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Study to Establish Cost Predictions for the Production of Redox Chemicals

Paul R. Ammann, Michael Loreth,
and W. W. Harvey
Charles River Associates

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Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center
Under Contract DEN 3-251

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U.S. DEPARTMENT OF ENERGY
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FOREWORD

The chromium and iron chloride chemicals are significant first costs for the NASA Redox Energy Storage System. This study was carried out to determine the lowest cost at which chromium and iron chlorides could be obtained for a complex of Redox Energy Storage Systems. In addition, since the solutions gradually become intermixed during the course of operation of Redox units, it was an objective of this study to evaluate schemes for regeneration of the operating solutions. Three processes were evaluated for the production of chromium and iron chlorides. As a basis for preliminary plant design and economic evaluation, it was assumed that the plant would produce about 25,000 tons of contained chromium as $CrCl_3$ and an equivalent molar quantity of $FeCl_2$. Preliminary plant designs, including material and energy balances and sizing of major equipment, were prepared and capital and operating costs were estimated.

This project was carried out by Paul R. Ammann, Michael Loreth, and W. W. Harvey (a consultant to CRA in hydrometallurgical processes).

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SUMMARY

The chromium and iron chloride chemicals are significant first costs for the NASA Redox Energy Storage System. For previous system cost study evaluations, chromium chloride was priced at $1.66 per pound of contained chromium. This present study was carried out to determine the lowest cost at which chromium and iron chlorides could be obtained for a complex of Redox Energy Storage Systems. In addition, since the solutions gradually become intermixed during the course of operation of Redox units, it was an objective of this study to evaluate schemes for regeneration of the operating solutions.

Three processes were evaluated for the production of chromium and iron chlorides. As a basis for preliminary plant design and economic evaluation, it was assumed that the plant would produce about 25,000 tons of contained chromium as $CrCl_3$ and an equivalent molar quantity of $FeCl_2$. Preliminary plant designs, including material and energy balances and sizing of major equipment, were prepared and capital and operating costs were estimated. In addition to the costs of production, plant location, transportation of the chemicals, and preparation of the Redox solutions at the user site must be considered in assessing the price of these chemicals. For this preliminary study, it has been assumed that the chemicals are prepared as two molar solutions in two molar HCl at the production plant and prices are calculated FOB this plant.

Chromite ore was considered as one raw material for the production plant since it is readily available at low cost. During the course of the study, it became apparent that ferrochrome, which contains both iron and chromium, but relatively few impurities as compared to chromite ores, could be an attractive source of these metals.

Three manufacturing processes were considered for making chromium and iron chlorides. Two are hydrometallurgical processes in which either chromite ore or ferrochromium is dissolved in hydrochloric acid, and the desired metals are separated and recovered as purified two molar solutions. The third process employs the reduction-chlorination of chromite ores to generate iron and chromium chloride vapors which are separated, purified and recovered.

The economics for the three manufacturing processes are summarized in Table 1-1. The hydrometallurgical process based on ferrochrome has the lowest investment at an estimated $40 million or $0.80 per annual pound of contained chromium in the chromium chloride product. The selling price required to provide the manufacturer with a 15 percent discounted cash flow rate of return is $.97 per pound of chromium, yielding a sales to investment ratio for a 25,000 ton per year plant of $1.20 revenue per dollar invested. Ferrochrome raw material is the major manufacturing cost. The plant design is based on known and commercially employed process equipment and technology.

A second process based upon the hydrometallurgical dissolution of chromite ore could produce chromium chloride for sale at about $1.10 per pound of contained chromium. This process has an advantage over the first in using low cost chromite ore as a raw material. However, the investment requirement is estimated at $69 million or $1.39 per annual pound contained chromium product. It is a feasible process, using mainly standard hydrometallurgical technology, though there are some development needs before it could be commercialized.

A third process is based on the chlorination of chromite ore. This process is technically more difficult than either of the other two processes. The investment for a 25,000 tons contained chromium per year plant is estimated at $95 million, or $1.91 per annual pound chromium. The cost of production is slightly lower than the other processes, but the large investment would require a selling price of $1.25 per pound contained chromium. The chlorination process has the highest technical uncertainty; we believe that an extensive development program would be required prior to commercialization.

It is projected that as the Redox systems are used over time, the iron and chromium chloride salts will exchange from one electrolyte to another, and both solutions will need regeneration. One approach to regeneration of the chromium and iron chloride solutions is to return quantities of solution periodically to the manufacturing plant. The incremental cost for regeneration is estimated to be 40 to 50 cents per pound of contained chromium (for both solutions); with 1,000 miles of shipping, another 50 cents per pound of contained chromium would be incurred. Although the regeneration cost is about equal to the original cost of manufacture, it may be less than the cost of environmentally acceptable discard of the solutions. Another alternative would be to have a small regeneration system at each Redox facility to save transportation costs. This alternative has not been costed.

Table 1-1

COMPARISON OF ALTERNATIVES FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES
(25,000 TONS CONTAINED CHROMIUM PER YEAR)

Process:	Hydrometallurgical		Reduction Chlorination
Source of Chromium:	Ferrochrome	Chromite Ore	Chromite Ore
Chrome material			
Consumption, tons/yr.	47,847	91,392	91,600
Cost, cents/lb. Cr	0.53	0.17	0.17
Energy consumption			
kWh/lb. Cr	0.12	0.24	0.24
Mil. BTU/lb. Cr	0.0043	0.067	0.03
Investment*, $ million			
Fixed Capital	27.1	59.5	82.5
Working Capital & other	12.8	9.9	12.8
Total	39.9	69.3	95.3
Unit Investment*, $/annual lb. Cr	0.80	1.39	1.91
Operating Cost,** $/lb. Cr	0.87	0.82	0.81
Required selling price,+ $/lb. Cr	0.97	1.10	1.25

*Investment accuracy is estimated at plus 30%, minus 0%.
**Operating cost accuracy is estimated at plus 10%, minus 10%.
+Price to achieve 15 percent DCF-ROR on investment.

SOURCE: Charles River Associates, December 1981.

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INTRODUCTION

The NASA Lewis Research Center has developed a Redox Energy Storage System which is designed to be used with stand-alone energy generators, such as photovoltaic or wind turbine installations, and for electric utility load leveling. The Redox system is based upon the transfer of energy between acidified chromous/chromic chloride solutions and ferric/ferrous (iron) chloride solutions.

The concept has been demonstrated in laboratory cells and in a 1 kW pre-prototype unit. In a cost analysis for an electric utility load-leveling application (10mW/100mWh capacity), it was estimated that the chromium chloride chemicals represented nearly 40 percent of the initial investment (NASA briefing, 1981). For this estimate, chromic chloride was priced at $1.66 per pound of contained chromium, or 25 percent of the current selling price, under the assumption that on a large commercial scale, chromite ores can be converted into chromium chloride chemicals at lower than current costs.

In support of the Redox Energy Storage System development, Charles River Associates (CRA) was commissioned to evaluate the commercial production of chromium chloride (25,000 tons contained chromium per year) and project the prices at which the reagent could be available on a large scale.

CRA proposed to examine three candidate processes for commercial production of chromium chloride chemicals, assess their technical and economic feasibility, estimate their capital and operating costs, and calculate selling prices that would provide a reasonable return on investment to a manufacturer. In its original proposal, CRA identified three processes which employed chromite ore as the source of chromium. During the course of this study, it became apparent that ferrochromium, though 10 times higher in cost per unit of chromium than chromite ore, could produce the desired product at probably the lowest cost. With the concurrence of NASA, a process based on ferrochromium was substituted for one of the concepts originally proposed; this process has been evaluated to be technically superior and of lower cost than the others.

This report presents a description of each of the three processes, including preliminary design information, capital and operating costs, and profitability calculations.

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CHROMIUM SOURCE MATERIALS

The chromium ore selected as the starting material in this study for production of chromium chloride is based on the type of ore used currently in the production of chromium chemicals. Ore from the Transvaal area of South Africa is the primary source for this use. Therefore, since substantial ore reserves exist, Transvaal ore is recommended as the basis for the preliminary plant designs and economic analysis. The reported compositions of Transvaal ore are shown in Table 3-1.

The listed composition values differ very little from each other. The ore composition given by Udy was selected to be used in determining the costs for production of chromium chloride and iron chloride, as it is representative of raw material for chromium chemical manufacture, including domestic chromite concentrates.

Domestic ores from Montana, Oregon, and California would produce concentrates similar in composition to the Transvaal ore, but with a lower chromium content (38 to 40 percent Cr_2O_3). In times of necessity, the domestic deposits could supply sufficient ore for chromium chemical manufacture in the United States. The cost of chromium chemicals from domestic ores would likely be higher, due to their lower grade and to higher mining costs.

Several alternative source materials for the production of chromium chloride are listed in Table 3-2. A domestic supply of chromium would involve reopening small mines, and long lead times might be required to bring production on stream. Other sources include chromite from the U.S. stockpile, stainless steel and superalloy scrap, and imported ferrochromium. However, because of higher costs and potential technical problems, stainless steel and superalloy scrap were not considered in this study for production of chromium chloride.

Table 3-1

COMPOSITION OF TRANSVAAL GRADE B FRIABLE CHROMITE
Percent (Dry Basis)

Type	Cr_2O_3	Total Iron As Fe	SiO_2	Al_2O_3	MgO	CaO	V	Cr/Fe	Source
1	44.3	19.2	3.5	14.2	10.1	0.4	0.2	1.58	Udy
2	44.5	19.2	3.9	15.0	10.1	0.3	--	1.58	Sully & Brandes
3	44.5	19.2	3.9	15.0	10.0	0.3	--	1.57	Northern Miner
4	44.5	19.3	3.2	15.4	10.7	--	0.3	1.58	Kirk - Othmer

SOURCES: Udy, M. J., Chromium, Vol. 1, Chemistry of Chromium and Its Compounds, Reinhold Publishing Corp., New York, 1956.

Sully, A. H., and Brandes, E. A., Chromium, second edition, Plenum Publishing Corp., New York, 1967.

Northern Miner, November, 1978.

Kirk-Othmer Encyclopedia of Chemical Technology, Vol. 5, Editorial Board, Herman F. Mark, et al., third edition, New York, Wiley, 1978.

Table 3-2

ALTERNATIVE SOURCES OF CHROMIUM FOR PRODUCTION OF $CrCl_3$

Domestic podiform chromite deposits: mined intermittently in the past; low grade but probably suitable for $CrCl_3$ manufacture; would require opening or reopening a number of small mines; long lead times required.

Domestic stratiform chromite deposits (principally the Stillwater Complex in Montana): constitute a larger domestic resource; sampled and evaluated metallurgically, but not mined commercially in the past (except for stockpile additions); future mining for platinum metals may provide access to mined chromite; in common with other domestic chromites, this resource is low grade.

Chromite tailings from domestic nickeliferrous laterites: reduction-roast/ammoniacal-leach process for Ni and Co recovery leaves Cr as chromite in the tails; concentration would be required since only about 2 percent Cr; one planned operation would provide 20,000 tpy Cr in 70,000 tpy concentrate; one to two additional U.S. operations feasible; concentration costs substituted for mining costs; very low grade.

Chromite from the U.S. strategic stockpile: inventories of chemical, metallurgical, and refractory grades of chromite are below goal; however, the stockpile contains some low-grade domestic chromite that FEMA may wish to replace with higher grade.

Stainless steel and superalloy scrap: since these constitute the major end-uses of Cr, resources would be adequate; recycling has been and is being studied; the chemistry and metallurgy for $CrCl_3$ production would be different as compared with chromite starting materials.

Imported ferrochromium: higher base price, about 50¢/lb. contained Cr.

SOURCE: Charles River Associates, 1981.

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HYDROMETALLURGICAL PROCESSES

The dissolution of either chromite ore or ferrochrome in hydrochloric acid produces a solution of chromium and iron chloride which, after separation and purification, would be directly suitable for the Redox system. While chromite ores contain appreciable quantities of MgO, Al_2O_3, SiO_2, etc. (Table 3-1) in addition to the desired components, ferrochromium is enriched in chromium and iron and is essentially free of undesired components. Consequently, use of ferrochromium as starting material can result in a process with lower overall costs.

4.1 HYDROMETALLURGICAL FERROCHROMIUM PROCESS

Ferrochrome is produced in many countries throughout the world. Western world production in 1979 was 2.3 million tons, far below installed capacity of over 3 million tons. Thus, there would be ample supply of ferrochromium for production of Redox chemicals. There are several grades of ferrochrome; all would be suitable for production of chromium chloride. The largest impurity is carbon, which should be insoluble under the process conditions. Accordingly, use of the lower-priced high carbon ferrochrome is assumed.

Conceptually, ferrochrome is crushed and dissolved with concentrated hydrochloric acid to produce chromous and ferrous chlorides according to the principal reaction shown below:

$$Fe_{0.6}Cr + 3.2HCl(aq) = 0.6FeCl_2(aq) + CrCl_2(aq) + 1.6H_2(g) \qquad (4.1)$$

The iron and chromium chlorides are separated, purified and concentrated to product specification.

A conceptual process flowsheet is presented in Figure 4-1 and overall materials flow is shown in Figure 4-2. While only a schematic diagram is shown in this report, a complete process flowsheet including all major equipment sizes and process flows was prepared to assess technical feasibility and to determine costs.

4.1.1 PROCESS DESCRIPTION

The ferrochrome is assumed to be received in 30-gallon drums containing about 1,000 pounds of material. The drums are opened and the material transferred to a crusher rated at 25 tons per hour. The crushed material is passed over screens which are sized at 20 mesh. The oversize material is returned to the crusher and the undersize material transferred by conveyor to the storage bin, which has a four-hour holdup, or 32 tons. The ferrochrome is fed continuously to two 15,000 gallon mechanically-stirred reactors where it is contacted with concentrated hydrochloric acid at a rate of about 90 gallons per minute. In order to maintain concentration of the iron and chromium in solution, 75 gallons per minute of water must also be added continuously. The holdup of the reactor is 15,000 gallons, or three hours. A second reactor of equal size is placed in series with the first tank, providing an additional three hours of holdup time.

