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CAUSTIC SODA PRODUCTION

by

J.R. Tuffley

Investigated by: Chemical Metallurgy Section
Officer in Charge: J.E.A. Gooden

F.R. Hartley. Director

THE AUSTRALIAN MINERAL DEVELOPMENT LABORATORIES
Adelaide South Australia

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SUMMARY

Background

A scheme for producing caustic soda from sodium chloride and sulphur dioxide was proposed to the South Australian Department of Mines by Mr S.B. Dickinson in 1972. After discussions with the BHAS Pty Ltd and a preliminary investigation of the chemical feasibility of the processes involved, Amdel was commissioned to conduct a feasibility study of establishing the overall scheme at Port Pirie assuming that the necessary sulphur dioxide would be obtained from waste smelter gases.

Objectives

The aim of the investigation was to determine the economic feasibility of establishing a plant at Port Pirie to produce 80 000 tonnes/a NaOH in a 50% caustic soda solution, using NaCl and sulphur dioxide from waste gases as the principal raw materials.

Summary of Work Done

The overall process involves the conversion of NaCl to NaOH via the intermediate compounds Na_2SO_4, Na_2S and Na_2CO_3. Details of process equipment, utilities, conversion efficiencies, etc., for each process stage were obtained from published information. For the production of Na_2SO_4 and Na_2S, equipment has been assumed that will allow large-scale, continuous operation rather than the small-scale, batch operation used previously. Equipment for recovering sulphur from waste gases is based on a process piloted successfully by the US Bureau of Mines, but not yet in commercial operation.

Mass balances were calculated for the process on the basis of a production rate of 80 000 tonnes/a NaOH in a 50% caustic soda solution. These gave a raw materials requirement as follows:

	tonnes/a
Salt	150 000
Limestone	133 700
Coke	8 100
Coal	86 250
Kerosene	375

The equipment was then sized and costed. Utilities requirements were calculated to be as follows:

Power	33.66×10^6	kWh/a
Water	493 000	tonnes/a
Fuel oil	6 420	tonnes/a

A summary of costs is given below:

1. Capital

	$
Equipment, delivered	5 053 620
Installation, piping, foundations etc.	7 984 720
Total direct costs	13 038 340
Eng. construction and contractors' fees	2 607 668
Contingency	3 129 202
Working capital	3 236 336
Total Capital Investment	22 011 546
say,	22 000 000

2. Operating

	$/annum
Raw materials	3 558 600
Utilities	671 680
Direct labour	740 000
Maintenance	1 502 017
Supervision	148 000
Operating supplies	74 000
Direct Manufacturing Costs	6 694 297
Indirect Manufacturing Costs	1 324 943
Fixed Manufacturing Costs	1 689 767
Total Manufacturing Costs	9 709 007
Non-Manufacturing Costs	1 747 620
Total Production Costs	11 456 627

3. Sales

80 000 tonnes/a NaOH	$11 200 000
Loss	256 627

However, if the whole HCl production of 88 900 tonnes/a could be sold for $100 per tonne, the income from sales would increase by $8 890 000.

This would result in a pre-tax profit of $8 633 370, or 39.2% pa on total capital investment.

Conclusions

1. It would be essential that a market for hydrochloric acid or chlorine be established before any plant is built.
2. Unless there is a market for the greater part of the hydrochloric acid byproduct the production rate considered in this Report, viz. 80 000 tonnes/a NaOH, is too small for economic operation. A higher production rate (say, 200 000 tonnes/a NaOH) plus sales of a smaller proportion of the HCl product is another possible economic alternative.
   For example, the following two cases both yield a 25% Rate-of-Return (before tax):
   - i. 80 000 t/a NaOH plant producing at design capacity and selling 57 570 t/a of HCl (65% of HCl production);
   - ii. 200 000 t/a NaOH plant producing at design capacity and selling 54 360 t/a of HCl (24% of HCl production);

   and other cases can be interpolated or extrapolated. However, it will be noted that in both cases cited, a market must be established for around 55 000 t/a of HCl.
3. Sulphur recovered from waste gases at the BHAS Pty Ltd would become more expensive as the production rate of caustic soda increased above 80 000 tonnes/a.
4. The equipment and processes in the sulphur recovery, sodium sulphate and sodium sulphide sections would require extensive piloting before they could be incorporated in a commercial plant.

Recommendations

1. Establish the extent of the market for hydrochloric acid or chlorine in Australia or overseas.
2. Consider the economic viability of alternative cases as follows:
   - i. An independent plant burning imported sulphur.
   - ii. A plant sited adjacent to a smelter which currently vents strong SO_2 gases to atmosphere.

3. If a case can be made for siting the process at Port Pirie, hold discussions with the BHAS Pty Ltd to determine whether the establishment of a caustic soda plant at Port Pirie to utilise smelter waste gases would be an acceptable proposition.

4. If a set of conditions is found which make the process economically attractive, conduct pilot-plant work in sulphur recovery, sodium sulphate production and sodium sulphide production.

1. INTRODUCTION

A scheme for producing caustic soda from sodium chloride and sulphur dioxide was proposed to the South Australian Department of Mines by Mr S.B. Dickinson in 1972. It was thought originally that this would provide a convenient outlet for strong SO_2 gases from the Works of the Broken Hill Associated Smelters Pty Ltd (BHAS) at Port Pirie, at a time of oversupply of sulphuric acid.

Discussions were held in October 1973 with the Chief Metallurgist of BHAS Pty Ltd, Mr F. Whitworth, who indicated that the acid plant was running at full capacity and all the acid produced was being sold. No slackening of demand was forecast within the foreseeable future. Hence, high grade SO_2 gases and sulphuric acid at Port Pirie are fully committed.

However, approximately 14 000 tonnes of sulphur are emitted to atmosphere every year in stack gases which range in composition from 0.11% to 0.5% SO_2. If SO_2 recovery units were installed, sufficient sulphur could be recovered to provide the required make-up for a caustic soda plant. This sulphur would be more expensive than that contained in strong SO_2 gases from the sinter machine.

2. BASIS

Cost estimates have been made for a plant to produce 80 000 tonnes/a NaOH in a 50% solution. It has been assumed that the plant will be situated at Port Pirie as part of the BHAS Pty Ltd smelter complex. Most auxiliary buildings and services will therefore already be available, although extensions to these may be required.

The plant will operate continuously and an operating year is assumed to be 330 days. The life of the plant is assumed to be the same as the life of the smelter, for which a minimum figure of 20 years is assumed.

3. PROCESS DESCRIPTION

A flow sheet illustrating the processes described in this section is presented in Fig.1.

