

# Recovery of Potassium Chloride, Potassium Sulfate and Boric Acid from the Salar de Atacama Brines

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## ABSTRACT

*A solar evaporation process has been designed for the extraction of potassium chloride, potassium sulfate and boric acid from the brine deposits of the Salar de Atacama in northern Chile. The brine will be concentrated in a series of ponds. A sequence of three stages was defined for the crystallization of halite, sylvinite and mixed sulfate salts, containing schoenite, kainite and lithium-potassium sulfate. Potassium chloride is produced by flotation of the harvested sylvinite salts. Potassium*

*sulfate is obtained from the harvested mixed sulfate salts. Boric acid is recovered by acidification from the end brines of the sulfate ponds. The harvested halite is discarded.*

*The Chilean Development Corporation (CORFO) has supported the development of a project to produce 520,000 tons per year of potassium chloride, 150,000 tons per year of potassium sulfate and 30,000 tons per year of boric acid. The project design and the economic estimates will be presented here.*

## INTRODUCTION

The high evaporation rates and minimal rainfall at the Salar de Atacama in northern Chile make it possible to use solar evaporation ponds to concentrate the brines of this large salt deposit. The phase chemistry studies of evaporating brines have demonstrated that it is possible to recover potassium salts from the mixed salts. In a series of ponds the sequence of salt crystallization is: halite, sylvinite and sulfate salts.

The potassium salts to be produced are potassium chloride and potassium sulfate. The raw materials are harvested salts from the sylvinite and sulfate ponds, respectively. Boric acid will also be recovered from the end brines of the sulfate ponds. Lithium production was not considered in this process design.

It was found experimentally that weather conditions alter both the quantity of the solar deposit and composition of the mixed salts that crystallize in the solar ponds. Therefore, the evaporation process design considers two different end brine compositions for each evaporation stage; one for "summer" (September to March) and the other for "winter" (April to August).

The production of potassium chloride from sylvinite is a well known process and can use technologies proven in commercial operations. Flotation is the most important method of separating sylvite from the sylvinite. The salt

one used by Great Salt Lake Minerals & Chemicals Corp. in the U.S.A. to produce potassium sulfate. This similarity allows an analogy to be made with their operation. However, the lithium content in the harvested sulfate salts has to be adjusted in order to use the Great Salt Lake process.

The boric acid production by acidification of the final concentrated brines also has some industrial precedent from a previously employed operation at the Chilean nitrate industry.

Experimental work was carried out to demonstrate the feasibility of producing  $KCl$  and  $K_2SO_4$  from the solar evaporation salts, and  $H_3BO_3$  from the end brines of the pond system. The steps involved in the commercial operation (Figure 1) are the following:

- Pumping of brines from production wells to solar evaporation ponds
- Concentration of brines in solar ponds and harvesting of the crystallized salts
- Production of potassium salts and boric acid in chemical plants.

A chemical complex to produce annually 520,000 tons of  $KCl$ , 150,000 tons of  $K_2SO_4$  and 30,000 tons of  $H_3BO_3$  was evaluated.

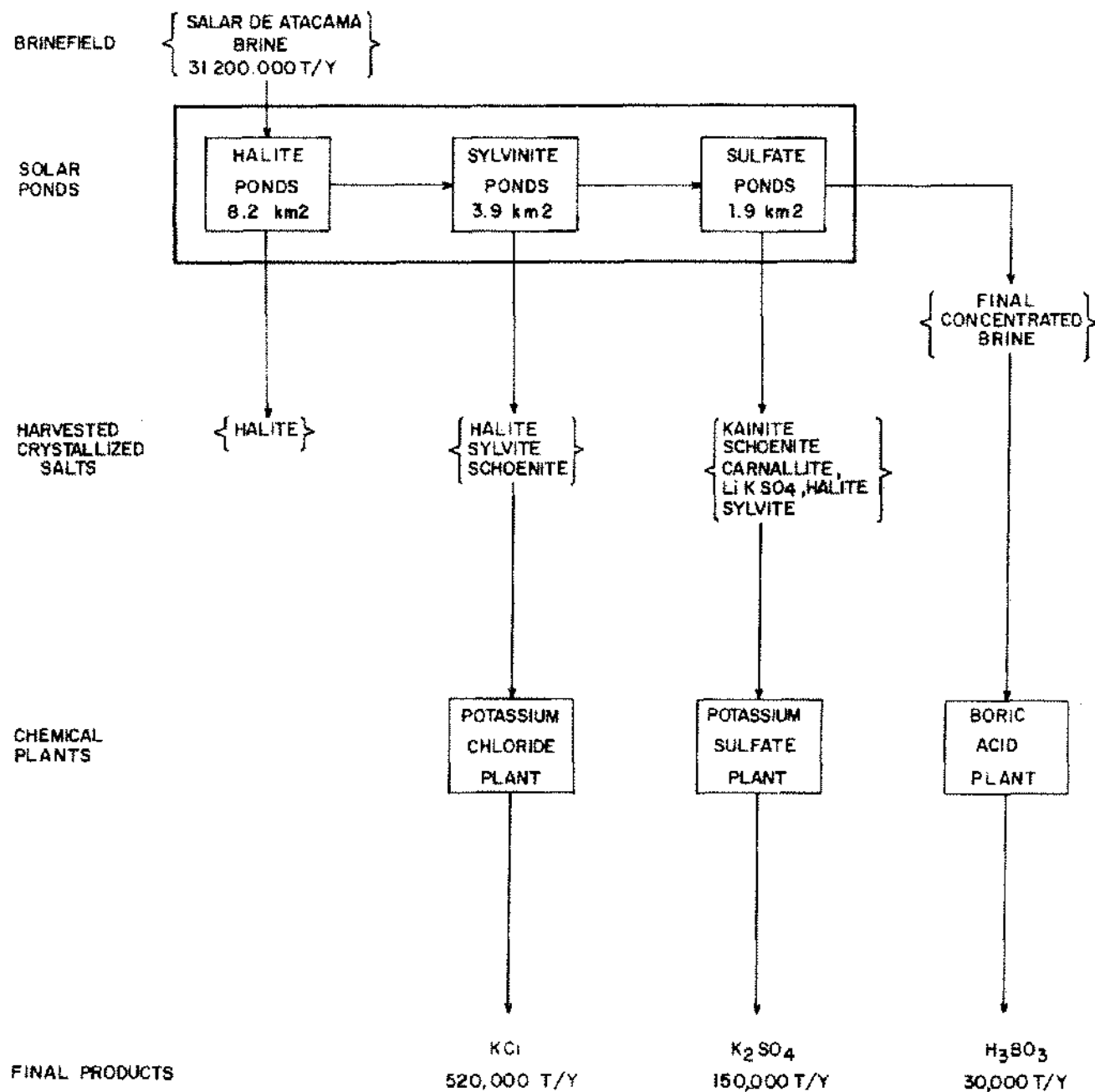


Figure 1. General flow diagram of a chemical complex to produce potassium salts and boric acid at the Salar de Atacama

### PROCESS DESIGN

**Brinefield.** Pumping tests performed in the Salar nucleus allow a brine well design based on flow of 30 l/sec. According to the plant sizes selected, the feed requirement to the solar ponds is about 31.2 million tons/year of brine. This represents an annual average flow rate in the order of 800 l/sec. Nevertheless, a higher flow is needed to meet summer evaporation rates plus the requirements

during peak filling operation. Forty production wells, which would be 16" diameter holes drilled down to 30 meters, will be necessary.

The brine delivery system will consist of a single pipeline with booster pumps extending 25 kilometers from the brinefield toward the pond area.

**Concentration of Brines in Solar Ponds.** Experimental studies carried out in the field with small metal pans has provided basic information on evaporation rates and the

phase chemistry of the Salar de Atacama brines. This information was used to define the number of stages in an evaporation process for the extraction of potassium salts, and to calculate the area of the pond system.

Brine pumped from the production wells will be concentrated in three groups of solar evaporation ponds. In the first, sodium chloride is crystallized during concentration to potassium saturation. The brine is then transferred to a second group of ponds where a mixture of halite and sylvite, with some schoenite, crystallizes. Further evaporation in a third group of ponds crystallizes potassium in the form of kainite, schoenite, potassium-lithium sulfate, potassium chloride and some carnallite, together with sodium chloride. The salts harvested from all these ponds, except the halite, will be transported to chemical plants to be processed.

The potassium content in the starting brine is about 1.8 per cent. The enormous amount of water which must be evaporated would make the process uneconomic were it not for the possibility of using solar evaporation. Furthermore, the occurrence of good quality clays on the north side of the central nucleus of the Salar permits the construction of large, inexpensive solar ponds. An estimated total pond area of 14 km<sup>2</sup> is required to evaporate about 18 million tons per year of water to recover the desired tonnage of products. It is important to remark that the utilization of solar energy to evaporate all this water would save approximately 1.8 million tons/year of Fuel Oil No 6.