The reaction between hydrochloric acid and ferrochromium is exothermic. The heat is removed by circulating fluid external to the tank through a heat exchanger, and then back into the tank. The reactions will generate hydrogen, as shown in Equation 4.1 and, as a result of the temperature rise, some water vapor will also be produced. The gases from the reactor are exhausted through a condenser which will allow for water and hydrochloric acid to condense and return to the tank.

In the second tank, air is added in order to help complete the solubilization of the ferrochrome and also to oxidize the iron to the ferric state for subsequent separation. It is assumed that the air is 50 percent reacted in this tank; the nitrogen and unreacted oxygen exit through the water vapor and hydrochloric acid condenser. In the second tank, the dissolution of ferrochrome is virtually complete. Small amounts of unreacted material plus the carbon and other insoluble components of the ferrochrome form a residual solid phase. The slurry is pumped to a thickener to allow for separation of solids. These are estimated to comprise about 10 percent of the input material and consist mainly of iron, chromium, silica, and carbon. The 25-foot diameter thickener allows settling of a large fraction of the undissolved material. The overflow liquor is pumped through a pressure filter to remove additional solids and subsequently to a polish filter prior to subsequent processing. The solution at this point contains 2 molar or 104 grams per liter chromium, and about one molar or 60 grams per liter iron in the ferric state. The hydrochloric acid concentration is about 73 grams per liter.

Figure 4—1

CONCEPTUAL PROCESS FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM FERROCHROME

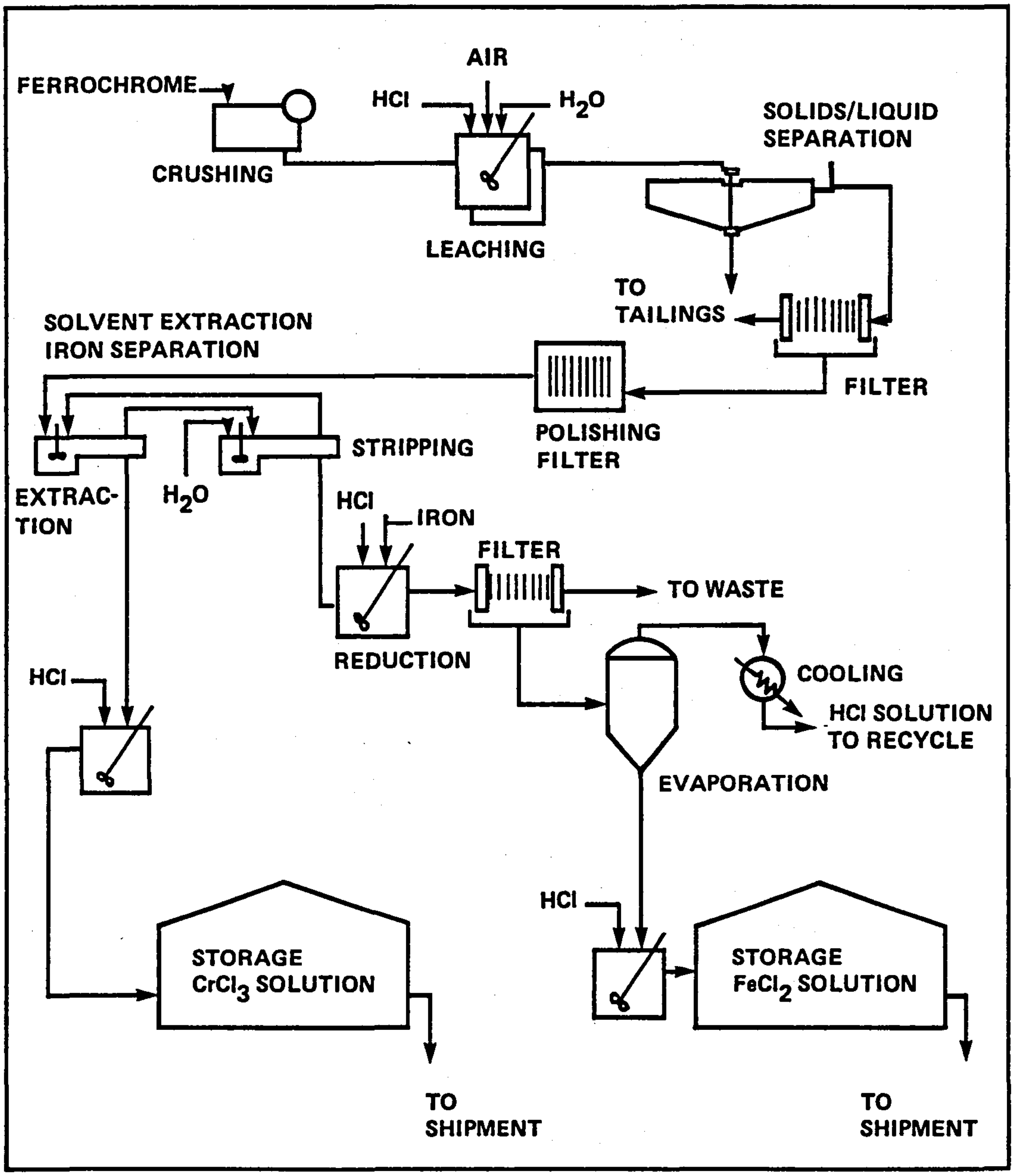
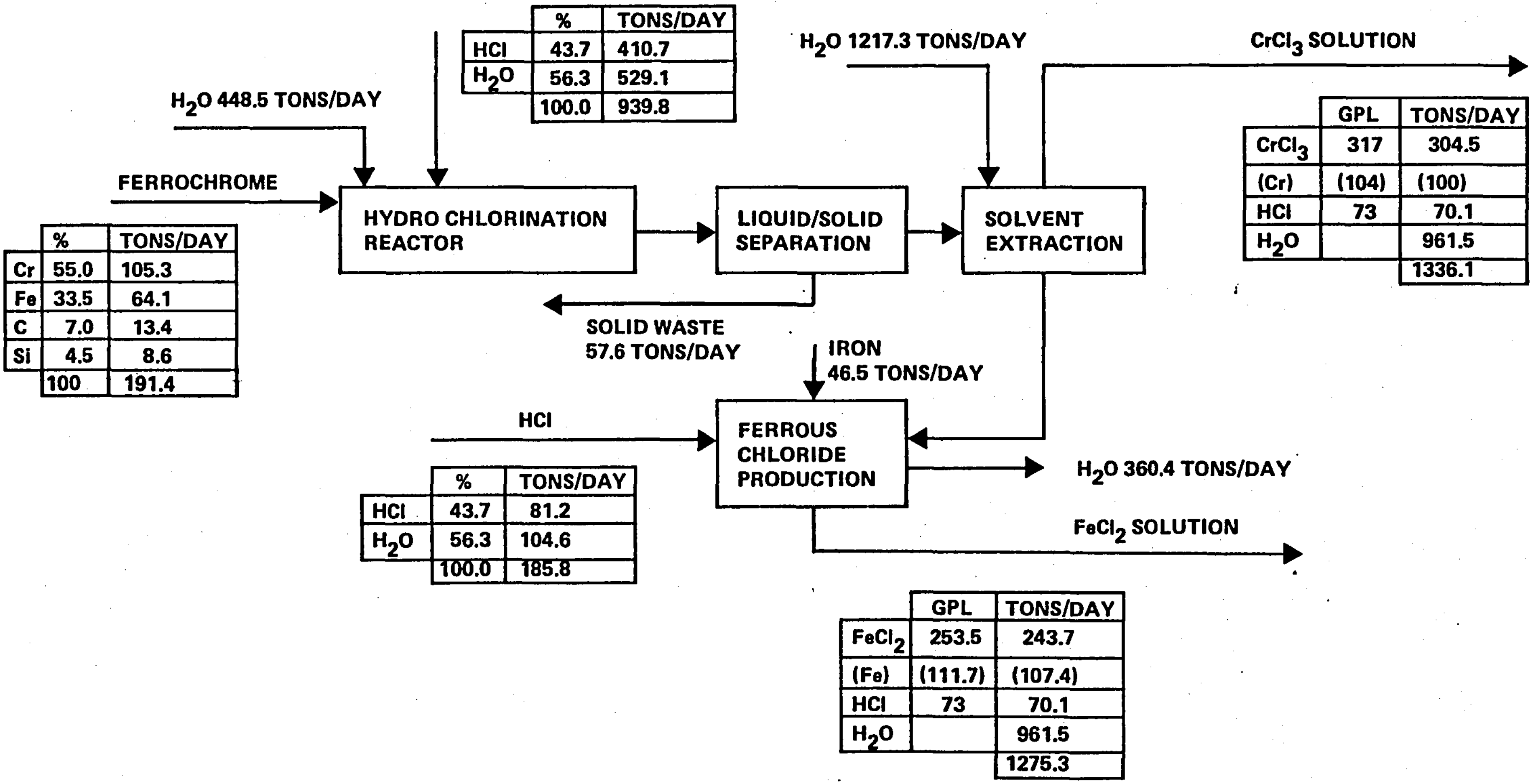


Figure 4—2

PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM HIGH-CARBON FERROCHROME



Ferrochrome feed:

	%	TONS/DAY
Cr	55.0	105.3
Fe	33.5	64.1
C	7.0	13.4
Si	4.5	8.6
	100	191.4

Feed to hydro chlorination reactor:

	%	TONS/DAY
HCl	43.7	410.7
H_2O	56.3	529.1
	100.0	939.8

$CrCl_3$ solution:

	GPL	TONS/DAY
$CrCl_3$	317	304.5
(Cr)	(104)	(100)
HCl	73	70.1
H_2O		961.5
		1336.1

HCl feed to ferrous chloride production:

	%	TONS/DAY
HCl	43.7	81.2
H_2O	56.3	104.6
	100.0	185.8

$FeCl_2$ solution:

	GPL	TONS/DAY
$FeCl_2$	253.5	243.7
(Fe)	(111.7)	(107.4)
HCl	73	70.1
H_2O		961.5
		1275.3

SOURCE: Charles River Associates, October 1981.

An 8,000 gallon surge tank is placed after the polish filter to allow for variation in the flow in the plant. The chromic-ferric solution is then pumped into a solvent extraction circuit in which the ferric chloride is selectively extracted into tributyl phosphate, leaving the chromic chloride in the aqueous phase. The chromium solution from solvent extraction is pumped to a 3,000 gallon tank where the hydrochloric acid concentration is adjusted to 2 molar, and then pumped to a one million gallon storage tank.

The ferric chloride which is extracted into tributyl phosphate is stripped with water to provide an acidic ferrous chloride solution. From the stripping section of the solvent extraction circuit, the ferric chloride solution is pumped to a 10,000 gallon tank where hydrochloric acid and metallic iron are added in order to bring the acid and iron concentrations to 2 normal. After the solution is filtered in a filter press to remove any undissolved iron or other extraneous material, it flows to an evaporator in order to concentrate the iron and hydrochloric acid to their final values. Following evaporation, the solution is pumped to a one million gallon storage tank, from which it is shipped.

By this design, both the chromium and iron chloride solutions are prepared to specifications as two molar in the metal chloride prior to direct shipment to the end user.

4.1.2 ECONOMICS

Capital and operating costs were developed using the flow sheet just described for a plant designed to produce 50 million pounds per year of contained chromium as chromium chloride. A major equipment list (Table 4-1) was prepared for the plant, and equipment costs developed.

The total capital cost was estimated using the installed equipment cost as a base and factors were applied for offsites, engineering, field expense, fees and contingencies. The installed equipment cost is about $10 million. The fixed capital cost is estimated at $27 million (Table 4-2), or about $.50 per annual pound of contained chromium. The working capital, which is made up principally of ferrochrome inventory, finished goods inventory, and accounts receivable (see Table 4-3), is about $12 million. The total investment for this process is almost $40 million, or $.80 per annual pound of chromium production.

The annual operating costs and unit operating costs are presented in Table 4-4. Raw materials in the form of ferrochromium and hydrochloric acid represent $.74 a pound of contained chromium product. Utilities, labor and maintenance and other direct costs are relatively small. The total production cost is estimated at $.85 per pound of contained chromium, with a total cost of $.87 including property taxes, sales, and G&A. The total annual cost for a plant producing 50 million pounds of contained chromium would be $43 million.

Table 4-1

MAJOR EQUIPMENT FOR THE PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM FERROCHROME

Item	Size	Quantity
Water Storage Tank	500,000 Gal.	1
HCl Storage Tank	1 Million Gal.	2
Ferrochrome Storage Bin	160 ft^3	1
Screen	3 ft. by 6 ft.	2
Jaw Crusher	25 ton/hr.	1
Weigh Belt Feeder	8 ton/hr.	1
Leach Tanks	15,000 Gal.	4
Leach Liquor Cooler		4
HCl Condensers		2
Thickener	25 ft. dia.	1
Filter Press	80 $ft.^2$	2
Polishing Filter	160 GPM	1
Holdup Tank	8,000 Gal.	1
Hoist		1
Forklift		2
Front End Loader		1
Organic Mixer	200 Gal.	1
Organic Surge Tank	3,000 Gal.	1
Organic Makeup Tank	3,000 Gal.	1
Reduction Tank	10,000 Gal.	1
Filter Press		1
Evaporator		1
HCl Addition Tank	3,000 Gal.	2
$FeCl_2$ Storage Tank	1 Million Gal.	2
$CrCl_3$ Storage Tank	1 Million Gal.	2
Solvent Extraction Equip.		
Lined Tailings Pond	6 Million $ft.^3$	1
Pumps and Agitators		

SOURCE: Charles River Associates, October 1981.