3.1 Sodium Sulphate Production[1,2,3]

Sodium sulphate can be produced by reacting NaCl with either H_2SO_4 (Reaction 1) or with SO_2, air and steam (Reaction 2).

$$2NaCl + H_2SO_4 \longrightarrow Na_2SO_4 + 2HCl \quad \text{.. .. ..} \quad (1)$$

$$2NaCl + SO_2 + H_2O + \tfrac{1}{2}O_2 \longrightarrow Na_2SO_4 + 2HCl \quad \text{.. .. ..} \quad (2)$$

The old established methods for producing Na_2SO_4 were in common use 30 to 50 years ago. They were labour intensive and suitable mainly for small-scale production. In one process, acid and salt were fed to a Mannheim furnace, consisting of an externally heated, rabbled cast-iron pan. The reaction temperature was 700°C. Batches of up to 1 tonne were processed by this method and the exit gases contained about 30% HCl. In another process used in Europe and Florida a mixture of air, steam and SO_2 is passed through a series of towers which are externally heated to 700°C and packed with salt briquettes. The exit gases contain about 10% HCl.

A recent innovation for larger-scale production is the use of a steel-lined fluid-bed reactor for producing Na_2SO_4 from salt and acid. It would appear that a fluid-bed reactor could also be used for reacting salt with a mixture of SO_2, air and steam. This process will be assumed for use in the present exercise but experimental confirmation will be required. The acid process cannot be considered because an extra acid plant would be required to produce acid from re-cycled SO_2. The cost of doing this would be prohibitive.

Mass and heat balances for the process are set out in Appendixes A and B respectively, assuming a reaction temperature of 700°C. This information in turn is used to calculate the size of the reactor, assuming a nominal residence time of one hour. The reaction is exothermic and additional heat is provided by burning fuel oil in the windbox. The exit gases contain about 15% HCl.

3.2 Hydrochloric Acid Production[1,2,3]

The hot gases are cooled, filtered in a coke filter to remove H_2SO_4 mist, and the HCl is absorbed in water. Absorption liberates 1630 kJ/kg of HCl absorbed and hence a cooling system must be provided. The product from 15% HCl gas will contain 25 to 30% HCl.

If there is no market for the HCl, it will be neutralised with $CaCO_3$ sludge from the causticising circuit and CaO from the lime kiln. The $CaCl_2$ product will be discharged to waste.

3.3 Sodium Sulphide Production[2,3]

The hot Na_2SO_4 product is charged with coal to a rotary reverberatory furnace where it is reduced to Na_2S at 900° to 1000°C. Descriptions of the apparatus used for this reaction are not detailed and refer to the formation of a rather pasty product. Experimental confirmation of the type of equipment to be used in this stage will be required. The product is water-leached to produce a 15% Na_2S solution. Any undissolved solids are removed by settling and filtration.

3.4 Carbonation[1,2,3]

A limestone-coke mixture is burnt in a conventional, vertical lime kiln to produce gases containing about 40% CO_2. These gases are compressed and passed to carbonating towers where they react with the Na_2S solution (Reaction 3) to produce a 20% Na_2CO_3 solution at 98% yield.

$$Na_2S + CO_2 + H_2O \rightarrow Na_2CO_3 + H_2S \quad .. \quad .. \quad .. \quad (3)$$

CaO produced in the lime kiln is used for causticising the Na_2CO_3 solution. Some CaO may also be required for neutralising HCl.

3.5 H_2S Treatment[4,5]

Off-gases from the carbonating towers are contacted with monoethanolamine (MEA) in absorber towers to extract H_2S from the gases. The solution is then heated to drive off a high-grade H_2S gas and regenerate MEA for reuse. About two-thirds of the H_2S is combusted with air in a conventional package boiler to raise steam and produce SO_2. The other third is used in the sulphur recovery stage for precipitating elemental sulphur (Section 3.6). Combustion gases from the boiler contain about 14% SO_2 and are re-cycled to the Na_2SO_4 productions process.

3.6 Sulphur Recovery

Sulphur used in the process will be recycled but additional sulphur will be required to make up losses. This will be obtained by recovering sulphur from the stack gases by one of the many processes being considered for commercial development.

In general, these processes consist of absorption of SO_2 from the gas stream followed by regeneration of the absorbing medium with simultaneous release of the sulphur as SO_2, H_2S or elemental sulphur.

Three absorbents which appear feasible for use in the present exercise are alkalised alumina, magnesia slurry, and sodium citrate solution.[6,7,8,9] The citrate method[6] has processing stages which would conveniently dovetail in with other processes in the plant under consideration, and hence reduce capital and operating costs. It has therefore been selected for use in this project. Detailed costing and a survey of recovery processes would be necessary before a final choice of process is made.

In the citrate process, waste gases from the smelter are cleaned and cooled in a packed scrubber tower. The SO_2 is then absorbed in a solution of sodium citrate, citric acid, and sodium thiosulphate. Some of the H_2S recovered from the carbonating towers is used to precipitate elemental sulphur by Reaction 4. The citrate solution is regenerated simultaneously:

$$2H_2S + SO_2 \longrightarrow 3S + 2H_2O \quad \ldots \quad \ldots \quad \ldots \qquad (4)$$

Kerosene equal to 2½% of the weight of sulphur is added to the slurry to float the sulphur which is skimmed off as a cake and concentrated by melting. The sulphur is burnt in a package boiler to produce concentrated SO_2 for Na_2SO_4 production and raise steam for use elsewhere in the plant.

3.7 Caustic Soda Production[1,2,3]

CaO from the lime kiln is slaked in the Na_2CO_3 solution using a slaker-classifier. Unburnt $CaCO_3$ grit is separated out in the classifier and is either discarded or used for neutralisation of HCl. The slurry of slaked lime in Na_2CO_3 solution is passed to a causticising stage where it is heated with steam to 85°C for one hour. Caustic soda and a precipitate of $CaCO_3$ are produced by Reaction 5:

$$Na_2CO_3 + Ca(OH)_2 \longrightarrow 2NaOH + CaCO_3 \quad \ldots \quad \ldots \quad \ldots \qquad (5)$$

Slurry from the causticising stage contains about 11% NaOH and 13% solids. The solids are settled in a thickener and then filtered and washed to produce a $CaCO_3$ sludge. This is not suitable for re-use in the lime kiln because it is very fine and still contains a small quantity of NaOH. It is either discarded or used for HCl neutralisation.

The NaOH solution is passed to a multi-effect evaporator to concentrate it to 50% NaOH. The solubility of Na_2CO_3 in 11% NaOH solution is 1.7%. In 50% NaOH solution the solubility falls to 0.15%. As a result, Na_2CO_3 is formed as a precipitate during the evaporation stage and is separated from the NaOH solution by settling and filtration. The solids are returned to the Na_2CO_3 solution.