**Solar Pond Harvesting.** The ponds will be harvested after the desired crystallized salt has formed a 0.30-metre-thick layer over a previously formed protective salt floor base (0.30 m). The harvesting operation will consist of first draining all superficial solution from the pond and then trenching the salts for drainage. The harvesting system selected will be similar to the one used in the solar salt industry, where a specially designed harvester removes the salt from the floor, conveys it and loads a truck running with equal speed alongside the harvesting machine. Different equipment may be required for the mixed salts. Additional study will be necessary for the final design.

Auxiliary equipment (motor graders, bulldozers, front-end loaders, etc.) will be used to windrow the salt, and for cleanup and bottom smoothing. The meteorological conditions at the Salar de Atacama allow harvesting throughout the whole year.

**Sodium Chloride Production.** The first group of ponds is used to crystallize approximately three quarters of the sodium chloride present in the brine. The halite salts will be harvested and discarded.

The brine density changes from 1.226 to 1.262 g/cm<sup>3</sup> in this stage, crystallizing approximately 330 kg of sodium chloride per 1,000 kg of evaporated water.

Chemical analyses of crystallized salts in test pans have shown that in the halite stage potassium precipitates

earlier in winter. To minimize the potassium losses different end brine compositions were defined for summer and winter, as indicated in Table 1. Therefore, the crystallized salts in the halite ponds are practically pure sodium chloride.

**Potassium Chloride Production.** The sylvinite salts harvested from the second group of ponds will be the raw material for the potassium chloride plant. The type of salts that crystallize in these ponds are slightly different for the summer and winter. For this reason, the end brine compositions were defined to avoid the crystallization of lithium as KLiSO<sub>4</sub> in summer and to keep the crystallization of schoenite in winter as low as possible. The average composition for these brines and salts are presented in Table 2. In the sylvinite ponds about 60% of the entering potassium will crystallize. For each 1,000 kg of evaporated water over 450 kg of salts precipitate.

Potassium chloride crystallizes together with sodium chloride as a salt mixture called sylvinite. The process to recover potash from this salt is well known and involves the flotation of the sylvite from the sylvinite. A schematic flow diagram is shown in Figure 2.

The sylvinite salts are first subjected to a wet grinding to get to the liberation size. Laboratory work on the flota-

TABLE 1

Halite Pond Brine Composition  
(Moles/1000 moles H<sub>2</sub>O)

	Original Brine	Summer End Brine	Winter End Brine
Na <sub>2</sub> Cl <sub>2</sub>	41.57	30.40	37.43
K <sub>2</sub> Cl <sub>2</sub>	5.75	11.80	13.50
Li <sub>2</sub> Cl <sub>2</sub>	2.84	5.82	4.71
MgCl <sub>2</sub>	5.36	11.00	9.21
MgSO <sub>4</sub>	4.97	10.20	10.32
H <sub>3</sub> BO <sub>3</sub>	1.58	3.24	2.73
Density (g/l)	1,226	1,264	1,258

TABLE 2

Average Composition of End Brines  
and Salts in the Sylvinite Ponds

	Brine (moles/1000 moles H <sub>2</sub> O)			Salt (Weight %)	
	Summer	Winter		Summer	Winter
Na <sub>2</sub> Cl <sub>2</sub>	15.8	17.0	NaCl	68.2	66.9
K <sub>2</sub> Cl <sub>2</sub>	9.9	9.1	KCl	25.6	22.5
Li <sub>2</sub> Cl <sub>2</sub>	12.2	9.5	Schoenite	—	10.4
MgCl <sub>2</sub>	23.0	21.0	Kainite	5.2	0.2
MgSO <sub>4</sub>	19.6	15.3	KLiSO <sub>4</sub>	—	—
H <sub>3</sub> BO <sub>3</sub>	6.3	5.5			
Density (g/l)	1,289	1,286			

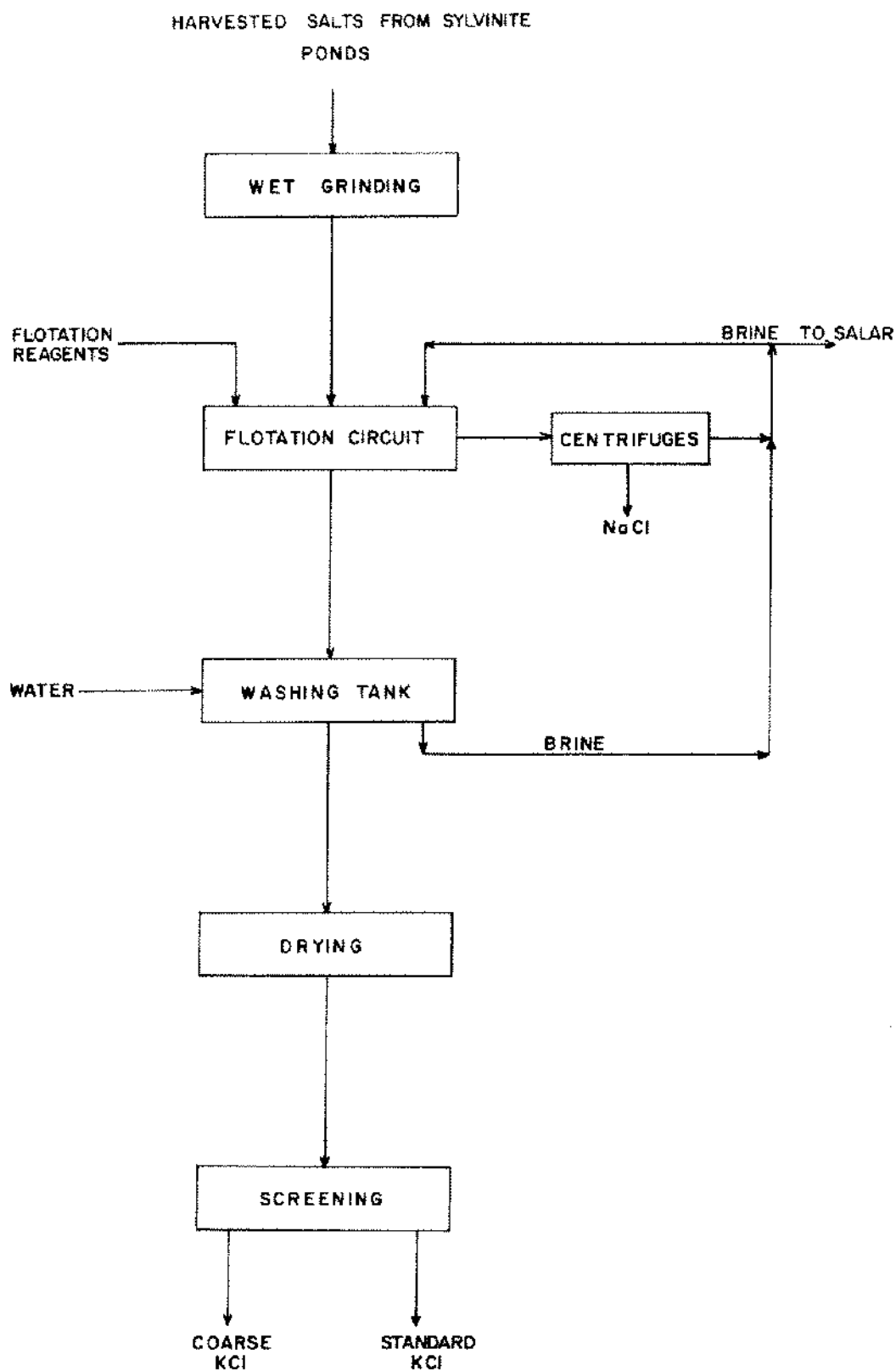


Figure 2. Schematic flow diagram of potassium chloride production

tion of salts harvested from test metal pans reveals that a 35-mesh grind will be adequate to separate the sylvite from the halite. The salts will next be treated with a fatty acid-amine flotation reagent and the potash floated in conventional flotation cells.

The product from the finishing cells will contain 90 to 95 per cent KCl, and will be given a brief leach to dissolve enough of the sodium chloride to bring the purity to 96 per cent, which is equivalent to the industry minimum standard of 60% K<sub>2</sub>O. The leached product will be washed, dried and screened into a coarse and standard fraction. An 86% total potash yield has been assumed in the plant design.

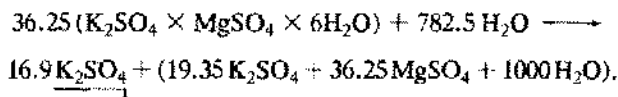
**Potassium Sulfate Production.** The production of potassium sulfate involves the conversion to schoenite of the potassium salts contained in the mixed salts harvested from the sulfate ponds. These mixed salts, depending upon the seasonal conditions, will consist of several of the following species: halite, sylvite, schoenite, kainite, carnallite and potassium-lithium sulfate. To achieve the best composition of mixed sulfate salts for the conversion it is necessary to keep the end brine compositions at the level shown in Table 3. Under this adjustment, the lithium concentration in the mixture of summer and winter salts will be less than 1.0 per cent. In this stage about 60% of the entering sulfate will crystallize. For each 1,000 kg of evaporated water over 550 kg of salts crystallize (Parada-Frederick, 1982).