Table 4-2

CAPITAL COST FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM FERROCHROME
(25,000 TPY contained chromium)

	COST (THOUSANDS OF 1981 DOLLARS
INSTALLED EQUIPMENT COST	10140.
OFFSITES	6300.
DIRECT PLANT COST (DPC)	16440.
ENGINEERING AND FIELD EXPENSE (25.00 % DPC)	4110.
SUBTOTAL	20550.
CONTRACTOR'S FEE (7.00 % SUBTOTAL)	1439.
CONTINGENCIES (25.00 % SUBTOTAL)	5138.
FIXED CAPITAL COST (FCC)	27126.
WORKING CAPITAL COST	12474.
LAND	300.
TOTAL CAPITAL COST	39900.

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - OCT. 1981

Table 4-3

WORKING CAPITAL COST FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM FERROCHROME
(25,000 TPY contained chromium)

	COST (THOUSANDS OF 1981 DOLLARS)
PROCESS MATERIALS INVENTORY (CHEMICALS), 15 DAYS	627.
FERROCHROME INVENTORY, 60 DAYS	6316.
WORK IN PROCESS INVENTORY, 1 DAYS	174.
FINISHED GOODS INVENTORY, 15 DAYS	2608.
MATERIALS AND SUPPLIES, 3.00 % OF FIXED CAPITAL COSTS	814.
CASH (PAYROLL AND MISCELLANEOUS), 30 DAYS	191.
ACCOUNTS RECEIVABLE, 30 DAYS	6960.
ACCOUNTS PAYABLE, 30 DAYS	-5215.
TOTAL WORKING CAPITAL	12474.

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - OCT. 1981

Table 4-4

OPERATING COST FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM FERROCHROME
(25,000 TPY contained chromium)

				ANNUAL COST	
	NAME OF UNIT	QUANTITY PER YEAR	UNIT COST	THOUSANDS OF 1981 DOLLARS	DOLLARS PER UNIT OF PRIMARY PRODUCT
RAW MATERIAL	TONS	47847.	550.00	26316.	0.53
PROCESS MATERIALS					
HCl (CONTAINED)	TONS	123000.	75.00	9225.	0.18
IRON	TONS	11625.	100.00	1163.	0.02
ORGANIC				60.	0.00
TOTAL PROCESS MATERIALS				10448.	0.21
UTILITIES					
POWER	KWH	6000000.	0.04	240.	0.00
WATER	1000 GAL	150000.	0.50	75.	0.00
STEAM	1000 LB	216000.	3.00	648.	0.01
TOTAL UTILITIES				963.	0.02
LABOR DEPENDENT					
DIRECT LABOR (DL)	MAN-HOURS	68640.	14.50	995.	0.02
SUPERVISION	(20.00 % DL)			199.	0.00
TECH, ADMIN, AND GEN EXPENSES	(40.00 % DL)			398.	0.01
TOTAL LABOR DEPENDENT				1592.	0.03
MAINTENANCE AND REPAIRS					
LABOR	(2.00 % FCC)			543.	0.01
SUPPLIES	(3.00 % FCC)			814.	0.02
TOTAL MAINTENANCE AND REPAIRS				1356.	0.03
OTHER CASH COSTS				1600.	0.03
TOTAL PRODUCTION COST				42275.	0.85
PROPERTY TAX	(2.00 % FCC & LAND)			549.	0.01
INSURANCE	(0.50 % FCC)			136.	0.00
SELLING, GENERAL, AND ADMIN.				500.	0.01
TOTAL CASH COST				43459.	0.87
					$1.91 kg/Cr

NOTES :: PRIMARY PRODUCT IS CHROMIC CHLORIDE
EXPRESSED IN UNITS OF POUNDS

'FCC' REPRESENTS FIXED CAPITAL COSTS

COMPONENTS MAY NOT ADD TO TOTALS DUE TO ROUNDING

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - OCT. 1981

The economics of a commercial plant are summarized in Table 4-5. If the chromium chloride selling price is $1 per pound of contained chromium, the revenues are $51 million per year. The value of the ferrous chloride is calculated using present prices, at about $7 million per year. Total revenues are $58 million. The pre-tax profit is calculated at $14.5 million, the net income at $6 million, or 16 percent of total investment, and the cash flow is $9.1 million. The discounted cash flow profitability is shown in Figure 4-3 as a function of selling price and the cost of ferrochrome. The graph shows that chromic chloride can be produced at prices of $0.95 to $1.10 per pound contained chromium, given ferrochrome prices between $0.50-0.75 per pound of contained chromium.

4.1.3 TECHNICAL AND ECONOMIC DISCUSSIONS

The hydrometallurgical treatment of ferrochrome has many attractive features. First, ferrochrome consists mainly of chromium and iron, both of which are required for the Redox system. Ferrochrome should be readily soluble in hydrochloric acid, with a minimum of residue or undesirable products, and those should be readily separated or filtered from the ferrous chloride and chromic chloride solutions. The key to technical and economic success is an effective process for the separation and purification of the chromium and the iron chlorides. For the purposes of this report, solvent extraction of ferric chloride from chromic chloride has been assumed. It is highly likely that by control of the pH and of the metal concentrations, a complete separation can be made. However, it is recognized that development work would be required to complete the design parameters for such a step. The final concentration of the chromium and the iron chloride solutions and removal of any undesirable elements, such as vanadium, should be reasonably straightforward.

The flowsheet shows very standard metallurgical equipment and technology. For this reason, it is highly likely that this process could be adopted for commercial production of chromium and iron chloride solutions, with a minimum of development and a minimum of technical risk. In addition, it should be possible by acquisition of only small amounts of additional technical data (particularly in solvent extraction) to develop accurate capital and operating costs. The largest uncertainty in this project would be the availability and cost of ferrochrome, since this represents one of the higher cost raw materials for making these chloride products.

Table 4-5

SUMMARY OF PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM FERROCHROME
(25,000 Tons/Year Contained Chromium)

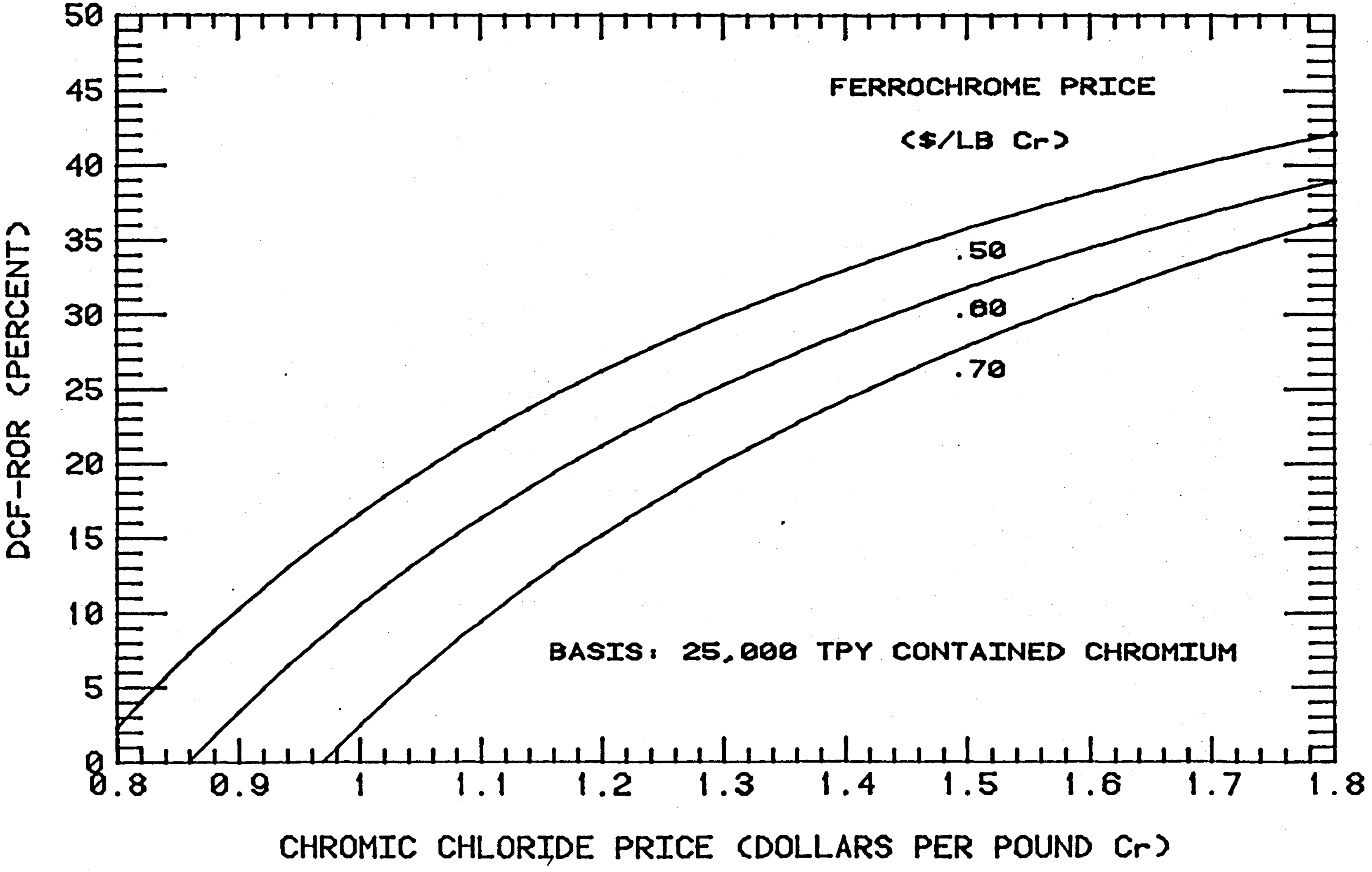
Revenues	Annual Amount ($ 000/year)	
Chromium Chloride Solution	51,320*	
Iron Chloride Solution	6,690	
TOTAL		58,010
Production Costs		43,460
Pretax Profit		14,550
Depreciation		2,710
Taxable Income		11,840
Net Income		6,390
Cash Flow		9,100

*Based on $1.00 per pound contained chromium.

SOURCE: Charles River Associates, October 1981.

Figure 4–3

PROFITABILITY OF CHROMIUM AND IRON CHLORIDE PRODUCTION FROM FERROCHROME



SOURCE: CHARLES RIVER ASSOCIATES, OCTOBER 1981.

4.2 HYDROMETALLURGICAL PROCESS WITH CHROMITE ORE

Chromite ore is the lowest cost source of chromium. In addition, it also contains iron which is desirable for the iron chlorides. However, chromite is difficult to digest or dissolve, and a number of undesirable components are present.

The principal desired reactions are:

$$FeO \cdot Cr_2O_3 + 8HCl = FeCl_2 + 2CrCl_3 + 4H_2O$$

However, chromite ores consist of several components and a more general reaction is described as:

$$(Fe,Mg)O \cdot (Cr,Al,Fe)_2O_3 + 8HCl = nFeCl_3 + mFeCl_2 + pCrCl_3 + qMgCl_2 + rAlCl_3 + 4H_2O$$

The ore tends to be unreactive, but can be dissolved in strong HCl at temperatures of 160°C and higher. At these conditions, a pressure of two or more atmospheres is required. The reaction is exothermic, i.e., according to calculations based on individual component oxides, and heat must be removed from the system to maintain the desired operating conditions. The estimated energies of the reaction are shown in Table 4-6.

In this process, magnesium and aluminum chlorides are put into solution along with the iron and chromium chlorides. Thus, an important step involves 1) separation of $MgCl_2$ and $AlCl_3$ from the chromium and iron chlorides and 2) separation of the iron and chromium from each other. Solvent extraction appears to be a suitable method for achieving these separations commercially. Our review of the solvent extraction technology suggests that this approach may be satisfactory; however, a technical development program would be required to specify process conditions and demonstrate the concept.

After separation, the concentration and storage of the iron and chromium chloride solutions is similar to the ferrochromium process. However, there are solutions of magnesium and aluminum chlorides which must be treated. It is proposed that these solutions are concentrated to near saturation and then hydrolyzed at high temperature by spray roasting to produce discardable metal oxides and regenerate hydrochloric acid.

4.2.1 PROCESS DESCRIPTION

The process is shown in Figure 4-4 and the overall material balance is presented in Figure 4-5. Twelve normal hydrochloric acid is received in railroad cars and stored in two storage tanks, each containing 500,000 gallons of acid. Chromite ore is received by railroad, screened, crushed to

Table 4-6

REACTIONS AND ENERGY BALANCE FOR DISSOLUTION OF CHROMITE ORE

Component	Material Flow (000 Pounds per Hour)	Reaction	Energy Balance (Million Btu/Hr)
Cr_2O_3	13.5	$Cr_2O_3 + 6HCl\ T\ 2CrCl_3 + 3H_2O$	- 7.5
FeO	6.0	$FeO + 2HCl\ T\ FeCl_2 + H_2O$	- 6.3
Fe_2O_3	1.7	$Fe_2O_3 + 6HCl\ T\ 2FeCl_3 + 3H_2O$	- 0.4
MgO	3.1	$MgO + 2HCl\ T\ MgCl_2 + H_2O$	- 4.4
Al_2O_3	4.3	$Al_2O_3 + 6HCl\ T\ 2AlCl_3 + 3H_2O$	- 2.9
CaO	0.1	$CaO + 2HCl\ T\ CaCl_2 + H_2O$	- 0.1
Subtotal	28.7	Subtotal	-21.6
HCl (recycle)	48.4		- 3.1
H_2O (recycle)	74.8		+ 0.5
Other Materials	0.6		+ 8.1
			-16.1