The 50% NaOH solution is the final product for market and contains 80 000 tonnes NaOH per annum.

4. DESIGN CALCULATIONS

Mass balances for the process are set out in Appendix A, while Appendix B contains the design calculations for the sizing of major items of equipment.

4.1 Schedule of Equipment

The equipment schedule, together with the estimated purchase cost, is listed below:

Section/Item	No.	Size	Cost $
1. Sodium Sulphate:			
fluid-bed reactor	2	5.3 m dia.	417 000
bucket elevators	2	20.3 × 12.7 cm buckets	14 000
cyclones	4	40 000 kl/h, high efficiency	4 000
blowers	7	8000 kl/h @ 50 kPa	10 000
			445 000
2. Hydrochloric Acid:			
absorber incl. cooler	1	2.5 m dia. × 4 m packed height	50 000
neutralising tanks	3	2.5 m dia. × 3 m, rubber lined	17 000
agitators	3	2 kW turbine	6 000
			73 000
3. Sodium Sulphide:			
rotary reverberatory furnace	1	2 m dia. × 40 m	325 000
leach tanks	2	2.8 m dia. × 3 m stainless steel	18 000
agitators	2	2.5 kW, turbine, - ditto -	4 000
thickener	1	10 m dia.	15 000
filter	1	25 m², rotary vacuum	58 000
			420 000
4. Carbonation:			
compressors	2	4000 m³/h	313 000
carbonating towers	2	3 m dia. × 25 m	255 000
			568 000
5. Limestone Calcination:			
lime kiln	1	4.5 m dia. × 12 m working height	544 000

Section/Item	No.	Size	Cost $
6. H_2S Treatment:			
absorption tower	1	1 m dia. × 10 m	22 000
distillation tower	1	1 m dia. × 10 m	22 000
packaged boiler	1	31 500 kg/h @ 2000 kPa	92 000
			136 000
7. Sulphur Recovery:			
scrubbers	4	3.5 m dia. × 6 m	622 000
absorbers	2	3.8 m dia. × 9 m	513 000
reactor vessel	1	3 m dia. × 3.5 m - closed	7 000
agitator	1	3 kW turbine	1 000
thickener (incl. skimmers)	1	5 m dia.	8 000
melting furnace	1	500 litre capacity	30 000
air blowers	16	8000 kl/h @ 50 kPa	23 000
			1 204 000
8. Caustic Soda:			
slaker-classifier-causticisers	3	4 m dia. × 2½ m	20 000
thickeners	3	30 m dia.	208 000
filters	2	28 m^2, rotary vacuum	121 000
thickener	1	22 m dia.	42 000
filter	1	25 m^2, rotary vacuum	58 000
evaporators	2	350 m^2, forced circulation	637 000
			1 086 000
9. Auxiliaries:			
caustic storage tanks (4 weeks)	3	20 m dia. × 10 m	300 000
fuel oil storage tank (4 weeks)	1	10 m dia. × 8 m	40 000
Process pumps (including installed spares)	2	15 l/min oil	600
	2	50 l/min water	900
	6	500 l/min water	3 000
	1	600 l/min slurry	750
	2	600 l/min HCl	2 100
	9	500 l/min liquor	6 750
	5	400 l/min MEA liquor	3 000
	5	600 l/min water	3 500
	2	250 l/min caustic	1 500
	3	800 l/min liquor	2 900
			365 000

4.2 Schedule of Power Requirements

The power requirements are as tabulated below:

Item	No.	Power kW	kWh/day
Bucket elevators	2	1	48
Blowers	22	150	79 200
Agitators:	3	2	144
	2	2.5	120
	1	3	72
Rotary reverberatory furnace	1	16	384
Thickeners:	1	1	24
	3	2	144
Slaker-classifiers	3	1	72
Lime kiln ancillaries	1	5	120
Compressors	2	360	17 280
Filters	4	20	1 920
Pumps: oil	1	0.25	6
water	1	0.5	12
water	5	1.5	180
slurry	1	2	48
HCl	1	2	48
liquors	7	2	336
MEA liquor	4	1	96
water	3	6	432
caustic	1	5	120
liquor	2	3	144
			100 950
Assumed for lighting and miscellaneous			1 050
		Total	102 000

4.3 Direct Labour Requirements

The direct operating labour requirements are as follows:

Materials handling,	2 men, day shift only
Na_2SO_4 and Na_2S production,	6 men per shift
Lime kiln	2 men per shift
H_2S treatment	4 men per shift
Sulphur recovery	6 men per shift
Caustic soda production,	6 men per shift
Total	2 men, day shift only.

26 men per shift for 4 shifts, i.e., 104 men.

4.4 Raw Materials Requirements

Salt:	150 000 tonnes/a
Limestone:	133 700 tonnes/a for lime kiln
Coke:	8 100 tonnes/a for lime kiln
Coal:	86 250 tonnes/a for Na_2S production
Kerosene:	375 tonnes/a for S flotation

4.5 Utilities Requirements

Power:	102 000 kWh/day, or 33.66×10^6 kWh/a from Section 4.2	
Water:	in final product	80 000 tonnes/a
	in HCl or $CaCl_2$ waste	356 000 tonnes/a
	gas cleaning and cooling	57 000 tonnes/a
	Total	493 000 tonnes/a
Fuel oil:	for fluid bed	6420 tonnes/a
Steam:	nil - plant is self-sufficient.	

5. TOTAL CAPITAL INVESTMENT

The total capital investment was estimated by the factored method and is based on estimates of the total delivered cost of the major items of equipment and applying experience factors for all other cost items.