The conversion can be done easily if the salts are brought into contact with a low magnesium chloride brine at 25°C. It was found experimentally that all the lithium went into solution when the lithium content of the mixture of salts was less than 1.0 per cent. Furthermore, it is possible to utilize the phase diagram for the reciprocal salt pairs K<sub>2</sub>, Mg//Cl<sub>2</sub>, SO<sub>4</sub> (Figure 3) to represent the conversion step of the potash salts into schoenite.

The resulting schoenite can be readily separated from halite by flotation. Several flotation collectors were tested at the laboratory with the ground mixture of salts at -48

mesh. The best results were obtained with Duomac T and the sulfonate reagents, Aeropromoter 801-R and 830-R. Over 94% of schoenite was recovered with a purity of 95% in the first flotation stage.

The potassium sulfate is produced as a solid phase by leaching the schoenite with water at 50°C. The phase chemistry of this reaction is given in the diagram of the system MgSO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>O at 50°C, shown in Figure 4. At this temperature the reaction of the schoenite with water is represented by the equation:



A potassium yield of 44% was achieved from laboratory experiments. The water consumption was 4.85 per unit of K<sub>2</sub>SO<sub>4</sub> produced. The resulting end liquor from the leaching is recycled to be used as the conversion brine in the prior schoenite production.

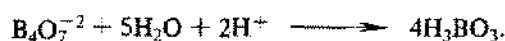
It is possible to increase the potassium sulfate production by using a solution of KCl instead of pure water. This reaction known as metathesis, is represented in Figure 5. Depending on the amount of KCl used in this reaction more potassium sulfate could be produced. A commercial grade product (50% K<sub>2</sub>O minimum) can be obtained after dewatering, washing and drying.

Figure 6 indicates a schematic flow diagram of the potassium sulfate production.

**Boric Acid Production.** Two potential methods of producing boric acid from the Salar de Atacama brines were studied: solvent extraction and crystallization by acidifying the brine with sulfuric acid. Although the yield is not high, the last method appears to be the simplest and with the lower overall cost, essentially, because it requires low acid consumption.

The amount of boric acid recovered by acidification is primarily dependent upon the initial concentration of boric acid in the brine, as illustrated in Figure 7. Boron is present in the brine as borate and boric acid, but its concentration is all calculated as boric acid. As the boric acid concentration increases in the brine, the pH decreases, as shown in Figure 8. Laboratory tests performed at a temperature in the order of 18°C have demonstrated that boric acid crystallizes by acidification when the concentration is higher than 21 g/l.

The chemical reaction involved is the following:



For the acidification the optimum pH for crystallizing the boric acid appears to be about 2.0 for brines having a boric acid concentration between 25 g/l and 50 g/l. Figure 9 shows that the sulfuric acid consumption increases

TABLE 3

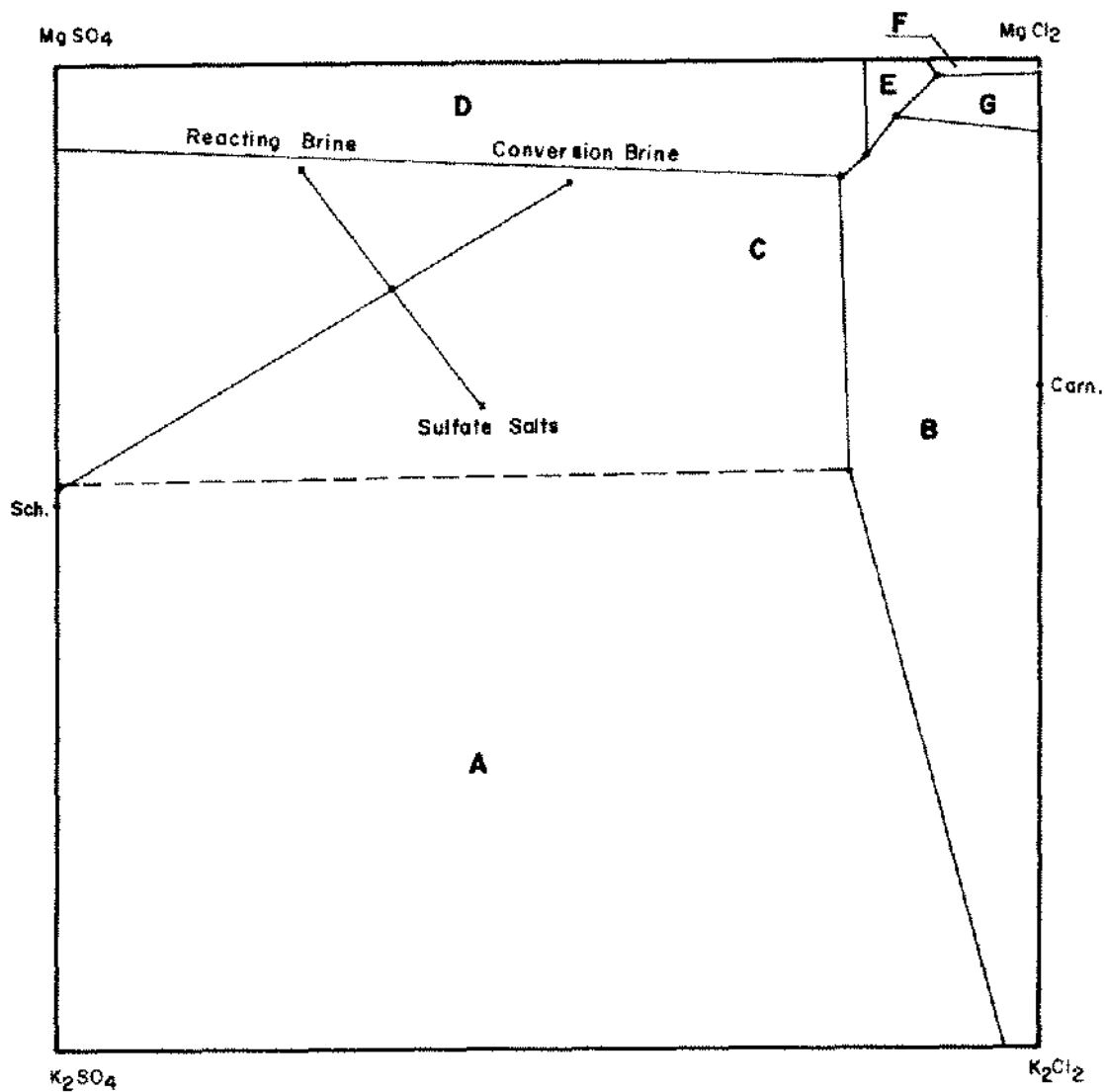
Average Composition of End Brines  
and Salts in the Sulfate Ponds

	Brine (moles/1000 moles H <sub>2</sub> O)			Salt (Weight %)	
	Summer	Winter		Summer	Winter
Na <sub>2</sub> Cl <sub>2</sub>	1.76	4.60	NaCl	40.99	40.50
K <sub>2</sub> Cl <sub>2</sub>	4.10	4.90	KCl	3.82	6.33
Li <sub>2</sub> Cl <sub>2</sub>	13.65	21.30	Kainite	24.77	33.89
MgCl <sub>2</sub>	51.00	52.00	Schoenite	—	19.28
MgSO <sub>4</sub>	12.11	11.60	Carnallite	3.01	—
H <sub>3</sub> BO <sub>3</sub>	10.32	12.30	KLiSO <sub>4</sub>	27.41	—
Density (g/l)	1,304	1,304			

# DIAGRAM

$K_2, Mg, // SO_4, Cl_2$

$T = 25^\circ C$



A =  $K_2SO_4$

B =  $K_2Cl_2$

C =  $K_2SO_4 \cdot MgSO_4 \cdot 6H_2O$  (Sch)

D =  $MgSO_4 \cdot 7H_2O$

E =  $MgSO_4 \cdot 6H_2O$

F =  $MgCl_2 \cdot 6H_2O$

G =  $KCl \cdot MgCl_2 \cdot 6H_2O$  (Carn.)