Figure 4—4

CONCEPTUAL HYDROMETALLURGICAL PROCESS FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES

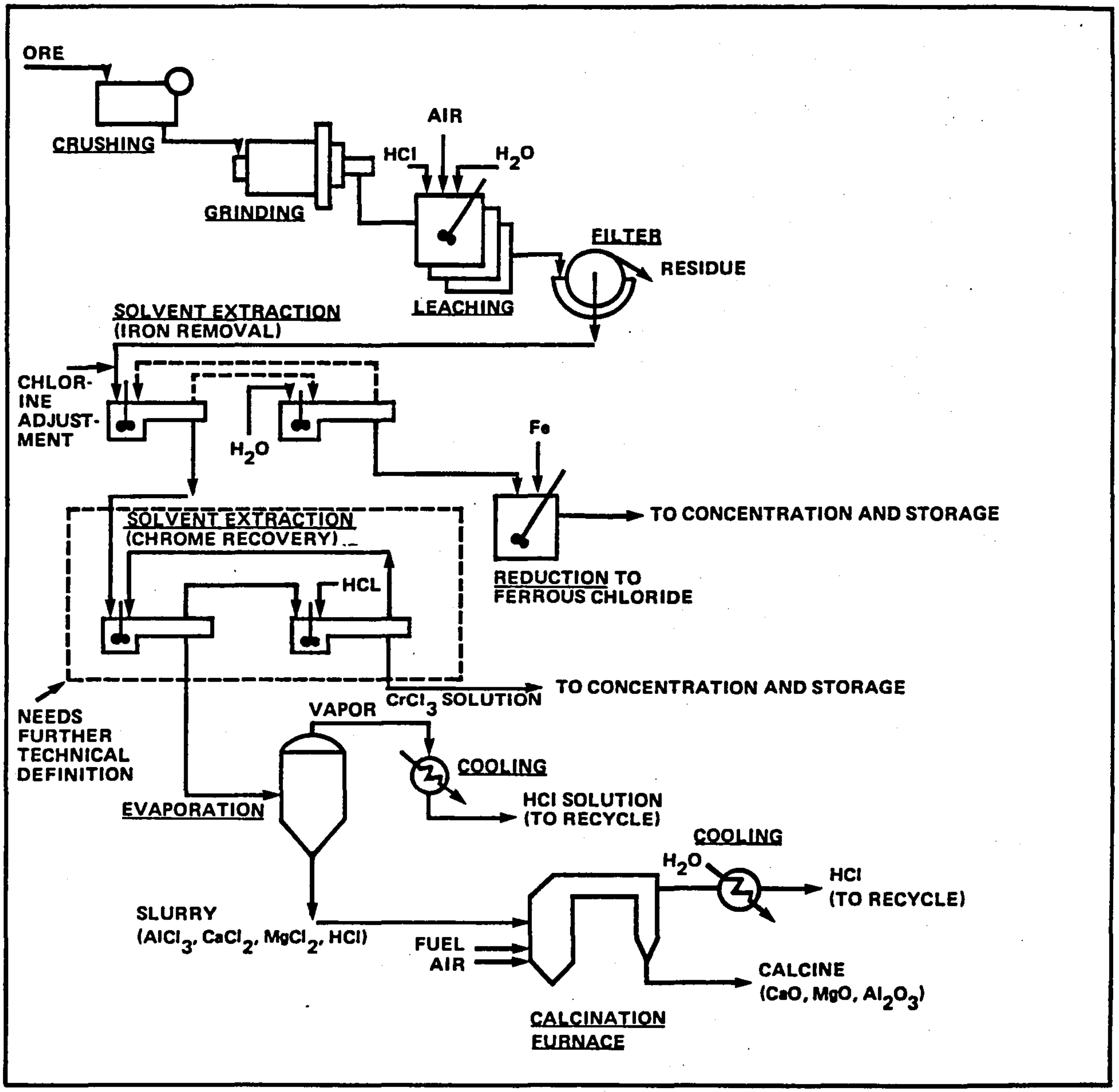
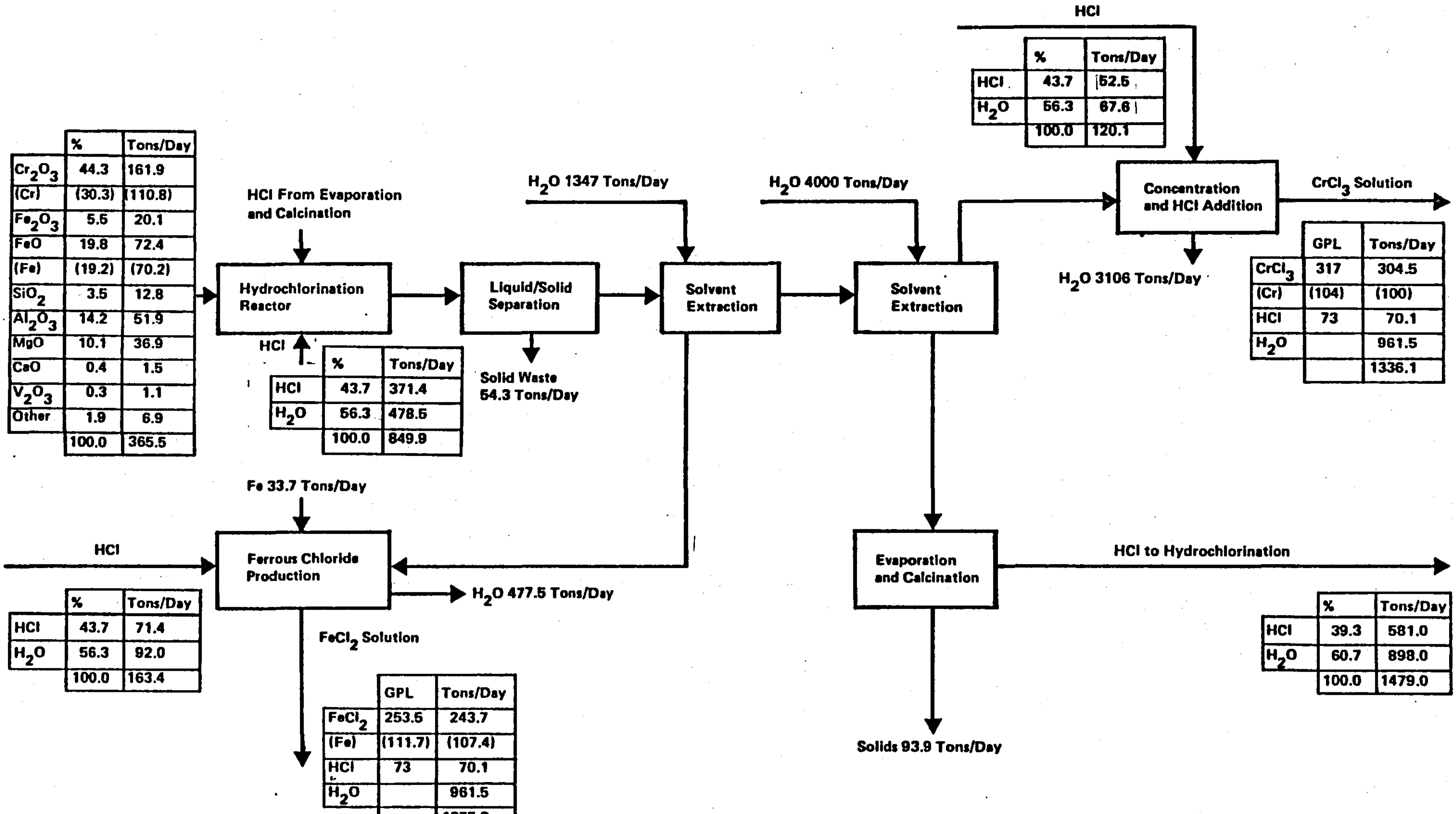


Figure 4–5

HYDROMETALLURGICAL PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM CHROMITE ORE



Ore feed to Hydrochlorination Reactor:

	%	Tons/Day
Cr_2O_3	44.3	161.9
(Cr)	(30.3)	(110.8)
Fe_2O_3	5.5	20.1
FeO	19.8	72.4
(Fe)	(19.2)	(70.2)
SiO_2	3.5	12.8
Al_2O_3	14.2	51.9
MgO	10.1	36.9
CaO	0.4	1.5
V_2O_3	0.3	1.1
Other	1.9	6.9
	100.0	365.5

HCl to Hydrochlorination Reactor:

	%	Tons/Day
HCl	43.7	371.4
H_2O	56.3	478.5
	100.0	849.9

HCl to Concentration and HCl Addition:

	%	Tons/Day
HCl	43.7	52.5
H_2O	56.3	67.6
	100.0	120.1

$CrCl_3$ Solution:

	GPL	Tons/Day
$CrCl_3$	317	304.5
(Cr)	(104)	(100)
HCl	73	70.1
H_2O		961.5
		1336.1

HCl to Ferrous Chloride Production:

	%	Tons/Day
HCl	43.7	71.4
H_2O	56.3	92.0
	100.0	163.4

$FeCl_2$ Solution:

	GPL	Tons/Day
$FeCl_2$	253.5	243.7
(Fe)	(111.7)	(107.4)
HCl	73	70.1
H_2O		961.5
		1275.3

HCl to Hydrochlorination:

	%	Tons/Day
HCl	39.3	581.0
H_2O	60.7	898.0
	100.0	1479.0

SOURCE: Charles River Associates, December 1981.

size in a cone crusher and further screened. The minus 200-mesh material is conveyed to a storage bin, the oversize is put through a rod mill to reduce its size, the minus 200-mesh material is transferred to an intermediate storage bin in preparation for charge to the leach tanks.

There are two lines of leach tanks, each consisting of two 15,000 gallon reactors in series, providing a total of six hours residence time of the solids in solution. These vessels are agitated and operate at temperatures of 160°C or above and two or more atmospheres of pressure. In the first of the two reactors, hydrochloric acid reacts with the ore, producing hydrogen gas which is vented along with steam and a solution of metal chlorides. The fluid is circulated external to the tank, and heat is exchanged to remove energy. The solution and solids overflow continuously to the second reactor, where the dissolution is completed. Air is sparged into the system in order to oxidize the iron to the ferric state. The temperature in this tank is approximately 140°C. The off-gases from the tank contain nitrogen, unreacted oxygen, water vapor and small amounts of hydrogen. These gases pass through a condenser that returns the water and are then vented. The solution exiting from the second reactor contains a small amount of undissolved solids. It is heat-exchanged with incoming water and hydrochloric acid to remove the heat and reduce the temperature to about 100°C. The solution passes into a thickener to remove large particulates and the solution overflows to a filter press to remove the final solids content before passing into an intermediate storage tank prior to solvent extraction. The thickener underflow containing most of the solids passes through a filter press to remove the solids and the liquid phase and wash liquor are recycled back to the solution tanks.

The chloride solution contains two moles per liter of chromium salt and the corresponding concentrations of iron, magnesium and aluminum chlorides. In the solvent extraction step, the first stage is designed to extract the iron selectively with tributyl phosphate. A second extraction stage with di-2-ethyl hexaphosphoric acid (DEHPA) extracts the chromium and rejects the magnesium and aluminum chlorides. Alternatively, iron and chromium chlorides are co-extracted with Aliquot 336, an amine reagent. Ferric chloride is stripped from the TBP with water and the chromium chloride is stripped from the DEHPA. By either procedure, the chromium chloride, at two moles per liter, passes into an intermediate tank where hydrochloric acid concentration is adjusted to the required two molar and the solution is then pumped to the storage tank prior to shipment.

The ferric chloride solution is concentrated, iron is added to make up the required amount of iron chloride and reduce the ferric to ferrous, and hydrochloric acid is added to make up the required composition. This solution is pumped to the storage tank for shipment.

The magnesium and aluminum chloride solution is concentrated by evaporation and pumped into a spray roasting furnace in order to hydrolyze the chlorides, to magnesium and aluminum oxide. The hydrochloric acid vapors are condensed and recirculated back to the dissolution tank. The magnesium and aluminum oxides may then be sold for their oxide values or discarded safely.

4.2.2 ECONOMICS

The capital costs were developed by using factored costs based on installed major equipment (Table 4-7). The investment costs are summarized in Table 4-8. The total installed equipment is estimated at $22.2 million. With off-sites, engineering and field expense, contractors fee and a contingency of 25 percent to allow for process and cost uncertainties, the total fixed cost is estimated at $59 million, or $1.19 per annual pound of contained chromium in the product. Working capital and land add another $10 million, so the total investment is calculated at $69 million, or $1.38 per annual pound of installed capacity. Working capital, based primarily on an inventory of chrome ore, finished goods, materials and supplies and accounts receivable, is estimated at $9.6 million (Table 4-9).

The operating costs are estimated in Table 4-10. Chrome ore is estimated at $.17 per pound of contained chromium in the products. Other process materials, such as hydrochloric acid and iron, add $.21 per pound and utilities $.23 per pound. Most of the utility costs lie in energy as steam and fuel required for evaporation of solution and calcination of the magnesium and aluminum salts. Labor is $.05 per pound, maintenance and repairs another $.06 per pound, and adding property tax, selling, G&A and etc. the total direct operating cost is calculated at $.82 per pound.

Profitability is sensitive to the selling price of the chromium chloride product and to a smaller degree the cost of chromite ore. The economics of a commercial plant are summarized in Table 4-11. The sensitivities are shown in Figure 4-6. The figure shows that a 15 percent discounted cash flow rate of return would be realized by the producer if the chromium chloride were sold at $1.06 per pound of contained chromium.

4.2.3 TECHNICAL AND ECONOMIC DISCUSSION

One of the attractive features of this process is the use of low-cost chromite ore as a source of the chromium and iron for the chloride solutions. However, the ore also contains extraneous elements which will report to the leach solution. The process, therefore, must include separation and recovery equipment for these undesirable elements.