The estimate is summarised as follows:

Item		Cost $
1a. Purchased Equipment Cost:		
1. Sodium sulphate		445 000
2. Hydrochloric acid		73 000
3. Sodium sulphide		420 000
4. Carbonation		568 000
5. Limestone calcination		544 000
6. H_2S treatment		136 000
7. Sulphur recovery		1 204 000
8. Caustic soda		1 086 000
9. Auxiliaries		360 000
	Total purchase	4 836 000
1b. Delivery and Location Costs: 4.5% of $4 836 000		217 620
1. Equipment cost, delivered		5 053 620
2. Installation,	30% of equipment cost	1 516 086
3. Instrumentation,	15% of equipment cost	758 043
4. Piping	35% of equipment cost	1 768 767
5. Electrical	15% of equipment cost	758 043
6. Buildings	10% of equipment cost	505 362
7. Foundations and structures	30% of equipment cost	1 516 086
8. Land		nil
9. Yard improvements	3% of equipment cost	151 609
10. Utilities	20% of equipment cost	1 010 724
11. Total Direct Costs		13 038 340
12. Eng. construction and contractors fees	20% of Direct Costs	2 607 668
13. Total Direct and Indirect Costs		15 646 008
14. Contingency	20% of Direct and Indirect Costs	3 129 202
15. Fixed Capital Investment		18 775 210
16. Working capital:	4 months' manu. costs	3 236 336
17. Total Capital Investment		22 011 546
	say,	22 000 000

6. TOTAL PRODUCTION COSTS

The total production costs are summarised as follows:

Item			Cost	
			$/annum	$/tonne NaOH
1.	Raw Materials:			
	Salt	150 000 tonnes/a @ $8/t	1 200 000	15.00
	Limestone	133 700 tonnes/a @ $3/t	401 100	5.01
	Coke	8 100 tonnes/a @ $25/t	202 500	2.53
	Coal	86 250 tonnes/a @ $20/t	1 725 000	21.56
	Kerosene	375 tonnes/a @ $80/t	30 000	0.38
		Total raw materials	3 558 600	44.48
2.	Utilities:			
	Power	33.66×10^6 kWh/a @ 0.8¢/kWh	269 280	3.37
	Water	493 000 tonnes/a @ 10¢/t	49 300	0.62
	Fuel oil	6420 tonnes/a @ $55/t	353 100	4.41
		Total utilities	671 680	8.40
3.	Direct Labour:			
	2 men, day shift only @ $6000/a		12 000	0.15
	104 men, shifts, @ $7000/a		728 000	9.10
		Total direct labour	740 000	9.25
4.	Maintenance: 8% of fixed capital		1 502 017	18.78
5.	Supervision: 20% of direct labour		148 000	1.85
6.	Operating Supplies: 10% of direct labour		74 000	0.93
7.	Royalties and Patents		nil	nil
8.	Direct Manufacturing Costs (Items 1 to 7)		6 694 297	83.69

Item	Cost	
	$/annum	$/tonne NaOH
9. Payroll overhead: 20% direct labour	148 000	1.85
10. Plant overhead: 125% of direct labour	925 000	11.56
11. Process control: 25% of direct labour	185 000	2.31
12. Packaging: 1% of direct costs	66 943	0.84
13. Indirect Manufacturing Costs (Items 9-12)	1 324 943	16.56
14. Depreciation 8% of fixed capital	1 502 017	18.78
15. Property taxes and insurance: 1% of fixed capital	187 750	2.35
16. Fixed Manufacturing Costs (Items 14-15)	1 689 767	21.13
17. Manufacturing costs (Items 8+13+16)	9 709 007	121.38
18. Administrative expenses: 3% of manufacturing costs	291 270	3.64
19. Distribution and marketing expenses: 10% of manufacturing costs	970 900	12.14
20. Research and development 5% of manufacturing costs	485 450	6.07
21. Financial expenses assumed as share capital	nil	nil
22. Non-manufacturing costs (Items 18-21)	1 747 620	21.85
23. Total Production Costs (Items 17+22)	11 456 627	143.23

7. PROFITABILITY

The profitability of the venture can be assessed assuming the current price for caustic soda of $140 per tonne.

	Cost	
	$/annum	$/tonne NaOH
Total sales: 80 000 tonnes/a @ $140/t	11 200 000	140.00
Total production costs	11 456 627	143.23
Loss	256 627	3.23

Therefore, the venture as outlined in this Report is not economically viable. However, this is based on the assumption that there is no available market for HCl. If the whole HCl production of 88 900 tonnes/a could be sold for $100/tonne, the income from sales would increase by $8 890 000. This would result in a pre-tax profit of $8 633 370, or 39.2% pa on total capital investment.

Hence, profitable operation could probably be achieved either by increasing the size of plant or by establishing a market for HCl.

Cost figures for plants producing 150 000 tonnes/a and 200 000 tonnes/a caustic soda respectively have been calculated by factoring from the costs for the 80 000 tonnes/a plant and are listed below:

		Production, t/a	
		150 000	200 000
Total capital investment,	$	32 700 000	39 200 000
Total production costs,	$/a	18 700 000	23 636 000
Total sales (NaOH only)	$/a	21 000 000	28 000 000
Pre-tax profits,	$/a	2 300 000	4 364 000
Pre-tax profitability,	% $/a	7.0	11.1

Even at a production rate of 200 000 tonnes/a, a market for HCl would be required to make the venture economically attractive.

8. DISCUSSION

1. Although most of the equipment and processes described in this Report involve known and commercially established technology, some of the equipment is innovative or in the experimental stages. The Sections involving sulphur recovery, sodium sulphate production and sodium sulphide production fall into the latter category and would require extensive pilot-plant investigations.

2. Sulphur available in waste gases at BHAS Pty Ltd comes from four sources at four different concentrations. If a plant to produce more than 80 000 tonnes/a caustic soda is envisaged, then sulphur would have to be recovered from poorer grade waste gases at a higher cost per unit of sulphur.

3. The economic viability of the process would be greatly improved if strong SO_2 gases could be obtained direct from the sinter machine. At BHAS Pty Ltd this could be done only at the expense of acid production. Hence the aim should be to establish the process at a site adjacent to a smelter which currently vents strong SO_2 gases to atmosphere.

9. CONCLUSIONS

1. It would be essential that a market for hydrochloric acid or chlorine be established before any plant is built.

2. Unless there is a market for the greater part of the hydrochloric acid byproduct the production rate considered in this Report, viz., 80 000 tonnes/a NaOH, is too small for economic operation. A higher production rate (say, 200 000 tonnes/a NaOH) plus sales of a smaller proportion of the HCl product is another possibly economic alternative. For example, the following two cases both yield a 25% Rate-of-return (before tax):

   (i) 80 000 t/a NaOH plant producing at design capacity and selling 57 570 t/a of HCl (65% of HCl production);

   (ii) 200 000 t/a NaOH plant producing at design capacity and selling 54 360 t/a of HCl (24% of HCl production);

   and other cases can be interpolated or extrapolated.
   However, it will be noted that in both cases cited, a market must be established for around 55 000 t/a of HCl.

3. Sulphur recovered from waste gases at the BHAS Pty Ltd would become more expensive as the production rate of caustic soda increased above 80 000 tonnes/a.

4. The equipment and processes in the sulphur recovery, sodium sulphate and sodium sulphide sections would require extensive piloting before they could be incorporated in a commercial plant.

10. RECOMMENDATIONS

1. Establish the extent of the market for hydrochloric acid or chlorine in Australia or overseas.

2. Consider the economic viability of alternative cases as follows:

   (i) An independent plant burning imported sulphur.

