

ALUMINA PROCESS FEASIBILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN

**Task 3 Report:
Preliminary Design of 25 Ton Per Day Pilot Plant**

VOLUME I PROCESS TECHNOLOGY AND COSTS

**KAISER ENGINEERS, INC. - CONTRACTOR
KAISER ALUMINUM & CHEMICAL CORPORATION - SUBCONTRACTOR
OAKLAND, CALIFORNIA**

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FOREWORD

The Task 3 report, consisting of six volumes, was prepared by Kaiser Engineers Inc., Oakland, California, under USBM Contract number J0265048. The contract was initiated under the Metallurgy Program. It was administered under the technical direction of Reno Research Center with Gerald B. McSweeney acting as Technical Project Officer. Ronald J. Simonich was the contract administrator for the Bureau of Mines. This report is a summary of the work recently completed as a part of this contract during the period September 1976 to November 1979. This report was submitted by the authors in November 1979. The report contains no patentable features.

Because of the voluminous size of the Task 3 report, and an expected limited interest in volumes 2 to 6, only volume 1 has been placed with NTIS. Volumes 2 to 6 are on open file, and are available for public reference during regular working hours at the National Library of Natural Resources, U.S. Department of the Interior, 18th and C Streets, NW, Washington, D.C., and at the following Bureau of Mines facilities: Albany Research Center, 1450 West Queen Street, Albany, Oregon; Avondale Research Center, 4900 LaSalle Road, Avondale, Maryland; Boulder City Engineering Laboratory, 500 Date Street, Boulder City, Nevada; Denver Research Center, Building 20, Denver Federal Center, Denver, Colorado; Pittsburgh Research Center, Cochran's Mill Road, P. O. Box 18070, Pittsburgh, Pennsylvania; Reno Research Center, 1605 Evans Avenue, Reno, Nevada; Rolla Research Center, 13th and Bishop Streets, Rolla, Missouri; Salt Lake City Research Center, 729 Arapeen Drive, Salt Lake City, Utah; Spokane Research Center, East 315 Montgomery Avenue, Spokane, Washington; Tuscaloosa Research Center, Capstone Drive, University of Alabama Campus, Tuscaloosa, Alabama; Twin Cities Research Center, Country Road 62 and Highway 55, Fort Snelling, Minnesota.

ALUMINA PROCESS FEASIBILITY STUDY AND
PRELIMINARY PILOT PLANT DESIGN

U.S. BUREAU OF MINES CONTRACT No. J0265048
Kaiser Engineers Job No. 76161-003

Task 3, Volume I

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NOTE

Kaiser Engineers originally submitted their report on Task 3 of contract J0265048 in the form of six volumes: Volume I, Process Technology and Costs; Volume II, Appendix A - Equipment List, Specifications and Quotations (Areas 1 through 7); Volume III, Appendix A - Equipment List, Specifications and Quotations (Areas 8 through 25); Volume IV, Appendix B - General Specifications; Volume V, Appendix B - General Specifications; and Volume VI, Appendix C - Drawings. The pagination for this volume has been renumbered consecutively for distribution through the National Technical Information Service and is not the same pagination as for the open file report.

1.0 INTRODUCTION

1.1 The Bureau of Mines awarded Contract No. J0265048 to Kaiser Engineers in Oakland, California, on September 30, 1976. With Kaiser Aluminum & Chemical Corporation as subcontractor, the objective of the project was the design of a 10 to 50 ton per day alumina from domestic resources pilot plant. The contract, entitled "Alumina Process Feasibility Study and Preliminary Pilot Plant Design," called for this work to be performed in three distinct, separate, and consecutive tasks, with approval by the Bureau of Mines required upon completion of each task before proceeding to the next.

The first of the three task reports was issued in September, 1977. The objective of the first task was to reduce the number of candidate processes from six to two. The six processes were:

- Clay/Nitric Acid
- Clay/Hydrochloric Acid using Evaporative Crystallization
- Clay/Hydrochloric Acid using Gas-Induced Crystallization
- Clay/Sulfurous Acid
- Anorthosite/Lime-Sinter
- Alunite/Reduction Roast - Bayer Extraction

On the basis of comparative capital and operating cost estimates, together with a technical analysis of each process and other considerations, the Bureau of Mines selected the Clay/Nitric Acid and Clay/Hydrochloric Acid (Gas-Induced Crystallization) processes and directed Kaiser Engineers to further study these two processes in the second task.

The second task report, issued in February 1978, recommended to the Bureau of Mines the process technology of the two selected in the first task that had the greater potential for supplying cell-grade alumina from kaolinic clay. To reach the objective of Task 2 required the expansion and refinement of the technical and comparative economic factors considered in the previous task, and the utilization of additional data developed in the Bureau of Mines mini-plant program. This program has been conducted on an on-going basis, as a jointly funded research project, by the Bureau of Mines and cooperating aluminum companies.

Based on the results of the Task 2 study, the Bureau selected the Clay/Hydrochloric Acid Extraction-Gas Induced Crystallization Process as the most promising technology for the preliminary design of a pilot plant in Task 3.

1.2 This report is concerned with the preliminary design and costs of a pilot plant to produce cell-grade alumina from clay, using the hydrochloric acid-gas induced crystallization process. Notice to proceed with the third task was given by the Bureau of Mines in February 1978. In the first few months of work on this task, the specification for maximum impurity levels in the product alumina was established by the Product Subcommittee of the Industrial Steering Committee in cooperation with the Bureau of Mines. Simultaneously, research work at the Boulder City Metallurgy Engineering Laboratory indicated that a single crystallization

step might not be expected to meet these specified levels under presently known techniques of crystallization.

Other refinements of the Task 2 process necessitated a review of the comparative costs reported in Task 2. A section of this Task 3 report was therefore added to address the differences in comparative costs for a commercial size (500,000 TPY) alumina plant using the Task 3 process.

Further refinements or confirmation of earlier assumptions to the Task 2 process have been made as a result of new data developed by research work conducted by the laboratories of the Bureau of Mines and private vendors. These have been included in the Task 3 preliminary pilot plant design.

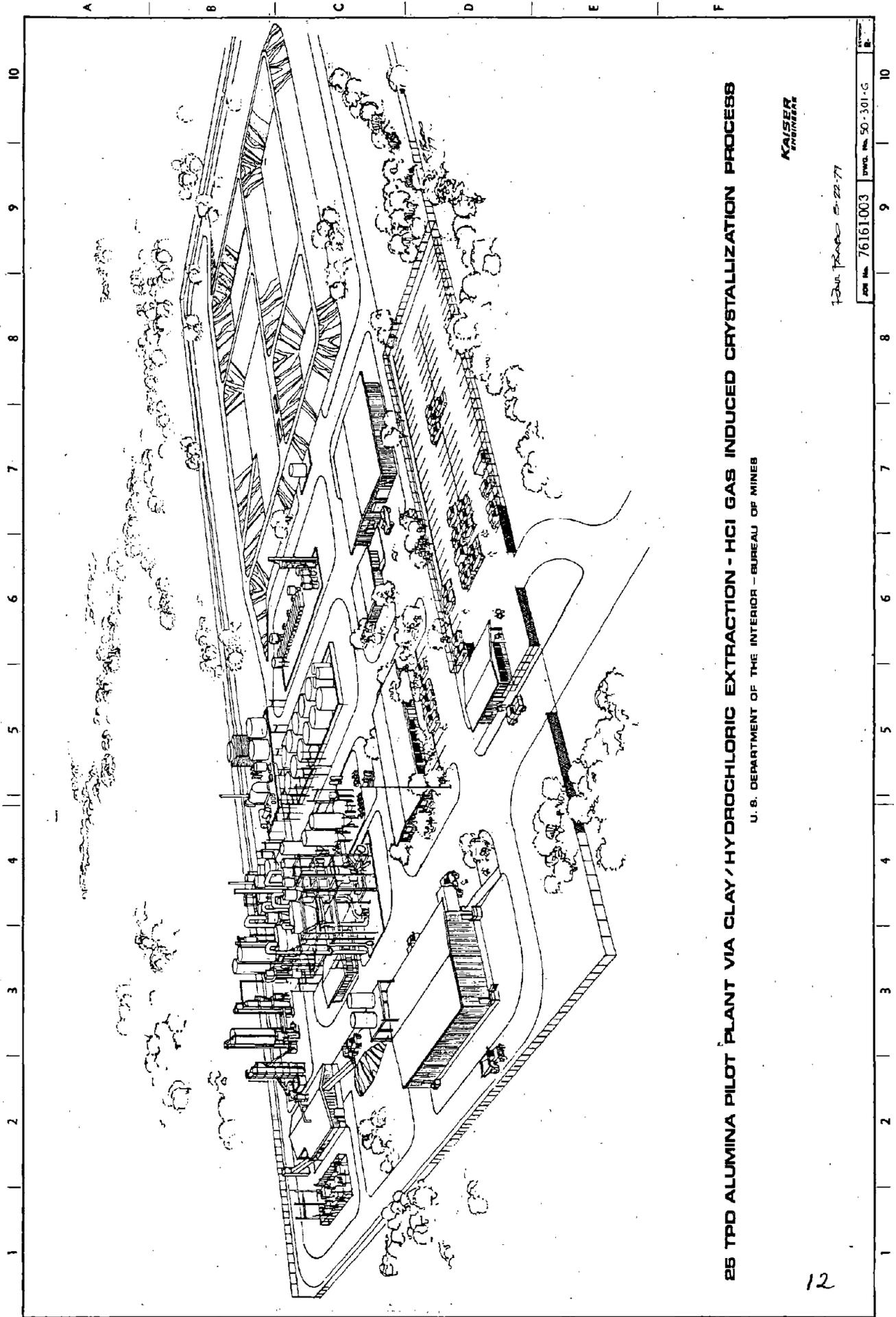
The size of the pilot plant was determined in consultation with the Bureau. The criterion was that a 25 TPD alumina pilot plant, when operated continuously for extended periods, would provide data suitable for scale up of the process technology to commercial size. An important consideration in making this determination was that by suitable measurement and data collection, the process heat utilization and heat recovery and losses could be interrelated in a manner comparable to expectations for a full-size plant. Lastly, the process equipment and areas have been fully enclosed to permit evaluations of the environmental impact the plant would have on a site considered appropriate for this process.

The estimated capital cost for the pilot plant is approximately 53.5 million dollars, exclusive of fee and escalation but including an allowance of approximately 1 million dollars to cover such items as land, right-of-ways, permits, capital spare equipment, taxes and the like.

This Task 3 report contains the technology, costs, and process drawings necessary to convey the intent of the plant design to the Bureau of Mines, Industry Cooperators, Contractors, and Suppliers. Prior to construction,

- a site must be provided
- detailed process and engineering design and procurement commenced
- federal, state, and local permits acquired
- right-of-ways obtained
- soils investigations and testing performed.

Included in this section are a general arrangement and a linear perspective of the pilot plant and its facilities. The plot is approximately 825 feet by 1,485 feet long, and occupies approximately 28 acres.



25 TPD ALUMINA PILOT PLANT VIA CLAY / HYDROCHLORIC EXTRACTION - HCl GAS INDUCED CRYSTALLIZATION PROCESS

U. S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

KAISER
ENGINEERING

Draw. No. 50-22-77

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2.0 SUMMARY

2.1 Pilot Plant Design Requirements

The purpose of the pilot plant is to demonstrate the feasibility of the process and provide the necessary data for the future design of a commercial size plant. In order to fulfill this purpose many factors affecting the design had to be considered. The major ones are outlined below:

Scale-Up

The size of the equipment must be such as to facilitate scale-up to commercial size. See Section 2.2 for details on plant sizing.

Alumina Testing

The pilot plant must be large enough to provide a sufficient quantity of alumina for testing in full scale aluminum cells in order to demonstrate that the production of on-grade aluminum from this alumina is feasible.

The decision was made to provide 9000 T of alumina for this purpose. See Section 2.2 for details.

Product Quality

The required alumina product quality, as recommended by a subcommittee of the Industry/USBM Mini-plant Program, is given below:

<u>Component</u>	<u>Impurity Levels in Wt. Percent of Product Alumina</u>
SiO ₂	0.015 max.
Fe ₂ O ₃	0.015 max.
Na ₂ O	0.40 max.
CaO	0.04 max.
B ₂ O ₃	0.001 max.
K ₂ O	0.005 max.
P ₂ O ₅	0.001 max.
NiO	0.005 max.
Ga ₂ O ₃	0.02 max.
MnO	0.002 max.
MgO	0.002 max.

<u>Component</u>	<u>Impurity Levels in Wt. Percent of Product Alumina</u>
Cr ₂ O ₃	0.002 max.
ZnO	0.02 max.
CuO	0.01 max.
V ₂ O ₅	0.002 max.
TiO ₂	0.005 max.
Residual Chloride	0.1 max., 0.05 target

The process has been designed to meet these specifications. In particular solvent extraction is used to remove the iron and the product is crystallized, dissolved and recrystallized to meet the specifications of all the other elements in the list. In addition, the bleed stream process section is provided to continuously purge the impurity elements from the process in order to prevent the build-up of impurity levels in process liquors and the product.

Tests indicate that the specified chloride content in the product can be achieved at reasonable temperatures and holding times in a fluid bed calciner. The required physical properties of the product alumina include α - alumina content 10-25 percent and B.E.T. surface area approximately 45 m²/gm.

Typical Clay Analysis

The chemical analysis of the clay feed (dry basis) used for the pilot plant design is as follows:

<u>Component</u>	<u>%</u>
SiO ₂	46.4
Al ₂ O ₃	36.5
L.O.I.	13.54
TiO ₂	2.23
Fe ₂ O ₃	0.86
MgO	0.082
CaO	0.042
K ₂ O	0.095
Na ₂ O	0.046
SO ₄	0.10
P ₂ O ₅	0.069
F	0.022
Other	0.04

The raw clay feed to the process contains 18.5% free moisture.

Data Collection

One of the primary functions of the pilot plant will be to collect data for future use in the design of a commercial plant. The plant has been instrumented to obtain this data and a small computer system has been provided to collect, organize and store this information. Long term storage of this data will be in the form of a computer print-out.

The computer system will be capable of displaying process data on cathode ray tubes and of running other programs without interfering with its data collection functions. Details of this system are contained in Section 4.2, Data Collection.

Flexibility of Operation

One of the prime requisites of any pilot plant is flexibility of operation in order that various modes of operation may be examined. In order to achieve the desired level of flexibility the design includes:

- Acid recovery and preparation equipment which will permit operation of the crystallizer section for limited periods when the ACH decomposition section is shut down and vice versa. See Section 3.2.9 Acid Recovery for details.
- Excess evaporating capacity in the evaporation section to remove additional water (compared to normal operation) from the process, if necessary.
- Excess bleed stream capacity which will permit processing a clay with double the design impurity level, if necessary.
- Liquor surge capacity between areas to permit shutdown of various process sections for up to 72 hours without affecting the operation of the rest of the plant.
- Spare equipment where feasible to maintain production during equipment maintenance, e.g., all the pumps on the main process liquor circuit have a non-operating spare.
- Excess capacity was provided in Iron Removal Section which will permit removal of up to 3.5 weight percent of Fe_2O_3 .

Environmental

Four different lined impoundment ponds are provided to receive liquid and solid waste from the plant. Two of these ponds provide the ability to test 2 alternatives for the disposal of mud from the mud washing system. One pond will receive a 50% solids filter cake, the other will receive the mud as a 10-25% slurry. A third pond, equipped with a lime pit for acid neutralization, will receive process spills which cannot be returned to the process. A fourth pond will settle solids and allow analysis of effluent before discharge to environment.

Airborne material from the plant is controlled by providing dust collectors where needed and HCl scrubbing systems connected to the vents on all vessels and equipment which may emit hydrochloric acid vapors.

Each process area containing acids and slurries has been provided with corrosion resistant coatings over concrete slabs; curbs prevent accidental spills from migrating to other areas; sumps and pumps remove spills to waste disposal and treatment.

2.2 Plant Size Determination

2.2.1 Objectives

It was decided at the outset that the pilot plant must be sized to meet several critical requirements, namely:

- Provide the technical data required for the design and operation of a commercial size plant.
- Provide performance data which would enable industrial participants to develop their own capital and operating costs of a commercial size plant.
- Demonstrate the commercial viability of the process through continuous, successful operation of the whole pilot plant for an extended period of time.
- Provide sufficient alumina for testing in full scale aluminum cells.

2.2.2 Conclusions

It was concluded that a pilot plant sized to produce 25 TPD of alumina would be the optimum size for the following reasons:

- A 25 TPD plant would use equipment of such a size that it could be readily scaled-up to commercial size. This fact will greatly facilitate the development of technical and economic data for a commercial size plant. Equipment vendors and other authorities were polled to establish permissible scale-up factors for all the critical equipment items.
- A 25 TPD plant would produce about 9000 tons of cell grade alumina for subsequent testing in full scale aluminum cells. The consensus of opinion among mini-plant cooperators was that this much alumina would be sufficient for test purposes.
- A 25 TPD plant would use equipment which is big enough to convincingly demonstrate the commercial viability of the process. In other words, the pilot plant will look more like a small-scale commercial plant than an enlarged bench-scale facility.

- A 25 TPD plant would be the minimum size pilot plant which would accomplish all of the objectives listed in the section above.

2.3 Process Description

2.3.1 Clay Preparation and Calcination

Run of mine clay will be delivered to the plant, crushed to -2 inch and transported to the raw clay storage building. Raw clay is then reclaimed from this building by front end loader which dumps to a conveyor transporting the clay to a secondary crusher.

The crusher product is screened in order to supply a nominal -4 and +20 mesh material to the clay calciner. The fines are pelletized and also fed to the clay calciner. Provision is made for varying the size of the clay feed to calcination in order that the effect on subsequent mud settling and filtration can be measured.

The clay calciner consists of a 3 stage fluid bed reactor with coal firing into the bed. The clay is calcined for 0.1-2 hours at about 1450°F in order to activate approximately 99 percent of the alumina content in the clay for subsequent leaching. The basic reaction is:



2.3.2 Clay Leaching

The calcined clay is metered out of a storage bin to 3 leach tanks operating in series. Leach acid containing about 25% HCl is metered to the leach tanks at a rate designed to provide a slight excess of HCl in the effluent. The reaction is exothermic which maintains the slurry at the boiling point of about 220°F at atmospheric pressure. About 95% extraction of the available alumina takes place in 1-2 hours under these conditions. The main reaction is:



The effluent from the leach tanks is then flash cooled under a slight vacuum to 140°F in order to prevent damage to the polymer lining of subsequent mud settlers and other vessels.

2.3.3 Mud Separation and Washing

The cooled leach slurry is fed to a rubber lined settler to separate the solids from the liquor. The overflow from the settler is pumped through a sand filter to remove trace amounts of solids and supply a clear liquor to the iron removal section.

The mud in the underflow from the settler is then subjected to a counter current water wash, first in settlers and then on a horizontal belt filter to recover almost all the AlCl_3 before being transported to the waste impoundment area.

2.3.4 Iron Removal

The polished pregnant liquor from the sand filters is first treated by the injection of gaseous chlorine to convert all ferrous iron to ferric iron before entering the iron removal section.

In this section the iron is removed from the treated liquor by solvent extraction with an organic extractant containing Alamine 336, decyl alcohol and kerosene.

2.3.5 Evaporation

The raffinate (purified pregnant liquor) is next evaporated to raise the $AlCl_3$ concentration close to the saturation point at about 30% $AlCl_3$ in order to maximize the yield of aluminum chloride hexahydrate (ACH) in the subsequent crystallization step.

2.3.6 Crystallization

In this section the concentrated $AlCl_3$ liquor from the evaporators is first fed to the primary crystallizers, where ACH crystals are produced by sparging HCl gas into the liquor. These ACH crystals are then separated from the mother liquor on a horizontal belt filter and redissolved in a combination of process liquors.

The ACH is thereafter recrystallized in a secondary crystallization step, again utilizing HCl gas sparging, followed by deliquoring and washing of the crystals on a centrifuge.

This method of crystallizing and washing the crystals will produce a sufficiently pure ACH crystal to meet the high degree of purity specified for the product.

2.3.7 Decomposition

This section of the process decomposes the ACH crystals produced in the crystallizers to alumina with the simultaneous production of HCl and H_2O vapors as represented by:



The decomposition is accomplished in 2 steps. In the first the ACH crystals are about 90% decomposed in an indirectly heated fluid bed reactor at 400°F to 750°F. A molten salt heating system is used to supply heat through tubes immersed in the bed.

In the second step the partially decomposed ACH is further calcined at 1600°F - 2000°F in a direct oil fired fluid bed reactor to obtain a product alumina with less than 0.1% residual chloride.

The off-gases from the decomposition section are processed in the acid recovery section of the process.

2.3.8 Acid Recovery

The purpose of the acid recovery section of the process is to receive the liquid and gaseous HCl containing effluents produced in various sections of the process and convert them to the required physical state and HCl concentration for recycle to the process. This section receives vapors from the ACH decomposer, and produces HCl gas for sparging use in the crystallizers as well as an uncontaminated 20% HCl solution for washing ACH crystals.

The HCl gas generated in this area is combined with the gas from the main ACH dissolver and the bleed stream HCl stripper to provide the total HCl gas required for crystallizer sparging.