Figure 3. Conversion of Mixed Salts to Schoenite

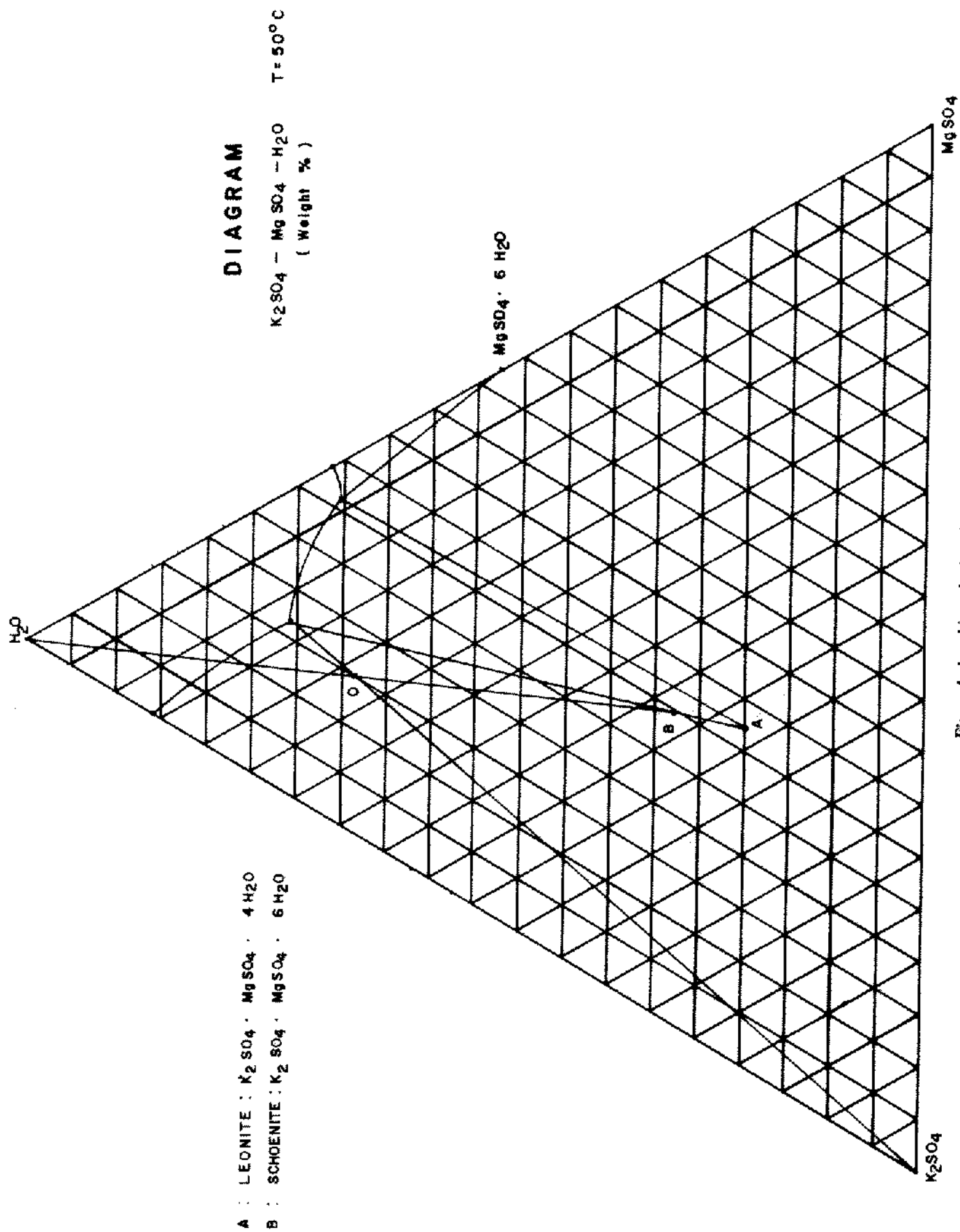
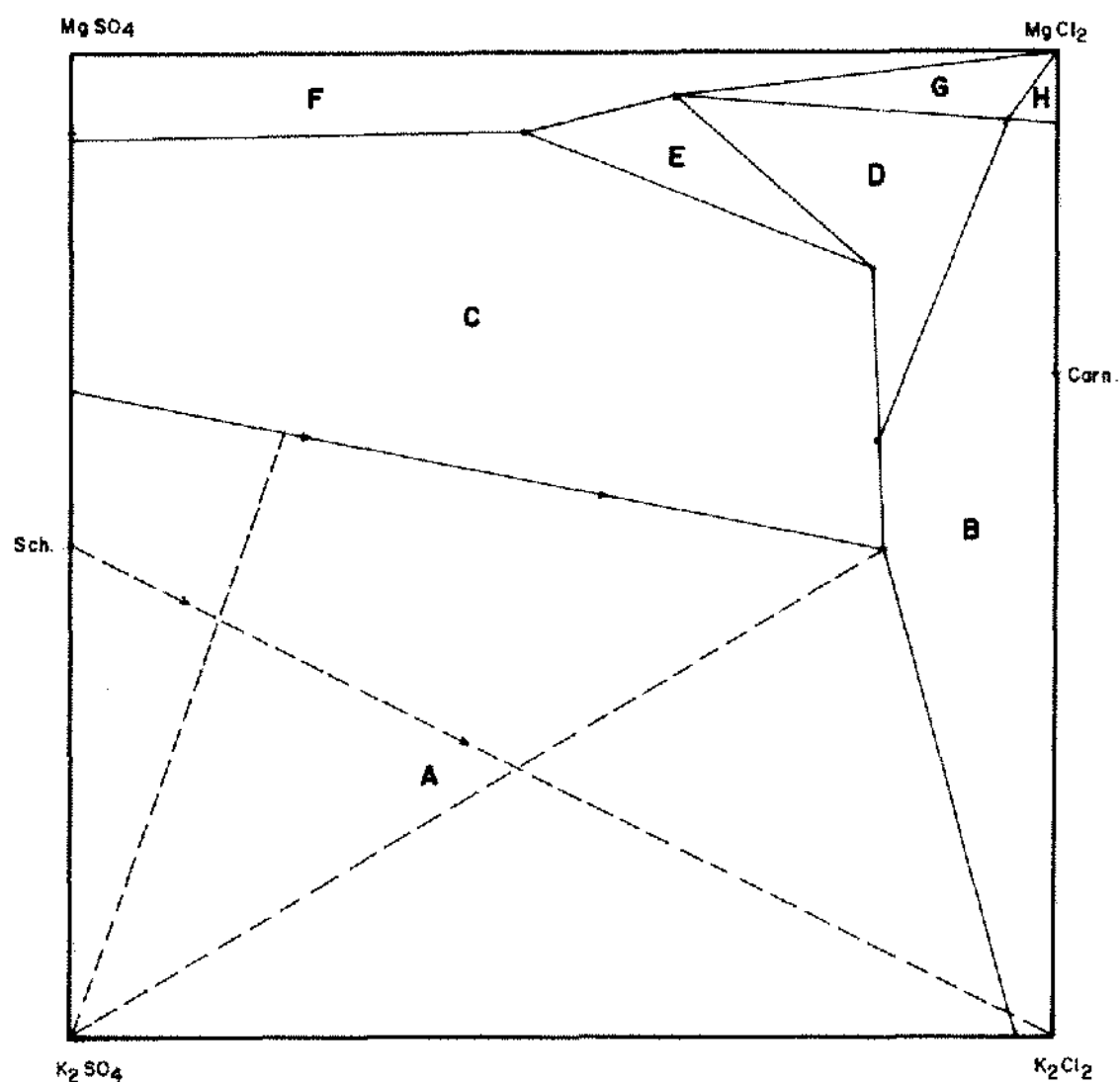


Figure 4. Leaching of schoenite

# DIAGRAM

$K_2Mg // SO_4, Cl_2$

$T = 55^\circ C$



- |  |                                    |
|--|------------------------------------|
| A = $K_2SO_4$                          | E = $K_2SO_4 \cdot 2MgSO_4$        |
| B = $K_2Cl_2$                          | F = $MgSO_4 \cdot 6H_2O$           |
| C = $K_2SO_4 \cdot MgSO_4 \cdot 4H_2O$ | G = $MgSO_4 \cdot H_2O$            |
| D = $KCl \cdot MgSO_4 \cdot 2,75H_2O$  | H = $KCl \cdot MgCl_2 \cdot 6H_2O$ |