The dissolution of chromite, which is known to be difficult, requires some relatively high-temperature, high-pressure conditions. Work conducted on lateritic-type materials indicated that this dissolution can be accomplished. Additional technical work would be required to assure virtually complete dissolution of the chromite under the assumed conditions. Chlorides are notably difficult to work with, and corrosion is an important aspect of chloride-based plants. The equipment selected for this process through the dissolution is a standard metallurgical type, and materials storage and handling can be sized accurately based on the plant throughput. The dissolution reactors can be sized and costed, but additional technical

Table 4-7

MAJOR EQUIPMENT LIST FOR HYDROMETALLURGICAL PLANT USING CHROMITE ORE

Item	Size	Quantity
H_2O Storage Tank	500,000 Gal.	2
HCl Storage Tank	1 Million Gal.	2
Ore Storage Bin	22,000 ton	1
Screen	50 ton/hr.	4
Cone Crusher	60 ton/hr.	1
Rod Mill	40 ton/hr.	1
Ball Mill	20 ton/hr.	1
Ore Sized Storage	60 ton	1
Weigh Belt Feeder	15 ton/hr.	1
Leach Tanks	15,000 Gal. (3 hr.)	4
Leach Liquor Cooler		4
HCl Condensers		2
Thickener	25 ft. dia.	1
Filter Press		2
Polishing Filter		1
Holdup Tank	8,000 Gal.	1
Hoist		1
Forklift		2
Front End Loader		1
Solvent Extraction-Iron		1
Solvent Extraction-Chrome		1
Reduction Tank and Mixer	10,000 Gal.	1
Filter Press		1
HCl Addition Tank - Fe	3,000 Gal.	2
$FeCl_2$ Storage Tank	1 Million Gal.	2
$CrCl_3$ Storage Tank	1 Million Gal.	2
Lined Tailings Pond	15 Million ft^3	1
HCl Addition Tank-Cr		1
Compressors		
Pumps		
Leach Agitators		
Evaporators		
Waste Water Cooler	5,000 ft^2	2
Recycle Cooler	9,000 ft^2	4
Calcination Furnace	133 Million Btu	1
Cyclone	80,000 ACFM	1
Spray Cooler	80,000 ACFM	1
Stack	100 feet	1

SOURCE: Charles River Associates, December 1981.

able 4-8

APITAL COST FOR THE HYDROMETALLURGICAL PRODUCTION F CHROMIUM AND IRON CHLORIDES FROM CHROMITE ORE
25,000 TPY contained chromium)

	COST (THOUSANDS OF 1981 DOLLARS)
NSTALLED EQUIPMENT COST	22230.
FFSITES	13820.
IRECT PLANT COST (DPC)	36050.
NGINEERING AND FIELD EXPENSE (25.00 % DPC)	9013.
UBTOTAL	45063.
ONTRACTOR'S FEE (7.00 % SUBTOTAL)	3154.
ONTINGENCIES (25.00 % SUBTOTAL)	11266.
IXED CAPITAL COST (FCC)	59483.
ORKING CAPITAL COST	9559.
AND	300.
OTAL CAPITAL COST	69341.

OURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - DEC. 1981

Table 4-9
WORKING CAPITAL COST FOR THE HYDROMETALLURGICAL PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM CHROMITE ORE
(25,000 TPY contained chromium)

	COST (THOUSANDS OF 1981 DOLLARS
PROCESS MATERIALS INVENTORY (CHEMICALS), 15 DAYS	627.
CHROME ORE INVENTORY, 60 DAYS	1994.
WORK IN PROCESS INVENTORY, 2 DAYS	327.
FINISHED GOODS INVENTORY, 15 DAYS	2453.
MATERIALS AND SUPPLIES, 3.00 % OF FIXED CAPITAL COSTS	1784.
CASH (PAYROLL AND MISCELLANEOUS), 30 DAYS	318.
ACCOUNTS RECEIVABLE, 30 DAYS	6960.
ACCOUNTS PAYABLE, 30 DAYS	-4905.
TOTAL WORKING CAPITAL	9559.

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - DEC. 1981

Table 4-10

OPERATING COST FOR THE HYDROMETALLURGICAL PRODUCTION OF CHROMIUM AND IRON CHLORIDES FROM CHROMITE ORE (25,000 TPY contained chromium)

	NAME OF UNIT	QUANTITY PER YEAR	UNIT COST	ANNUAL COST THOUSANDS OF 1981 DOLLARS	ANNUAL COST DOLLARS PER UNIT OF PRIMARY PRODUCT
RAW MATERIAL	TONS	91392.	90.93	8310.	0.17
PROCESS MATERIALS					
HCl (CONTAINED)	TONS	123825.	75.00	9287.	0.19
IRON	TONS	10000.	100.00	1000.	0.02
ORGANIC				150.	0.00
OTHER				10.	0.00
TOTAL PROCESS MATERIALS				10447.	0.21
UTILITIES					
POWER	KWH	12000000.	0.04	480.	0.01
FUEL	MIL. BTU	800000.	4.00	3200.	0.06
STEAM	1000 LB	2535700.	3.00	7607.	0.15
COOLING WATER	1000 GAL	3486000.	0.02	70.	0.00
MAKEUP WATER	1000 GAL	174000.	0.05	9.	0.00
PROCESS WATER	1000 GAL	264000.	0.50	132.	0.00
TOTAL UTILITIES				11498.	0.23
LABOR DEPENDENT					
DIRECT LABOR (DL)	MAN-HOURS	114400.	14.50	1659.	0.03
SUPERVISION	(20.00 % DL)			332.	0.01
TECH, ADMIN, AND GEN EXPENSES	(40.00 % DL)			664.	0.01
TOTAL LABOR DEPENDENT				2654.	0.05
MAINTENANCE AND REPAIRS					
LABOR	(2.00 % FCC)			1190.	0.02
SUPPLIES	(3.00 % FCC)			1784.	0.04
TOTAL MAINTENANCE AND REPAIRS				2974.	0.06
OTHER CASH COSTS				3000.	0.06
TOTAL PRODUCTION COST				38883.	0.78
PROPERTY TAX	(2.00 % FCC & LAND)			1196.	0.02
INSURANCE	(0.50 % FCC)			297.	0.01
SELLING, GENERAL, AND ADMIN.				500.	0.01
TOTAL CASH COST				40876.	0.82

NOTES :: PRIMARY PRODUCT IS CHROMIC CHLORIDE
EXPRESSED IN UNITS OF POUNDS

'FCC' REPRESENTS FIXED CAPITAL COSTS

COMPONENTS MAY NOT ADD TO TOTALS DUE TO ROUNDING

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - DEC. 1981

Table 4-11

SUMMARY OF HYDROMETALLURGICAL PRODUCTION
OF CHROMIUM AND IRON CHLORIDES FROM CHROMITE ORE
(25,000 tons/year Contained Chromium)

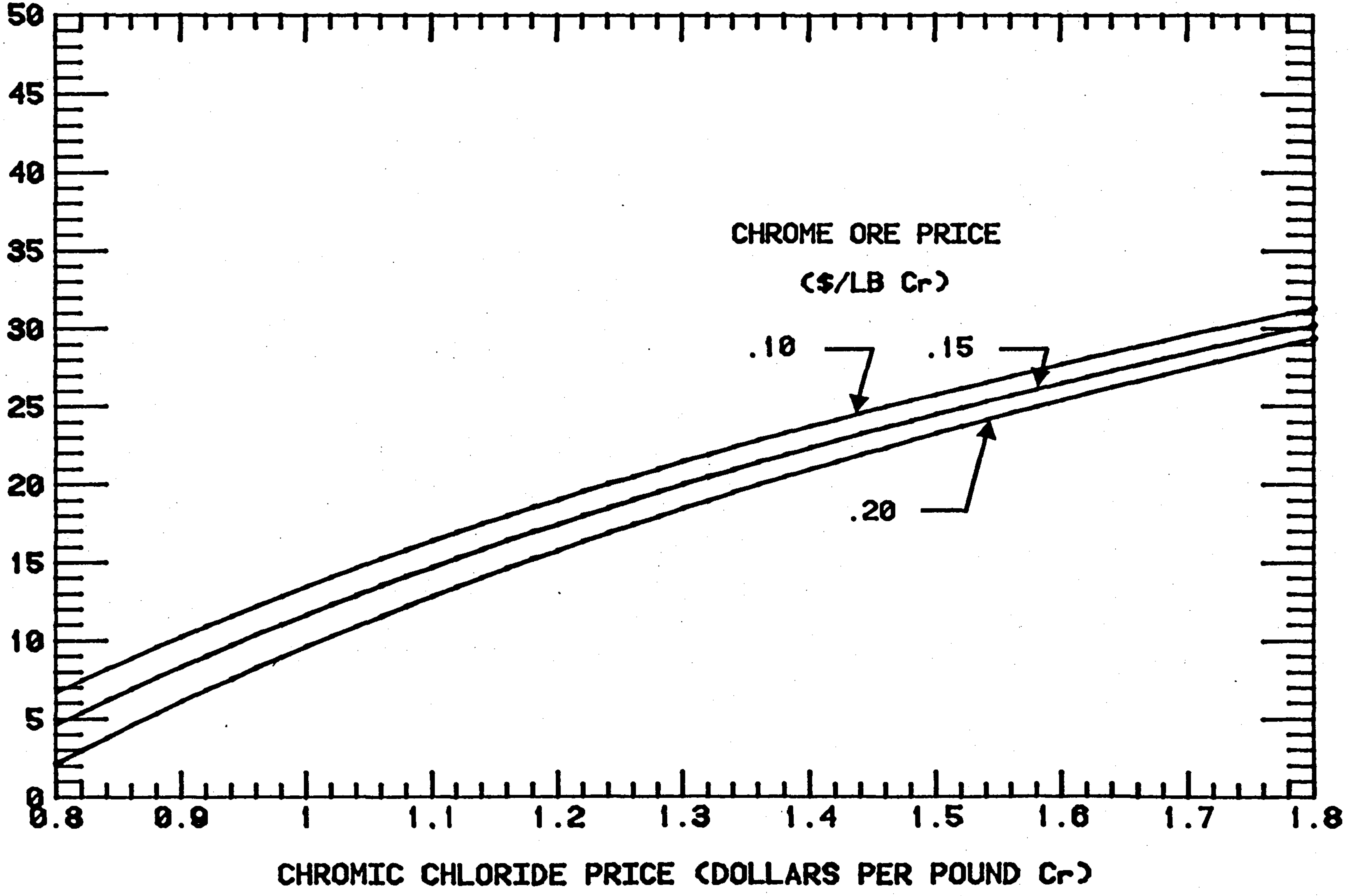
Revenues	Annual Amount ($ 000/year)	
Chromium Chloride Solution	51,320*	
Iron Chloride Solution	6,690	
TOTAL		58,010
Production Costs		40,880
Pretax Profit		17,130
Depreciation		5,950
Taxable Income		11,180
Net Income		6,040
Cash Flow		11,990

*Based on $1.00 per pound contained chromium.

SOURCE: Charles River Associates, December 1981.

Figure 4–6

HYDROMETALLURGICAL PRODUCTION OF Cr AND Fe CHLORIDES FROM CHROMITE ORE



SOURCE: CHARLES RIVER ASSOCIATES, JANUARY 1982.

information would be required to prepare a preliminary plant design. The separation of the iron and chromium chlorides from the magnesium and aluminum and any other undesirable components would require considerable technical development. While such a solvent extraction separation process is not assured, it is likely, based on available information, that this approach could be made successful after some development work. The separation and recovery of chromium and iron chlorides, one from another, can be confirmed reasonably well on the laboratory and small-pilot scale. The calcination of magnesium and aluminum chlorides is based on known technology and equipment at a scale comparable to those employed for this process.

That the capital cost for a hydrometallurgical plant using chromite ore is significantly higher than a plant using ferrochromium reflects the fact that the impurities in the ore must be separated and rendered innocuous prior to discard or sale. Consequently, there is a cost penalty associated with using this less expensive raw material. Energy is a significant operating cost and it is recognized that the process outlined here has not been optimized in this respect. It appears that some optimization of the energy balance could be achieved, but without critical impact on the overall production cost.

5

REDUCTION CHLORINATION OF CHROMITE ORE

The Bureau of Mines has worked on a process concept to pre-mix chromite ore with coke and to contact this material with chlorine at an elevated temperature. The resulting high-temperature reactions produce chromium, iron, magnesium, calcium and aluminum chlorides. The idea is to take the metal chloride vapors and to cool these gases in several steps in order to condense one metal chloride at a time. By cooling the reactor gases to 600°C, chromium chloride with some magnesium is first condensed. The calcium and magnesium chlorides are mainly liquid and remain largely in the reactor with the unreacted residues. By cooling at a lower temperature of 250°C, iron chloride is condensed and then by finally cooling to 50°C or below, aluminum chloride is condensed.

An important factor in this process concept is the feasibility for selective condensation and separation of each of the metal chlorides. Some of the metals form several metal chloride species with varying vapor pressures.

The chlorination reaction is endothermic. In the reactor concept oxygen or air is blown into the reactor to burn some of the coke, providing the heat for reaction. This means that the off-gases also contain nitrogen, unreacted oxygen, and chlorine as well as the metal chlorides. The process development has not proceeded beyond a laboratory scale, and the data obtained are not sufficient to develop a complete process flowsheet with any degree of specification. In order to assess its potential, we have developed a flowsheet using existing process equipment where known, and making assumptions of the design and cost of certain other equipment where new technology is required.