In addition, a 25% leach acid is prepared by adjusting the HCl concentration of the crystallizer mother liquor with low HCl concentration condensates.

2.3.9 Bleed Stream Treatment

The purpose of this section of the process is to systematically purge impurities from the process in order that the product will not contain excessive impurities. The clay feed contains trace amounts of many metals, most of which are dissolved to some extent by the leach acid to form chlorides.

In order that these impurity metal chlorides do not build up in the process liquor a portion of the mother liquor from ACH crystallization is taken from the process as a "bleed stream". The ACH content of this bleed stream is recovered in evaporative crystallizers. The HCl content is recovered in the bleed stream HCl stripper and the remaining liquor is calcined in a fluid bed with the addition of SO₂. The impurities are discharged from the fluid bed reactor in the form of dry, insoluble pellets for disposal.

2.4 Pilot Plant Operations

2.4.1 Pilot Plant Costs

The total cost for operating the pilot plant to produce 9000 T of cell grade alumina is estimated to be \$40,916,190. The major components of this cost are: 1) Raw materials, \$1,012,000 2) Processing supplies, \$833,995, 3) Fuel and utilities, \$4,318,430, 4) Labor, \$14,703,400, 5) Miscellaneous including R & M supplies and services, taxes and insurance, \$10,529,000, 6) Operating contractor off-site expenses and fees, \$2,700,000 and 7) Contingency, \$6,819,365. The detailed breakdown of these costs is presented in Table 4.7.1. These costs represent those costs incurred by the operating contractor generally cover the 3½ year period between plant construction and plant demolition. Costs incurred by the government or participating companies are not included.

2.4.2 Pilot Plant Manning

The manning for the pilot plant will be according to that shown of Figure 4.5.1. The period of employment is shown on Figure 4.5.2. The total plant manpower requirement is for three construction inspectors, sixteen management and technical persons, an administrative and supervisory staff of fourteen and seventy-four operating, maintenance and laboratory technicians, all of which are employees of the operating contractor. A contract maintenance staff of fifty-one and a contract security/janitorial staff of eleven is provided. The operating contractor will also provide home office support of seven man-years in various skills.

2.5 Capital Cost

The estimated capital cost for the 25 TPD pilot plant is \$52,311,000 expressed in 1979 dollars excluding fee and escalation. An allowance of \$1,000,000 should be added to this cost to cover land acquisition, permits, taxes, capital equipment spares and the like. A composite contingency factor was developed, based on evaluations of selected quotations and quantities. The contingency factor of 13 percent, is included in the estimate.

The duration of the project, from start of detailed engineering to the point of commissioning the plant is 32 months. The item controlling this project duration is the aluminum chloride decomposer/calcliner.

2.6 Comparative Costs for Commercial Plant

The Task 2 report issued earlier under this contract included capital and operating cost estimates for a 500,000 TPY alumina plant based on the Clay/Hydrochloric Extraction - HCl Gas Induced Crystallization process. Several revisions have been made to the process to improve product quality based on recent information developed by the Bureau of Mines and these revisions are incorporated in the process design for the 25 TPD pilot plant.

The capital and operating costs for a 500,000 TPY plant including these revisions are presented in Section 6.0 of this report.

As a result the capital and operating costs for a 500,000 TPY alumina plant have been increased by \$18.5 million and \$12.07 per ton of alumina respectively. It may be possible to reduce the additional operating costs from \$12.07 per ton of aluminum to as low as \$4.92 per ton of alumina by recovering additional waste heat from the process. A discussion of potential waste heat recovery methods are discussed in Section 6.0.

2.7 Process Development Recommendations

A considerable amount of development work has been done to provide the basic design for this process. However, as with any process at this

stage of development, there remain significant opportunities for optimization of the process and reduction in process costs.

Further study is recommended on improvement of the settling and filtration characteristics of the leach residue, the fundamental chemistry of the crystallization process, the decomposition of aluminum chloride hexahydrate, and the mechanism of the bleed stream treatment process.

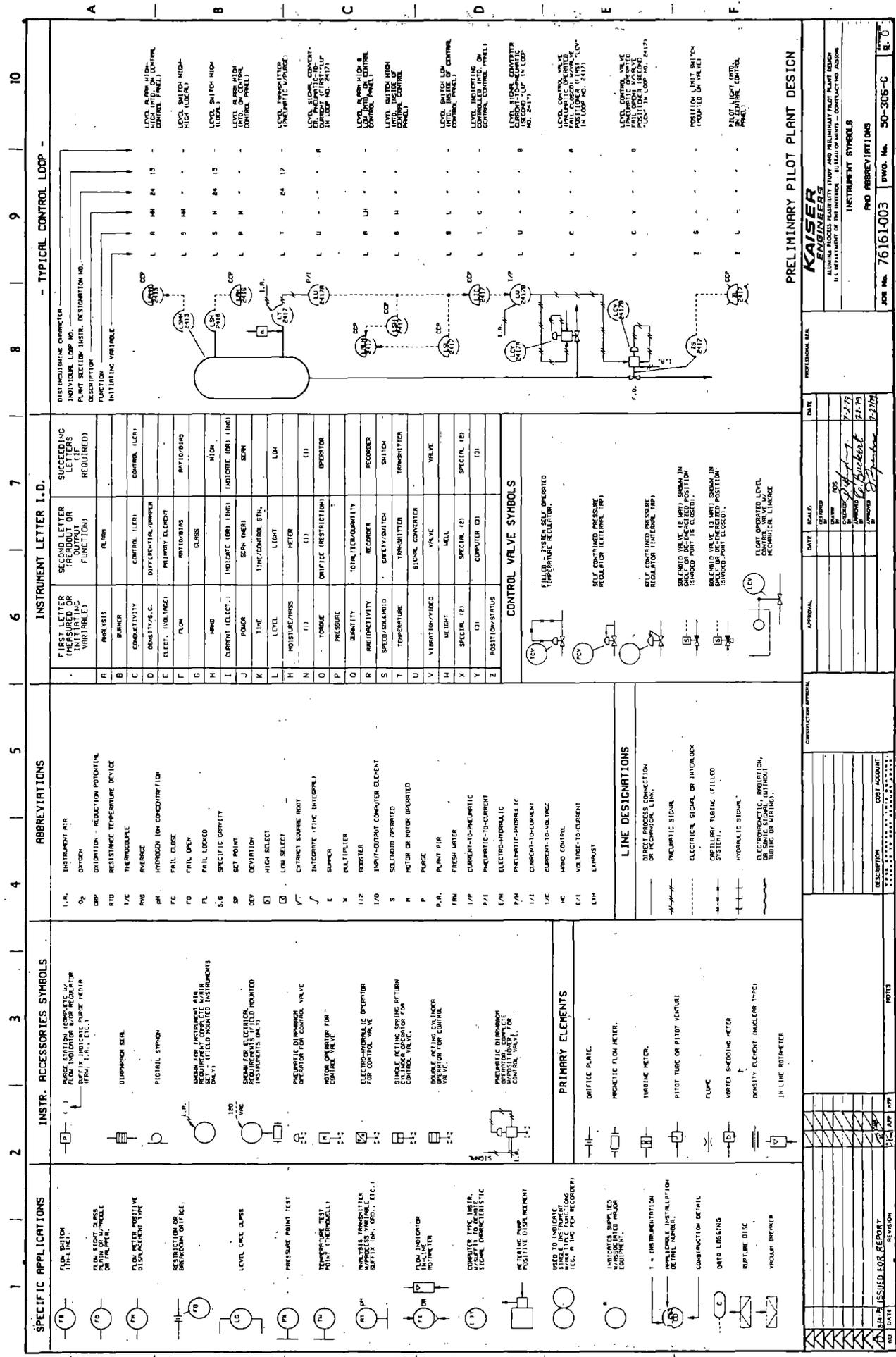
3.0 PROCESS DESCRIPTIONS

3.1 Process Flow and Material Balance for Pilot Plant

Block Flow Diagram - Drawing No. 50-302-G

Material Balance - Drawing No. 50-303-G

This section of the report addresses process descriptions of the pilot plant and the process flow and material balances. For ease of reference, smaller scale copies of the process flow diagrams have been included with the respective process descriptions.



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SPECIFIC APPLICATIONS	INSTR. ACCESSORIES SYMBOLS	ABBREVIATIONS	INSTRUMENT LETTER I.D.	ABBREVIATIONS	INSTRUMENT LETTER I.D.	ABBREVIATIONS	ABBREVIATIONS	ABBREVIATIONS	ABBREVIATIONS	
<p>1. FLOW SWITCH</p> <p>2. FLOW METER</p> <p>3. FLOW METER POSITIVE</p> <p>4. FLOW METER POSITIVE WITH ORANGE</p> <p>5. FLOW METER POSITIVE WITH RED</p> <p>6. FLOW METER POSITIVE WITH GREEN</p> <p>7. FLOW METER POSITIVE WITH BLUE</p> <p>8. FLOW METER POSITIVE WITH PURPLE</p> <p>9. FLOW METER POSITIVE WITH BROWN</p> <p>10. FLOW METER POSITIVE WITH BLACK</p> <p>11. FLOW METER POSITIVE WITH WHITE</p> <p>12. FLOW METER POSITIVE WITH GREY</p> <p>13. FLOW METER POSITIVE WITH SILVER</p> <p>14. FLOW METER POSITIVE WITH GOLD</p> <p>15. FLOW METER POSITIVE WITH BRASS</p> <p>16. FLOW METER POSITIVE WITH COPPER</p> <p>17. FLOW METER POSITIVE WITH ZINC</p> <p>18. FLOW METER POSITIVE WITH ALUMINUM</p> <p>19. FLOW METER POSITIVE WITH MAGNESIUM</p> <p>20. FLOW METER POSITIVE WITH CALCIUM</p> <p>21. FLOW METER POSITIVE WITH SODIUM</p> <p>22. FLOW METER POSITIVE WITH POTASSIUM</p> <p>23. FLOW METER POSITIVE WITH AMMONIUM</p> <p>24. FLOW METER POSITIVE WITH NITRATES</p> <p>25. 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PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
 ALUMINA PROCESS FEASIBILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN
 U. S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONSTRUCTION DIVISION

INSTRUMENT SYMBOLS AND ABBREVIATIONS

JOB No. 76161-003 DWG. No. 50-305-C R. 0

APPROVAL	DATE	SCALE	REVISION
DESIGNED BY	7/2/77	AS SHOWN	
CHECKED BY	7/2/77		
APPROVED BY	7/2/77		
DATE	7/2/77		

26

3.2 Process Description for Individual Process Areas

3.2.1 Clay Preparation and Calcination - Areas 1 & 2 - Drawing No. 1/2-100-M

3.2.1.1 Process Description - Clay Preparation

Summary

The clay preparation section of the demonstration plant is designed to receive, store and size clay for calcination. It is designed to operate 8 hours a day and 5 days per week.

The clay is received, sized and conveyed to the raw clay and coal storage building where 1,400 tons of it is stockpiled. The clay is reclaimed, and is conveyed to the secondary crusher for final sizing prior to being fed to the clay calciner. The sized clay is screened and the oversized clay is returned to the secondary crusher. The undersized clay is pelletized and transported back to the screens. 700 tons of clay feed to the calciner is stored in a bin.

This section is also designed to receive, convey and store coal for use as the main source of fuel of the clay calciner. The coal is received and conveyed to the open area of the raw clay and coal storage building, where 100 tons of coal can be stockpiled. Reclaimed coal is transported to the 15 ton capacity coal day bin.

The equipment in this section includes:

- hoppers and belt conveyors
- screeners
- a clay and coal storage building
- crushers
- a pelletizer
- bucket elevators
- a tramp iron magnet and an automatic sampler
- dust control equipment
- storage bins
- weigh feeders

Raw Clay Receiving and Storage

Run of mine clay, less than 12 inches in size, is received in a 15 ton hopper from 10 ton trucks. A ramp is provided to allow trucks to dump directly to the hopper. Beneath the hopper, a 30 tons per hour vibrating grizzly allows minus 2 inch clay to pass through directly to the 24 inch wide rubber belt conveyor. Plus 2 inch size clay on the grizzly drops into a double roll primary clay crusher with a capacity of 30 tph.

The minus 2 inch clay from the crusher drops into a hooded 30 tph capacity belt conveyor. The raw clay and coal distributing conveyor has a walkway and is equipped with a tramp iron magnet and six manually operated plows. The plows allow clay to be stored in one of two sections provided for clay storage.

The raw clay and coal storage building is divided into three sections. Two sections, each with a storage capacity of 700 tons, are provided to store two types of clay. This area is totally enclosed except for conveyor openings. A baghouse dust collection system with a design air volume of 25,800 CFM, is provided to collect dust from the crusher system, the conveyor and the storage building. The dust collector is equipped with a fan, a screw conveyor and a rotary seal valve. Collected clay dust is returned to the stockpile. Capacity of the clay storage enables the calciner to operate at normal capacity for 15 days. This system operates 5 days per week and 8 hours per day.

Coal Receiving and Storage

Coal less than 1 inch in size is also received in the truck dump hopper from coal dump trucks. The vibrating grizzly allows the coal to pass through to the belt conveyor. The coal is conveyed and stored in the third section of the storage building. Storage capacity is 100 tons enabling the calciner to operate for 8 days. Coal is reclaimed with a front-end loader and transported to the raw clay surge bin bucket elevator and into the coal day bin. The coal day bin discharges to a weigh feeder which meters the flow of coal to the air swept pulverizer. Filling of the coal day bin is done once a day 5 days a week.

Raw Clay Sizing

Raw clay is reclaimed from the storage building by a front-end loader, dumped into the 20 ton raw clay reclaim hopper and conveyed out by the raw clay transfer conveyor. This conveyor is covered, 24 inches wide and has a capacity of 21 tph. Clay is transferred to the secondary crusher feed belt conveyor which is also covered, 24 inches wide and has a capacity of 21 tph. The clay is fed into the 40 tph hammermill secondary crusher. The secondary crusher reduces the clay from minus 2 inches to a 4 mesh x 0 product. Product clay drops into a covered, 40 tph, 24 inch wide, secondary crusher discharge belt conveyor, and is lifted by the clay screens bucket elevator, which has a capacity of 40 tph. The clay is then screened in a double decked 6 ft x 16 ft spring mounted, covered screen. Oversized clay is chuted back to the secondary crusher and the undersized clay is chuted into the pelletizer or allowed to pass along with the sized clay. The sized clay is chuted into a hopper, and drops into a 2 ft x 6 ft long 50 tph vibrating feeder. Sized clay is then lifted to a 700 ton capacity raw clay surge bin by a bucket elevator which has a capacity of 30 tons per hour. The discharge chute of the bucket elevator is equipped with a diverter gate to permit discharge into the coal day bin when the system is being used to transfer coal. The raw clay surge bin is provided with a vibrator to prevent bridging at the bottom discharge. The clay is metered by a 2 to 8 tph

weigh feeder. After transfer to a covered, 24 inch wide, 10 tph belt conveyor, the clay is dropped into a chute, sampled, and then raised by the 10 tph raw clay bucket elevator into the calciner. Feed into the calciner is normally 10,545 lbs/hr. The raw clay surge bin has a 7 day capacity. This is in addition to the 15 days capacity of the raw clay storage building.

This section is designed to operate 8 hours a day, 5 days per week. The raw clay weigh feeder, the automatic sampler and the raw clay bucket elevator will operate 24 hours per day, 5 days per week.

Undersized clay is fed into a pelletizing system as required. The pelletizer is a 12 ft. diameter disc-type with a 15 tph capacity. The pelletizer will agglomerate the clay with a water spray and is plowed to produce a minus 4 mesh plus 20 mesh product from a minus 20 mesh feed. A suitable binder material may be added at this point to facilitate agglomeration. The product overflows from the covered pan to the discharge chute and into the covered, 21 tph, 24 inch wide pelletizer belt conveyor. The pellets are fed directly to the calciner.

A baghouse dust collection system with a design air volume of 11,150 CFM is provided to collect dust from the raw clay transfer conveyor to the fines pelletizer. This dust collector is equipped with a fan and rotary seal valve that discharges dust into the pelletizer. Another baghouse dust collection system with a design air volume of 8,400 CFM is provided to collect dust from the raw clay surge bin bucket elevator. This dust collector is equipped with a fan and rotary seal valve that discharges dust into the raw clay weigh feeder.

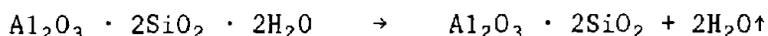
A cascading electrical control system is provided to shut down equipment downstream of any equipment that has a failure. The cascading shutdown system is provided from the raw clay surge bin bucket elevator downstream to the raw clay transfer conveyor. The raw clay bucket elevator will be interlocked with the clay calciner and a cascading shutdown system is provided from the raw clay bucket elevator downstream to the weigh feeder.

3.2.1.2 Process Description - Clay Calcination

Summary

The clay calcination section of the demonstration plant is designed to receive wet raw clay from storage, meter, dry, calcine, cool, transport and store clay at a production rate of 7400 lb/hr of calcined clay. Major design objectives include minimum particle attrition, high fuel efficiency and lot-to-lot segregation of clays.

The hydrochloric acid leaching process for the recovery of alumina from clay requires calcination of the clay for 0.1 to 2 hours in the temperature range 1,200-1,500°F in order to render the clay suitably reactive to the leaching acid. The reaction is:



$$\Delta H = 624 \text{ Btu/lb Al}_2\text{O}_3$$

Each clay particle being calcined must reach temperature within the above limits in order for the alumina to be rendered acid soluble, but conversion of alumina to the acid soluble form is very rapid once the specified temperature range is reached. The required residence time in calcination is therefore largely determined by the size of particles calcined and by the rate of heat transfer to individual particles. Calcined clay particles may be held within the specified temperature range for reasonable periods of time without loss of alumina reactivity, but heating them above this range will cause rapid deactivation of the alumina. Calcination also removes free and combined water and destroys any organic materials which may be present in the clay as mined.

When coal is used for calcination, small particles of ash which may remain in the calcine are not expected to interfere with subsequent processing, because this processing must, in any case, provide for separation of impurities that might be introduced through the leaching of ash and because the particles of ash are expected to be rendered largely inert by the high temperature they will selectively attain during combustion.

The design of the fluid bed dryer/calciner is based on feasibility tests conducted by an equipment vendor for the Bureau of Mines. Fluid bed calcination will offer improved heat utilization versus rotary kilns and the tests indicate an acceptably low level of product attrition.

Equipment Description

The clay calcination section is designed to operate continuously 24 hr/day, 5 days/week. Raw wet clay is received from storage at a nominal rate of 10,545 lb/hr. The clay feed rate is controlled by a weigh feeder with $\pm 2\%$ accuracy and an operating range of 2,500 lb/hr to 12,000 lb/hr. After weighing, the wet clay is conveyed to a bucket elevator for lift to the dryer-calciner feed port.

The calcination of the clay is effected in a pulverized coal or fuel oil fired, three stage fluidized bed reactor. The top bed receives and dries the wet clay at approximately 250°F. Supplemental heat can be added to this bed to assure drying temperatures are maintained. Dried clay transfers to the middle bed of the unit where a calcination temperature of approximately 1400°F is maintained by the addition of either coal or oil. The residence time in this bed is nominally one hour at design capacity. Heat recovery is accomplished by cooling the clay in the third (bottom) bed with incoming combustion air. The clay leaves the calciner unit at approximately 900°F and is transferred to a fluid bed aftercooler. A water spray and air cools the clay to 150°F. The clay calcination unit is equipped with cyclones and a baghouse for dust control and an SO₂ scrubber for sulfur dioxide emission suppression.

The clay calciner is designed to handle both coarse (85% - 4 mesh and 97% + 20 mesh) and fine (95% - 20 mesh and 90% + 200 mesh) clay. This will permit preparation of various feed materials to test particle size and attrition effects in leach and mud separation.

Calcined clay leaving the calciner system is transferred to storage by two pneumatic transport lines. The system is designed to separate the fine fraction of clay collected in the product cooler cyclone and bag filter from the coarse fraction, if so desired. This permits use of one or more of the following modes of operation:

- recycling fines to the disc pelletizer
- disposal of fines with leaching of "fines-free" clay
- FeCl_3 treatment with fines only
- leaching of total clay including all fines.

Two 300 T silos are provided for calcined clay storage. The silos have enough capacity for approximately seven days' operation at design rate. Two silos are provided in order to permit calcination of 1 lot of clay while a second (and different) lot of clay is being leached. A baghouse and exhaust fan is provided for the calcined clay silo to control dust emissions.