Figure 5. Potassium sulfate production by metathesis



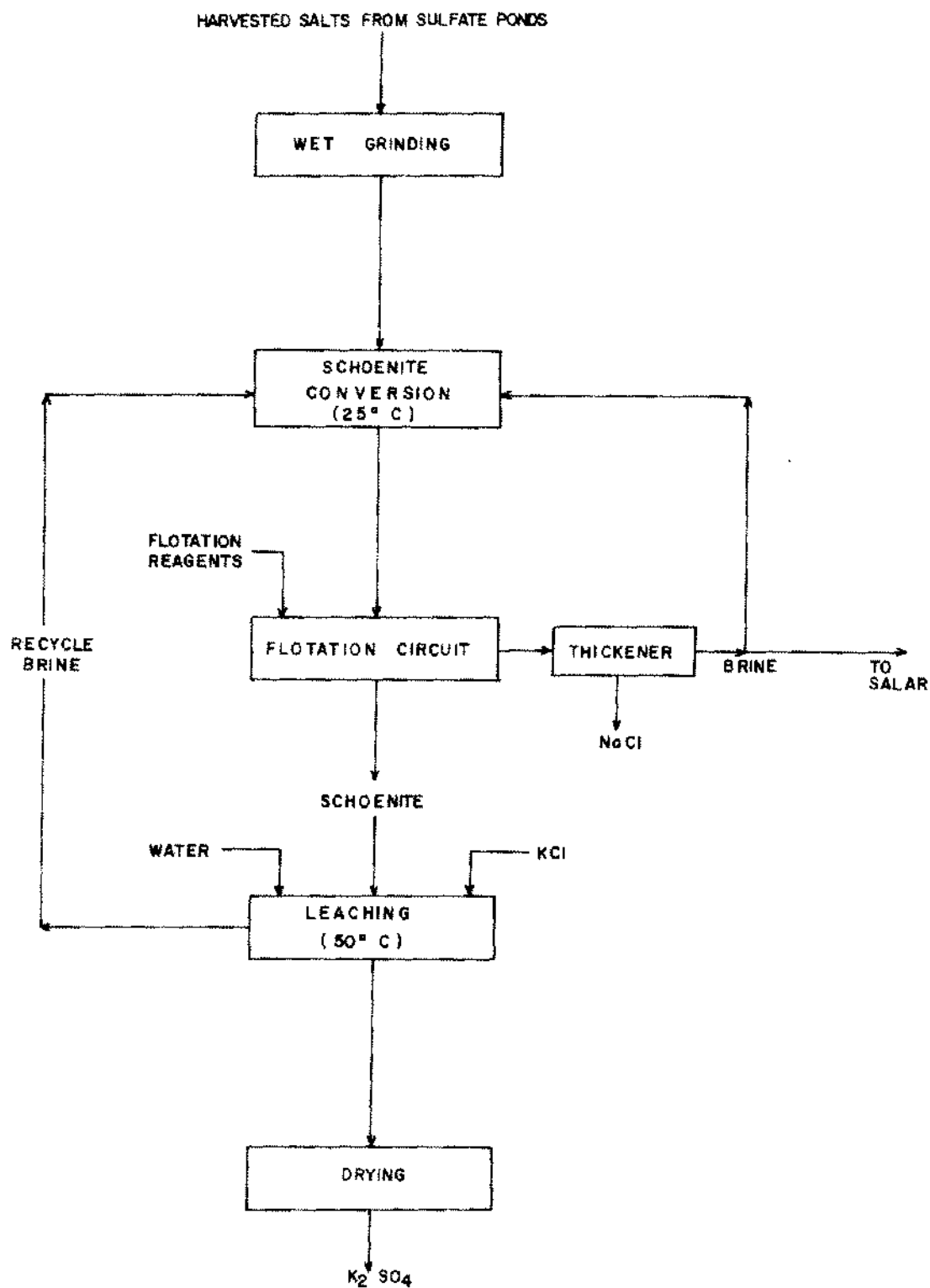


Figure 6. Schematic flow diagram of the potassium sulfate production

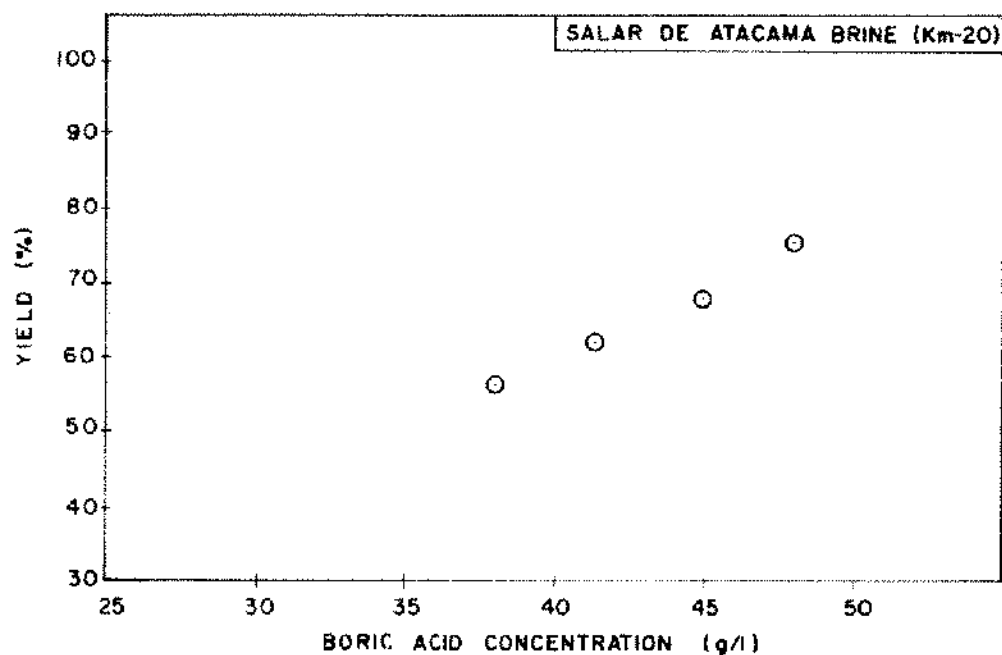


Figure 7. Relationship between boric acid recovery and initial boric acid concentration in the brine

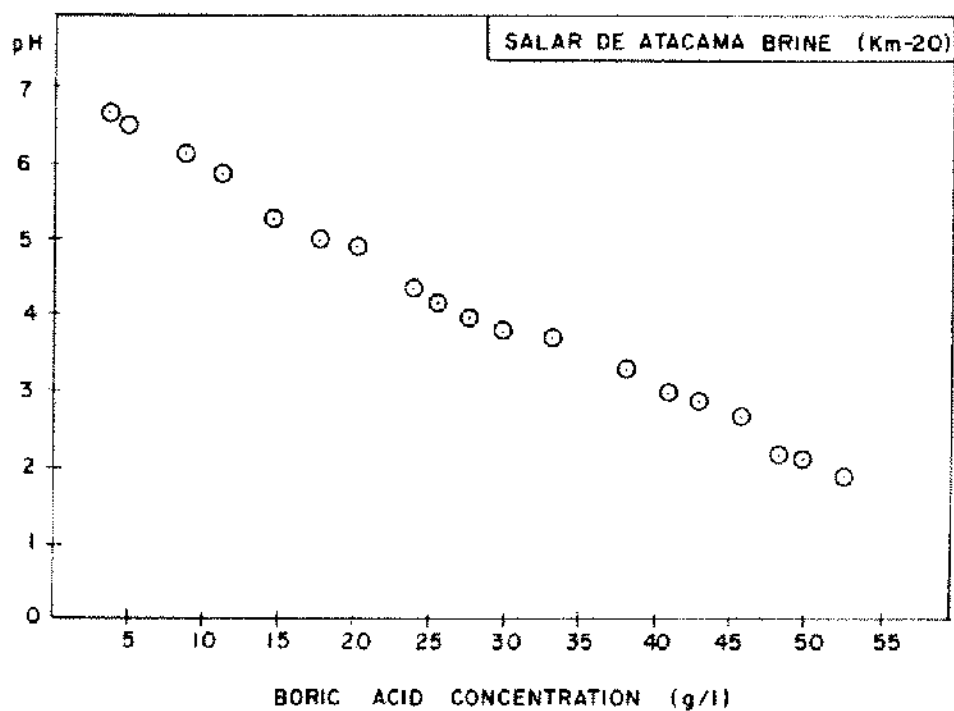


Figure 8. Variation of pH with boric acid concentration in the brine

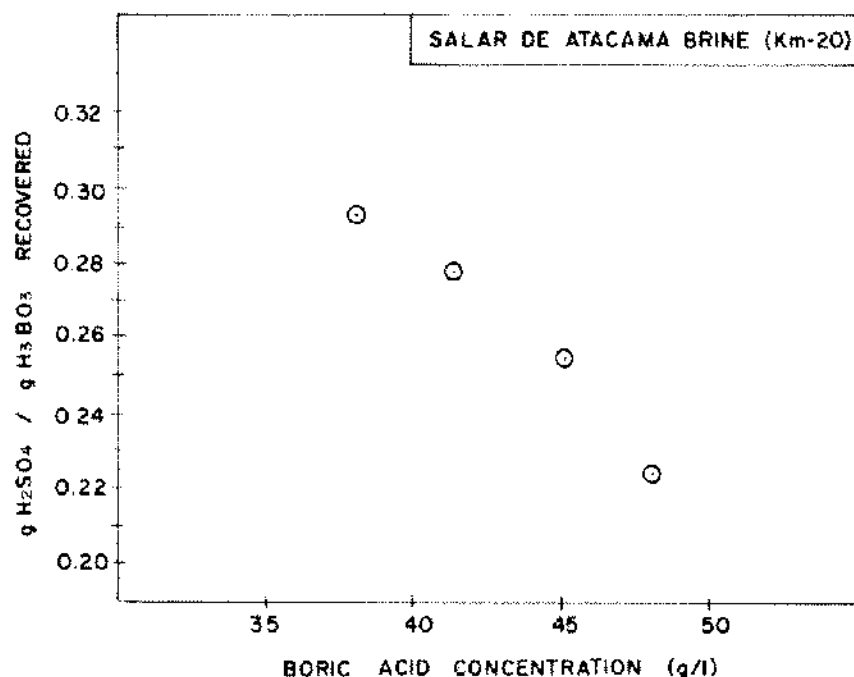


Figure 9. Variation of sulfuric acid consumption with boric acid concentration in the brine

when the brine is less concentrated in boric acid. The acid requirement is expressed per unit of  $H_3BO_3$  recovered.