   (ii) A plant sited adjacent to a smelter which currently vents strong SO_2 gases to atmosphere.

3. If a case can be made for siting the process at Port Pirie, hold discussions with the BHAS Pty Ltd to determine whether the establishment of a caustic soda plant at Port Pirie to utilise smelter waste gases would be an acceptable proposition.

4. If a set of conditions is found which make the process economically attrative, conduct pilot-plant work in sulphur recovery, sodium sulphate production and sodium sulphide production.

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APPENDIX A

MASS BALANCE CALCULATIONS

1. PRODUCT

The product is 80 000 tonnes/a NaOH contained in a 50% solution which has sp.gr. 1.52 at 20°C and contains 7.65 g per litre NaOH and 0.15% Na_2CO_3.[10†]

$$\text{Volume of product} = \frac{80000 \times 10^6}{765 \times 330 \times 24 \times 60}$$

$$= 220 \text{ litres/min}$$

$$Na_2CO_3 \text{ in product} = 0.15\% \text{ of } 160\ 000 \text{ tonnes/a}$$

$$= 240 \text{ tonnes/a}$$

2. SOLUTION PRIOR TO EVAPORATION

The 11% NaOH solution prior to evaporation has sp.gr. 1.08 at 85°C (operating temperature) and 1.12 at 20°C and contains 123.6 g/litre NaOH and 1.7% Na_2CO_3 at 20°C.

$$\text{At 85°C, NaOH concentration} = 123.6 \times \frac{1.08}{1.12}$$

$$= 119 \text{ g/litre}$$

$$\text{Volume of liquor (85°C)} = \frac{80000 \times 10^6}{119 \times 330 \times 24 \times 60}$$

$$= 1412 \text{ litres/min.}$$

$$Na_2CO_3 \text{ in solution} = 1.7\% \text{ of } 80000 \times \frac{100}{11}$$

$$= 12\ 360 \text{ tonnes/a.}$$

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† References in Section 11 of the Report.

Na_2CO_3 precipitated during evaporation = 12360 -240

= 12 120 tonnes/a

Solids in suspension to final thickener $= \frac{12120 \times 100}{(160000 + 12120)}$

= 7%.

Water evaporated = 1412 -220

= 1192 l/min.

3. CAUSTICISER

The causticising reaction is expressed as follows:

$$Na_2CO_3 + Ca(OH)_2 \rightarrow 2NaOH + CaCO_3 \quad .. \quad .. \quad (A\text{-}1)^*$$

For 80 000 tonnes/a NaOH, CaO required

if 10% excess is used $= 56000 \times \frac{11}{10}$

= 61 600 tonnes/a

Limestone required @ 95% utilisation $= 61600 \times \frac{100}{56} \times \frac{100}{95}$

= 115 800 tonnes/a

Grit separated out in slaker-classifier = 5% of 115 800

= 5800 tonnes/a

$CaCO_3$ mud formed = 100 000 tonnes/a $CaCO_3$

+ 7400 tonnes/a $Ca(OH)_2$

= 107 400 tonnes/a

Steam required for causticisers is 2700 tonnes for every 2000 tonnes NaOH in solution.[2]

∴ Steam = 108 000 tonnes/a, or 13 640 kg/h.

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* Reaction 5, Section 3.7 of the Report.

4. LIME KILN

Coke required in a lime kiln is about 70 tonnes for every 1000 tonnes of limestone.

Limestone required = 115 800 tonnes/a

∴ Coke required (85% carbon) = 115.8 × 70

= 8100 tonnes/a

CO_2 produced:

(a) from limestone if 95% is calcined $= \frac{44}{100} \times \frac{95}{100} \times 115800$

= 48 300 tonnes/a

(b) from coke @ 85% carbon $= \frac{44}{12} \times \frac{85}{100} \times 8100$

= 25 200 tonnes/a

Total CO_2 = 73 500 tonnes/a

$$\text{Air required @ 10\% excess} = \frac{25200 \times 10^6}{330 \times 24 \times 60 \times 10^3} \times \frac{32}{44} \times \frac{22.4}{32} \times \frac{100}{21}$$

= 130 std kl/min

CO_2 content of exit gas = 40% approx.

5. CARBONATING TOWERS

For 80 000 tonnes/a NaOH, $NaCO_3$ required is 120 000 tonnes/a[2]† in a 20% solution (sp.gr. 1.2).

$$\text{Volume of solution} = \frac{600000 \times 10^6}{1.2 \times 10^3}$$

$= 500 \times 10^6$ litres/annum

or 1050 l/min

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† Reference in Section 11 of Report.

The stoichiometric CO_2 requirement is determined from Reaction 3 (Equation A-2):

$$Na_2S + CO_2 + H_2O \longrightarrow Na_2CO_3 + H_2S \qquad \ldots \qquad \ldots \qquad (A-2)$$

CO_2 required $= 120000 \times \frac{44}{106}$

$= 50\ 000$ tonnes/a

CO_2 produced in lime kiln $= 73\ 500$ tonnes/a

∴ 47% excess CO_2 is available.

Na_2S required in carbonating towers at 98% conversion

$= 120000 \times \frac{100}{98} \times \frac{78}{106}$

$= 90\ 000$ tonnes/a

6. Na_2S PRODUCTION

Na_2S is produced by reduction of Na_2SO_4 as in Reaction 6 (Eq. A-3):

$$Na_2SO_4 + 2C \longrightarrow Na_2S + 2CO_2 \qquad \ldots \qquad \ldots \qquad (A-3)$$

Na_2SO_4 required at 95% conversion $= 90000 \times \frac{142}{78} \times \frac{100}{95}$

$= 172\ 500$ tonnes/a

Coal required for reduction and fuel is 1 tonne for every 2 tonnes Na_2SO_4.[11]†

∴ Coal required = 86 250 tonnes/a.

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† References in Section 11 of Report.