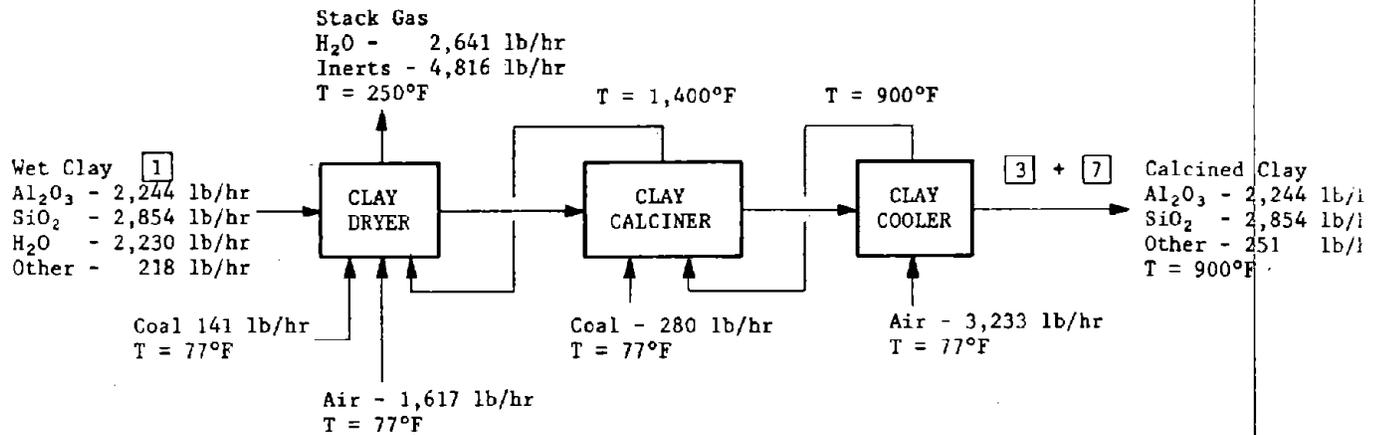
Clay is discharged from the bottom of each of two (2) calcined clay storage silos by clay transfer belt conveyor which intermittently moves approximately 20 tph of clay to a bucket elevator. The bucket elevator raises the clay to dump into the top of a nominal 32 T capacity (10 ft x 10 ft straight side with 60° cone) calcine clay feed bin which is located over the leach reactors. Clay transfer from the storage silos to the feed bin is normally done once every 8 hour shift.

The calcine feed bin is equipped with a baghouse dust collector and exhaust fan which will keep slight negative pressure on the bin and elevator. A vibrator is installed in the bin interlocked with the weigh feeder, to prevent bridging of the calcine. A slide valve is installed in the bin cone outlet to permit positive shutoff of clay flow for downstream maintenance.

**CLAY CALCINATION
HEAT AND MATERIAL BALANCE**

3.2.1.3

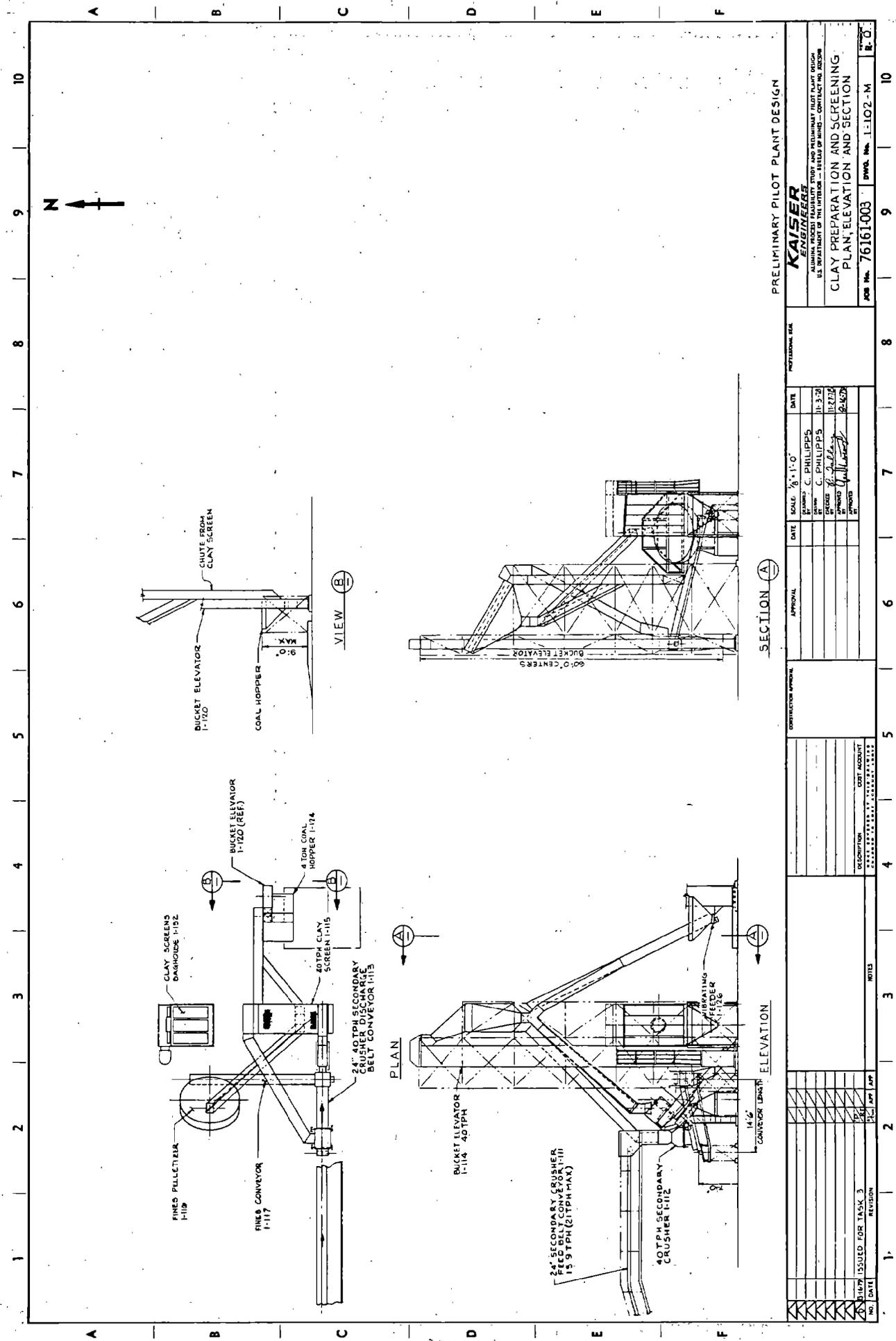
<u>HEAT IN</u>		<u>HEAT OUT</u>	
<u>Stream</u>	<u>Btu/hr</u>	<u>Stream</u>	<u>Btu/hr</u>
<u>1</u> Wet Clay	-0-	Calcination Reaction	1,400,000
Combustion Air	-0-	Water Vaporization	2,260,000
Coal, 11,600 Btu/lb	4,880,000	Stack Gas Sensible Heat	430,000
		<u>3</u> + <u>7</u> Calcined Clay	390,000
		Radiation Loss	400,000
Total	4,880,000	Total	4,880,000



Base Temp = 77°F

Note: Stream numbers where shown correspond to stream numbers on Dwg 50-302-G, Block Flow Diagram

The mass flow rates shown are based on 24 hr/day 7 day/wk operation.



10 9 8 7 6 5 4 3 2 1



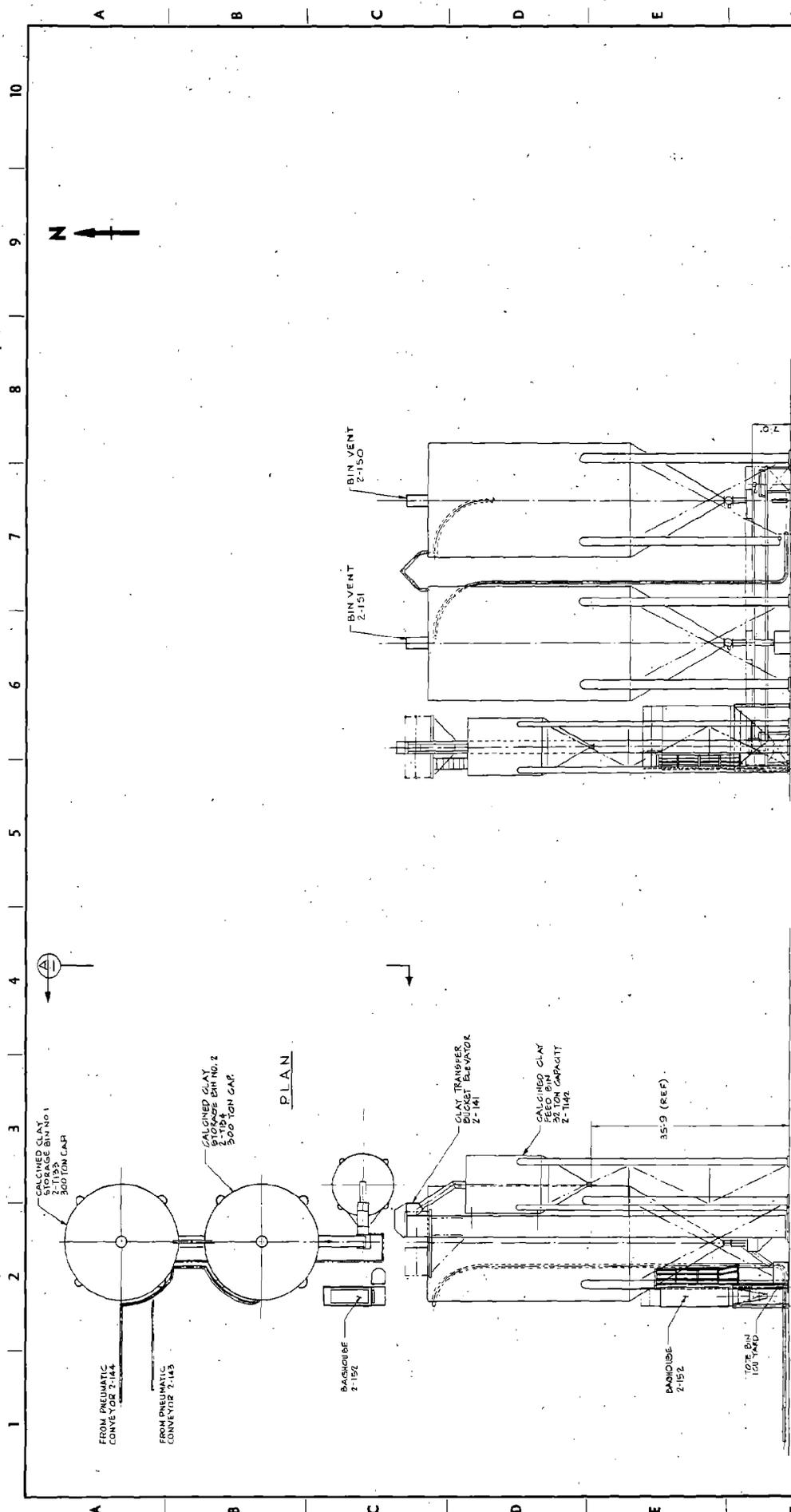
PRELIMINARY PILOT PLANT DESIGN

KAISER ENERGY SERVICES	
ALUMINA REFINERY FEASIBILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN CLAY PREPARATION AND SCREENING	
U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. ROOM	PLAN, ELEVATION AND SECTION
JOB No. 76161-003	DRAW. No. 1-102-M
	8.0

DATE	SCALE	DATE	DATE
APPROVAL	1/2" = 1'-0"	DATE	DATE
CONTRACTOR APPROVAL	BY: C. PHILIPPS	DATE	DATE
	BY: C. PHILIPPS	DATE	DATE
	BY: C. PHILIPPS	DATE	DATE
	BY: C. PHILIPPS	DATE	DATE
	BY: C. PHILIPPS	DATE	DATE
	BY: C. PHILIPPS	DATE	DATE

NO.	DATE	DESCRIPTION
1	11/27/72	ISSUED FOR TASK 3
2	12/1/72	REVISION
3	12/1/72	REVISION
4	12/1/72	REVISION
5	12/1/72	REVISION
6	12/1/72	REVISION
7	12/1/72	REVISION
8	12/1/72	REVISION
9	12/1/72	REVISION
10	12/1/72	REVISION

NO. 1	DATE	DESCRIPTION	NOTES
2			
3			
4			
5			
6			
7			
8			
9			
10			



ELEVATION

SECTION A

NO.	DATE	REVISION	ISSUED FOR	APP.	DATE	DESCRIPTION	COST ACCOUNT
1			ISSUED FOR L.A.S. 3				
2							
3							
4							
5							
6							
7							
8							
9							
10							

PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
 1100 P.O. BOX 10000
 ALBUQUERQUE, NEW MEXICO 87110
 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 45309

CALCINED CLAY STORAGE AND FEED BIN
 PLAN ELEVATION AND SECTION

JOB No. 76161-003 DWG. No. 2-103-M

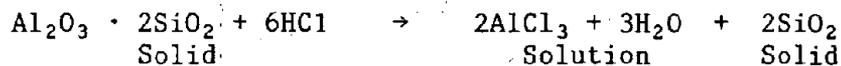
3.2.2 Leaching - Area 3-Dwg. No. 3-100-P

3.2.2.1 Process Description

Summary

The clay leaching section of the demonstration plant is designed to extract aluminum from calcined kaolin clay by hydrochloric acid. Hydrochloric acid and calcined clay are metered into leach vessels wherein alumina is dissolved as aluminum chloride by the acid. Additional elements such as iron, magnesium, sodium, etc., are also dissolved. An insoluble fraction of the kaolin, primarily silica, remains as solid. The extraction is exothermic and a boiling slurry results (approximately 220°F). After extraction has been completed (nominally 2 hours), the slurry is flashed cooled to approximately 140°F under vacuum. The cooled slurry is then pumped to the mud separation and washing section of the plant.

The primary leaching reaction is:



$$\Delta \quad H = 1446 \text{ Btu/lb Al}_2\text{O}_3$$

Other impurities go into solution to a greater or lesser extent.

Major design objectives include high aluminum extraction efficiency, low hydrogen chloride loss, and minimum fines (slime) generation. Equipment included in this section are:

- feed acid pumps and acid preheater
- calcined clay screw conveyor elevators and feed hopper, clay weigh clay
- agitated leach vessels, reflux condenser and defoamer addition system and,
- vacuum flash tank with condensers, steam jets and transfer pumps.

Calcined Clay Feed System

The calcined clay feed system is designed to transfer and meter 5,237 lbs/hr of calcined clay from the storage bins to the first of three leach reactors. Clay feed to the leach reactor is adjustable from 1,000 to 6,000 lbs/hr. Clay feed is controlled by an automatic continuous weigh feeder with a 1% accuracy at full speed. The clay from the weigh feeder discharges into a seal screw conveyor which conveys the clay into the leach reactor and provides a fume seal to prevent HCl vapors from venting into the atmosphere. A double rotary valve is provided to assure a positive seal.

Leach Acid Feed

Leach acid consisting of 24.8% HCl, 7.4% ACH and a small amount (.2%) of other salts is metered at 34.4 gpm from leach acid storage into the leach reactors. Two lined centrifugal pumps with a capacity of 50 gpm each supply the leach acid to the reactors. The pumps have double mechanical seals with water purge. Flow is controlled by a magnetic flow meter with tantalum probes. Normally acid flow is set to 5% above stoichiometric requirements based on alumina in the kaolin.

For startup only, an impervious graphite heater is installed to preheat either the feed acid or recycled leach liquor from the thickener overflow pumps in mud separation (Area 4). The heater will use approximately 2,068 lbs/hr of 75 psig steam when in service and will have a heat duty of 1,850,000 Btu/hr when heating a 100°F stream at 35 gpm.

Leach Reactors

Alumina is leached out of the calcined clay by hydrochloric acid in three agitated leach reactors operating in series. The reactors are made from Haveg 41 or equal and each has a contained operating volume of 1,865 gallons. This provides approximately 2 hours residence time. Liquid overflow height is 6.5 ft on the straight side. The reactors are totally closed and are equipped with agitators capable of providing gentle agitation sufficient to maintain the solids in motion but not to cause unnecessary particle attrition. The agitator motor and drive are designed to startup when the paddle blades are covered by settled solids after shutdowns. The agitator is equipped with a variable speed drive.

Feed entry into each reactor vessel is by dip leg extending at least 3 feet below the liquid level to minimize bypassing. The reactors, agitators and internals are constructed of fiberglass reinforced Haveg 41. The reactors are protected from external damage.

The heat of the leaching reaction causes the reaction mass temperature to rise to the boiling point of approximately 220°F and causes boil-off of substantial HCl and water vapor. The boil-off gases from the three reactors are ducted to a single impervious graphite reflux condenser. The heat duty of the condenser is 995,000 Btu/hr and condenses approximately 1,071 lbs/hr of 18.9% HCl vapors. The condenser requires approximately 80 gpm of 95°F cooling tower water. The condenser is vented to a fume scrubber system.

A 10 gallon steel pressure tank is provided for defoamer storage. The defoamer solution is piped to each reactor and remotely activated manual solenoid valves are provided to add defoamer during "foaming" conditions. Defoaming is provided to prevent solids entering the condenser system and fouling or plugging it.

Each reactor is provided with level, temperature, and pressure indication and alarm. The third reactor level is controlled by a control valve feeding the flash cooler.

The slurry is pumped from the reactors to a flash tank at 38.9 gpm by two (2) lined centrifugal slurry pumps with a nominal 50 gpm capacity. The pumps have a double mechanical seal.

Flash Cooling System

The hot leach slurry is pumped at 24,451 lbs/hr and 220°F to a flash tank where it is flash cooled at approximately 56 mm.Hg (abs) to 140°F. The 5.5' diameter with 60° cone flash tank is constructed of Haveg 41. Approximately 1,561 lbs/hr of vapor containing 7.3% HCl are flashed off to effect cooling. This cooling is required to protect downstream FRP or rubber lined equipment against excessive temperature.

The flashing vapors are ducted to an impervious graphite tube-in-shell water cooled condenser to condense essentially all of the H₂O/HCl stream at approximately 105°F. This condenser requires water 85°F or cooler. The heat duty of the condenser is 1,511,000 Btu/hr and 300 gpm of 85°F water is required. Pressure drop through the condenser for the vapor must be small. The condensed 7.3% HCl solution flows at 3 gpm by gravity to a 50 gal Haveg 41 surge pot and then is pumped to the acid recovery area (Area 10).

The non-condensable portion of the HCl/H₂O stream passes to a steam jet eductor for compression to 1 atm and then to a water cooled barometric condenser. The effluent from the barometric condenser discharges to an FRP lined hot well and then is pumped to the cooling tower.

The cooled leach slurry at 140°F is pumped from the flash tank to solid/liquid separation by identical pumps to the hot slurry pumps at 35.3 gpm.

In order to minimize vacuum sealing and pumping problems, the flash tank, HCl/H₂O condenser and barometric condenser is mounted 35 feet above grade to provide a barometric leg.

Level indicating and control instrumentation are provided for the leach slurry in the flash tank and the condensed HCl from the condenser surge pot. Temperature and pressure indication are required on all streams as well as a high temperature alarm on the cooled leach slurry stream.

SUPPLEMENTAL
MATERIAL BALANCE
AREA 3

PROCESS STREAM	3A	3B	3C*						
Al ₂ O ₃	---	---	---						
AlCl ₃ .6H ₂ O	---	---	19293						
FeCl ₃	---	---	168						
Fe ₂ O ₃	---	---	---						
SiO ₂	---	---	---						
HCl	202	114	189						
H ₂ O (FREE)	869	1447	20826						
OTHER	---	---	118						
ORGANIC	---	---	---						
Cl ₂		---	---						
GAS (NON-CONDENSIBLE)	---	---	---						
FUEL	---	---	---						
TOTAL	1071	1561	40594						
TEMPERATURE °F	---	140	80						
DENSITY, lb/gal (lb/ft ³)	---		11.2						
FLOW RATE, GPM	---	(508)	60						

* Intermittent

3.2.3 Mud Separation and Washing - Area 4 - Dwg No. 4-100-P

3.2.3.1 Process Description

Summary

The mud separation and washing section of the pilot plant is designed to receive cooled AlCl_3 leach slurry containing 12.7% solids from the leaching area and remove the spent solids, primarily silica, from the solution in a thickener. The clarified AlCl_3 solution overflows from the thickener and is pumped to the filtration section. The solids are removed as a 20% solids underflow and pumped to a six washer counter-current decantation (CCD) circuit and a horizontal belt filter operating in series. The cake from the filter at 50% solids is sent to mud disposal. Hot wash water is used to wash the filter cake. The mud washing system is designed to recover 99.5% of the AlCl_3 in the leach slurry.

A secondary purpose of this section is to receive 2.9 gpm of AlCl_3 containing slurry from the iron removal section and recover the AlCl_3 values while rejecting the spent solids to waste disposal. This slurry is fed to the 6th washer in the CCD train.

The mud separation and wash section includes the equipment required to thicken and wash by CCD and filtration the spent solids while clarifying the AlCl_3 solution prior to filtration. Major equipment items include:

- a thickener with pumps and flocculant addition
- six washers with pumps and flocculant addition
- a 150 ft² horizontal belt filter with all auxiliaries

Design of this section has been based on data collected at the USBM Boulder City mini-plant.

Leach Slurry Thickener

The leach slurry thickener is designed to receive 35.3 gpm of 12.7% solids slurry from the leaching section and 27.5 gpm of wash liquor from the CCD washers. The total feed contains 3,096 lbs/hr of solids. Based on tests at the Bureau of Mines, the thickener is sized for 3.25 ft²/TPD of solids which is equivalent to a total area of 121 ft² or 12.5 ft diameter tank. A standard unit of 14 ft diameter is provided. The thickener is fully enclosed and all internals and wetted parts are rubber-lined. The thickener cone bottom slope is 2 in/ft. The rake drive is of adequate power to startup with 30% solids filling the thickener. The thickener design allows for handling solids which are 80% + 35 mesh and 20% - 200 mesh.