The boric acid production in this process design has been conceived by taking the end liquor from the mixed sulfate ponds as raw material. This brine contains an average boric acid concentration of 35 g/l, so that about 48% yield will be obtained with a consumption of 0.34 g  $H_2SO_4$ /g  $H_3BO_3$  crystallized. By increasing the boric acid content of the brine over 35 g/l, the yield would grow, but it also would increase the crystallization of lithium in the ponds as  $Li_2SO_4 \times H_2O$ , together with carnallite. This situation has to be avoided in order to keep most of the lithium in solution to return it to the Salar. By concentrating the brine over 90 moles  $Mg Cl_2$ /1000 moles  $H_2O$ , boric acid precipitates in meaningful amounts, together with other salts.

A schematic flow diagram for the boric acid process is indicated in Figure 10. Basically, only a crystallizer and its supporting equipment are required. Care must be taken to disperse the sulfuric acid gently and thoroughly into a large volume of recirculating slurry and to maintain the pH control accurately. An excellent product should result directly without recrystallization. As some lithium sulfate crystallizes in the acidification by a salting-out reaction, it is necessary to leach the product before drying.

### ECONOMIC ANALYSIS

**Background.** The state-owned Chilean Development Corporation (CORFO) has developed two projects for the

exploitation of the Salar de Atacama mineral wealth: a lithium project and a potash project. In the first, following the successful completion of a feasibility study carried out by Foote Mineral Co. of the U.S.A., a joint venture called Sociedad Chilena de Litio Ltda. (SCL) was established in 1980. Foote, one of the two leading producers of lithium in the world, owns 55% of SCL, while CORFO holds 45%.

A lithium carbonate plant, which is designed to produce 14 million pounds per year and will cost approximately US \$60 million, is being constructed by SCL. This company will operate in a concession area located at the south of the Salar nucleus (Figure 11). The plant itself, to be built near the port city of Antofagasta, will convert a concentrated brine produced at the Salar through a solar evaporation process into lithium carbonate. This plant is scheduled to come on stream in mid-1984.

The second project corresponds to the chemical complex for the production of 520,000 t/y of potassium chloride, 150,000 t/y of potassium sulfate and 30,000 t/y of boric acid. Feasibility studies, already completed, were conducted by CORFO's Mixed Salts Committee, with the assistance of Saline Processors Inc. of California.

The nominal plant sizes aforementioned were selected in accordance with the world market possibilities and the estimated brine reserves. Brazil is the main potential client. Reserves in the area intended for the project amount to 47 million tons of potassium, 2.3 million tons of boron, 24.5 million tons of magnesium and 3.6 million tons of lithium.

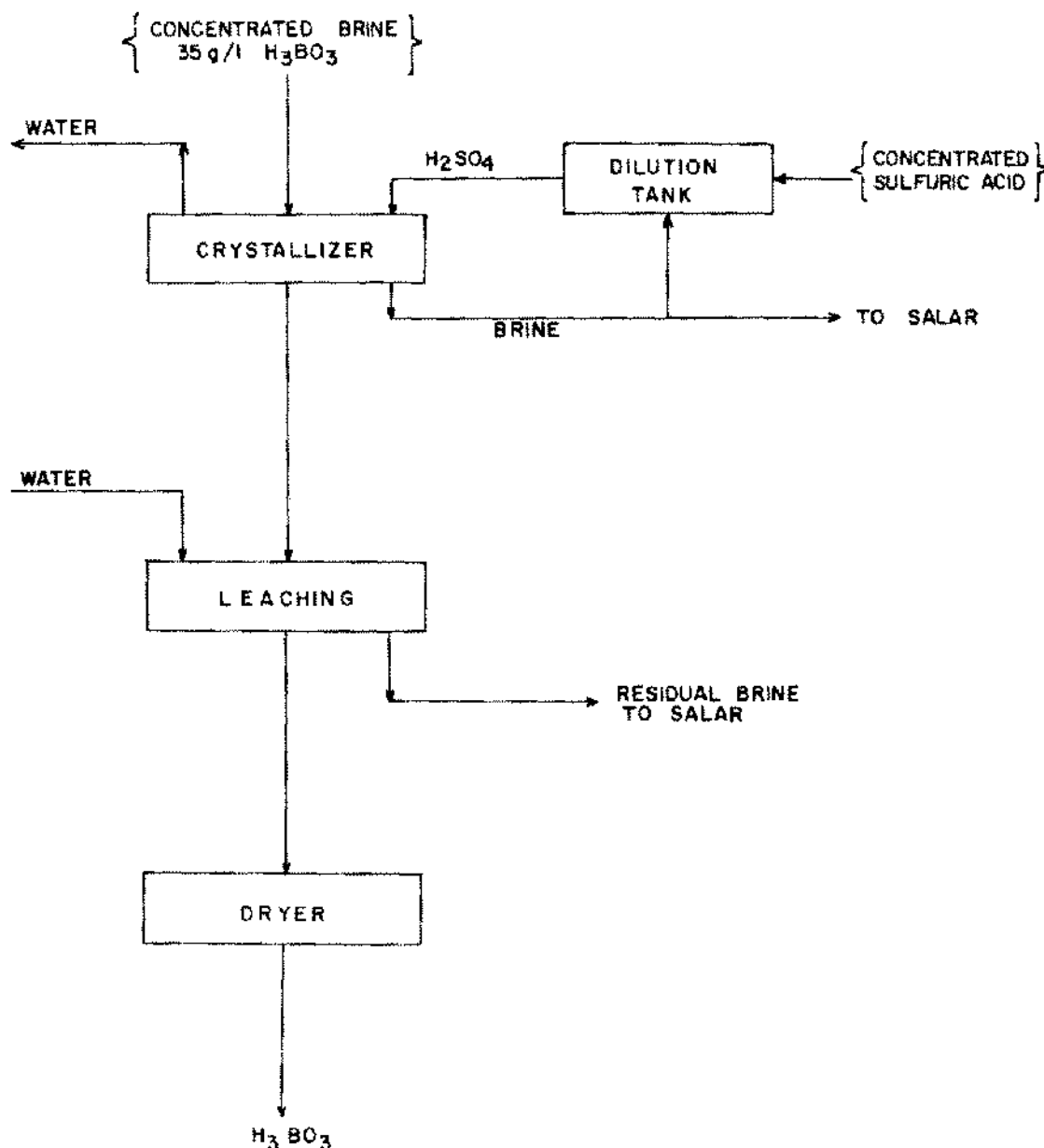


Figure 10. Schematic flow diagram of boric acid production

Lithium production was not considered in the process design of the potash project.

**Basis for Evaluating the Potash Project.** The proposed flow sheets for each plant are shown in Figures 12, 13, and 14 (Saline Processors Inc., 1979).

Although the KCl plant contemplates the production of 520,000 t/y, an amount of 500,000 t/y will be sold, and the remaining 20,000 t/y is required to achieve the desired  $K_2SO_4$  annual tonnage.

The project design and cost estimates are based upon the following general considerations:

1. The solar ponds and chemical plants will be located in the north of the Salar (Figure 11). The plants are 5 km away from the ponds.
2. Pond construction (estimated 14 km<sup>2</sup>) will consist only of constructing dikes, since an area of good quality clays will provide the impervious bottom.