7. Na_2SO_4 PRODUCTION

Na_2SO_4 is produced by Reaction 2 (Equation A-4):

$$2NaCl + SO_2 + H_2O + \tfrac{1}{2}O_2 \rightarrow Na_2SO_4 + 2HCl \quad \text{..} \qquad \text{..} \quad (A-4)$$

NaCl required at 95% conversion

$$= 172500 \times \frac{117}{142} \times \frac{100}{95}$$

$$= 150\ 000 \text{ tonnes/a}$$

H_2O required at 5% excess

$$= 172500 \times \frac{18}{142} \times \frac{105}{100}$$

$$= 23\ 000 \text{ tonnes/a, or } 2900 \text{ kg/h.}$$

Oxygen required at 5% excess

$$= 172500 \times \frac{16}{142} \times \frac{105}{100} \times \frac{10^6}{32} \times \frac{22.4}{1000 \times 330 \times 24 \times 60}$$

$$= 30.0 \text{ std } k\ell/\text{min}$$

SO_2 required at 95% conversion

$$= 172500 \times \frac{64}{142} \times \frac{100}{95}$$

$$= 82\ 000 \text{ tonnes/a, or } 60.4 \text{ std } k\ell/\text{min.}$$

8. H_2S TREATMENT AND SULPHUR RECOVERY

H_2S from carbonating towers

$$= \frac{34}{106} \times 120000 \times \frac{98}{100}$$

$$= 39\ 300 \text{ tonnes/a.}$$

Some of this is burnt to produce SO_2 by Reaction 7 (Equation A-5):

$$H_2S + 1\tfrac{1}{2}O_2 \rightarrow H_2O + SO_2 \qquad \text{.. .. . (A-5)}$$

The remainder is used in the sulphur recovery section. However, all the sulphur is burnt to produce SO_2, so that all the sulphur in the H_2S eventually reports as SO_2.

SO_2 produced from H_2S

$$= \frac{64}{34} \times 39300$$

$$= 72\ 000 \text{ tonnes/a.}$$

∴ Make-up SO_2 required

$$= 10\ 000 \text{ tonne/a.}$$

(5000 tonnes/a sulphur)

Stack gases at BHAS Pty Ltd contain about 14 000 tonnes/a sulphur.

In the citrate process, sulphur is precipitated from solution by Reaction 4 (Equation A-6):

$$2H_2S + SO_2 \rightarrow 3S + 2H_2O \qquad \text{.. .. (A-6)}$$

∴ For 5000 tonnes/a make-up sulphur from recovered SO_2, a total of 15 000 tonnes/a sulphur is precipitated.

Kerosene required for flotation

$$= 2\tfrac{1}{2}\% \text{ of } 15\ 000 \text{ t/a}$$

$$= 375 \text{ t/a.}$$

H_2S required $= 15000 \times \frac{68}{96}$

$$= 10\ 600 \text{ tonnes/a.}$$

∴ H_2S burnt in the boiler = 39300 -10600

$$= 28\ 700 \text{ tonnes/a.}$$

Steam produced by oxidation of H_2S is 7 kg at 300 psi per kg S oxidised.[4,5]

$$\therefore \text{ Steam produced} = 28700 \times \frac{32}{34} \times \frac{1000 \times 7}{330 \times 24}$$

$$= 23\ 900 \text{ kg/h.}$$

Steam produced from oxidation of elemental sulphur is 4 kg per kg S oxidised by comparison of heats of reaction.[12]

$$\text{Steam produced} = \frac{15000 \times 1000 \times 4}{330 \times 24}$$

$$= 7600 \text{ kg/h.}$$

Total steam produced

$$= 31\ 500 \text{ kg/h.}$$

9. HCl RECOVERY

$$\text{HCl produced} = 150000 \times \frac{95}{100} \times \frac{36.5}{58.5}$$

$$= 88\ 900 \text{ tonnes/a.}$$

This will be contained in a 25 to 30% solution. If there is no market for HCl, neutralisation by Reaction 8 (Equation A-7) will be necessary:

$$2HCl + CaO \rightarrow CaCl_2 + H_2O \qquad .. \quad .. \qquad (A\text{-}7)$$

CaO required at 5% excess

$$= 88900 \times \frac{56}{73} \times \frac{105}{100}$$

$$= 71\ 600 \text{ tonnes/a.}$$

CaO available in $CaCO_3$ mud

$$= \frac{56}{100} \times 100000 + \frac{56}{74} \times 7400$$

$$= 61\ 600 \text{ tonnes/a.}$$

∴ Extra limestone required to be calcined

$$= 10000 \times \frac{100}{56}$$

$$= 17\ 900 \text{ tonnes/a.}$$

∴ Total limestone for calcination

$$= 17900 + 115800$$

$$= 133\ 700 \text{ tonnes/a.}$$

APPENDIX B

SIZING OF EQUIPMENT

1. Na_2SO_4 PRODUCTION

Na_2SO_4 will be produced in a fluid bed reactor. The following assumptions are made:

(i) NaCl is fed to the reactor in granular form at ambient temperature (20°C).

(ii) Gases leave the reactor at 700°C and no heat recovery is practised.

(iii) The solid product leaves the reactor at 700°C and is fed hot to the following process stage.

(iv) Water is injected directly into the bed.

(v) Fuel oil is burned in the windbox.

(vi) Air and the gases containing SO_2 are at ambient temperature, although it is possible in practice that the SO_2 gases may be somewhat hotter.

Feed to the Reactor

NaCl: 150 000 tonnes/a, or 18 900 kg/h

H_2O: 2900 kg/h

SO_2: 60.4 std kl/min in a gas containing about 14% SO_2 and 86% inert gas (mainly N_2).

Associated N_2: $60.4 \times \frac{84}{14}$

$= 371.0$ std kl/min

O_2: 30.0 std kl/min

Associated N_2: $30.0 \times \frac{79}{21}$

$= 112.9$ std kl/min.

Plus fuel oil and combustion air — to be determined.

Product from Reactor

Na_2SO_4: 172500 tonnes/a + 7500 tonnes/a unreacted NaCl
total 180 000 tonnes/a, or 22 700 kg/h

N_2: (371.0 + 112.9) std kℓ/min, or 36 400 kg/h + N_2 from combustion air and products of combustion.

O_2: 5% of 30.0 = 1.5 std kℓ/min
or 130 kg/h

SO_2: 5% of 60.4 = 3.02 std kℓ/min
or 520 kg/h

H_2O: 5% of 2900 kg/h = 3.0 std kℓ/min
or 140 kg/h

HCl: 114.8 std kℓ/min,
or 11 220 kg/h

Heat In

		kJ/h
1.	Sensible heat in solids	0
2.	Sensible heat in gases	0
3.	Heat of reaction, from published thermochemical data[12][†]	33.2×10^6
4.	Heat of combustion of fuel $= \frac{17500}{1.8} \times 4.187 \times A$	40 700 A

where A = kg oil/h.

Total heat in = 33.2×10^6 + 40700 A kJ/h.