Thickener underflow pumps are capable of handling up to a 30% solids slurry. Two centrifugal pumps (1 operating and 1 spare) are used. One centrifugal thickener overflow pump is provided.

A 1% solution of Dow Chemical Separan MGL flocculant is added to the thickener to flocculate the fines. Normal addition rates of 0.5 lbs of Separan MGL per ton of solid in the slurry are planned. A 100 gallon mix tank is provided to prepare 1% stock solutions of Separan. Details of mix tank design are furnished in Dow form 192-404-75 titled "Separan Polymers Settle Process Problems". Stock solutions are transferred to a 950 gallon feed tank which supplies flocculant solution to the flocculant metering pumps. The flocculant metering pumps are positive displacement with variable speed control. The normal feed rate is 10.4 gallons per hour (.17 gpm) of 1% stock solution. The stock solution is metered into the overflow stream returning from the first washer overflow. An in-line mixer is provided to assure the flocculant is well mixed with the slurry feed to the thickener.

Countercurrent Decantation Washers

The underflow from the mud thickener is first washed in a CCD washer train containing six washers each identical to the thickener described in the above section. The washers are operated in series and remove 86% of the soluble salts from the mud slurry. Each washer receives 22.5 gpm of 20% solids slurry from the underflow of the preceding washer and 27.5 gpm of overflow from the following washer.

An 11% solids slurry is fed to the sixth washer at 2.9 gpm. This slurry is the effluent from the iron removal section of the plant and is represented by stream 16 of the material balance.

Horizontal Belt Filter

The underflow from the sixth washer, containing 20% - 22% solids is pumped to the horizontal belt filter. The filter has an effective area of 150 ft². The filter is constructed of rubber-lined, FRP, and titanium parts where exposure to the cake or liquor is possible. The filter belt is encased in an FRP hood which is vented to a scrubber system to contain HCl fumes. The filter has 2 wash stages each receiving 6.7 gpm of the hot water. Polypropylene filter cloth is used and back wash is provided to clean the cloth. The filter has 2 receivers and filtrate pumps and a vacuum pump. Controls are mounted in a vapor-tight cabinet and the entire unit is platform mounted. The system was sized based on tests conducted by the Bureau of Mines.

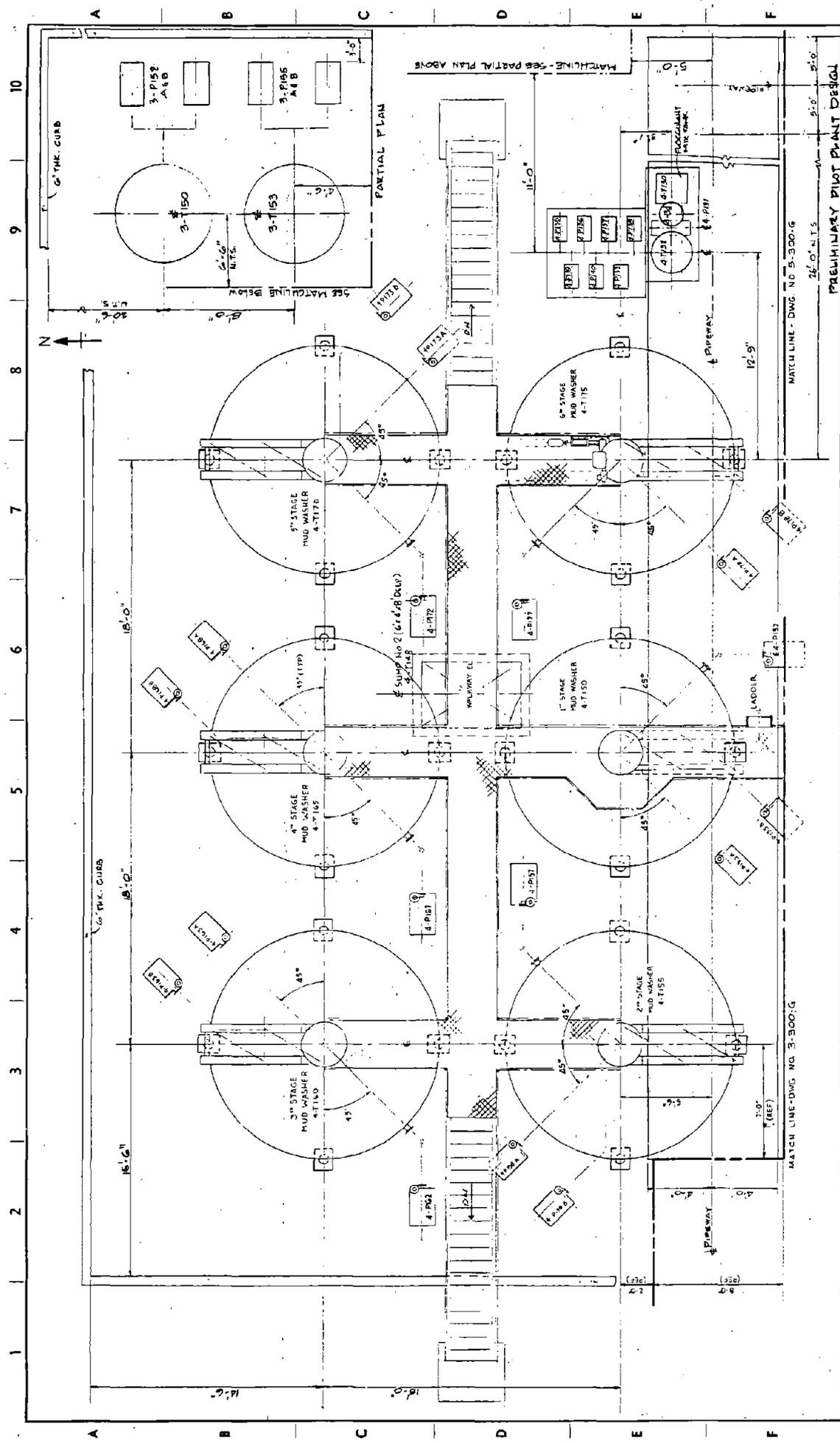
Final Cake Disposal

The belt filter will discharge 6,410 lbs/hr of wet cake containing 50% solids, 35 lbs/hr AlCl₃ and less than 1 lb/hr of HCl. This cake is dumped into a bin for transport to the solids impoundment pond or can be immediately repulped for pumping to the drainage test pond.

SUPPLEMENTAL
MATERIAL BALANCE
AREA 4

PROCESS STREAM	4A	4B							
Al ₂ O ₃	125	---							
AlCl ₃ ·6H ₂ O	620	588							
FeCl ₃	10	9							
Fe ₂ O ₃	52	---							
SiO ₂	2854	---							
HCl	10	9							
H ₂ O (FREE)	12173	15413							
OTHER	180	6							
ORGANIC	1	0							
Cl ₂	---	---							
GAS (NON-CONDENSIBLE)	---	---							
FUEL	---	---							
TOTAL	16025	16025							
TEMPERATURE °F	140	140							
DENSITY, lb/gal (lb/ft ³)	11.4	8.0							
FLOW RATE, GPM (SCFM)	22.5	33.4							

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NO.	DATE	ISSUED FOR REPORT	REVISION
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APPROVAL	DATE	SCALE	3/8" = 1'-0"
DESIGNED BY: E.M. CASTILLO	1/17/79		
DRAWN BY: E.M. CASTILLO	1/17/79		
CHECKED BY: J. B. BENTLEY	1/23/79		
APPROVED BY: J. B. BENTLEY	1/23/79		

CONTRACTOR APPROVAL	DATE

PROFESSIONAL SEAL	DATE

KAISER ENGINEERS 1500 P STREET, SUITE 1000, WASHINGTON, D.C. 20004 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 34304	
AREA - 364 MUD SEPARATION AND WASHING GENERAL ARRANGEMENT - PLAN JOB NO. 76161003 DWG. NO. 4300-G R.D.	

3.2.4 Filtration - Area 5 - Drawing No. 5-100-P

3.2.4.1 Process Description

Summary

The filtration section of the demonstration plant is designed to clarify AlCl_3 solution coming from the mud thickener overflow in mud separation. The clarification of this solution containing 70 ppm solids is effected by passing it through a sand bed filter. The filtrate, containing approximately 5 ppm solids is then chlorinated to convert ferrous ion to ferric ion and sent to the solvent extraction section.

This area receives feed from the mud thickener overflow pump and delivers filtrate to the polished pregnant liquor head tank in solvent extraction. At required intervals 50 gpm of filtrate is pumped from the polished pregnant liquor storage tank which backwashes the filter bed and is then sent to the first mud washer vessel 4-T150.

Filtration

The filtration section is designed to filter 37.9 gpm of 47.5% aluminum chloride hexahydrate solution containing 70 ppm solids and 0.5% HCl. The solution is normally at 140°F with a density of 10.6 lbs/gallon. The contained solids are silica particles primarily between 1 and 5 microns in size. This silica can be flocculated with Separan MGL. The filtration is effected in a high rate downflow sand filter (upflow permissible upon manufacturer's recommendation) which will remove at least 90% of the particles contained in the feed. The filtrate will contain less than 6 ppm solids.

The filtration system consists of a skid-mounted, fully automatic, two (1 operating and 1 spare) 3' diameter x 5' filter units. A flocculant (polyelectrolyte) metering/piping system is provided to assist filter efficiency. The medium in the filter beds is silica and is not attacked by HCl or AlCl_3 . The wetted parts of the filter system are rubber lined, of FRP construction and of titanium construction.

The filtration system is equipped with controls and valving such that the cleaning cycle will initiate automatically upon differential pressure buildup and/or elapsed time. The cleaning of the filter bed is effected by backwashing with up to 50 gpm filtrate from the polished pregnant liquor storage tank. The backwash slurry is sent to the first mud washer feed well.

The filter beds are sized to pass 125% of normal flow for short periods of time. The cleaning cycles are of a short duration to prepare the bed for return to service within 30 minutes assuming a normal on-line time of approximately 4 hours.

Instrumentation is provided to measure stream temperature and pressure as well as bed differential pressure. Indicator lights are provided to indicate cleaning/filtration cycle status.

Chlorination

The anionic chloride complex FeCl_4^- , which is the extractable iron species, is formed only from ferric iron. Any ferrous iron present in the leach liquor is therefore oxidized prior to solvent extraction with Cl_2 gas by the reaction



to produce a $\text{Fe}^{+++}/\text{Fe}^{++}$ ratio of approximately 5000:1.

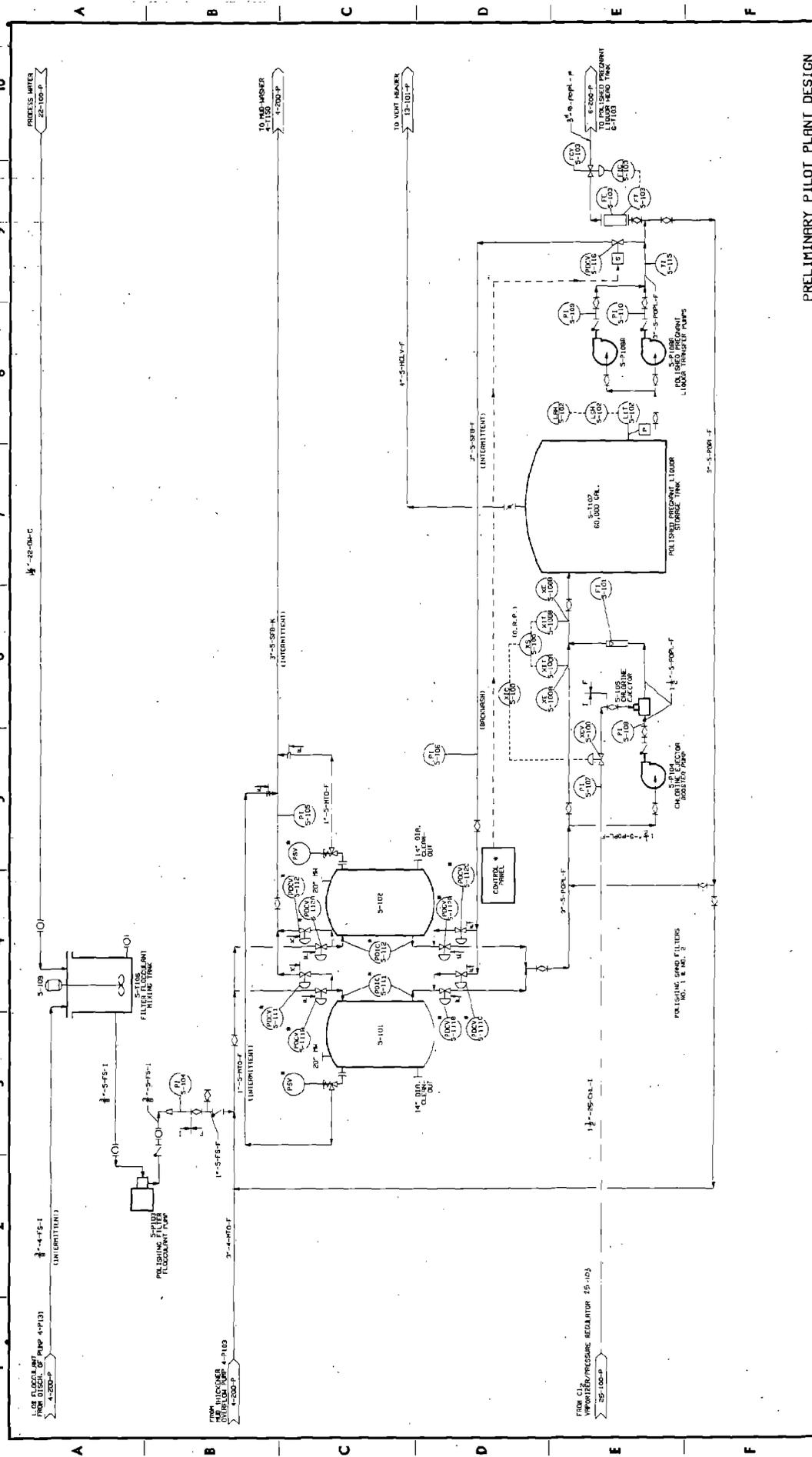
This reaction takes place rapidly at room temperature and is accomplished by direct injection of Cl_2 gas into the clarified liquor in a pipeline at a Reynolds number in the turbulent range with sufficient pipe length to provide a residence time of 5 seconds prior to discharge of the liquor into the polished pregnant liquor storage tank.

Liquid chlorine is received in 1 ton cylinders. The chlorine is filtered, vaporized, and pressure regulated in a commercial chlorinator injection system. It is fed into the polished pregnant liquor stream through an eductor. A booster pump is provided for the pregnant liquor to assure adequate flow and pressure drop through the eductor. Chlorine flow is controlled by the oxidation potential of the AlCl_3 solution leaving the eductor to assure complete conversion of ferrous ion to ferric ion.

A 60,000 gallon rubber-lined storage tank provides surge capacity between leaching/mud washing and the downstream process areas.

SUPPLEMENTAL
MATERIAL BALANCE
AREA 5

PROCESS STREAM	5A								
Al ₂ O ₃									
AlCl ₃ ·6H ₂ O	11459								
FeCl ₃	100								
Fe ₂ O ₃	---								
SiO ₂	---								
HCl	133								
H ₂ O (FREE)	12370								
OTHER	70								
ORGANIC	---								
Cl ₂	---								
GAS (NON-CONDENSIBLE)	---								
FUEL	---								
TOTAL	24132								
TEMPERATURE °F	140								
DENSITY, lb/gal (lb/ft ³)	10.6								
FLOW RATE, GPM (SCFM)	37.9								



PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
ALUMINA PROCESS FEASIBILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN
U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 33500

FILTRATION PIPING & INSTRUMENTATION DIAGRAM

JOB No. 76161003 DWG. No. 5-200-P

APPROVAL	DATE	SCALE	DATE
DESIGNER		AS SHOWN	
CHECKER		AS SHOWN	
APPROVER		AS SHOWN	
DATE	7/17/71		
BY	W. J. B. / J. P. B.		
DATE	7/17/71		
BY	W. J. B. / J. P. B.		

NO.	DATE	REVISION	NOTES
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3.2.5 Iron Removal - Area 6 - Drawing Nos. 6-100-P & 6-101-P

3.2.5.1 Process Description

Summary

The iron removal section includes a solvent extraction system designed to remove iron from the crystallization feed liquor. The process section in which iron is removed from clarified pregnant liquor includes:

- Removal of dissolved FeCl_3 from the polished pregnant liquor in 3 mixer-settler stages of solvent extraction.
- Storing the pregnant liquor from which iron has been removed (raffinate) in one or the other of two 60,000 gal tanks, each having the capacity to store one day's production, for the purpose of fully coalescing and final disengagement of any entrained organic solution.
- Stripping, in 12 stages of mixer-settlers, the iron from the loaded organic phase using 0.1% HCl.
- Adding any required makeup organic phase and recirculating the stripped organic to extraction.
- Storing, in one or the other of two 3,000 gal tanks having one day's capacity, the FeCl_3 extract to fully coalesce and disengage any entrained organic phase.
- Heating the FeCl_3 extract to 220°F in a heat exchanger.
- Reacting the hot FeCl_3 extract for 3 hours in a series of 3 stirred reactors with an amount of calcined clay containing alumina equivalent to 150% of the sum of the dissolved iron plus free HCl in the strip liquor.
- Sending the product slurry to the mud separation and washing section.

Design of this section has been based on information from the Bureau of Mines and selected vendors.

Equipment Description

Removal of Iron by Solvent Extraction

Three stages of countercurrent solvent extraction in mixer-settlers at an aqueous/organic ratio of approximately 4:1 are provided to reduce FeCl_3 to <.005% in the raffinate using Alamine 336 as the extractant. Oxidized liquor feed to solvent extraction may be at any temperature between ambient and about 140°F, although higher temperatures favor both the iron extraction and the phase separation. Cells are covered and vented.

The extractant is prepared by mixing together the following:

- 15 parts by volume Alamine 336, which is a water insoluble, symmetrical straight chain, saturated tertiary amine manufactured by General Mills Chemicals.
- 10 parts of commercial decyl alcohol comprising mixed isomers. The purpose of using the decyl alcohol is to prevent the formation of a second organic phase under certain conditions of iron loading.
- 75 parts of kerosene.
- Sufficient hydrochloric acid to provide HCl in excess of the amount required to convert all of the amine to the hydrochloride. (The average molecular weight of the tertiary amine is 392.)

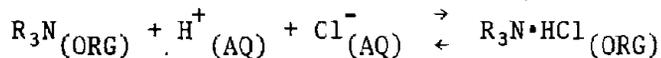
A 1,200 gallon capacity organic makeup mixing tank with an agitator is provided for organic makeup to the system. A batch mix of three 55-gallon drums of Alamine 336, two 55-gallon drums of decyl alcohol and fifteen 55-gallon drums of kerosene is made in this 6' dia. x 6' str. side tank.

The mixer-settler cells are designed to allow 2 minutes residence time for phase equilibration in the mixer and to provide 1 sq. foot per gallon per minute for phase separation. Proper phase separation is essential to successful operation of the iron removal section.

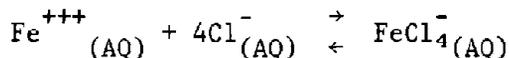
Organic and aqueous phases enter any given stage immediately below the center of the mixer turbine, which performs the dual functions of mixing the phases and providing the small hydraulic head required for flow of both phases through the stage. During startup the cells are successively filled with both phases and each mixer turbine started when an appropriate liquid level is reached in the mixing compartment. The operation may be shut down by stopping both liquid feeds and at the same time stopping the mixer turbines. Drain connections are provided to each cell for removing the total contents to the stripped organic surge tank. A drain connection is provided to drain the coalesced aqueous phase to a sump for recycle or disposal.

The length/width ratio of the settlers is 5.5:1, and phases discharge over weirs the full width of the settler. Provision is made in each mixer-settler for independently controllable internal recycle of each phase. The aqueous phase is recycled in Stage 1, from which the loaded organic leaves to make the aqueous phase continuous. Stage 2, and Stage 3, from which the purified pregnant liquor leaves, are operated with the organic phase continuous. This mode of operation is employed to maximize the phase separation, to minimize turbulence in the settler, and to minimize the amount of organic phase entrained in the purified pregnant liquor. This is because the entrained organic can destroy the polymeric lining intended for use on other items of equipment.

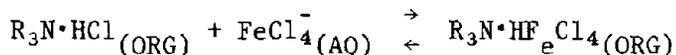
Alamine 336 reacts with hydrochloric acid as follows:



Ferric iron in the pregnant liquor in the presence of an excess of chloride ions forms an anionic chloride complex:



This complex reacts with the amine hydrochloride:



to form an iron-bearing addition compound which also is soluble in the organic phase. The second two reactions above can be reversed by contacting the iron-bearing addition compound with an aqueous phase containing only a small chloride concentration. This regenerates the extractant for reuse. Thus, it is seen that the driving force for iron removal is a change in chloride concentration. The kerosene serves as a carrier and diluent; decyl alcohol prevents the formation of a second organic phase in the presence of substantial organic iron loadings.