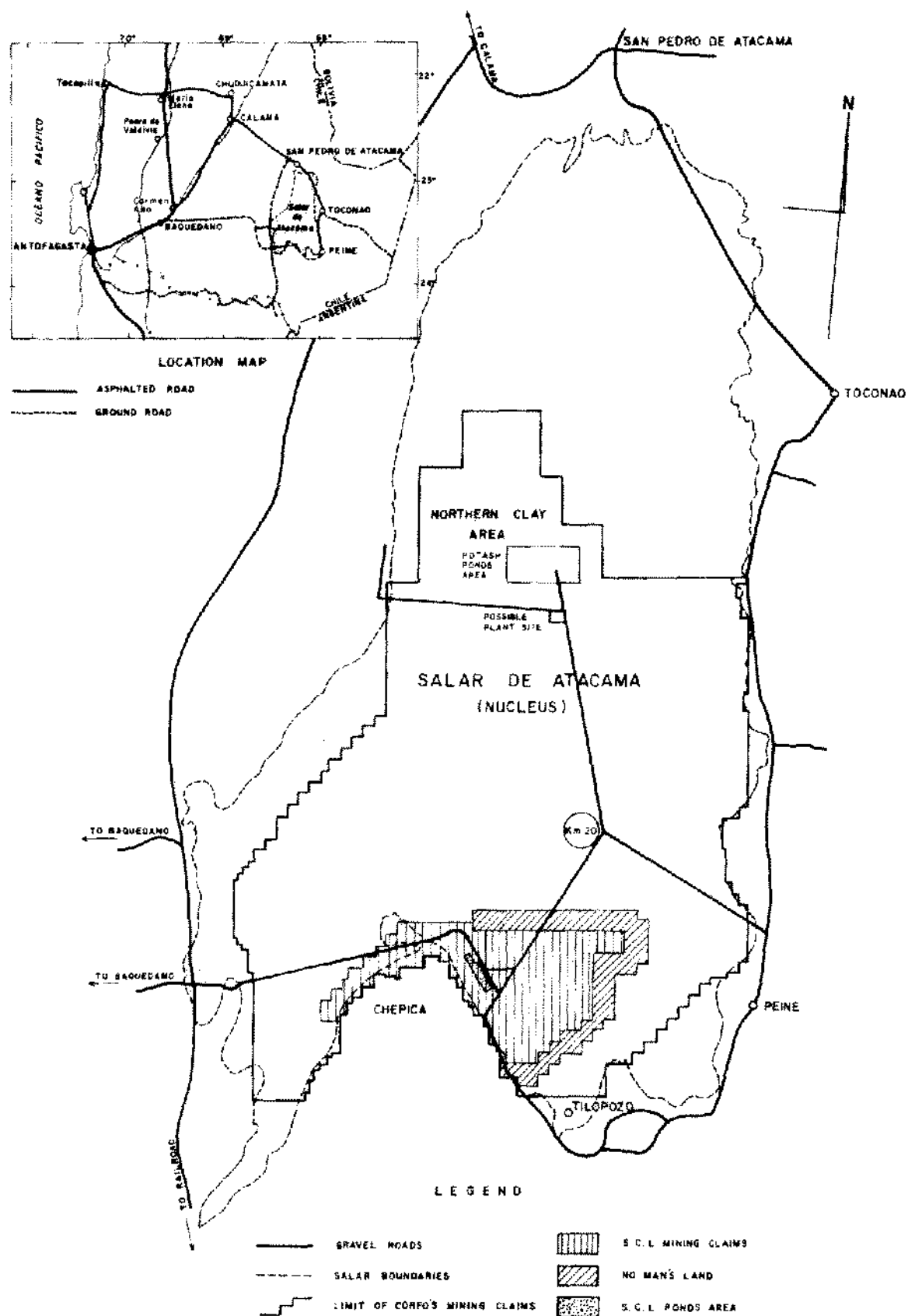


Figure 11. Map showing Salar de Atacama project site

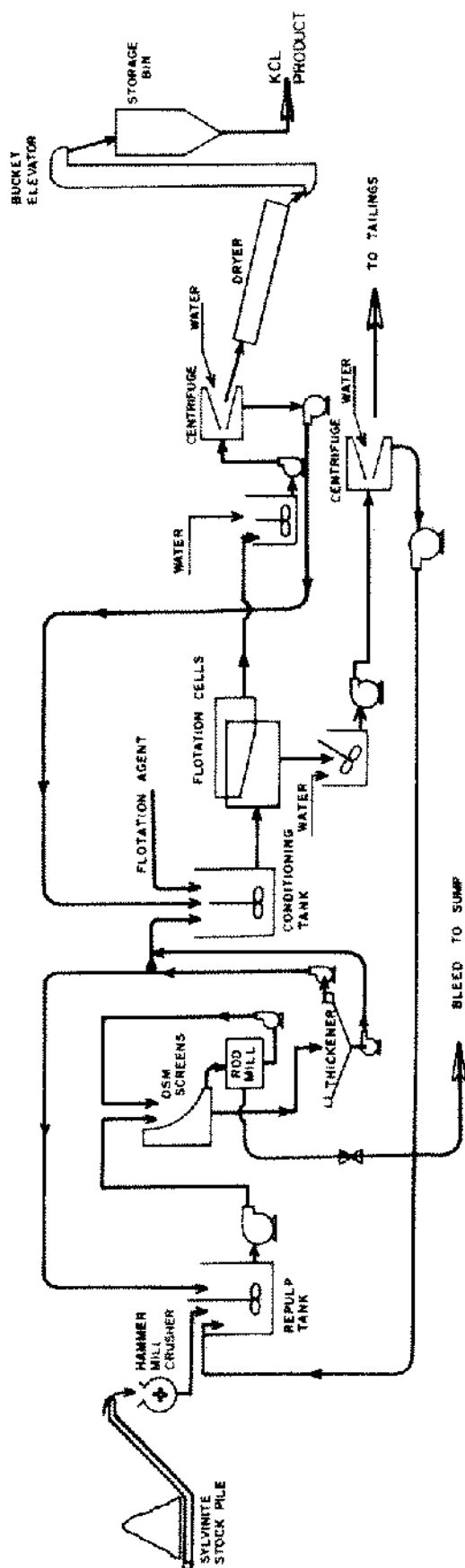


Figure 12. Flow sheet of potassium chloride plant

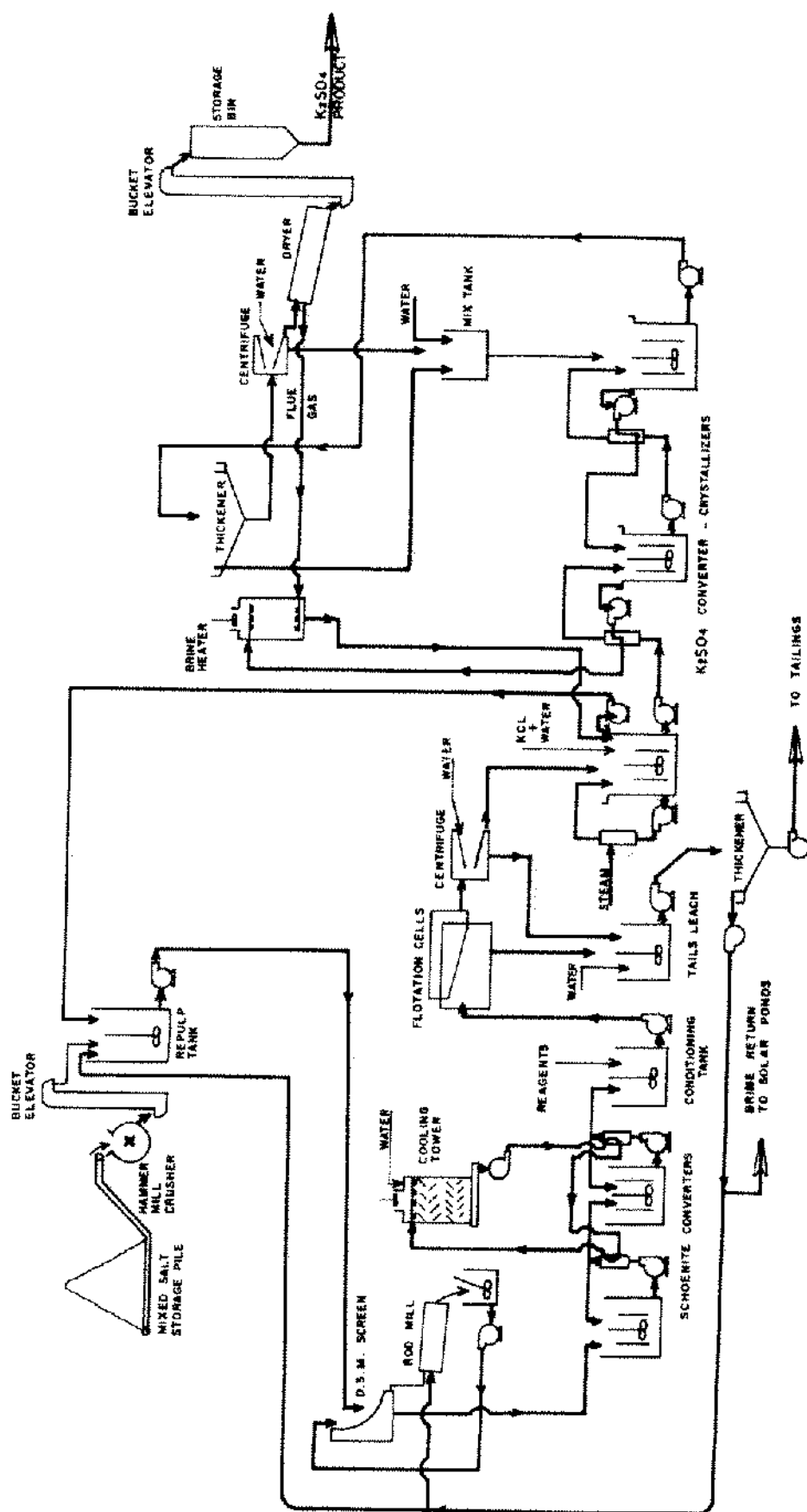


Figure 13. Flow sheet of potassium sulfate plant

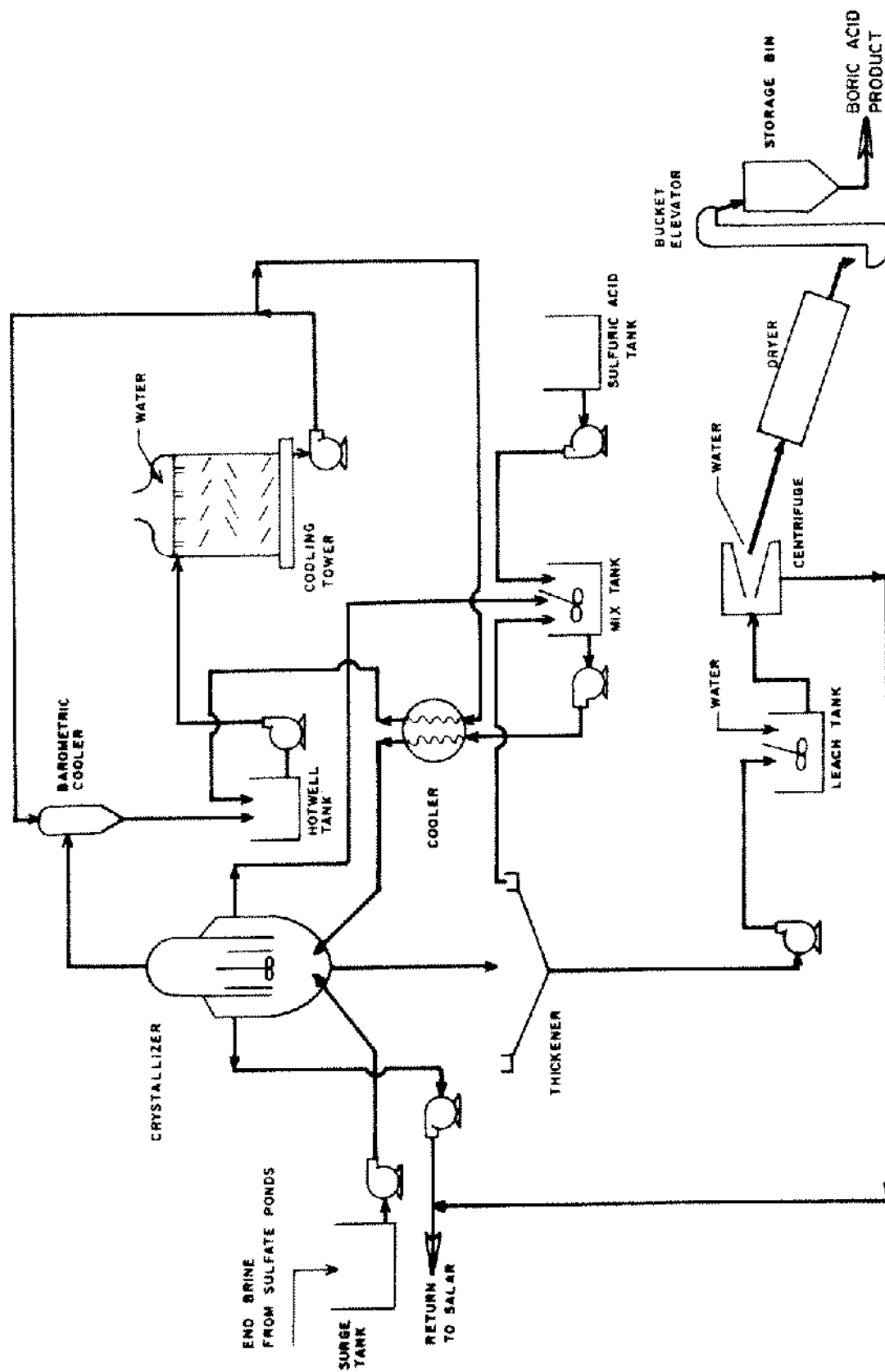


Figure 14. Flow sheet of boric acid plant



Material for the dikes will be hauled from a borrow pit 18 km away.

3. Km 20 is considered the brinefield site, 25 km away from the ponds.
4. Dry harvesting.
5. Truck hauling of potash salts from plant site (Salar de Atacama) to María Elena-Coya Sur (nitrate industry location), using public roads of the route Toconao-San Pedro de Atacama-Calama-María Elena (approximately 260 km), and a 30 km new road to be constructed from plant site to Toconao. It has been assumed that the trucks to be used will be owned by the company.
6. Railway shipment from Coya Sur to the port of Tocopilla (approximately 90 km). The present bulk loading facilities of this port, belonging to the nitrate industry, will be used.
7. A storage facility of 70,000 tons capacity for potash salts will be constructed in Tocopilla.
8. Boric acid will be truck hauled in bags to the port of Antofagasta (390 km away from the plant).
9. A township with auxiliary facilities will be set up near Toconao.
10. Electrical power (10 MW) will be provided by diesel-driven generators.
11. Process water, supplied from a source close to Toconao area, will be pumped through a 35 km-long pipeline.

**Estimated Capital Investment.** The project capital cost estimate, calculated in U.S. dollars of December 1982, is summarized in Table 4, and is based on delivered equipment cost (Osses, 1982). The capital investment required is estimated to be US \$134.8 million in fixed capital costs and US \$16.8 million in working capital for a total of US \$151.6 million. These figures include contingency and are current costs without allowance for escalation. The interest expenses during the construction phase also have not been taken into account. It is expected that the engineering and construction of the facilities will take 4 years, including the start-up.

Of the project cost, approximately 60% will be incurred in Chilean currency and the remaining 40% in U.S. dollars. The construction management expenses and contractor's fees are included in the cost of the plants and all other facilities. It should be noted that the infrastructure represents an important percentage of the investment.

**Production Cost Estimate.** The total annual cost of operations is estimated to be US \$38.0 million excluding depreciation of the production equipment and facilities. Table 5 gives a breakdown of the operating cost estimate. Although the solar evaporation system functions 365 days per year, projected on-stream time for the chemical plants is 320 days per year. The estimated total labor force, both

TABLE 4  