5.1 PROCESS DESCRIPTION

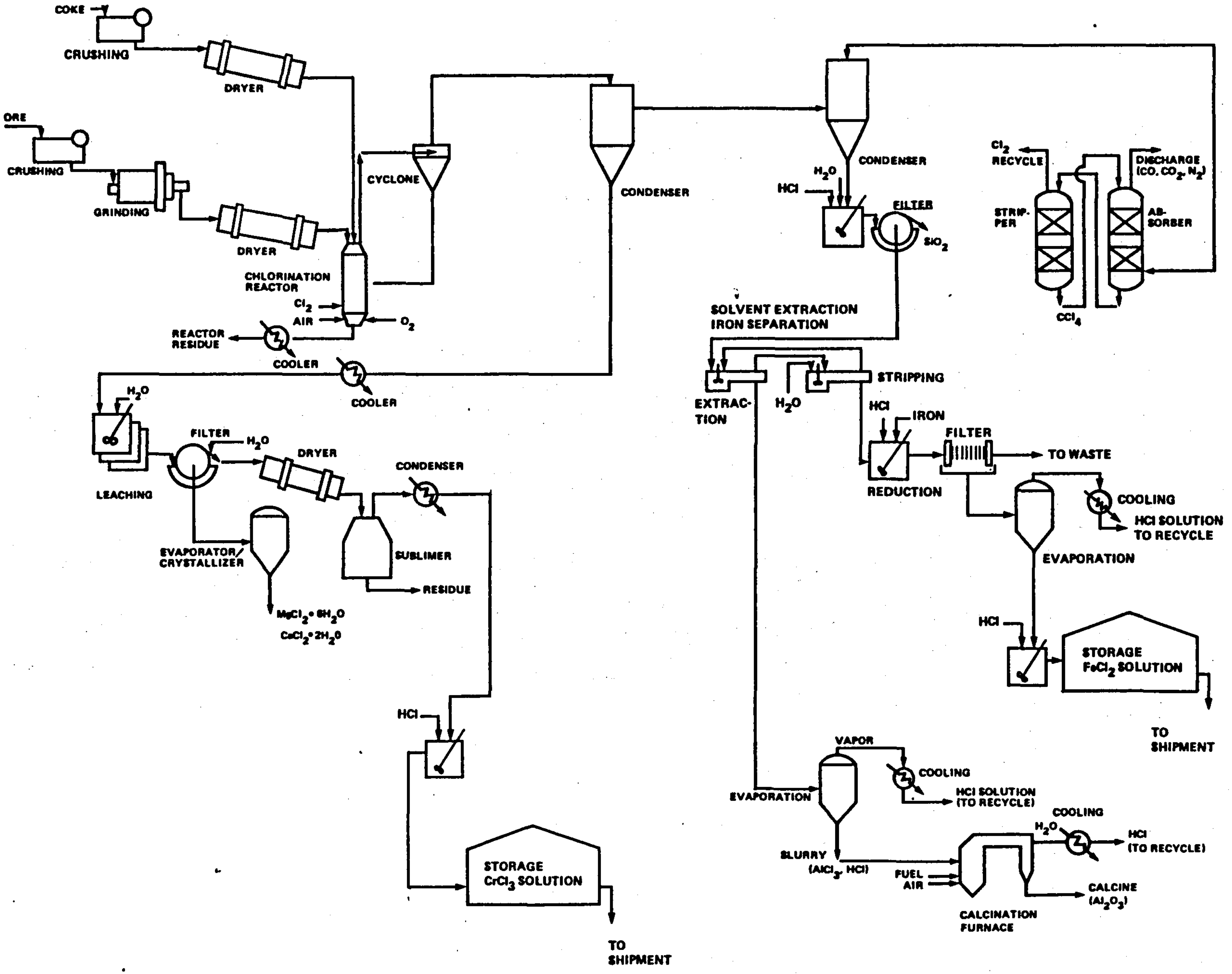
It is assumed that ore is received by rail and put on a storage pile (Figure 5-1). It is assumed that in preparation for chlorination, the ore must be crushed to a fine size, blended with coke, and then agglomerated into a form which can be charged to a vertical shaft-type furnace. Chlorine is received by tank car and transferred to storage tanks. Agglomerated feed is fed to the top of the chlorination reactor, and air/oxygen and chlorine are blown into the bottom of the reactor. The air/oxygen oxidizes some of the coke, generating heat, and the chlorine reacts with reduced metal oxides to form the metal chlorides. The vapors exit from the chlorination reactor at over 800°C. These gases will contain a certain amount of entrained dust. The gases are fed to a cyclone to remove most of the dust, which is recycled, and the gas is passed through a first condenser that removes chromium, magnesium and calcium chlorides at a temperature of 600°C. The condensed materials are cooled and then charged to a leaching system to solubilize magnesium and calcium chlorides. The undissolved chromium chloride is filtered and the magnesium and calcium chloride solution is fed to an evaporator to remove water and produce chloride salts for discard. There may even be a market for these materials, as road salt or as a chemical plant feed. The chromium chloride is dried and then charged in an electrically-heated subliming unit in order to vaporize the chromium chloride and remove any residual impurities. The chloride vapor must be condensed, fed to a mix tank where hydrochloric acid is added, and the chromium chloride-hydrochloric acid solution is made up.

The gases from the chromium/magnesium/calcium chloride condenser pass through a second condenser at 250°C, which removes iron and aluminum chloride. The cooled solids are charged to a mix tank where hydrochloric acid is added. Solids are filtered away, notably silica and dust materials, and the solution is sent to a solvent extraction circuit in order to separate the iron chloride from aluminum and any other materials. The ferric chloride solution is stripped away from the organic phase, iron is added to reduce the ferric to the ferrous state, and hydrochloric acid is added to make up the appropriate ratio of hydrochloric acid to iron chloride. The solution is filtered to remove any residual solids, evaporated to a two normal concentration of iron chloride, and pumped to a ferrous chloride storage tank prior to shipment. The aluminum chloride which is not extracted in the solid extraction phase is concentrated in an evaporator and then fired in a calcination furnace in order to make aluminum oxide for discard. From this furnace, the hydrogen chloride is condensed and recycled back into the process.

Finally, the off-gases from the low temperature condenser may contain chlorine and residual metal chlorides in addition to carbon oxides and nitrogen. These pass into a carbon tetrachloride absorber in order to remove residual chlorine. The chlorine is stripped from the carbon tetrachloride in concentrated form and returned back to the chlorination reactor.

Figure 5–1

CONCEPTUAL CHLORINATION PROCESS
FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES



5.2 ECONOMICS

For the very preliminary flowsheet, an equipment list has been made. Costs were developed for all of the major process units, and installed equipment costs estimated at $30 million as shown in Table 5-1. The total fixed capital cost, including engineering and field expense, contractor's fee and contingencies of 25 percent, is estimated at $82.5 million. The working capital, which is made up as shown in Table 5-2, is estimated at $13 million, most of which is in accounts receivable and inventory of finished goods and materials and supplies. The operating costs are summarized in Table 5-3. Chromite ore represents $.17 per pound of contained chromium in the finished products. Other process materials such as hydrogen chloride, coke, oxygen and, in particular, chlorine add another $.26 per pound of chromium. Utilities are $.12, and then labor, maintenance and repairs and other costs add an additional $.21 per pound. The direct operating costs, therefore, are estimated at $.81 per pound of contained chromium in the products.

The profitability of such a plant is shown in Table 5-4. While the direct operating costs are similar to that of the hydrometallurgical treatment of chromite ore, the higher investment requires a higher selling price in order to achieve an acceptable rate of return. The discounted cash flow rate of return for such a project is shown in Figure 5-2 as a function of the selling price of chromium chloride in the product and the purchase price of chrome ore. As shown in this figure, a 15 percent discounted cash flow rate of return would require a selling price of $1.20-1.25 per pound of contained chromium in the products.

5.3 TECHNICAL AND ECONOMIC DISCUSSION

This process employs two unproven steps: 1) the reduction chlorination of chromite ore at high temperature and 2) fractional separation of the volatile metal chlorides by condensation. The supporting data for equipment design is minimal at this point, and considerable development work will be required to show feasibility. Analysis of the vapor pressure data of the metal chlorides suggests that fractional separation of the desired and undesired metal chlorides may be extremely difficult (Figure 5-3). It is very likely that the chromium chloride will contain appreciable amounts of magnesium and calcium chloride and the iron chloride will contain appreciable amounts of particulates as well as aluminum and chromium chlorides. Therefore, it is not likely that such a process would be effective in making pure iron and chromium chloride products without subsequent purification steps. (On the other hand, the purity may be adequate for NASA's requirements.)

It may be necessary to redissolve these chlorides and conduct further purification. Under this situation, the process has no foreseeable advantages over either of the previous two discussed.

Table 5-1

CAPITAL COST FOR THE CHLORINATION OF CHROMITE ORE TO PRODUCE CHROMIUM AND IRON CHLORIDES
(25,000 TPY contained chromium)

	COST (THOUSANDS OF 1981 DOLLARS
INSTALLED EQUIPMENT COST	30000.
OFFSITES	20000.

DIRECT PLANT COST (DPC)	50000.
ENGINEERING AND FIELD EXPENSE (25.00 % DPC)	12500.

SUBTOTAL	62500.
CONTRACTOR'S FEE (7.00 % SUBTOTAL)	4375.
CONTINGENCIES (25.00 % SUBTOTAL)	15625.

FIXED CAPITAL COST (FCC)	82500.
WORKING CAPITAL COST	12529.
LAND	300.

TOTAL CAPITAL COST	95329.

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - DEC. 1981

Table 5-2

WORKING CAPITAL COST FOR THE CHLORINATION OF CHROMITE ORE
TO PRODUCE CHROMIUM AND IRON CHLORIDES
(25,000 TPY contained chromium)

	COST (THOUSANDS OF 1981 DOLLARS
PROCESS MATERIALS INVENTORY (CHEMICALS), 15 DAYS	778.
CHROME ORE INVENTORY, 60 DAYS	1999.
WORK IN PROCESS INVENTORY, 5 DAYS	809.
FINISHED GOODS INVENTORY, 15 DAYS	2426.
MATERIALS AND SUPPLIES, 3.00 % OF FIXED CAPITAL COSTS	2475.
CASH (PAYROLL AND MISCELLANEOUS), 30 DAYS	434.
ACCOUNTS RECEIVABLE, 30 DAYS	8460.
ACCOUNTS PAYABLE, 30 DAYS	-4852.
TOTAL WORKING CAPITAL	12529.

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - DEC. 1981

Table 5-3

OPERATING COST FOR THE CHLORINATION OF CHROMITE ORE TO PRODUCE CHROMIUM AND IRON CHLORIDES
(25,000 TPY contained chromium)

	NAME OF UNIT	QUANTITY PER YEAR	UNIT COST	ANNUAL COST	
				THOUSANDS OF 1981 DOLLARS	DOLLARS PER UNIT OF PRIMARY PRODUCT
RAW MATERIAL	TONS	91600.	90.93	8329.	0.17
PROCESS MATERIALS					
HC1 (CONTAINED)	TONS	35050.	75.00	2629.	0.05
COKE	TONS	32500.	60.00	1950.	0.04
OXYGEN	TONS	16000.	100.00	1600.	0.03
CHLORINE	TONS	45000.	125.00	5625.	0.11
IRON	TONS	10000.	100.00	1000.	0.02
ORGANIC				60.	0.00
OTHER				100.	0.00
TOTAL PROCESS MATERIALS				12964.	0.26
UTILITIES					
POWER	KWH	12000000.	0.04	480.	0.01
FUEL	MIL. BTU	700000.	4.00	2800.	0.06
STEAM	1000 LB	800000.	3.00	2400.	0.05
WATER				150.	0.00
TOTAL UTILITIES				5830.	0.12
LABOR DEPENDENT					
DIRECT LABOR (DL)	MAN-HOURS	156000.	14.50	2262.	0.05
SUPERVISION	(20.00 % DL)			452.	0.01
TECH, ADMIN, AND GEN EXPENSES	(40.00 % DL)			905.	0.02
TOTAL LABOR DEPENDENT				3619.	0.07
MAINTENANCE AND REPAIRS					
LABOR	(2.00 % FCC)			1650.	0.03
SUPPLIES	(3.00 % FCC)			2475.	0.05
TOTAL MAINTENANCE AND REPAIRS				4125.	0.08
OTHER CASH COSTS				3000.	0.06
TOTAL PRODUCTION COST				37867.	0.76
PROPERTY TAX	(2.00 % FCC & LAND)			1656.	0.03
INSURANCE	(0.50 % FCC)			413.	0.01
SELLING, GENERAL, AND ADMIN.				500.	0.01
TOTAL CASH COST				40436.	0.81

NOTES :: PRIMARY PRODUCT IS CHROMIC CHLORIDE
EXPRESSED IN UNITS OF POUNDS

'FCC' REPRESENTS FIXED CAPITAL COSTS

COMPONENTS MAY NOT ADD TO TOTALS DUE TO ROUNDING

SOURCE :: CHARLES RIVER ASSOCIATES, INCORPORATED - DEC. 1981

Table 5-4

SUMMARY OF CHLORINATION OF CHROMITE ORE
TO PRODUCE CHROMIUM AND IRON CHLORIDES
(25,000 tons/year Contained Chromium)