The control parameters in solvent extraction are the aqueous and organic flow rates, which are set at fixed values.

Storage of Purified Pregnant Liquor

It is essential that all entrained organic phase be removed from the purified liquor to prevent attack on the polymeric lining used on other items of process equipment and for other reasons. The process design calls for raffinate to be passed through a commercially available coalescing device and then delivered into the top of one or the other of two 21' dia. x 24' str. side FRP lined raffinate storage tanks, each providing one day's storage of liquor. Raffinate is withdrawn from the bottom of the tank and may be analyzed for both its organic and iron content before being pumped to evaporation. Any coalesced organic phase is drained from time to time off the surface of the liquid in the tank and returned to extraction. Raffinate, meeting specifications for iron and entrained organic is pumped to evaporation as required.

Stripping Loaded Organic

The chemical mechanism for stripping and regeneration of the organic extractant has been discussed above. A 0.1% solution of HCl is used as the stripping agent in order to prevent the precipitation of any basic compounds of iron, because the presence of solids of any kind would destroy the operability of the stripping operation. The stripping equilibrium curve given in USBM RI No. 8188, "Amine Extraction of Iron from Aluminum Chloride Leach Liquors", J. A. Eisele et al, 1976, shows that twelve stages of countercurrent stripping are required. This minimizes the amount of strip liquor to be subsequently treated and reintroduced into the process.

Recycle within the mixer-settler is employed so that the final cell producing regenerated organic is run aqueous continuous. All other stripping cells are operated organic continuous. The length/width ratio of the settlers is 4:1. The temperature of the strip acid and the loaded organic may vary between ambient and about 140°F, with lower temperatures making it somewhat easier to transfer the iron back into the aqueous phase. Higher temperatures facilitate separation of the phases.

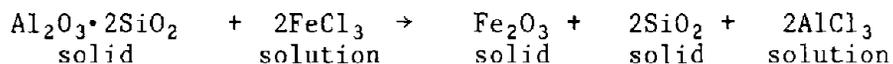
The control parameter is the same as in the case of extraction--operation with preselected and measured rates of loaded organic and strip acid.

Storage of the FeCl₃ Extract

The FeCl₃ extract is passed through a coalescer similar in design to that employed for the raffinate and then into one or the other of two 3,000 gal FRP tanks. These tanks each provide storage for about one day's production of FeCl₃ extract. FeCl₃ extract from which the organic phase has been fully removed by coalescence is withdrawn alternately from one tank or the other for further processing. Coalesced organic is withdrawn from the top of a filled tank as necessary and returned to stripping.

FeCl₃ Extract Treatment

The FeCl₃ extract is heated to 220°F in an impervious graphite heat exchanger and the FeCl₃ content of the extract reacts with a 150% excess of calcined clay to produce a solution of AlCl₃:



A total residence time of about 2 hours is provided in 3 cascaded stirred reactors having sufficient agitation to keep the solids in suspension.

Calcined clay in an amount containing alumina equivalent to approximately 150% of the FeCl₃ in the entering extract is added to the first reactor from a storage bin by means of a weigh feeder. Exact measurement of the calcined clay delivered is not critical. An operator, to control the conversion for a given constant flow rate of extract into the reactors and a predetermined corresponding addition rate of calcined clay addition, withdraws a sample of slurry from the third reactor and adds coagulant. A colorless supernatant liquor indicates satisfactory conversion. Small amounts of FeCl₃ which may not be converted to dissolved AlCl₃ are returned via waste solids washing to the process and will be reextracted.

Slurry from the third reactor is pumped to the 6th stage mud washer in the mud separation and washing area.

ADDENDUM TO 3.2.5.1 Iron Removal

Although the design of the pilot plant includes a solvent extraction system for the removal of iron from the crystallization feed liquor, there is indication that a process for the extraction of Al_2O_3 from clay via HCl extraction, which incorporates dissolving and recrystallization of the intermediate product $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (ACH), may be operated with or without iron removal by solvent extraction prior to the first crystallization. The separation of FeCl_3 achieved in two crystallizations is good enough so that ACH from the second crystallization may be decomposed to Al_2O_3 meeting the reduction-grade specification for Fe_2O_3 . If solvent extraction for iron removal is not used, acid-soluble iron in the entering raw material will be removed from the process in the bleed stream section of the pilot plant.

The advantages to using the solvent extraction system, as proposed for the pilot plant, for iron removal are:

- First crystallizer operation, although practical in the presence of FeCl_3 , is certain to be improved (and its cost lowered) by the prior removal of FeCl_3 , partly because the concentration of ACH entering crystallization can be greater.
- Bleed stream treatment is certain to be less costly in the absence of FeCl_3 .
- Available choices for materials of construction for the process will be widened; as one example, to include zirconium for the first crystallization and associated equipment.
- The process will have greater flexibility in regard to the economically acceptable $\text{Al}_2\text{O}_3:\text{Fe}_2\text{O}_3$ ratio in raw materials and the choice of the fraction of first crystallizer mother liquor taken as the bleed stream.
- The FeCl_3 extract, although dilute, is relatively pure. Separation of FeCl_3 in relatively pure form preserves the option of possibly further processing the material into some marketable form.
- The option is also preserved for possible eventual recovery of gallium by a relatively inexpensive process.

The disadvantages include:

- The cost--even though it is only a small percentage of both capital and operating cost.
- Possible damage to some polymeric corrosion-resistant equipment linings as a result of a process upset which might cause the presence of solvent in process sections where it is not desired.

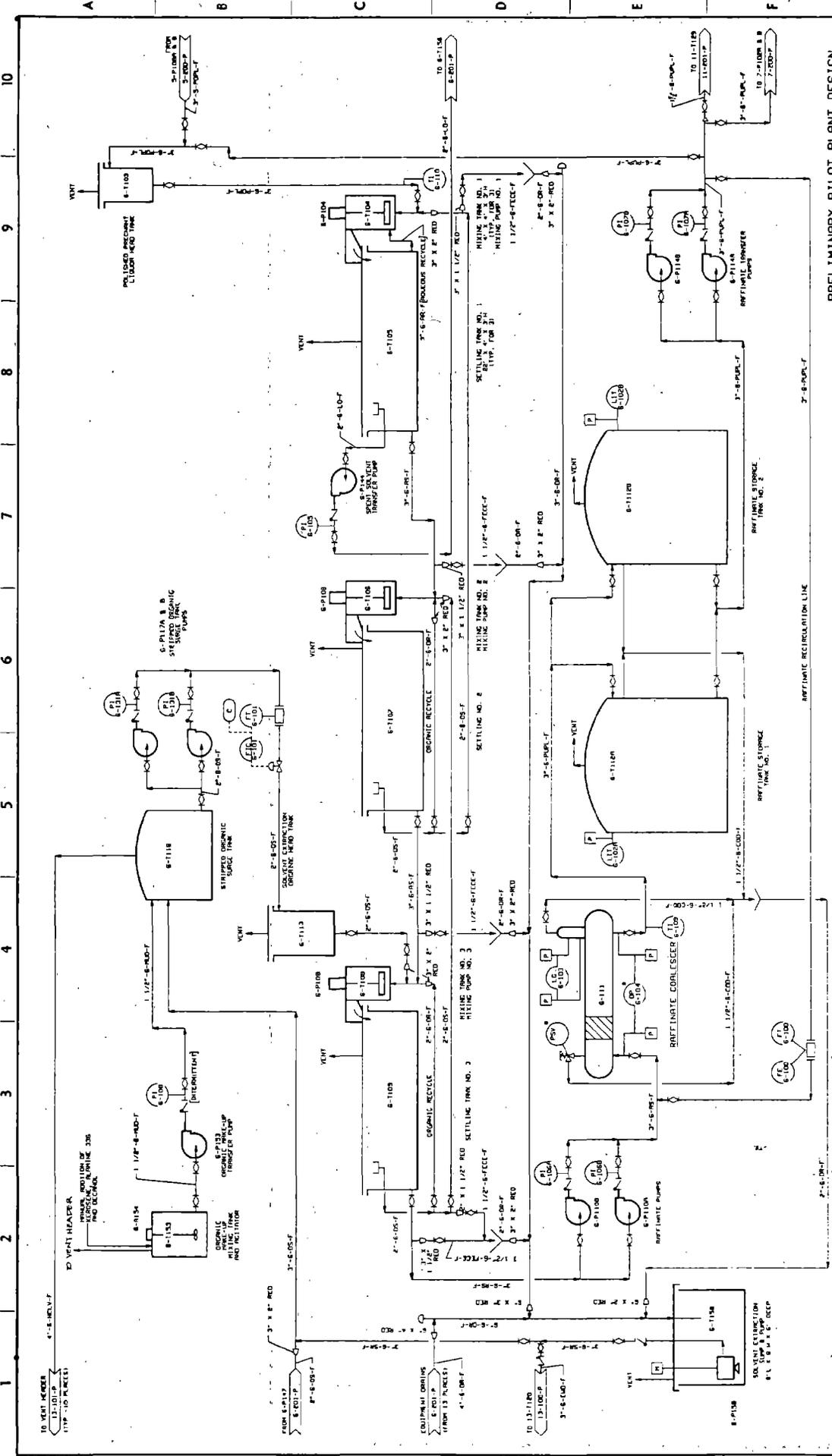
- The FeCl_3 extract is produced at a low concentration necessitating eventually a substantial amount of evaporation. The amount and cost of this evaporation is reducible by using extract converted to dilute AlCl_3 by reaction with calcined clay as a part of the wash H_2O for the main acid insoluble stream, and by using available waste heat for vacuum evaporation.

Accurate estimation of the value of the advantages in comparison with the modest cost of solvent extraction is difficult at the present state of development of the process. The first three items, however, are likely to provide savings at least offsetting the cost of the operation. It has therefore been concluded that removal of acid-soluble iron prior to crystallization should be examined in the pilot plant which has been engineered on this basis.

SUPPLEMENTAL
MATERIAL BALANCE
AREA 6

PROCESS STREAM	19	=	6A	+	6B					
Al ₂ O ₃										
AlCl ₃ ·6H ₂ O	11,421									
FeCl ₃										
Fe ₂ O ₃										
SiO ₂										
HCl	133									
H ₂ O (FREE)	12,370									
OTHER	70									
ORGANIC										
Cl ₂										
GAS (NON-CONDENSIBLE)										
FUEL										
TOTAL	23,994									
TEMPERATURE °F	140		140		140					
DENSITY, lb/gal (lb/ft ³)	10.6		10.6		10.6					
FLOW RATE, GPM	37.9		34.7		3.2					

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NO.		DATE	REVISION	DESCRIPTION	COST ACCOUNT	CONTRACTOR APPROVAL	APPROVAL	DATE	SCALE	DATE	REVISIONS	DATE	REVISIONS
1	1	7/1/79	1	ISSUED FOR PERMIT									
2	2	7/1/79	2	REVISED FOR PERMIT									
3	3	7/1/79	3	REVISED FOR PERMIT									
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5	5	7/1/79	5	REVISED FOR PERMIT									
6	6	7/1/79	6	REVISED FOR PERMIT									
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8	8	7/1/79	8	REVISED FOR PERMIT									
9	9	7/1/79	9	REVISED FOR PERMIT									
10	10	7/1/79	10	REVISED FOR PERMIT									

PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
 A DIVISION OF THE UNITED STATES STEEL CORPORATION
 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 20308

IRON REMOVAL -
 SOLVENT EXTRACTION -
 PIPING & INSTRUMENT DIAGRAM

JOB No. 76161-003 DWG. No. 6-200-P

REVISED FOR PERMIT

3.2.6 Evaporation - Area 7 - Drawing No. 7-100-P

3.2.6.1 Process Description

The evaporation section is provided to concentrate the pregnant liquor close to the saturation point (30% AlCl_3) before it is fed to the first crystallizer. Increasing the AlCl_3 concentration in this manner maximizes the yield of ACH crystals per unit of HCl gas sparged into the crystallizer.

The evaporator section also provides one of the means by which water is rejected from the process (in this case via the cooling towers) assisting in maintenance of the process water balance.

A portion of the effluent from Iron Removal is used to slurry ACH crystals from the Bleed Stream Treatment crystallizer. This resultant stream becomes one of the feed streams to evaporation. The remainder of the effluent from Iron Removal becomes the main feed stream to evaporation.

The evaporator is a single stage unit operating at 100 mm Hg and 160°F. The evaporator liquor is recirculated through an external vertical tube heat exchanger where 50 psig steam is used on the shell side. The recirculation rate of the liquor is designed to give approximately a 7°F rise across the heater for the maximum evaporation rate of 5400 lbs/hr. This means the normal temperature rise across the heater will be approximately 3.7°F corresponding to an evaporation rate of 2591 lbs/hr. The top tube sheet of the exchanger is located below the evaporator normal liquid level to minimize flashing in the tubes.

The main feed stream is introduced into the recirculating loop to maintain evaporator level. Steam is fed to the recirculation heat exchanger on flow control. The steam flow rate determines the evaporation rate. The 30 weight percent AlCl_3 solution is pumped from the bottom of the evaporator to the crystallizer feed surge tank using boiling point rise (BPR) control.

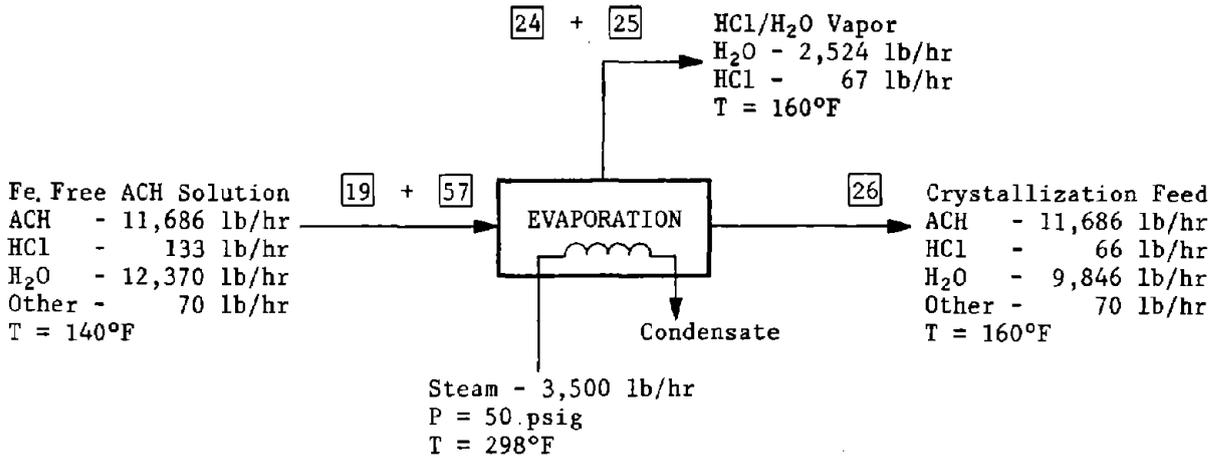
The evaporator overhead steam and HCl vapors enter the falling film partial condenser where a 10 weight percent HCl solution is condensed and pumped to Acid Recovery. Part of this HCl is recycled to the top of the partial condenser to improve the vapor-liquid contact area. The amount of cooling water to the condenser is varied to control condensed acid concentration at 10 weight percent. The condenser is designed for minimum tube side pressure drop.

The resultant uncondensed vapors passing through the partial condenser will be sent to a barometric condenser designed to condense 1921 lbs/hr vapor normally and 4800 lbs/hr vapor maximum. The steam jet is exhausted into the hotwell with the barometric effluent and the resultant liquor is pumped back to the cooling tower. The small amount of HCl in the hotwell requires the hotwell be designed with a hood with vapors being sent to the fume control system. The vapor flow is normally zero.

EVAPORATION
HEAT AND MATERIAL BALANCE

3.2.6.2

<u>Stream</u>	<u>HEAT IN</u>	<u>Btu/hr</u>	<u>Stream</u>	<u>HEAT OUT</u>	<u>Btu/hr</u>
[19] + [57] Fe Free ACH Solution		1,390,000	[24] + [25] Hcl/H ₂ O Vapor		
Steam		4,126,000	Sensible Heat		241,000
Total		5,516,000	Vaporization		2,570,000
			[26] Crystallization Feed		1,769,000
			Condensate		936,000
			Total		5,516,000



Base Temp = 77°F

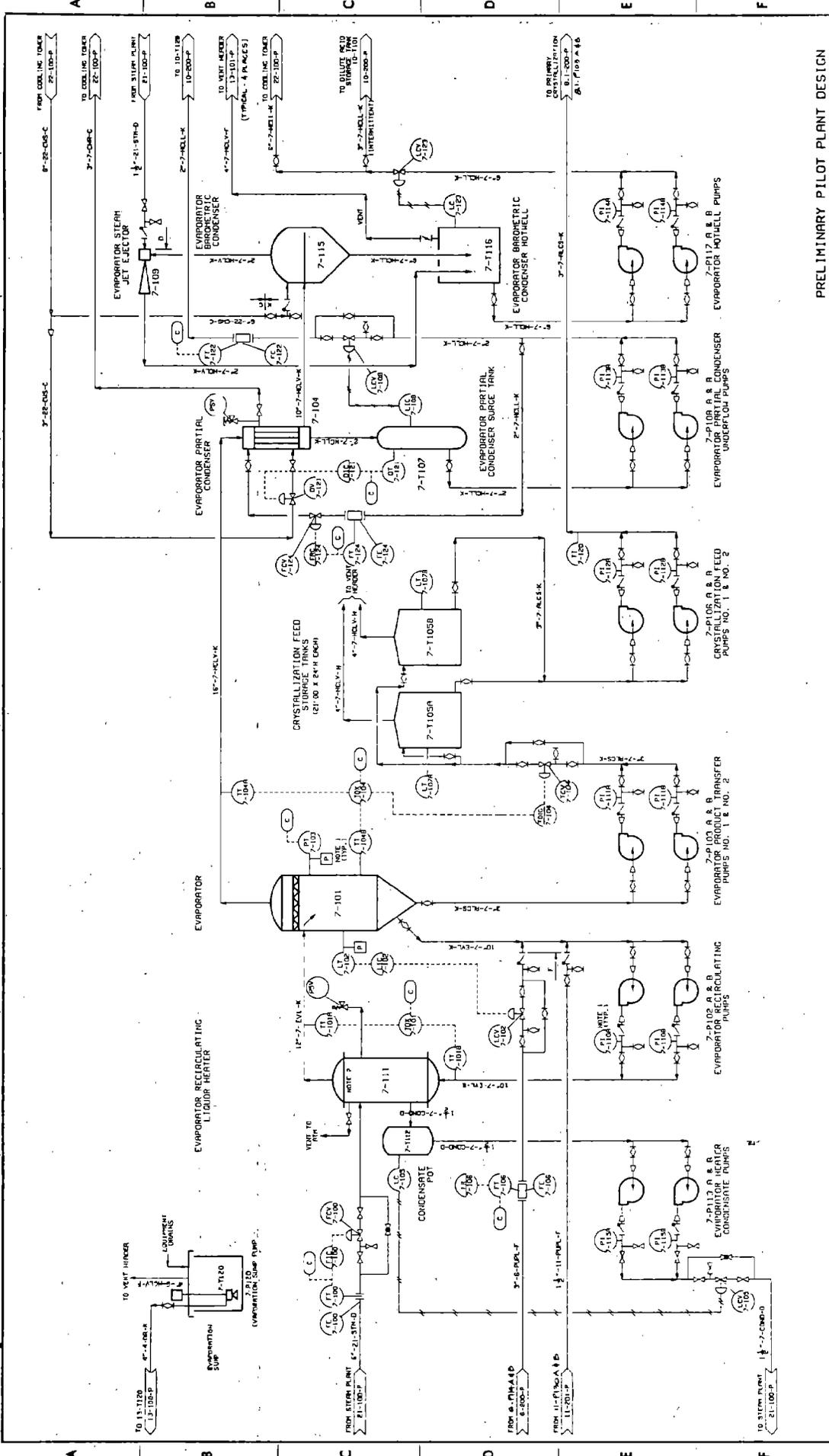
Steam Base Temp = 32°F

Note: Stream numbers where shown correspond to stream numbers on Dwg 50-302-G,
Block Flow Diagram

SUPPLEMENTAL
MATERIAL BALANCE
AREA 7

PROCESS STREAM	7A	+	7B	=	19	+	57			
Al ₂ O ₃										
AlCl ₃ ·6H ₂ O					11,421		265			
FeCl ₃										
Fe ₂ O ₃										
SiO ₂										
HCl					133					
H ₂ O (FREE)					12,370					
OTHER					70					
ORGANIC										
Cl ₂										
GAS (NON-CONDENSIBLE)										
FUEL										
TOTAL					23,994		265			
TEMPERATURE °F	140		140		140		160			
DENSITY, lb/gal (lb/ft ³)	10.6		10.6		10.6		(90)			
FLOW RATE, GPM	34.7		3.5		37.9		-			

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NO. DATE		REVISION		NOTES		APPROVAL		DATE		SCALE		JOB NO.		DATE	
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THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

1. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

2. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

3. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

4. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

5. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

6. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

7. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

8. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

9. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

10. THE HEATER OPERATOR SHOULD BE SECURED FIRST BEFORE THE EVAPORATOR IS STARTED. SEE PIPING & INSTRUMENT DETAIL AND DATA SHEET FOR COMPLETE LIST OF INSTRUMENTS AND CONNECTIONS OF OPERATIONAL SET.

PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS

STUDY AND INSTRUMENT DETAIL PILOT PLANT DESIGN
U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 48(10)

EVAPORATION & INSTRUMENT DIAGRAM

PIPE NO. 76161-003 DWG. NO. 7-800-P

NO. DATE

REVISION

NOTES

APPROVAL

DATE

SCALE

JOB NO.

3.2.7 Crystallization, Deliquoring and Dissolution - Area 8

Introduction

Certain properties of the system $AlCl_3/HCl/H_2O$ provide the basis for the operation of the crystallization process and its converse, the dissolving of crystals in hydrochloric acid solutions.

1) The solubility of $AlCl_3 \cdot 6H_2O$ (ACH) in HCl solutions is a function of the amount of HCl present, varying between about 58% at 0% HCl and 1% at 37% HCl. Over most of this range the molality with respect to total chloride remains almost constant.

2) The heat of absorption/desorption of HCl is substantially decreased to about 587 Btu/lb for solutions saturated with respect to both ACH and HCl. The molal heat of absorption/desorption for HCl therefore approaches the molal heat for the absorption/desorption of H_2O for these solutions. The heat of solution/crystallization of ACH from these solutions, although not precisely known, is very small.

3) The mole fraction HCl in HCl/ H_2O vapor which is in equilibrium with liquid that is saturated with respect to both HCl and ACH is 1.0, but is greater than the mole fraction HCl in the liquid for the entire range of ACH solubility in these solutions. The mole fraction HCl in the vapor approaches 1.0 when the mole fraction HCl in the liquid is ≥ 0.18 . The temperature at which the total vapor pressure above the liquid phase is one atmosphere decreases from about 260°F at 0% HCl to about 170°F for liquid mole fraction HCl of 0.2.

4) Aluminum chloride crystallizing from aqueous solution forms only one hydrate, $AlCl_3 \cdot 6H_2O$. The crystallization process is easily reversible, and attainment of equilibrium between the solid and liquid phases is very rapid. It is of particular importance that, even in the presence of relatively large amounts of other salts, $AlCl_3$ shows very little tendency to form double salts or for the inclusion of other salts in the $AlCl_3 \cdot 6H_2O$ crystal structure. The separation of $AlCl_3 \cdot 6H_2O$ from other salts by the crystallization process is undoubtedly enhanced by the fact that many other metallic chlorides, such as $FeCl_3$, form anionic chloride complexes in water solution whereas $AlCl_3$ does not.

Crystallization

A consequence of the above relationship is that a range of useful temperatures exists wherein HCl gas at one atmosphere pressure will dissolve readily in $AlCl_3$ solutions, causing the solubility of $AlCl_3$ to be reduced with consequent formation and growth of ACH crystals. The degree of supersaturation can be regulated to control both the formation of new crystal nuclei and the rate of growth of existing crystals. A controlled crystal size distribution is thereby obtained, and maximum separation between ACH and dissolved impurities is obtained. The crystallization process is mildly exothermic, the exotherm being the net of the heat of solution of HCl, the addition of sensible heat to liquor

entering crystallization, the small heat of crystallization of ACH, and heat losses from the equipment. The initial recovery of ACH by HCl-induced crystallization from a saturated solution of $AlCl_3$ may, if desired, be carried out at one atmosphere at a temperature that is much higher than towards completion of the crystallization, because of the higher partial pressure of HCl over the mother liquor at any given temperature when the HCl concentration in the liquid phase is higher towards completion of the crystallization. A possibility therefore exists in a staged crystallization for recovery at a useful temperature of a part of the heat released during crystallization.

Crystal Dissolution

The very severe limitations on amounts of impurities permitted in reduction grade alumina requires that ACH produced in a first crystallization be redissolved in H_2O containing only very small amounts of dissolved impurities and then recrystallized in order to finally obtain an ACH decomposable into alumina meeting product purity specifications. It is very desirable to dissolve the first crystals in condensed hydrochloric acid available within the process rather than in water from an external source. This is because the desorption of 1 lb HCl from 33% acid at a heat requirement of 587 Btu yields 2 lbs dissolver water, which if brought in from an external source would subsequently require approximately 2000 Btu for removal by evaporation. Condensates from within the process will also contain lesser amounts of dissolved impurities than most external sources of water.

Concentrated saturated solutions of $AlCl_3$ containing only a small mole fraction HCl will be in equilibrium with vapors containing much larger mole fractions of HCl, but there will be present in the vapor a substantial mole fraction of H_2O . The ratio of the equilibrium mole fraction HCl in the vapor to the mole fraction HCl in the liquid on a free H_2O basis at low to intermediate HCl concentrations decreases rapidly with decreasing concentration of $AlCl_3$ (solution not saturated). This is the reason for operating the dissolver always at saturation using a slurry of ACH crystals in dissolver liquid. The equilibrium mole fraction HCl in the vapor increases as the mole fraction HCl in the liquid increases (with a correspondingly smaller content of dissolved crystals at saturation). The dissolving operation must therefore be countercurrent in order to produce HCl gas containing very little H_2O and a concentrated solution of $AlCl_3$ containing very little dissolved HCl.

3.2.7.1 Primary Crystallization Process Description - Area 8.1 -
Dwg. 8.1-100-P

First Stage

Primary concentrated liquor from evaporation at a concentration of approximately 30% AlCl_3 is added continuously to liquor recirculated through the cooling heat exchanger, past the venturi provided for HCl addition, and back to the bottom of the crystallizer through the downcomer. Supersaturation due to the addition of HCl is relieved by growth of crystals suspended in the body of the crystallizer, the overflow liquor again returning to the circulating pump for another cycle. Crystal slurry is continuously withdrawn by means of a pump and sent to crystal slurry surge tank so as to maintain a constant inventory of liquor and crystals in the body of the crystallizer. The maintenance of steady-state operating conditions in the crystallization process is of utmost importance to maintaining a constant particle size distribution and the chemical purity of the ACH crystals produced.

A conservative operating temperature of 160°F has been specified for the first-stage crystallizer. Consistent with limitations of materials of construction, a higher operating temperature may produce purer crystals of ACH due to greater ionic mobility and enhance the separation of mother liquor from the crystals because of lower mother liquor viscosities. Alternatively, a higher rate of crystal production per unit crystallizer volume may be attainable.

The rate of HCl addition is controlled by the difference in temperature induced in the recirculating liquor stream by the HCl addition and is chosen so as to produce constantly only the desired degree of supersaturation in the recirculating liquor. The method of introducing the HCl is also important--the method specified in the design is at the throat of a venturi in the recirculating liquor line after the liquor leaves the heat exchanger. This method of HCl addition provides good mixing of the HCl into the liquor in the very turbulent liquid in the throat of the venturi, thereby minimizing any locally high HCl concentration. It also exposes the heat transfer surfaces in the heat exchanger to a minimum of supersaturation, thereby reducing any tendency towards scale formation.

The rate of addition of cooling fluid to the crystallizer heat exchanger is controlled by the temperature in the body of the crystallizer so as to maintain the preset temperature in that vessel.

An essentially straight-line relationship exists, for solutions saturated with both HCl and ACH, between dissolved ACH content and density. The latter value is therefore a primary control parameter in crystallizer operation. ACH crystals have a particle density of 1.629, considerably greater than liquor density for any composition. Slurry density is therefore utilized to control the rate of removal of the crystal slurry from the crystallizer body. The rate of addition of primary crystallizer feed liquor is controlled by the liquid level in the crystallizer body.

There will inevitably be, in a cycle process including thermal decomposition of ACH and which is operated for various reasons at slightly less than atmospheric pressure, some inert gases mixed with the HCl supplied to the crystallizer. These inert gases will not dissolve in the crystallizer mother liquor but will tend to collect in the space above the liquor in the crystallizer body. This space is vented through a scrubber to the atmosphere, thus providing a means of purging inert gases from the system.

Second Stage

Filtrate from the first-stage filter flows by gravity to the filtrate surge tank, which is provided to assure that feed liquor can be delivered to the second-stage crystallizer at a constant rate. The mode of operation and control of the second-stage crystallizer is exactly the same as for the first. Approximately the same amount of heat as in the first stage is available in the heat exchanger liquid cooling medium discharge, although at a lower temperature as determined by the operating temperature in the second stage.

3.2.7.2 Crystal Dissolution Process Description - Area 8.3 Dwg. No. 8.3-100-P

Crystals produced in the primary crystallization section must be redissolved prior to the second crystallization.

Acid being used for dissolving which is a mixture of secondary crystallizer mother liquor and various acid condensates, is mixed with ACH in a slurry tank and then is pumped to the top of the ACH crystal dissolver column. The slurry descends through the column, approaching equilibrium with the ascending vapor on each sieve tray. The vapor, produced originally by the reboiler and consisting mainly of steam as it progresses upwards through the column to trays where the liquid has successively higher HCl partial pressures and lower H₂O partial pressure, becomes enriched in HCl. The exit vapor normally used in the crystallizers, contains HCl > 95% by weight. An approximately corresponding number of moles of H₂O is absorbed into the descending liquid. The addition of this H₂O, as well as the removal of dissolved HCl, permits the ACH crystals to dissolve. Only half of the ACH crystals to be dissolved are added to the dissolver acid in the beginning to avoid working with more than 20% solids on the top tray, so liquid is withdrawn from the column at an appropriate tray and mixed in a tank with the remainder of the ACH. The resulting slurry is then pumped back to the appropriate point in the column and the stripping process continues. Concentrated AlCl₃ solution is withdrawn from the bottom of the dissolver and pumped to secondary crystallization. Thirteen sieve trays are specified.

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3.2.7.3 Secondary Crystallization Process Description - Area 8.2
Dwg. No. 8.2-100-P

First Stage

The first stage of the secondary crystallization operates exactly the same as the corresponding stage of the primary crystallization excepting that the feed is the relatively pure dissolver discharge liquor. The same operating temperature is used, and the same comments apply in regard to recoverable heat. A centrifuge is specified instead of a filter for ACH crystal recovery in order to reduce the amount of mother liquor remaining on the surface of the crystals to a very small percentage of crystal weight, and to obtain operating experience in this service with both types of equipment.

Second Stage

This stage operates in the same manner as the corresponding stage in the primary crystallization except that centrifuges are specified for ACH recovery rather than filters.

Control of the Second Crystallization Feed Liquor Impurity Level

It is possible to return the mother liquor from the secondary crystallization with very low levels of dissolved impurities by dissolving ACH from the primary crystallization in fresh condensate. To do so is somewhat costly in part because mother liquor returning to leach will carry with it a substantial amount of dissolved ACH which must recirculate through the entire process and be crystallized again in the primary crystallization. The preferred mode of operation will be to recirculate as large a fraction as possible of the secondary crystallization mother liquor to the dissolver in order to reduce the amount of dissolved ACH recycling to leach. The amount of condensates required in the dissolver will be correspondingly reduced. The limit on the amount of secondary crystallization mother liquor which may be returned to the dissolver will be set by the amount of dissolved impurity buildup which can be tolerated in the secondary crystallization feed liquor without causing permissible impurity levels to be exceeded in the secondary crystallization product ACH.

The crystallization section of the overall process in the event of a general power failure can be safely allowed to come to a halt. This event is unlikely as the pilot plant has an emergency electric generator incorporated into the design, as well as uninterruptable power supply for instrumentation/controls.

As a final note, the Primary Crystallization as well as the Secondary Crystallization sections have been designed so that their respective two stages operate in series flow. Provision has been made, however, to enable them to be operated in parallel.

3.2.7.4 Crystal Deliquoring Process Description - Area 8.1 and 8.2 -
Dwg. Nos. 8.1-101-P and 8.2-100-P

Summary

The crystal deliquoring sections of the pilot plant are designed to receive a nominal 20% solid slurry of aluminum chloride hexahydrate (ACH) from the 1st and 2nd stage crystallizers, to thicken and store thickened slurry, and to separate and wash ACH crystals from the slurry by filtration in the primary system and centrifugation in the secondary system. Washed crystals from the primary system are sent to ACH dissolution. Washed crystals from the secondary system are sent to the decomposition section for conversion to alumina. The final filtrate is split and sent to bleed stream treatment and to leach acid preparation. The final centrate is split and sent to primary crystal filtration and to ACH dissolution. 72 hours of storage capacity is provided in cone bottom tanks for ACH crystals as a 50% solid slurry. ACH separation and wash is effected on horizontal belt filters in the primary system and on pusher centrifuges in the secondary system.

The primary and secondary crystal deliquoring sections include the equipment necessary to thicken, store, transport and filter or centrifuge ACH slurry in order to prepare ACH crystals for dissolution or decomposition. Specifically the equipment is limited to one filtration and one centrifugation train of equipment, each with two stages. In each train, the first stage receives slurry from the first stage crystallizer and prepares feed liquor for the second stage crystallizer and the second stage receives slurry from the second stage crystallizer and makes filtrate or centrate which is sent either to bleed stream treatment and to leach acid preparation, or primary wash acid and ACH dissolution. Each train consists of two stages and each stage contains:

- a large slurry thickening and holding tank
- filter or centrifuge feed pumps
- slurry cyclones
- a horizontal belt filter or pusher-type centrifuge
- a filtrate or centrate tank with pumps
- used wash acid tank and pumps
- a vacuum pump and vapor scrubber are provided for the horizontal belt filters

Equipment Description

The ACH crystal deliquoring sections are both composed of similar equipment except for the substitution of centrifuges for filters in the secondary system train. The four equipment trains receive feed from the following:

- primary crystallization first stage
- primary crystallization second stage
- secondary crystallization first stage and
- secondary crystallization second stage.

In each train, crystal slurry is pumped from the crystallizer at a nominal 20% solids to a rubber-lined 17 ft dia. x 32 ft straight side conical bottom, crystal slurry tank. Crystals settle to the bottom of the tank and mother liquor overflows to the filtrate or centrate receiver tank. This tank provides 72 hours of crystal surge. It is equipped with an air injection port near the bottom of the cone which permits the use of plant air to agitate the tank in an emergency condition. The tank is also equipped with centrate injection ports to permit the injection of centrate to improve slurry flow.

Underflow from the crystal slurry tank is mixed with filtrate or centrate to improve pumping characteristics. The blended slurry is pumped by a lined pump to a cyclone to concentrate the solids to 60-70% for filter or centrifuge feed. Feed to the filter or centrifuge is set by a manually operated plug valve.

The slurries produced in primary crystallization are separated on horizontal belt filters. Tests conducted at the mini-plant indicate that filtration can yield cake moisture of about 10% and a washing efficiency of 85% when 0.2 lb of wash liquor per pound of ACH cake is used. Based on this data and some crystallizer performance data developed by the Bureau of Mines, a horizontal belt filter will result in adequate crystal purity. Since filtration equipment is much less expensive and probably more reliable than centrifugation equipment, it has been specified for the primary separation circuit. However, product purity obtained in the pilot plant is of great importance. Since pusher centrifuges can be expected to produce a cake with about one half of the residual cake moisture and to have better washing efficiency than filters, they would produce a cake of higher purity than filters. For this reason it is desirable to install centrifuges in the secondary separation circuit to obtain operating data. This data would be used to properly select equipment for any commercial plant.

Filtration is done on horizontal belt filters constructed of rubber-lined steel, FRP and zirconium where required. Cake washing is done with 25% hydrochloric acid as centrate from second stage ACH centrifugation and the filter is equipped to separate the spent wash acid from the filtrate (mother liquor). The filter vacuum pump is protected from acid fumes by a vapor scrubber. The filter is covered with an FRP fume hood vented to the scrubber system.

When centrifugation is employed, the centrifuge is a two stage pusher-type unit with a zirconium basket and rotating parts. Other wetted parts are of rubber-lined construction. The centrifuge is equipped for cake washing with 20% hydrochloric acid and for separation of the mother liquor and the wash liquor. The centrifuge is vented to the vent scrubber system.

The mother liquor is collected in a 500 gallon rubber-lined tank. The tank is equipped with a pair of lined centrifugal pumps.

The used wash acid is collected into a 50-gallon rubber-lined tank also equipped with two lined centrifugal pumps. The pumps deliver liquor to the destinations shown on the process flow diagram.

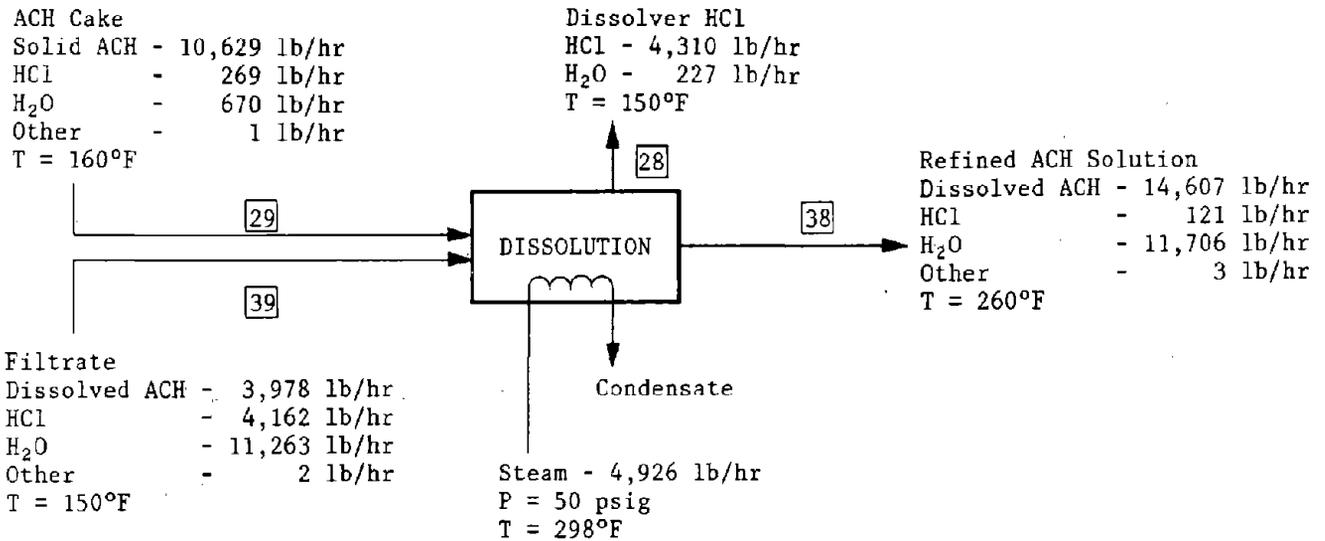
Solids level in the crystal slurry tank is indicated by an air purged probe. The centrate and used wash acid tanks are equipped with level controls and bleed rate is on flow control.