Heat Out

		kJ/h
1.	Sensible heat in solids $= 0.22 \times 22700 \times (973-293) \times 4.187$ =	14.2×10^6

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† References in Section 11 of the Report.

kJ/h

2. Sensible heat in gases

$= 0.26 \times 4.187 \times (973-293) \times (37190+13.83A)$

$+ 0.20 \times 4.187 \times (973-293) \times 11220 \quad = \quad 33.9 \times 10^6 + 10250\ A$

3. Latent heat of vaporisation of water

$= 540 \times 4.187 \times 2900 \quad = \quad 6.6 \times 10^6$

4. Heat losses: allow 5% of total heat in

$= 5\%$ of $33.2 \times 10^6 + 40700\ A \quad = \quad 1.7 \times 10^6 + 2000\ A$

Total heat out $= 56.4 \times 10^6 + 12250A$ kJ/h

$\therefore\ 33.2 \times 10^6 + 40700\ A = 56.4 \times 10^6 + 12250\ A$

$\therefore\ 28\,550\ A = 23.2 \times 10^6$

$A = 810$ kg oil/h

Annual oil requirement $= \dfrac{810 \times 330 \times 24}{1000}$

$= 6420$ tonnes/a.

Combustion air required

$= 13.84 \times 6420 \times 2.2 \times \dfrac{12.4}{330} \times \dfrac{28.3}{24 \times 60}$

$= 144.2$ std kl/min

Total exit gas flow $= 750.4$ std kl/min

$= 2670$ kl/min at 700°C

At an assumed fluidising velocity of 1 m/sec,

Cross-sectional area of reactor $= 44.5\ m^2$

Use 2 reactors, each $22.3\ m^2$ area or 5.3 m dia.

HCl in exhaust gases $= \frac{114.8}{750.4} \times 100$

$= 15.3\%$

Each reactor will require 2 high efficiency cyclones, 2.7 m diameter, and 3 blowers plus a common spare, each with a capacity of 8000 kl/h against a head of 50 kPa requiring 150 kW each.

2. HCl PRODUCTION

Production rate of HCl is 88 900 tonnes/a. Gas from the fluid-bed reactor contains about 15.3% HCl and has a total flow-rate of 750.4 std kl/min. The final acid solution contains 25 to 30% HCl and is produced at the rate of 40 700 kg/h. Using published graphs and nomograms[13] the absorption column required has an operating height of 4 m, a diameter of 2.5 m, and is assumed to be packed with 50 mm diameter Raschig rings.

3. NEUTRALISATION

If there is no market for HCl, neutralisation will be carried out using $CaCO_3$ sludge and CaO in agitated vessels. Assuming a reaction time of 1 hour and a slurry sp.gr. of 1.2, the total volume of the leach tanks will be 34 m^3. This will require 3 leach tanks each 2.5 m diameter and 3 m deep. Each tank will be equipped with a 0.8 m diameter turbine agitator requiring 2 kW power.

4. Na_2S PRODUCTION

Production rate is 90 000 tonnes/a or 11.4 tonnes/h.

It is assumed that a rotary reverberatory furnace will be used and that a residence time of 3 hours will be required.

∴ Mass in furnace = 34.2 tonnes

= 18.4 kl (sp.gr. = 1.85)

Assuming the furnace is full to one fifth of its diameter, the size of furnace required is 40 m long and 2 m internal diameter.

The Na_2S will be leached in 1050 litres/min water for an assumed residence time of 30 minutes.

∴ Volume of leach tanks = 31.5 m^3

Use 2 tanks each 2.8 m diameter and 3 m deep. Each tank will be equipped with an 0.9 m diameter turbine agitator requiring 2.5 kW power. The resulting solution will contain some undissolved solids, such as coal ash, which will be removed by settling and filtration. The actual amount of solids cannot be accurately calculated, but the size of thickener required is assumed to be 10 m diameter, and the rotary vacuum filter is assumed to have an area of 25 m^2.

5. CARBONATION

By comparison with the ammonia-soda process, two carbonation towers each 3 m diameter and 25 m high are required to produce 120 000 tonnes/a soda ash.

6. LIMESTONE CALCINATION

A vertical kiln must be used for lime calcination to obtain the high percentage CO_2 required in the off-gas.[3] Kilns of this type produce about 14.6 tonnes CaO/day/m^2.[13] The cross-sectional area required

$$= \frac{133500 \times 56}{300 \times 100 \times 14.6}$$

$$= 15.6 \text{ m}^2$$

∴ shaft kiln diameter is 4.5 m with an operating height of about 12 m.

7. H_2S TREATMENT

Effluent gas from the carbonating tower contains about 40% H_2S amounting to 39 300 tonnes/a.

$$\text{Volume of } H_2S = \frac{39300 \times 10^6 \times 22.4}{34 \times 10^3 \times 330 \times 24}$$

$$= 3270 \text{ k}l\text{/h}$$

$$\text{Total volume of gas} = 3270 \times \frac{10}{4}$$

$$= 8180 \text{ k}l\text{/h}$$

By comparison with published descriptions of H_2S absorption plants[5], the above flow-rate will require an absorption tower and a distillation tower each 10 m high and 1 m diameter.

8. SULPHUR RECOVERY

The sulphur contained in sinter machine tail gas at BHAS Pty Ltd is in excess of 9000 tonnes/a. The SO_2 content of this gas averages about 0.36% compared with 0.11% for blast furnace gas. Hence, only sinter machine tail gas will be treated for sulphur recovery.

Assuming 95% recovery, sulphur required in the gas stream

$$= 5000 \times \frac{100}{95}$$

$$= 5260 \text{ tonnes/a.}$$

$$\text{Volume of gases treated} = \frac{5260 \times 2 \times 1000 \times 22.4}{330 \times 24 \times 60 \times 64} \times \frac{100}{0.36}$$

$$= 2150 \text{ std } k\ell/\text{min}$$

By comparison with published design data[6], gas cooling and cleaning will require four packed scrubber towers each 3.5 m diameter and 6 m high, packed with 50 mm diameter Raschig rings.

The SO_2 concentration in solution when absorbing from an 0.36% gas is about 15 g/litre.[6]

$$\therefore \text{ Volume of liquor} = \frac{10000 \times 10^6}{330 \times 24 \times 60 \times 15}$$

$$= 1400 \text{ litres/min}$$

Using published graphs and nomograms[13], two absorption columns are required, each with an operating height of 9 m, a diameter of 3.8 m, and packed with 50 mm diameter Raschig rings.

Sulphur is precipitated in a closed, single stage, agitated vessel. Residence time is 15 minutes.

$$\text{Reactor volume required:} = \frac{1400 \times 15}{1000}$$

$$= 21.0 \text{ m}^3$$

Assume a reactor 3 m diameter and 3.5 m deep with a 3 kW agitator.