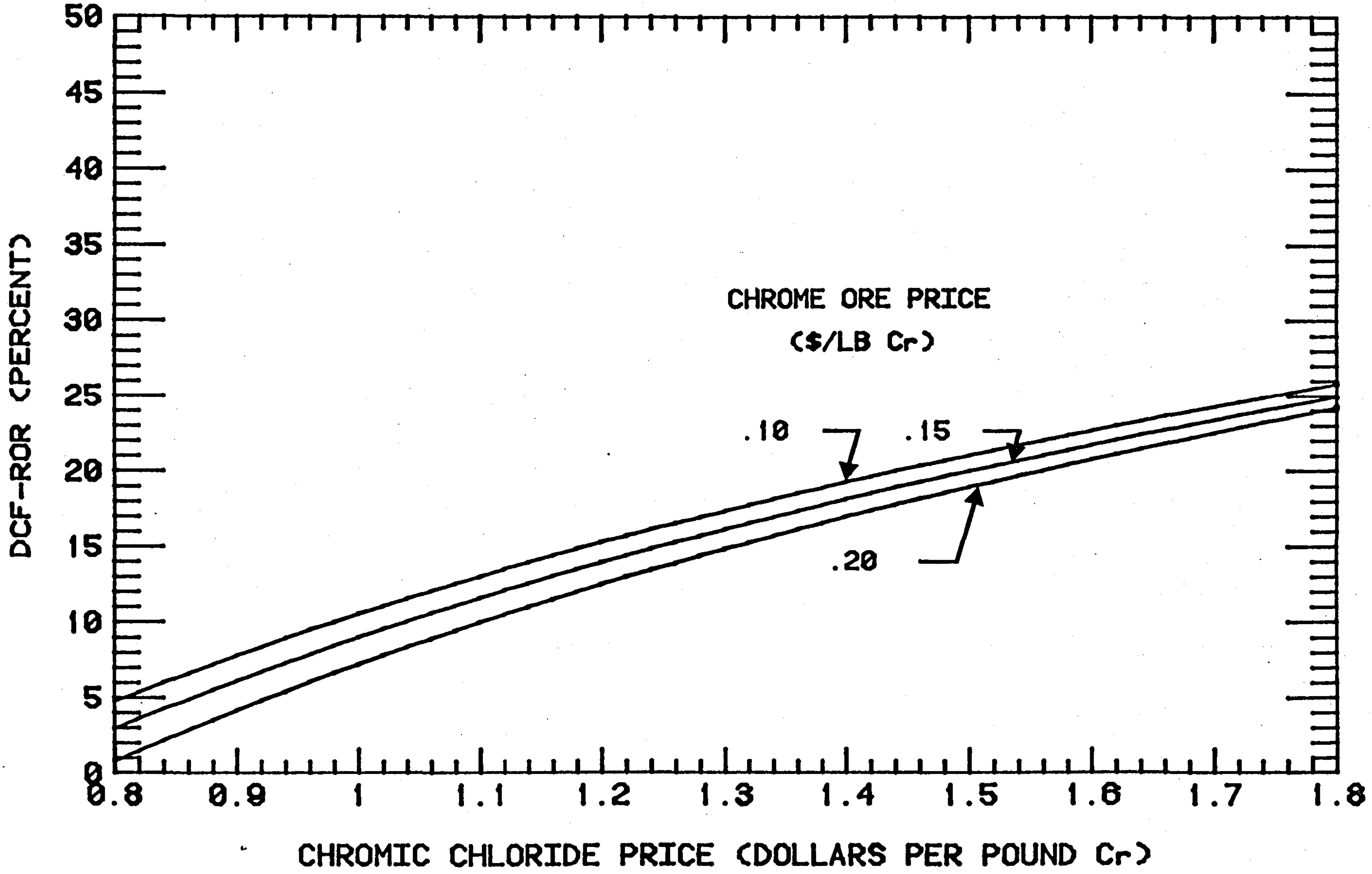
Revenues	Annual Amount ($ 000/year)	
Chromium Chloride Solution	51,320*	
Iron Chloride Solution	6,690	
TOTAL		58,010
Production Costs		40,440
Pretax Profit		17,570
Depreciation		8,250
Taxable Income		9,320
Net Income		5,030
Cash Flow		13,280

*Based on $1.00 per pound contained chromium.

SOURCE: Charles River Associates, December 1981.

Figure 5–2

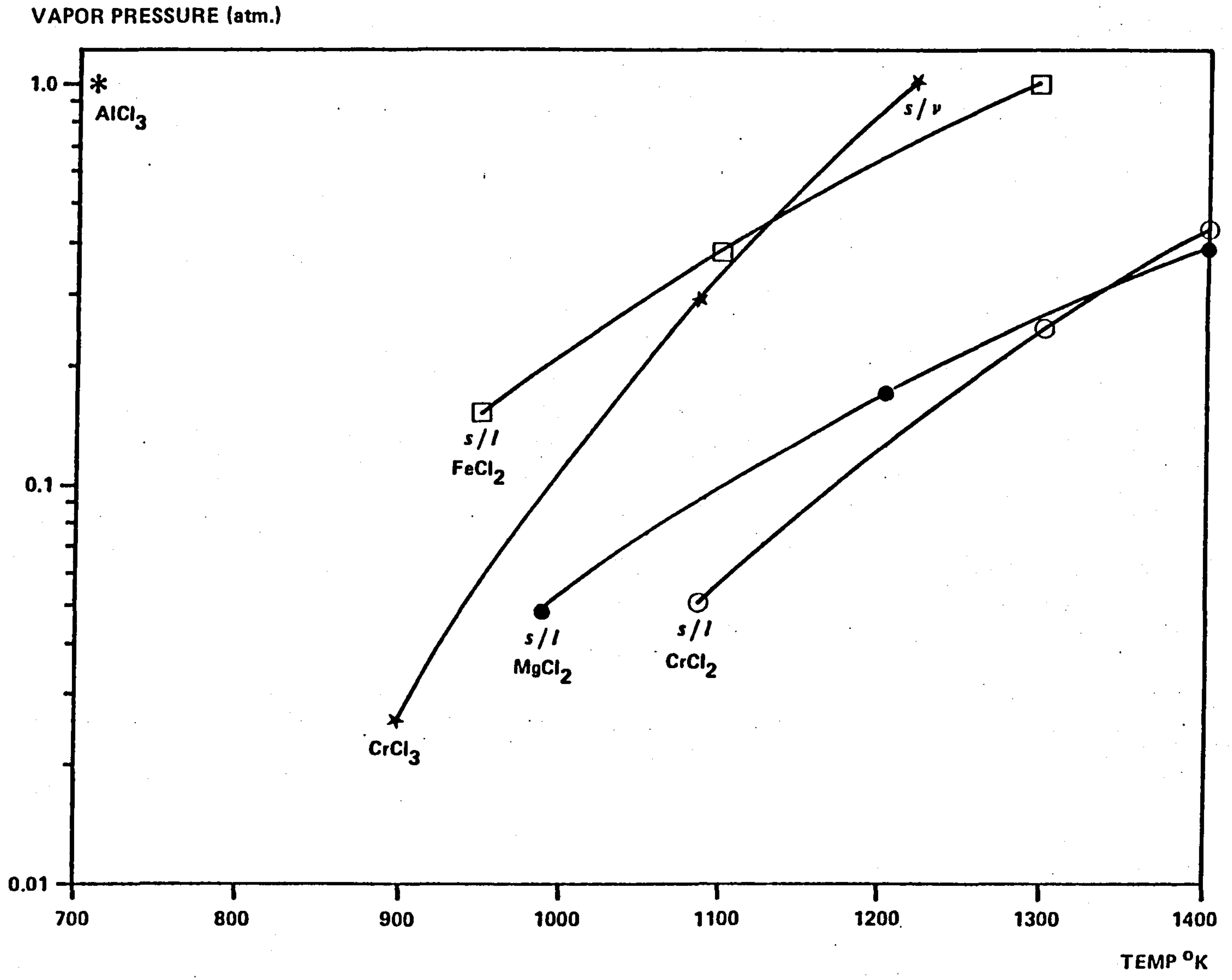
CHLORINATION OF CHROMITE ORE TO PRODUCE CHROMIUM AND IRON CHLORIDES



SOURCE: CHARLES RIVER ASSOCIATES, JANUARY 1982.

Figure 5–3

CALCULATED VAPOR PRESSURE OF METAL CHLORIDES VERSUS TEMPERATURE



SOURCE: Charles River Associates, October 1981.

The chlorination of metal oxides is commercially practiced, for example the chlorination of rutile to make $TiCl_4$. However, the chloride processes are highly corrosive and difficult to manage. Further, in the chlorination of chromite ore, the condensed chlorides are solid and the design of the condenser may be intricate compared to a condenser for liquid chlorides. Thus, there are uncertainties both in the design of the chlorination reactor and in the condensers. Apart from these areas, the remainder of the process may be straightforward. The high capital cost reflects some uncertainty as to viability of the chlorination process. Provision has been made for purification of the chromium chloride and the iron chloride streams. In addition, chlorides of undesirable metals will be generated, and provision has been made in the flow sheet to convert these to innocuous oxides and to recycle the chlorine back into the process.

6

REGENERATION OF REDOX SOLUTIONS

6.1 BACKGROUND

During operation of the NASA Redox Storage System, it is anticipated that slow intermixing of the chromium (anolyte) and iron (catholyte) solutions will occur. The mechanism is cross-diffusion of chromium and iron solute species through the anion-permeable membrane separator. Using physical data furnished by NASA, consideration is given to solution regeneration requirements and to an optimum regeneration procedure and associated costs.

For calculation of Redox-solution regeneration requirements, the following design criteria as established by NASA were employed:

1. Degree of intermixing allowed before regeneration of solutions required: 25 percent.
2. The membrane diffusion coefficient for chromium species does not exceed that for iron species, so that calculation of the rate of intermixing can be based on the latter.
3. Membrane diffusion coefficient for iron species: goal, $5\mu gFe/cm^2/h/mole/l$; diffusion coefficient attained with present membranes, $\sim 20\mu gFe/cm^2/h/mole/l$.

The time required for 25 percent intermixing was calculated by assuming simple, first-order diffusion. The applicable diffusion equation is:

$$-\frac{dm}{dt} = Dm\frac{A}{V} - D(m_0-m)\frac{A}{V} = \frac{DA}{V}(2m-m_0)$$

where:

D = mean diffusion coefficient for iron species in the membrane,

A = total active membrane area,

m = total mass of iron species in the catholyte, and

(m_0-m) = total mass of iron species in the anolyte, assumed to possess the same volume as the catholyte.

Rearrangement and integration yields

$$-\frac{dm}{2m-m_0} = -\frac{1}{2}\,d\ln(2m-m_0) = \frac{DA}{V}dt;\ \frac{1}{2}\ln\frac{m_0}{2m-m_0} = \frac{DA}{V}t$$

from which the time required for 25 percent intermixing can be calculated from

$$\frac{m}{m_0} = 0.75;\ \frac{1}{2}\ln\frac{m_0}{2m-m_0} = \frac{1}{2}\ln 2 = 0.34657^*$$

and known or assumed values of D, A and V.

Total active membrane area and total volume of anolyte or catholyte are given in the United Technologies Corp. final report for a 10MW/100MWh system as

$A = 152{,}646\,ft^2 (= 14{,}181\,m^2)$; $V = 1{,}213{,}000$ gal $(=4.5917\times10^6\,l)$.

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*If back-diffusion is neglected, the appropriate factor is $\ln(m_0/m)$ = 0.28768; if a constant diffusion rate is assumed, the corresponding factor is 0.25.

The target value of the membrane diffusion coefficient is

$$D = \frac{1}{\text{mole}}\ \frac{5\mu\text{g Fe}}{\text{cm}^2\ \text{h}} \times \frac{\text{mole}}{55.85\text{X}10^6\mu\text{g Fe}} \times \frac{10^4\text{cm}^2}{\text{m}^2} \times \frac{87.60\text{X}10^2\text{h}}{\text{yr}} = 7.8424\ \frac{1}{\text{m}^2\text{yr}}$$

$$t_{25\%} = 0.34657\ \frac{V}{DA} = \frac{0.34657 \text{ X } 4.5917 \text{ X } 10^6}{7.8424 \text{ X } 1.4181 \text{ X } 10^4} = 14.3 \text{ yr}$$

The estimated time for 25 percent intermixing, namely, 14.3 years, is so long that, averaged over the entire period, the volume of each solution to be regenerated would amount to only 232 gallons per day out of 1,213,000 gallons. Actually, if the indicated small volume of catholyte and of anolyte were bled from the system and regenerated on a daily basis, in 14.3 years the degree of intermixing resulting from membrane cross-diffusion would be appreciably less than 25 percent.

It should not be difficult to calculate the bleed-stream volume to regenerate Redox solutions at preselected intervals, or continuously, if that is the basis selected for regeneration. For present purposes, however, the important conclusion is that regeneration requirements for the NASA Redox System should be quite manageable. The obvious proviso is made that other sources of intermixing, such as perforations or cracks in the membranes and miscellaneous pathways leading to parasitic contacts between the two solutions contribute less than does membrane diffusion.

Since the time required for a given degree of intermixing by cross-diffusion through the membrane varies inversely as the magnitude of the effective diffusion coefficient, the calculated $t_{25\%}$ corresponding to the lowest rate of iron loss achieved as of the July 1980 industrial briefing, namely 14 μg Fe/cm^2/h/mole/l, is 14.3x5/14 = 5.10 yr. This shorter, approximately five-year period has been assumed in the estimation of regeneration requirements and costs, so that the results are presumably conservative. Because regeneration entails treatment of Redox solution external to the storage system proper, it is of economic significance that the volume of solution affected, averaged over time, is much less for regeneration than for rebalance.

6.2 REGENERATION PROCEDURE

Optimization of the procedure for regenerating the intermixed leach solutions was based on the following considerations:

1. No significant quantities of impurities are introduced during operation of the Redox Storage System, so that the paramount regeneration requirement is to separate iron species from chromium species.

2. The best separation scheme from many standpoints involves solvent extraction separation of ferric chloride by tributyl phosphate (TBP).

3. Net system oxidizing and reducing capacities must be preserved.

4. Precise adjustment of acid strength in the regenerated solutions is not essential, because self-adjustment can occur by permeation of HCl through the membranes.

Because of the incompatibility of the species $Fe^{3+}$and Cr^{2+}, only three of the four possible metal cations will be present in a given intermixed solution, namely, Fe^{3+}, Fe^{2+}, Cr^{3+} (catholyte) or Cr^{2+}, Cr^{3+}, Fe^{2+} (anolyte). In accordance with Condition 2 and in order to minimize reagent requirements for valence adjustment, the mainly-iron redox solution should be withdrawn from the system in its most highly charged state (Fe^{3+}/Fe^{2+} max.), and the mainly-chromium Redox solution should be withdrawn in its most highly discharged state (Cr^{2+}/Cr^{3+}min.). Both regenerated Redox solutions could be added back to their respective circuits directly, or the regenerated iron chloride solution, now entirely ferric, could be passed through the rebalance cells (Figure 6-1). In either case, overall system oxidizing and reducing capacities would be brought into balance by operation of the rebalance cells as appropriate.

The foregoing recommendation of solution withdrawal and replacement procedure presupposes that the system will not be completely shut down every five years for exchange of the entire solution inventory. If that course were followed, ferrous chloride and chromic chloride would presumably be supplied as for system startup. Instead of this, it is proposed that a suitable fraction of the Redox solution inventory be replaced at convenient intervals, say, annually or semiannually. It should be noted that shorter-interval solution replacement will result in a lesser average degree of intermixing but will also increase overall regeneration requirements (because of the thereby greater iron concentration).