ACH DISSOLUTION
HEAT AND MATERIAL BALANCE

3.2.7.5

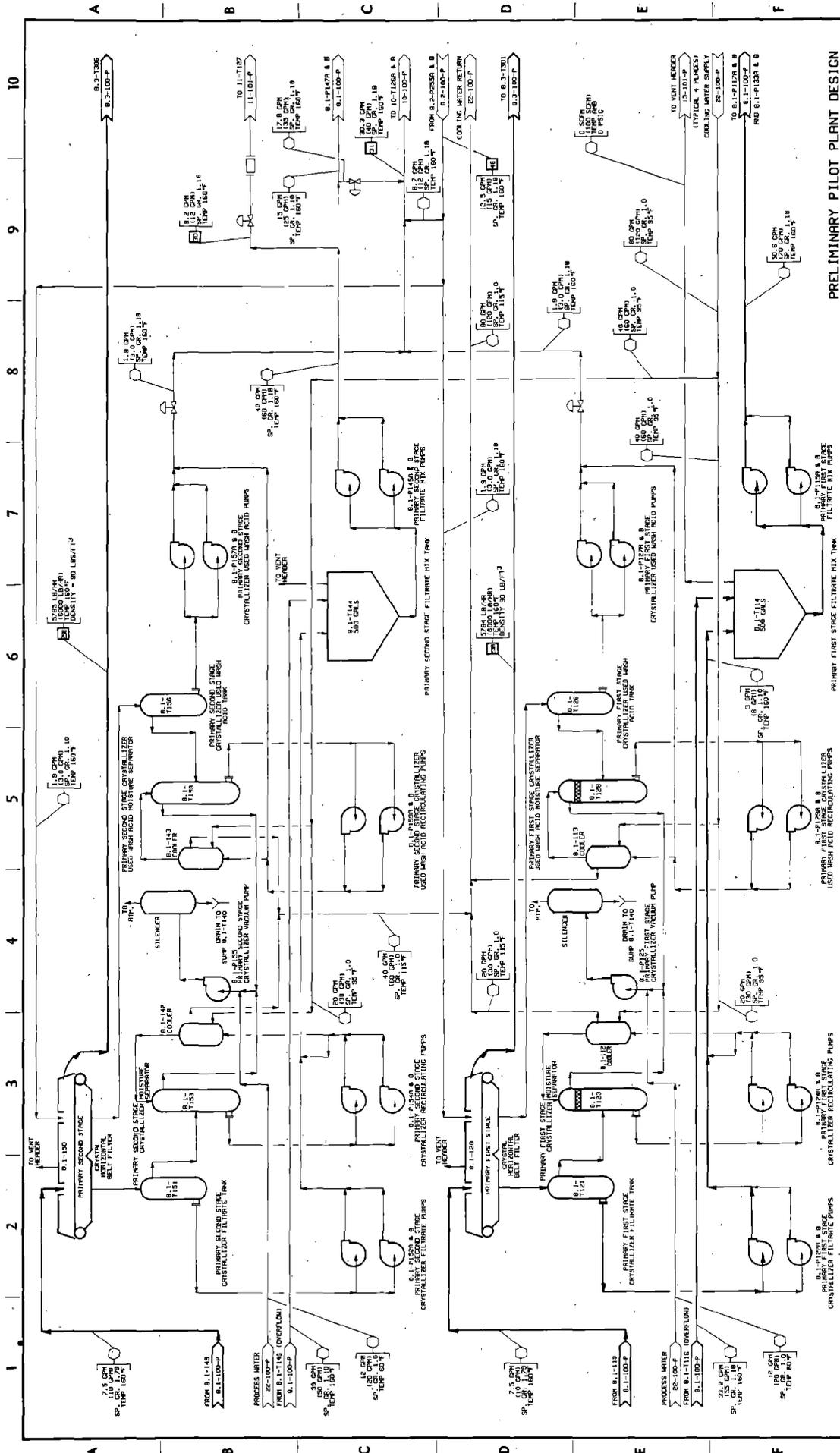
<u>HEAT IN</u>		<u>HEAT OUT</u>	
<u>Stream</u>	<u>Btu/hr</u>	<u>Stream</u>	<u>Btu/hr</u>
29	ACH Cake	28	Dissolver HCl Sensible
	520,000		330,000
39	Filtrate		Desorbtion
	1,570,000		2,530,000*
	Steam 50 psig	38	Refined ACH Solution
	5,808,000		5,723,000
	<u>Total</u>		Condensate
	7,898,000		<u>1,315,000</u>
			<u>Total</u>
			7,898,000

*Include heat of vaporization of H₂O in stream #28



Base Temp = 77°F
Steam Base Temp = 32°F

Note: Stream numbers where shown correspond to stream numbers on Dwg 50-302-G, Block Flow Diagram



NO DATE		REVISION		NOTES		DISCUSSION		COST ACCOUNT		CONSTRUCTION APPROVAL		APPROVAL		DATE		SCALE	
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PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS

ALUMINA PROCESS FACILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN
 U.S. DEPARTMENT OF THE INTERIOR, BUREAU OF MINES
 CRUSTAL FILTRATION
 PROCESS FLOW DIAGRAM

JOB No. 76161-003 DWG. No. 8.1-101-P

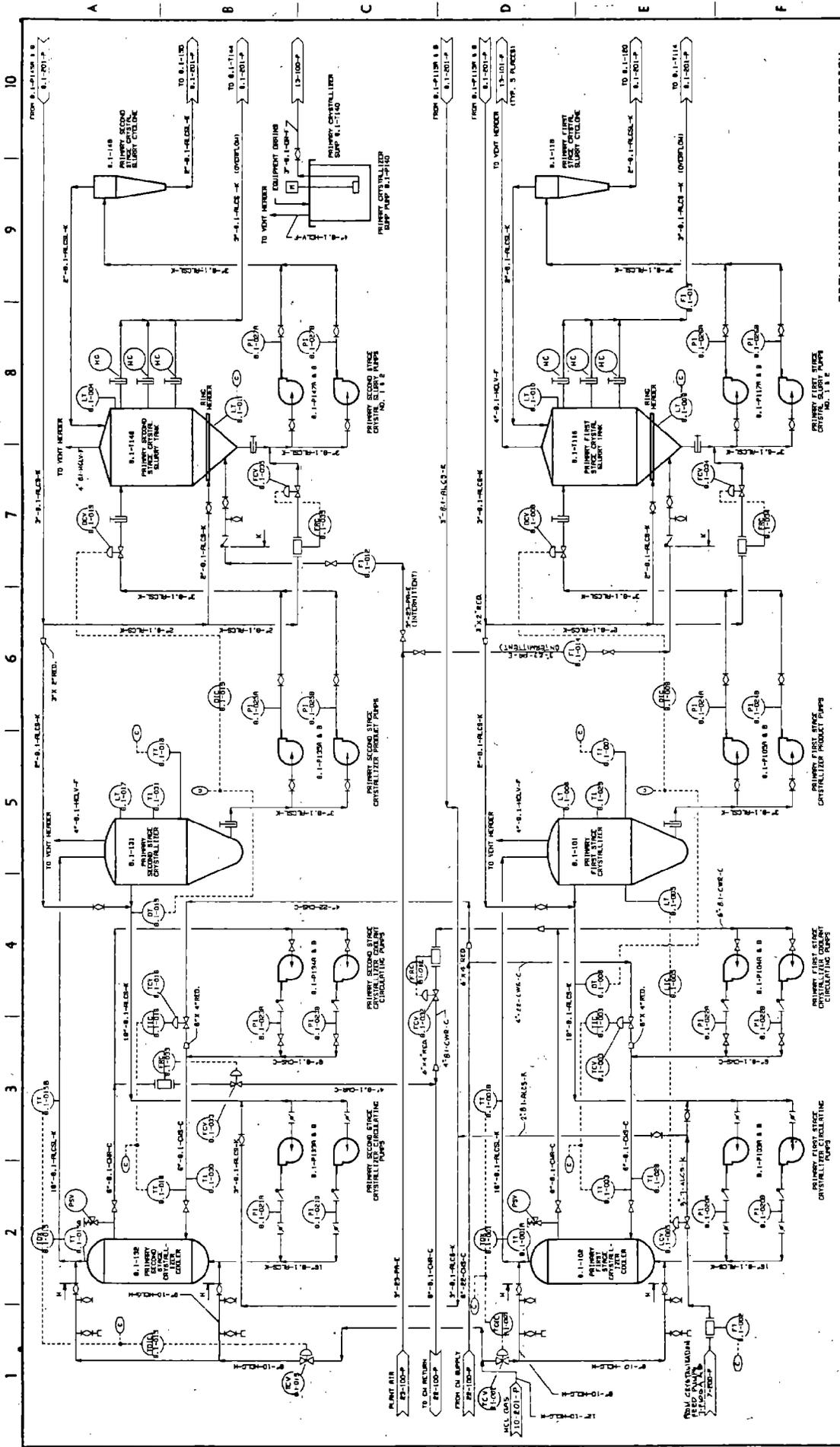
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PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
 CONSULTING ENGINEERS
 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 40008

PROJECT: PRIMARY CRYSTALLIZATION
 PIPING AND INSTRUMENT DIAGRAM
 SHEET NO. 76161-003
 DWG. NO. 8.1-200-P
 REV. NO. 1-1

DATE: 8.2.79
 DRAWN BY: [Name]
 CHECKED BY: [Name]
 APPROVED BY: [Name]

APPROVAL: [Signature]
 DATE: 8.2.79
 DRAWN BY: [Name]
 CHECKED BY: [Name]
 APPROVED BY: [Name]

DESCRIPTION: PRIMARY CRYSTALLIZATION
 PIPING AND INSTRUMENT DIAGRAM
 SHEET NO. 76161-003
 DWG. NO. 8.1-200-P
 REV. NO. 1-1

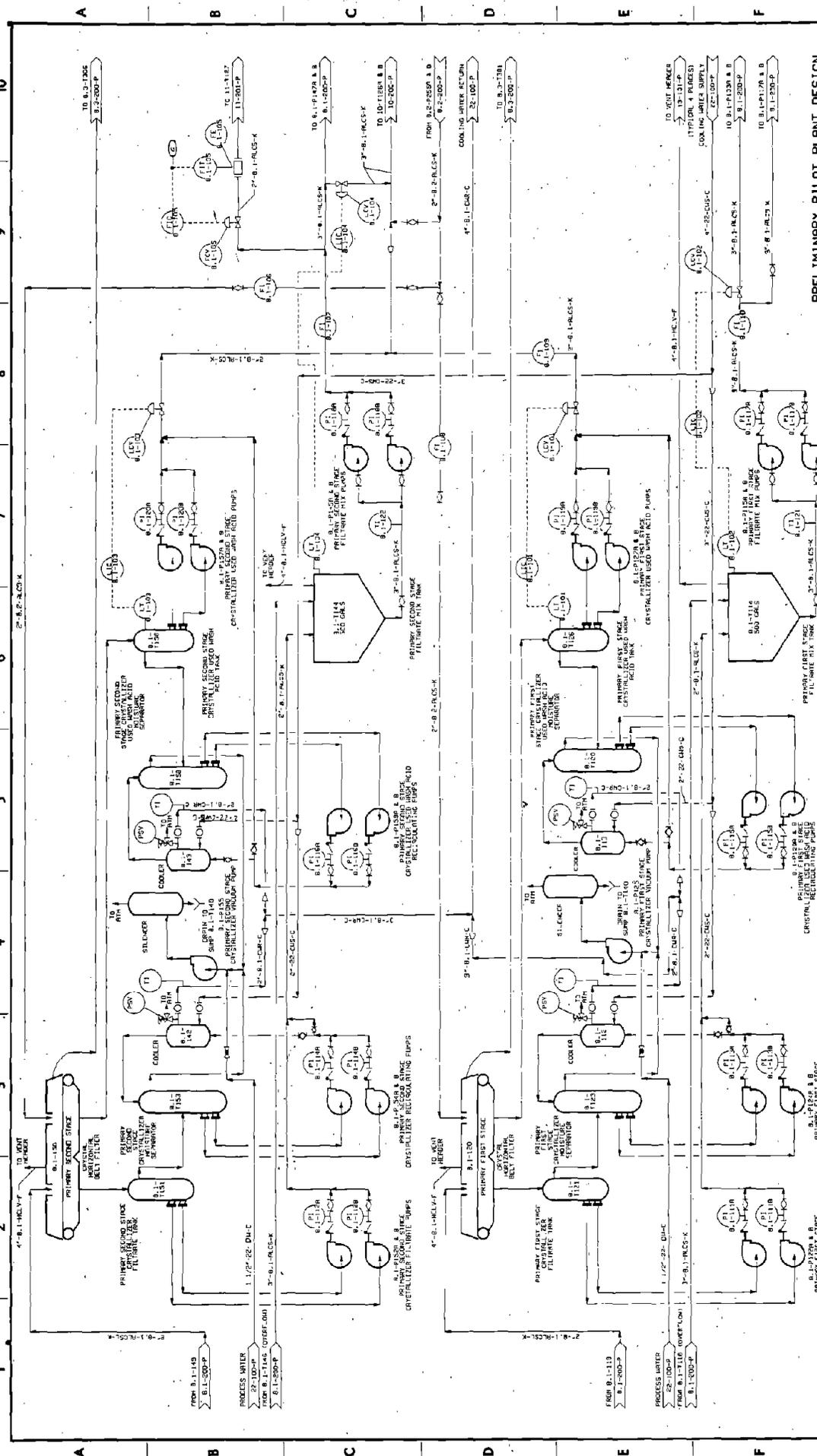
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NOTES: [List of notes]

CONTRACTOR APPROVAL: [Signature]

CONSTRUCTION APPROVAL: [Signature]

DATE: 8.2.79
 DRAWN BY: [Name]
 CHECKED BY: [Name]
 APPROVED BY: [Name]



NO. DATE		BY	CHK	APP	REV	DESCRIPTION	COST ACCOUNT	CONTRACTOR APPROVAL	APPROVAL	DATE	SCALE	SUNAK	DATE
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3										11/11			11/11
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PRELIMINARY PIPLING PLANT DESIGN

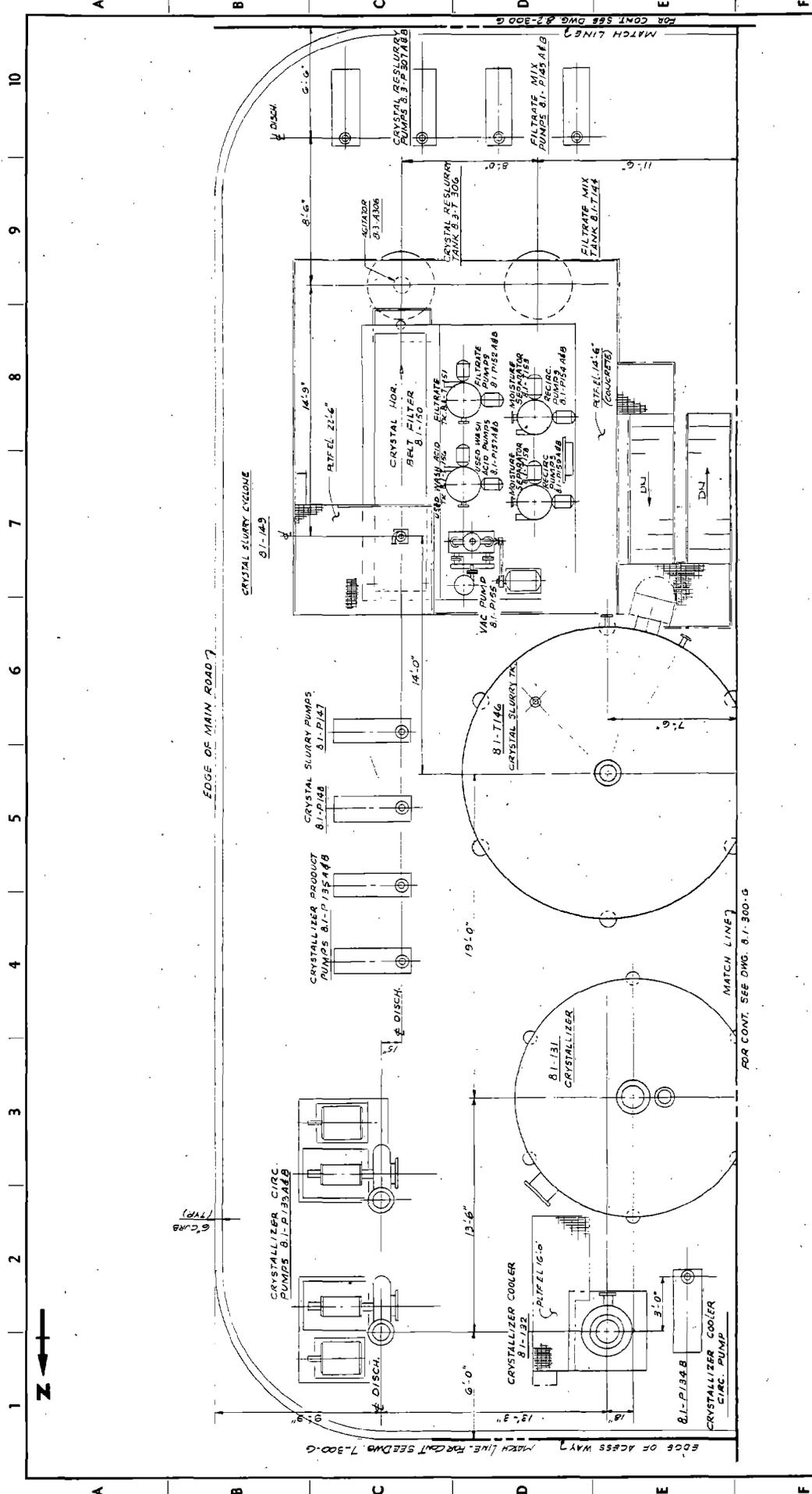
KAISER ENGINEERS

ALUMINA REFINERY FACILITY STUDY AND PRELIMINARY PLANT DESIGN
U.S. DEPARTMENT OF THE INTERIOR BUREAU OF MINES - CONTRACT NO. 40000

CRYSTALLIZER FILTRATION
PIPING & INSTRUMENTATION

JOE No. 76161003 DWG. No. B.1-201-P

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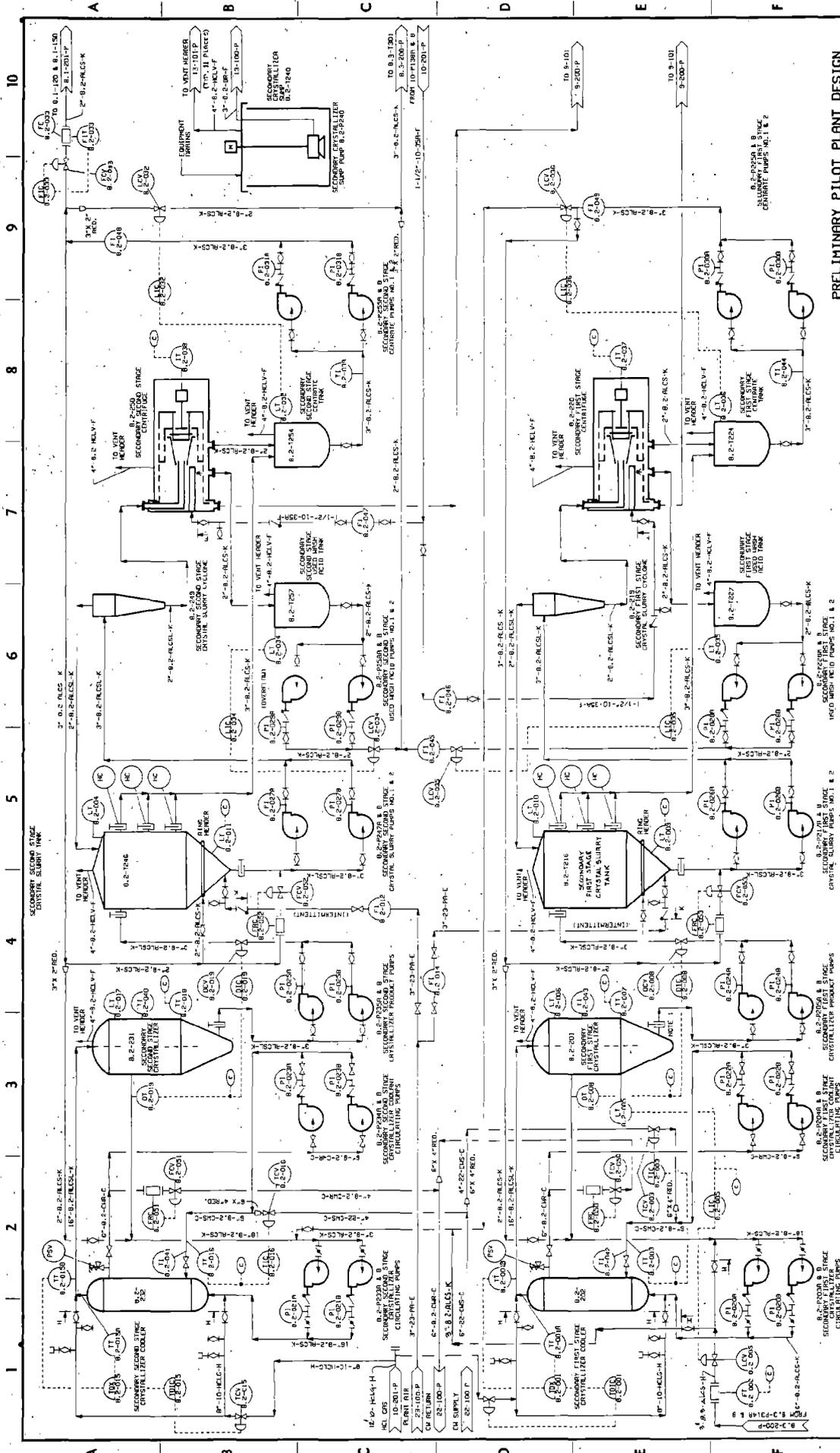
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DESCRIPTION	COST ACCOUNT

DATE	APPROVAL	SCALE	DATE
		3/8" = 1'-0"	5-31-79
		DESIGNED BY C. SINDRIN	7-19-79
		DRAWN BY C. W.	7-26-79
		CHECKED BY C. W.	7-26-79
		BY PROJECT MANAGER	8-1-79
		BY PROJECT ENGINEER	8-1-79

CONSTRUCTION APPROVAL	PROFESSIONAL SEAL

PRELIMINARY PILOT PLANT DESIGN	
KAISER ENGINEERS	
ALUMINA PROCESS FEASIBILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 20000	
AREA 8.1 (2ND STAGE) 8-3	
PRIMARY CRYSTALLIZATION/FILTRATION	
GENERAL ARRANGEMENT	
JOB No. 76161-003	DWG. No. 8.1-301-G



REVISION		DATE		APPROVAL		CONSTRUCTION APPROVAL		SCALE		DATE		APPROVAL	
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PRELIMINARY PILOT PLANT DESIGN

KAISER
 A LUMINA PROCESS EQUIPMENT SUPPLY AND PRELIMINARY PLANT DESIGN
 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 40000M

