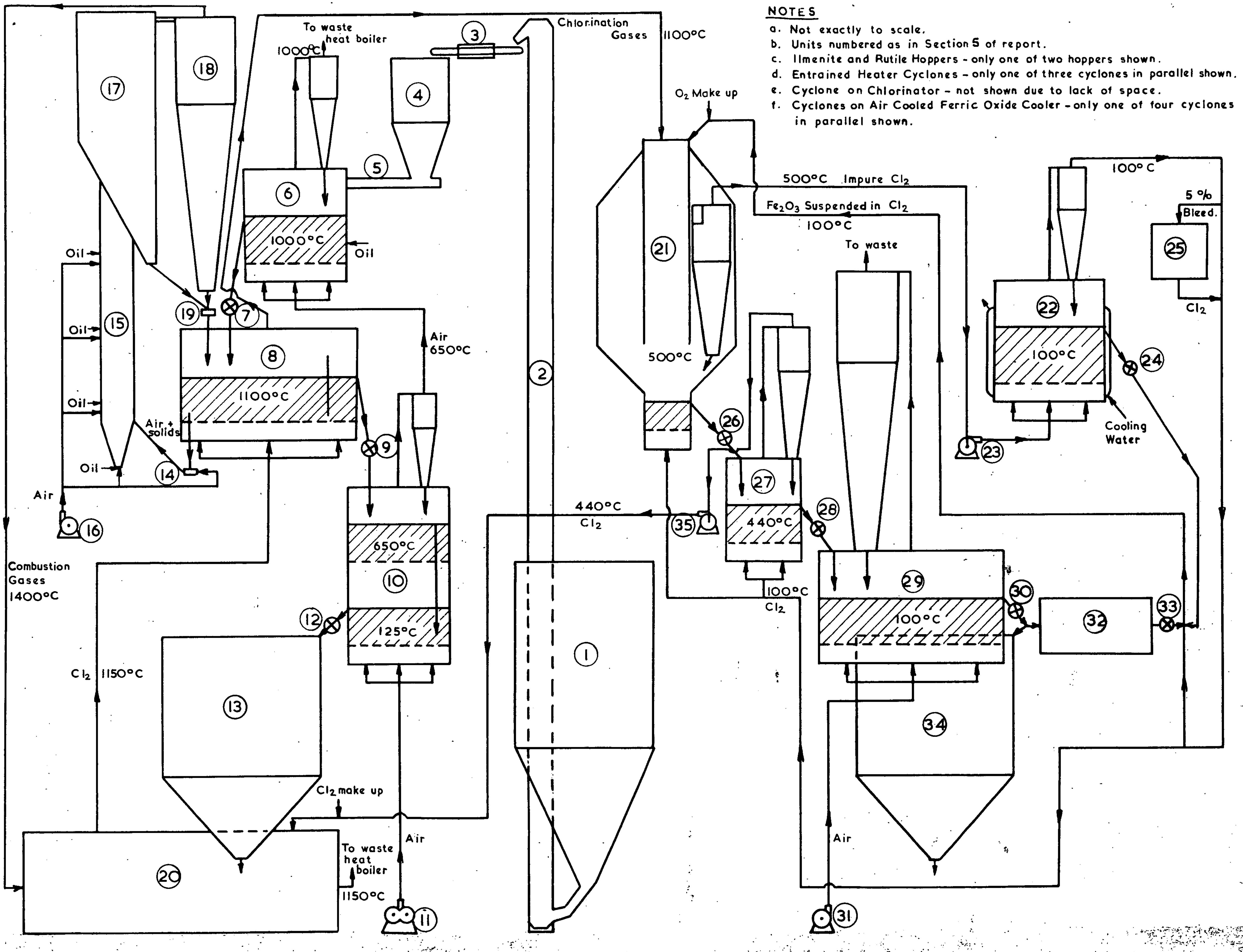


FIG. 5: SOLIDS CIRCULATION RATES
Dorrco Valve - Perspex Standpipe
Valve horizontal



NOTES

a. Not exactly to scale.
b. Units numbered as in Section 5 of report.
c. Ilmenite and Rutile Hoppers - only one of two hoppers shown.
d. Entrained Heater Cyclones - only one of three cyclones in parallel shown.
e. Cyclone on Chlorinator - not shown due to lack of space.
f. Cyclones on Air Cooled Ferric Oxide Cooler - only one of four cyclones in parallel shown.

FIG. 6. AMENDED FLOWSHEET FOR PRODUCTION OF TITANIA BY CHLORINATION OF ILMENITE