Kerosene is added to float the sulphur in a thickener without rakes, 5 m diameter. The sulphur cake is skimmed off the surface and melted in a steam-heated furnace to concentrate the sulphur. Residence time in the furnace is 15 minutes.

$$\text{Volume of sulphur melt} = \frac{15000 \times 1000 \times 20}{330 \times 24 \times 60 \times 2.07}$$

$$= 305 \text{ litres}$$

Some absorber liquor is also separated in this stage. Assume a furnace with a 500 litre capacity.

Sulphur and H_2S are burnt in a package boiler. A unit to produce 31 500 kg/h steam at 300 psi will be required.

9. CAUSTIC SODA PRODUCTION

The type of equipment and circuits used for slaking, classifying and causticising are described and illustrated in the literature.[6,7,8] A total residence time of one hour using 3 tanks in series has been assumed. Volumetric flow rate is 1050 litres/min liquor plus 227 litres/min steam.

$$\therefore \text{Total operating volume} = 1277 \times 60$$

$$= 76.5 \text{ m}^3$$

$$\text{or } 25.5 \text{ m}^3 \text{ per tank}$$

Size of each tank is 4 m diameter and 2½ m deep.

Feed to the thickeners contains about 13% solids. A settling area of 2 m²/tonne/day has been assumed to obtain 40% solids in the underflow.

$$\text{Area required} = \frac{109900 \times 2}{330}$$

$$= 665 \text{ m}^2$$

$$\text{Diameter of thickener} = 30 \text{ m.}$$

Three of these are required in series for washing purposes.

Solids from the third thickener are filtered and washed on a rotary vacuum drum filter. Assuming a medium filtration rate of 1 tonne/h/m², Filtration area $= \dfrac{109900}{330 \times 24}$

$= 13.9\ m^2$

Total surface area of filter drum $= 13.9 \times 4$

$= 55.6\ m^2$

The caustic liquor is concentrated to 50% NaOH in a double-effect evaporator. The required heat exchange area of each evaporation stage is calculated to be 350 m^2 using published methods for determining evaporation areas.[14]

A final thickener is used to settle precipitated Na_2CO_3. The liquid has a high specific gravity and a high viscosity, and hence the settling rate will be slow. A thickener requirement of 10 m^2/tonne/day has been assumed to settle 7% solids in the feed to form an underflow containing 25% solids.

Area required $= \dfrac{12140 \times 10}{330}$

$= 368\ m^2$

Diameter of thickener $= 22\ m$

The underflow is filtered on a drum filter.

Assuming a filtration rate of 250 $kg/h/m^2$,

Filtration area $= \dfrac{12140 \times 1000}{330 \times 24 \times 250}$

$= 6.1\ m^2$.

Total surface area of filter drum $= 6.1 \times 4$

$= 24.4\ m^2$

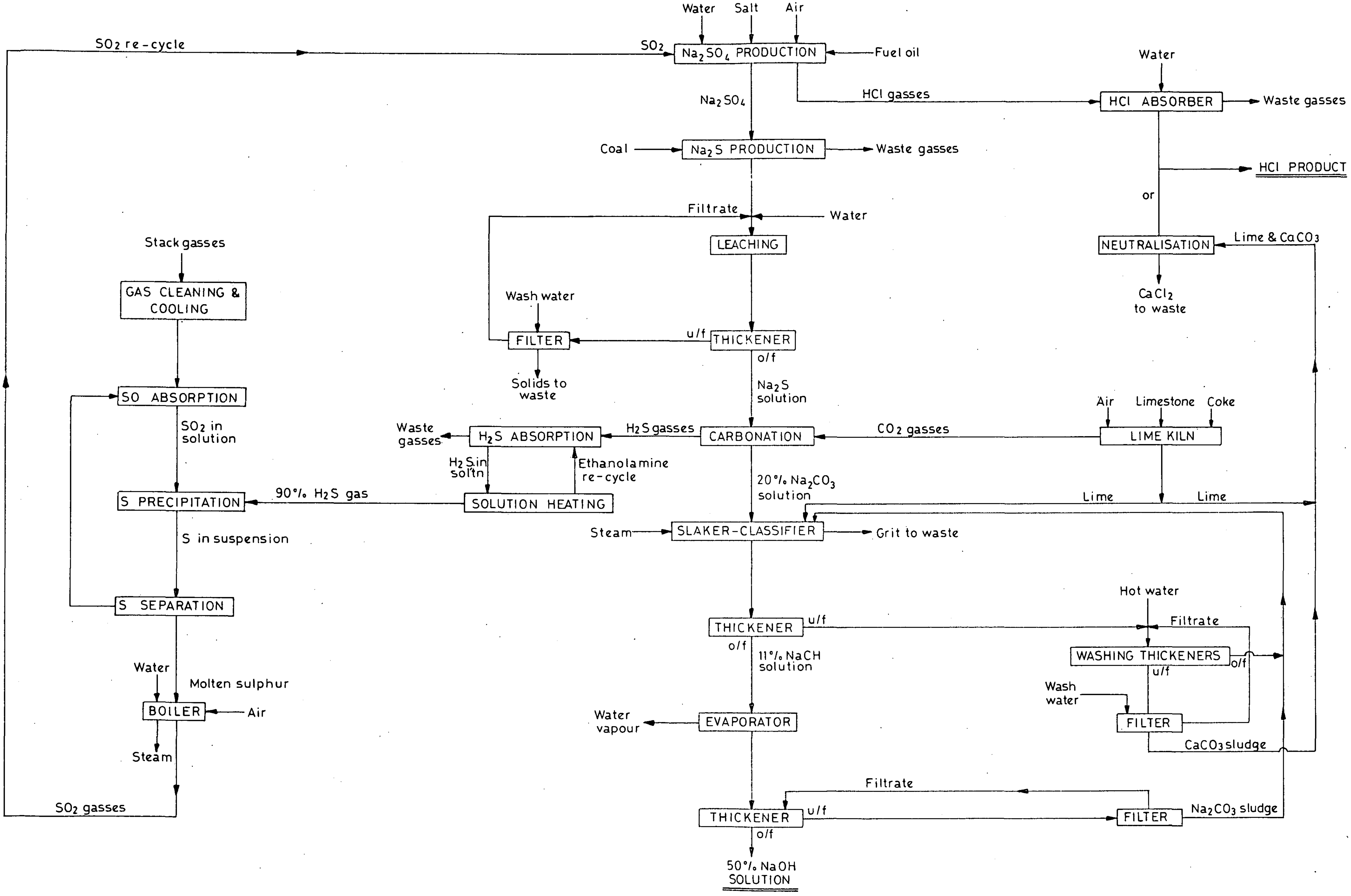

FIG.1: PROCESS FLOW SHEET