There is no evident advantage in regenerating the two intermixed Redox solutions separately. The total volume of solution to be treated and net oxidant requirement will be the same, as illustrated in Figure 6-2. The examples there given are for initially two molar iron chloride and chromium chloride solutions both 25 percent intermixed, withdrawn from a system cycling between 10 percent and 90 percent of discharge. In actual practice,

Figure 6–1

SCHEMATIC FLOW PATTERN OF THE REDOX STORAGE SYSTEM INCORPORATING REGENERATION AND REBALANCE FUNCTIONS. SYSTEM SHOWN ON CHARGE CYCLE.

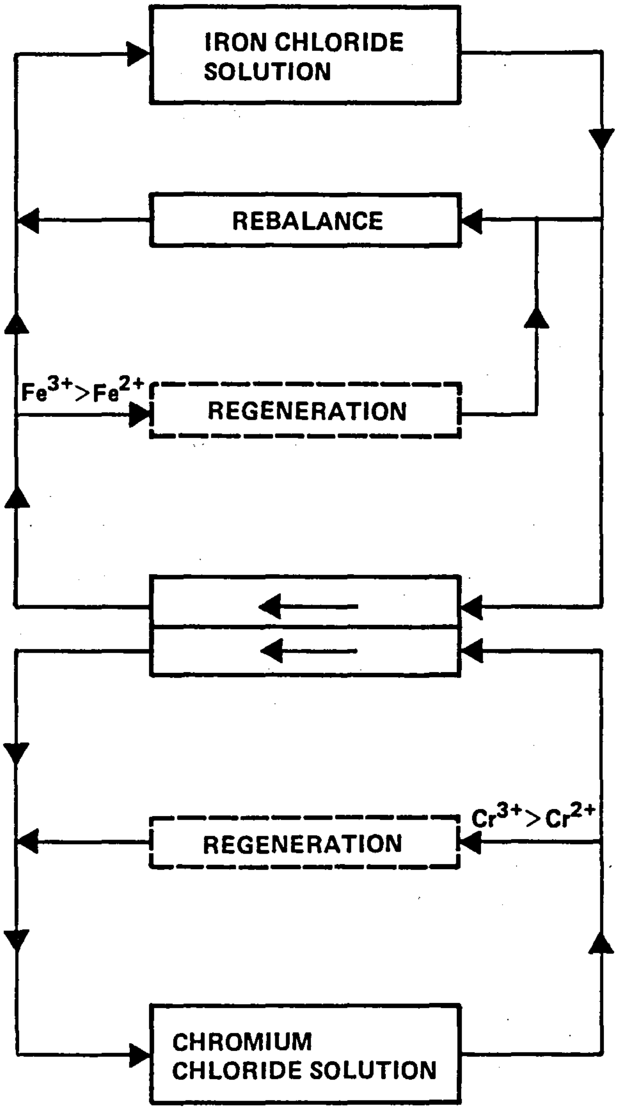
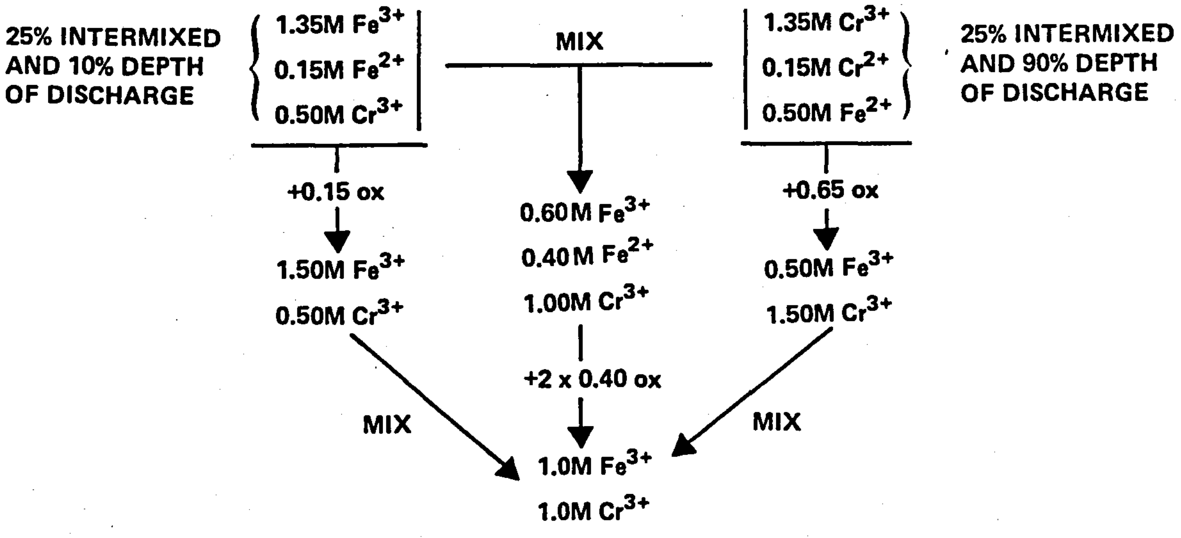


Figure 6–2

ILLUSTRATION OF REDOX SOLUTION OXIDATION PREPARATORY TO SOLVENT EXTRACTION



NOTE: "ox" = units of oxidant added

the degrees of intermixing will most probably not be the same for the two solutions, but the consequences of this for regeneration purposes are likely to be minor. If desired, appropriately proportioned volumes could be regenerated to maintain intermixing on both sides of the membrane within specified limits.

The Redox solutions to be regenerated are assumed to be mixed together, whereupon Cr^{2+} will be oxidized by Fe^{3+}, generating equivalent Fe^{2+}. The total ferrous content of the mixed solution would then be oxidized using air or oxygen, or chlorine, or a combination of the two oxidants. The choice depends in part upon any desired adjustments to total system acid and chloride:

$$4FeCl_2 + 4HCl + O_2 = 4FeCl_3 + 2H_2O$$

$$4FeCl_2 + 2Cl_2 = 4FeCl_3$$

Use of air or oxygen in place of chlorine might require a catalyst such as charcoal for rapid oxidation kinetics. The resulting ferric/chromic chloride solution would then be solvent extracted, whereby $FeCl_3$ and accompanying HCl would be separated, the iron essentially quantitatively, from $CrCl_3$ and the remaining HCl.

If this operation is carried out at a central location where initial pure Redox solutions are prepared from ferrochrome, the intermixed solutions from the Redox storage facility could be added to the clarified ferrochrome leach solution. Under some circumstances, it would be convenient to perform the regeneration function in-house, on a bleed-stream basis, employing a small solvent extraction unit. The applicable continuity equation for that mode of operation is

$$-\frac{dm}{dt} = \frac{DA}{V}(2m-m_0) - \frac{\delta V}{\delta t}\frac{m_0-m}{V}$$

where δV is the volume of iron chloride solution bled from the system in the short time interval δt or, if regeneration is performed on a continuous basis, $\delta V/\delta t$ is the volumetric flow rate of the bleed stream.

By either mode of operation, if the regenerated ferric chloride solution is passed through regeneration cells before entering the iron chloride circuit, the excess oxidizing capacity resulting from the regeneration treatment can be eliminated by electrochemical reduction with hydrogen:

$$2FeCl_3 + H_2 = 2FeCl_2 + 2HCl$$

In this connection, it will be recalled that the principal function of the rebalance cells, which operate in the iron chloride circuit, is to adjust for extraneous increases in system oxidizing capacity and concomitant decreases in system acid capacity that result from air oxidation of Fe^{2+} and Cr^{2+} and electrochemical reduction of H^+. Since the last effect is large, comprising about 1 percent of the chemical change occurring in the anolyte during the charging cycle, it should not be necessary to increase the capacity of the rebalance units above that designed into the original system.

6.3 COST OF REGENERATION

Regeneration of the Redox solutions can be accomplished most effectively by replacing a suitable fraction of each solution at convenient intervals. Regeneration can be carried out either at a central location where initial pure Redox solutions are prepared from ferrochrome, or in-house where the regeneration function is performed on a bleed-stream basis.

If the regeneration is performed at a central location, the required cost is estimated to be about 40 to 50 cents per pound of contained chromium in solution. Adding the cost for shipping the solution (1,000-mile round trip) brings the total cost to around $1.00 per pound of chromium. This cost includes regeneration and transport of the iron chloride solution as well as the chromium chloride solution. It may be possible to achieve a lower overall cost by regenerating the solution in-house, since there would be large savings in transportation. However, the regeneration unit would be built on a very small scale and therefore would be more expensive per unit of chromium due to economies of scale.

7

DISCUSSION OF RESULTS

Three manufacturing processes were considered for making chromium and iron chlorides. Two are hydrometallurgical processes in which either chromite ore or ferrochromium is dissolved in hydrochloric acid, and the desired metals are separated and recovered as purified two molar solutions. The third process employs the reduction-chlorination of chromite ores to generate iron and chromium chloride vapors which are separated, purified and recovered.

The economics for the three manufacturing processes are summarized in Table 7-1. The hydrometallurgical process based on ferrochrome has the lowest investment at an estimated $40 million or $0.80 per annual pound of contained chromium in the chromium chloride product. The selling price required to provide the manufacturer with a 15 percent discounted cash flow rate of return (DCF-ROR) is estimated at $0.97 per pound of chromium, yielding a sales to investment ratio for a 25,000 ton per year plant of $1.20 revenue per dollar invested. Ferrochrome raw material is the largest operating cost component at $0.53 per pound of contained chromium out of a total operating cost of $0.87 per pound.

A second process based upon the hydrometallurgical dissolution of chromite ore could produce chromium chloride for sale at about $1.10 per pound of contained chromium. This process has an advantage over the first in using low cost chromite ore as a raw material. However, the investment requirement is estimated at $69 million or $1.39 per annual pound contained chromium product.

Table 7-1

COMPARISON OF ALTERNATIVES FOR PRODUCTION OF CHROMIUM AND IRON CHLORIDES
(25,000 tons contained chromium per year)

Process	Hydrometallurgical		Reduction Chlorination
Source of Chromium	Ferrochrome	Chromite Ore	Chromite Ore
Chromium material consumption, tons/yr	47,847	91,392	91,600
cost, cents/lb	0.53	0.17	0.17
Energy consumption, kwh/lb Cr	0.12	0.24	0.24
Mil Btu/lb Cr	0.0043	0.067	0.03
Investment*, $ million			
Fixed capital	27.1	54.5	82.5
Working capital & other	12.8	9.9	12.8
Total	39.9	69.3	95.3
Unit Investment*, $/annual lb Cr	0.80	1.38	1.91
Operating cost**, $/lb Cr	0.87	0.82	0.81
Required selling price,+ $/lb Cr	0.97	1.10	1.25

*Investment accuracy is estimated at plus 30%, minus 0%.
**Operating cost accuracy is estimated at plus 10%, minus 10%.
+Price to achieve 15 percent DCF-ROR on investment.

SOURCE: Charles River Associates, January 1982.

A third process is based on the chlorination of chromite ore. This process is technically more difficult than either of the other two processes. The investment for a 25,000 ton contained chromium per year plant is estimated at $95 million, or $1.91 per annual pound chromium. The cost of production is slightly lower than the other processes, but the large investment would require a selling price of $1.25 per pound contained chromium.

There is excessive capacity to produce ferrochromium throughout the world, and the material should be readily available although the price should be sensitive to world energy costs. Although the hydrometallurgical process to convert ferrochrome to chromium and iron chlorides is not demonstrated, it is based on traditional metallurgical engineering and conventional process equipment. Hence, there is a high likelihood that the process can produce the chromium and iron chlorides at the projected price and investment cost.

The hydrometallurgical process for dissolution of chromite ore in hydrochloric acid appears to be technically feasible. The dissolution step requires elevated temperature and pressures. Once the ore is dissolved, the chloride solution will contain a number of components and an elaborate, unproven process will be required to separate and purify the desirable chromium and iron chlorides and to discard the wastes in an environmentally acceptable manner. While we foresee the process as technically feasible, a few years of research and development may be required to put such a process into commercial operation. We expect that the investment costs and the projected selling price would reflect a fully developed process and costs for the R&D are included in the financial calculations.

The direct chlorination of chromite ore is the most complex of the three processes, involving both high temperatures and a reducing/chlorine environment to react the ore. While titanium dioxide and silicon dioxides are commercially chlorinated, they are relatively pure raw materials compared to the chromite ores. The handling of solids in the chlorinator, and the cooling and separation of multicomponent chloride vapors, is anticipated to be technically difficult. Extensive R&D will be required to bring this type of process into commercial operation. There is some possibility that the investment and direct operating costs may be understated. The direct chlorination process is the most technically uncertain and expensive of the three that have been considered in this report.

8

CONCLUSIONS

It should be possible to produce chromium and iron chloride solutions commercially (i.e., on a scale of 25,000 tons of contained chromium per year), at a cost of \$0.97 to \$1.25 per pound chromium.

The lowest cost production would be achieved using ferrochromium as the basic raw material which is dissolved in hydrochloric acid. Though not demonstrated, this process is technically sound and it should be possibile to commercialize this method of manufacture with a minimum of research and development.

Two processes which use low cost chromite ore to produce chromium and iron chlorides have investments of \$1.38 to \$1.90 per annual pound of chromium and would require prices of \$1.10 to \$1.25 per pound of contained chromium. Because of the impurities in the chromite ore and the pressure/temperature conditions required to chlorinate/hydrochlorinate the ore, these processes are complex, have high investment cost, and may require several years of research and development before commercial operation is possible.

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16. Abstract

The chromium and iron chloride chemicals are significant first costs for NASA Redox energy storage systems. This study was performed to determine the lowest cost at which chromium and iron chlorides could be obtained for a complex of Redox energy storage systems. In addition, since the solutions gradually become intermixed during the course of operation of Redox units, it was an objective of this study to evaluate schemes for regeneration of the operating solutions. Three processes were evaluated for the production of chromium and iron chlorides. As a basis for the preliminary plant design and economic evaluation, it was assumed that the plant would produce about 25,000 tons of contained chromium as $CrCl_3$ and an equivalent molar quantity of $FeCl_2$. Preliminary plant designs, including materials and energy balances and sizing of major equipment, were prepared, and capital and operating costs were estimated.

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