Estimated Capital Investment

	US\$ (1000s)
KCl Plant, Equipment and Erection	13,900
K <sub>2</sub> SO <sub>4</sub> Plant, Equipment and Erection	16,200
H <sub>3</sub> BO <sub>3</sub> Plant, Equipment and Erection	4,800
Solar Evaporation Ponds	14,300
Harvesting Equipment	5,400
Production Wells and Brine Delivery System	6,600
Trucks	6,200
Storage Facilities in Tocopilla	6,000
Service Facilities (power plant, water supply system, etc.)	10,200
Township	3,500
Buildings (Office, maintenance, warehouse, laboratory, change house, medical, communications, etc.)	6,800
Other Infrastructure	3,600
Engineering	11,700
Start-up Expenses	3,200
Contingencies	22,400
Working Capital	16,800
Total Current Project Cost (December 1982 prices)	\$151,600

TABLE 5  
Estimated Annual Operating Cost Before Depreciation

	US\$ (1000s)
Operating and Maintenance Labor	2,500
Operating Supplies and Materials (sulfuric acid, flotation reagents, packaging materials, mobile equipment supplies, etc)	4,250
Fuels (plants, power and utilities, mobile equipment)	9,600
Maintenance Materials	2,800
Plant Overhead Costs	1,370
General Administrative Expenses	670
Insurance and Real Estate Taxes	870
Mineral Lease Payments	200
Transportation Costs for Potash Salts (Trucking from plant site to Coya Sur)	8,070
Potash Salts Rail Freight, Coya Sur to Tocopilla, and Ship Loading Fees	4,500
Transportation Costs for Boric Acid (Trucking to Antofagasta) and Port Fees	1,070
Contingencies	2,100
Total Operating Costs (including transportation to port and ship loading)	\$38,000

salaried personnel and hourly workers, required for the project amounts to 525 people. The principal operating costs are fuels and transportation costs.

**Profitability Evaluation.** The potash project profitability has been evaluated assuming no debt (100% equity), a fifteen-year period of service life, income taxes of 49%, no escalation in capital and operating costs, and rapid

depreciation (7 years). Although a sensitivity analysis has been made to determine the impact of various changes, the results of the base case are only given here. With 100% production capacity the annual income of the project is close to US \$90 million. This assumes the following FOB sales prices: KCl (US \$90/ton),  $K_2SO_4$  (US \$180/ton) and  $H_3BO_3$  (US \$520/ton).

The DCF rate of return of the base case is 16%. The net present worth, discounted at an interested rate of 12%, is US \$33 million.

### CONCLUSION

Detailed development studies to exploit the Salar de Atacama brine deposits have provided basic information to design a process for recovery of potash salts and boric acid. An economic evaluation concludes that it is feasible to set up a chemical complex. Several international and local companies are interested in participating in the project. CORFO has planned to invite bids during 1983.

### ACKNOWLEDGMENTS

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