SECONDARY CRYSTALLIZATION/CONFIGURATION
 PIPING & INSTRUMENT DIAGRAM
 JOB No. 76161003 DWG. No. 8.2-200-P

DATE: 8-2-79
 BY: RMB
 CHECKED: RMB
 APPROVED: C. G. GIBERT
 DRAWN: RMB
 REVISION: RMB

PROFESSIONAL SEAL

NO. 100

ISSUED FOR REPORT

REVISION

DATE

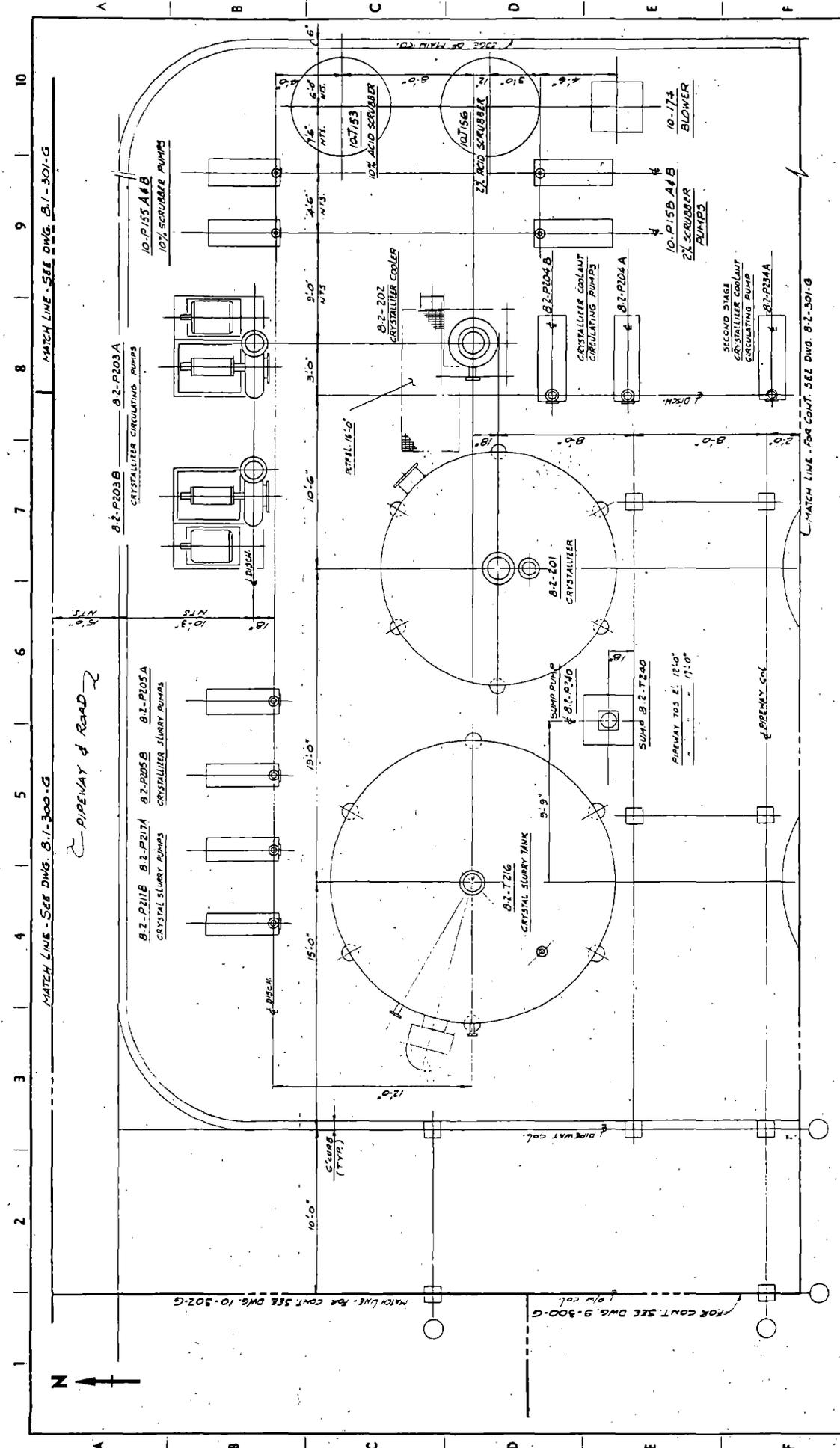
APPROVAL

CONSTRUCTION APPROVAL

SCALE

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APPROVAL



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MATCH LINE - SEE DWG. B.1-300-G

MATCH LINE - FOR CONT. SEE DWG. B.2-301-G

PIPEWAY & ROAD



REVISION		NOTES		APPROVAL		DATE		SCALE		DATE		DATE		DATE	
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10	DATE	BY	APP. BY	DESIGNER	CHECKED	APPROVED	DATE	3/18	1:1	3/18	3/18	3/18	3/18	3/18	3/18

PRELIMINARY PILOT PLANT DESIGN

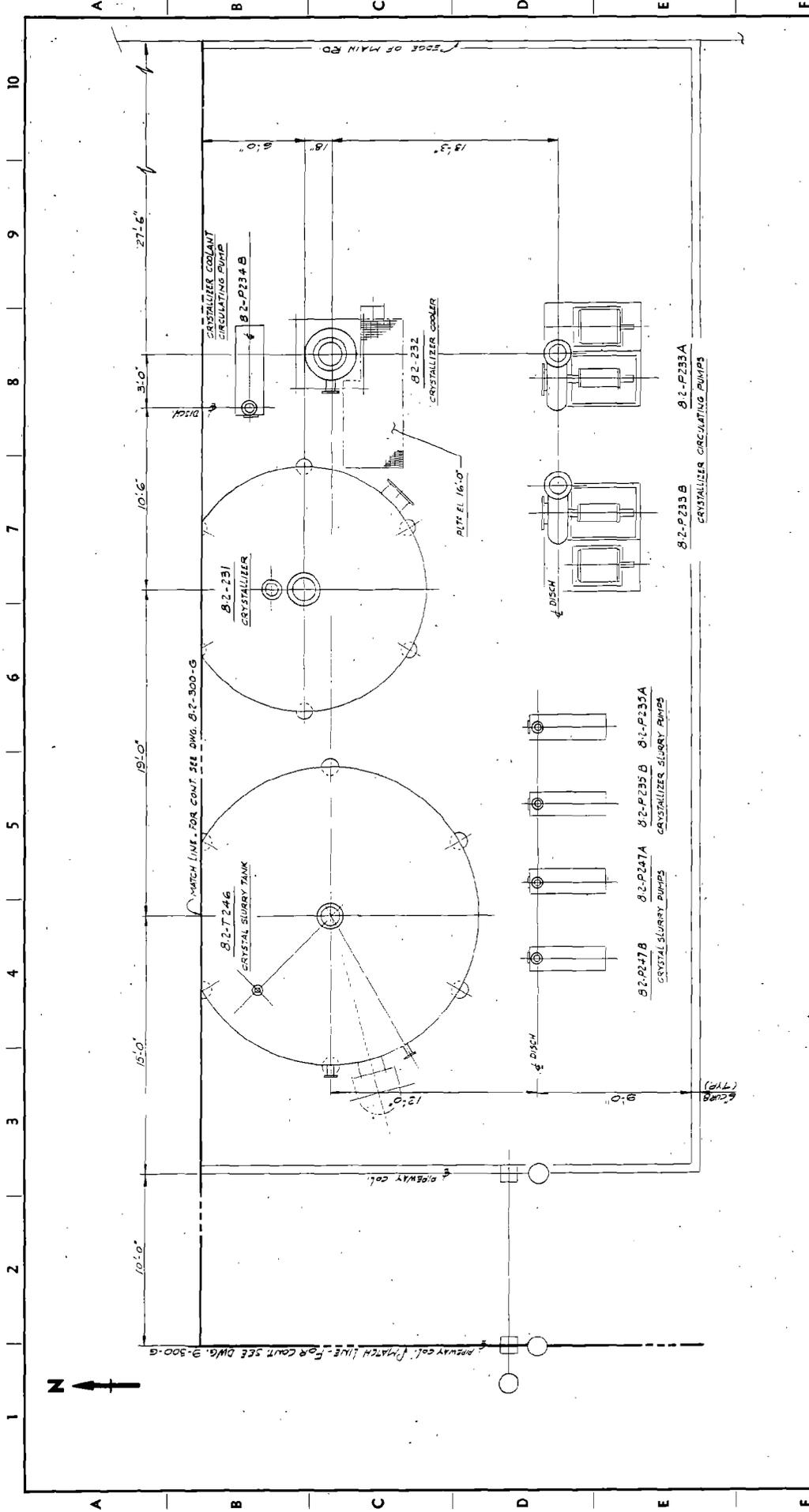
KAISER ENGINEERS

AREA 5-2 (11 STAGE) & 10
SECONDARY CRYSTALLIZATION/CENTRIFUGATION
GENERAL ARRANGEMENT

108 No. 76161003 DWG. No. B.2-300-G

R.O.

96



PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS

ALUMINA WAXES (ASBESTY STUDY AND RELIABILITY PILOT PLANT DESIGN)
 US INTERCOMMERCE TRADE SHOWS - CONTRACT NO. 48000
 AREA 2, ZONE 2, STAGE CENTRIFUGATION
 GENERAL ARRANGEMENT

JOB No. 76161-003 DWG. No. B 2-301-G

NO.	DATE	REVISION

DATE	SCALE	BY	DATE

DESCRIPTION	COST ACCOUNT

NO.	DATE	REVISION

1 2 3 4 5 6 7 8 9 10

3.2.8 Decomposition - Area 9 - Dwg. No. 9-100-P

3.2.8.1 Process Description

Summary

The ACH decomposition section of the demonstration plant is designed to receive aluminum chloride hexahydrate (ACH) crystals from secondary centrifugation, to reduce the average particle size to that which can be properly fluidized, and hold broken crystals in a surge bin for decomposition to alumina. Decomposition of ACH to alumina is done by heating the crystals in two fluid bed reactors in series, each of which produces an HCl containing gas stream which is sent to the acid recovery section. The first stage fluid bed is indirectly heated to between 450° and 750°F by molten salt circulating through coils submerged in the bed. The second stage reactor is directly fired to 1600°-2000°F by fuel oil. The intensive effort by the Bureau of Mines at their Reno Research Center, demonstrated the feasibility of this approach for the decomposition of $AlCl_3 \cdot 6H_2O$ to alumina.

The ACH decomposition section includes the equipment necessary to transport, crush, decompose and cool ACH and alumina as well as the HCl gas handling equipment required to clean and cool the gas prior to transfer to the acid recovery section.

The area receives ACH at the centrifuge discharge and finally feeds alumina to a pneumatic conveyor. It also delivers cleaned, cooled gas at approximately 1 atm. to acid recovery. The major equipment included in this area are:

- ACH Roll Crusher
- ACH Surge Bin
- ACH Weigh Feeder
- Venturi (Flash) heater to dry ACH crystals with calciner off-gas
- Calciner Off-Gas Cooler
- Recycle HCl Gas Blower
- Decomposer Off-Gas Cooler
- Decomposer Off-Gas Electrostatic Precipitator
- ACH Decomposer consisting of fluid bed reactor, dust recovery cyclones and internal heat exchanger
- Molten Salt Pumps

- Molten Salt Heater
- ACH Calciner including fluid bed reactor, holding time reactor and dust recovery cyclones
- Product fluid bed cooler
- Screw Conveyors

The design details of the Thermal Decomposition of ACH are based on a budget type quotation from Lurgi Chemie und Huettentechnik GmbH, for proprietary processes patented by Aluminum Pechiney (U.S. Pat. No. 4080437 and 4091085) and operated successfully in a pilot plant in France.

Since the design is proprietary, the processing details cannot be verified with information available at this time. The data and equipment have therefore been provided based on engineering judgment. The entrance and exit conditions are fixed by the process for this pilot plant.

Description

The first stage reactor is indirectly heated by molten salt to between 400°F and 750°F and accomplishes about 90% of the decomposition. The second stage reactor is directly fired with fuel oil to between 1600°F and 2000°F.

Crushed ACH at approximately 160°F is metered from a surge bin to a venturi (or flash) heater where it is pre-dried by direct contact with calciner off-gas at about 1800°F. The venturi heater discharges into two high efficiency cyclones in series. The gas leaving the cyclones is cooled in a heat exchanger to between 225° and 300°F and sent to acid recovery. The dry ACH flows into the indirectly fired fluid bed reactor. This reactor contains a corrosion resistant heating coil. Heat is provided by an oil-fired molten salt circulating system. The indirect fired decomposer off-gas passes through a cyclone and an electrostatic precipitator for solids removal and then to a heat exchanger to be cooled to between 225° and 300°F. Approximately 25% of this cooled gas is recycled back to the decomposer by a blower to maintain fluidization of the ACH. Approximately 1944 SCFM of a gas containing 53% HCl are sent to acid recovery. This gas must not be cooled to a temperature which will permit condensation of acid.

The partially decomposed solids are transferred to the direct fired ACH calciner. It is located alongside the direct fired calciner cyclone No. 1 which acts as a holding reactor where the final conversion to alumina takes place at approximately 1,800°F.

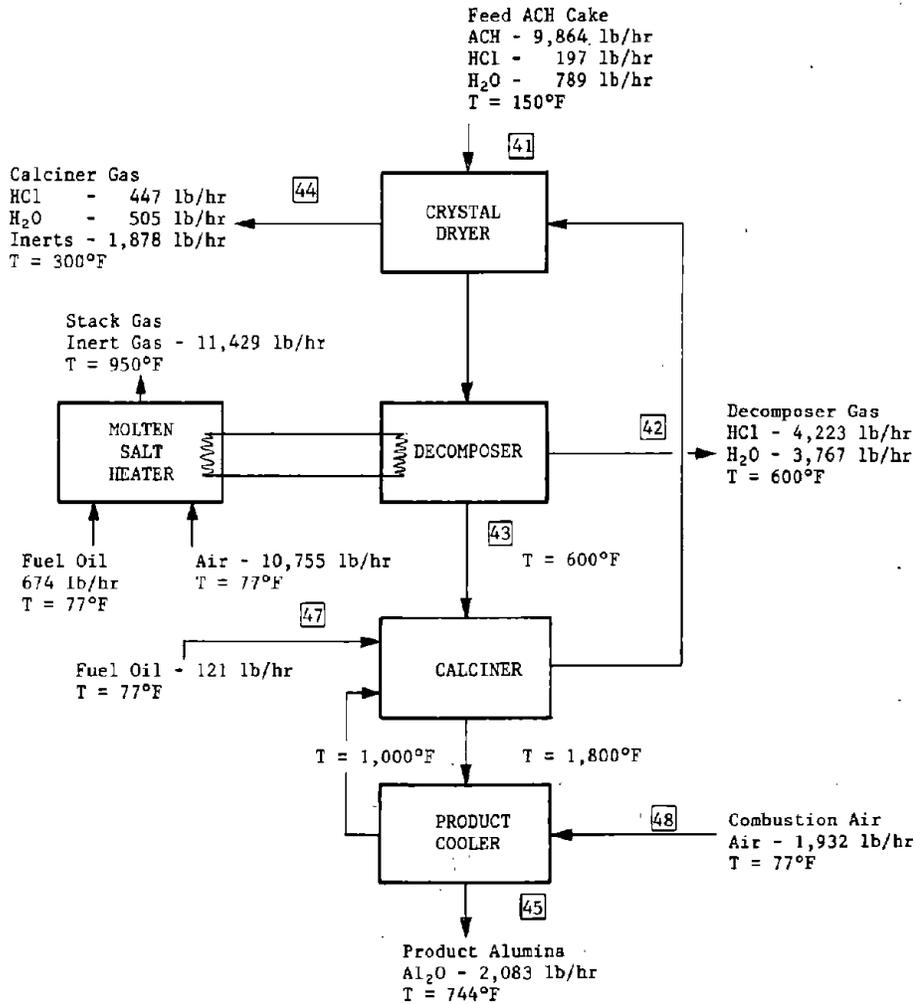
The product alumina at 2,083 lbs/hr is cooled to 150°F in a fluid bed cooler in which both combustion air and water are used to effect cooling.

The ACH decomposition system is expected to be equipped with temperature and pressure instrumentation at each transfer point between individual pieces of equipment and several points within individual pieces. Combustion control instrumentation is required for both burners. Substantial data will be recorded in the data logger.

ACH DECOMPOSITION AND CALCINATION
HEAT AND MATERIAL BALANCE

3.2.8.2

HEAT IN			HEAT OUT		
Stream		Btu/hr	Stream		Btu/hr
[41] ACH Sensible Heat		200,000	Decomposer Section		
Molten Salt Heater			Reaction		7,910,000
Fuel Oil		12,720,000	Vaporization		490,000
[47] Calciner Fuel Oil		2,280,000	Product Heat		290,000
[48] Combustion Air		-0-	[42] Decomposer Gas Heat		580,000
			Radiation Loss		720,000
			Molten Salt Heater		
			Stack Gas Heat		2,930,000
			Calcination Section		
			Reaction		880,000
			[45] Product Heat		780,000
			[44] Calciner Gas Heat		470,000
			Radiation Loss		150,000
			Total		15,200,000
Total		15,200,000	Total		15,200,000



Base Temp = 77°F

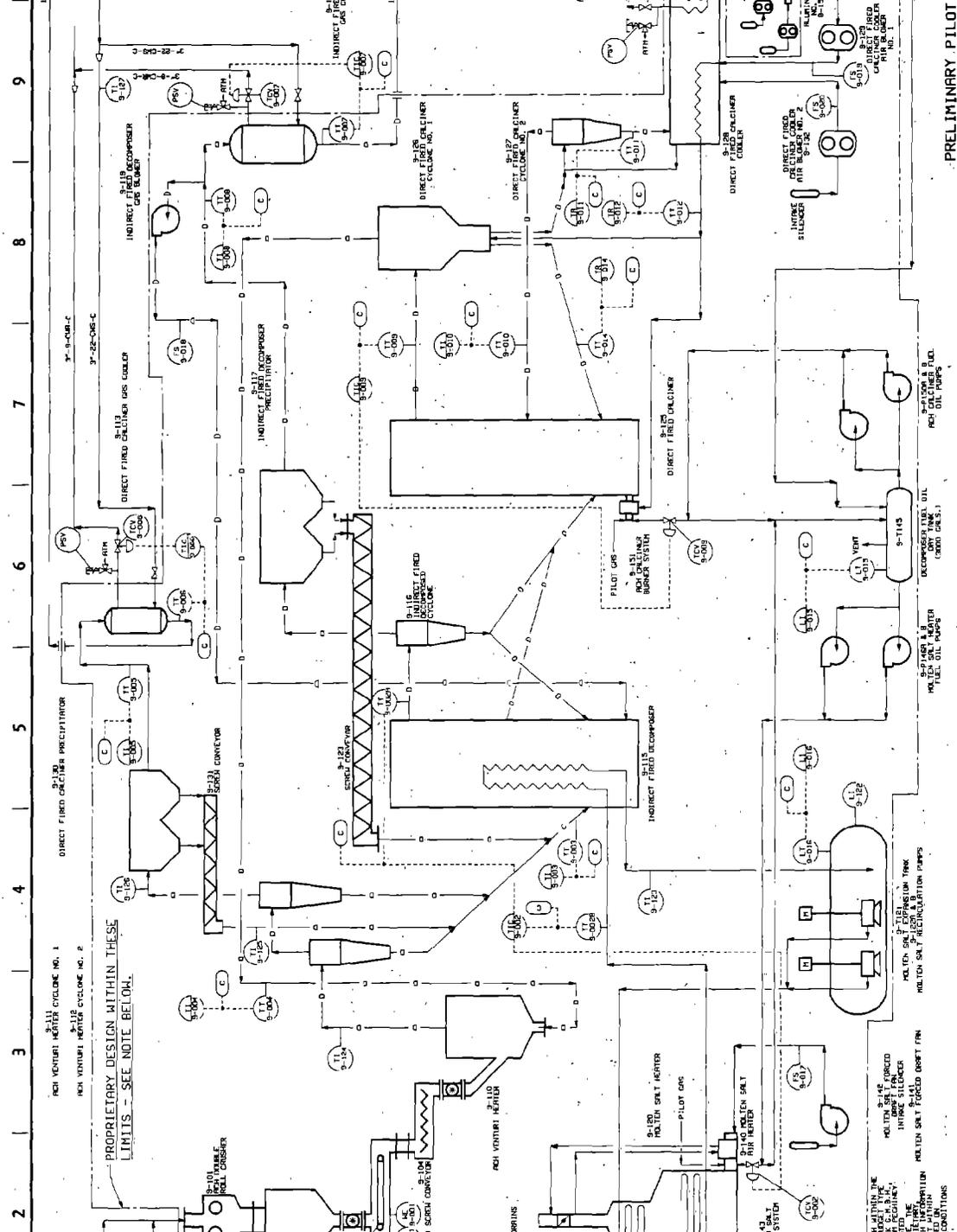
Fuel Oil Heat Value = 18,875 Btu/lb

Note: Stream numbers where shown correspond to stream numbers on Dwg 50-302-G, Block Flow Diagram

TO VENT HEADER 4'-3-1/2" H.C.F. TO 10-101-P TO 13-112-P TO 13-100-P

TO VENT HEADER 4'-3-1/2" H.C.F. TO 10-101-P TO 13-112-P TO 13-100-P

TO VENT HEADER 4'-3-1/2" H.C.F. TO 10-101-P TO 13-112-P TO 13-100-P



PRELIMINARY PILOT PLANT DESIGN

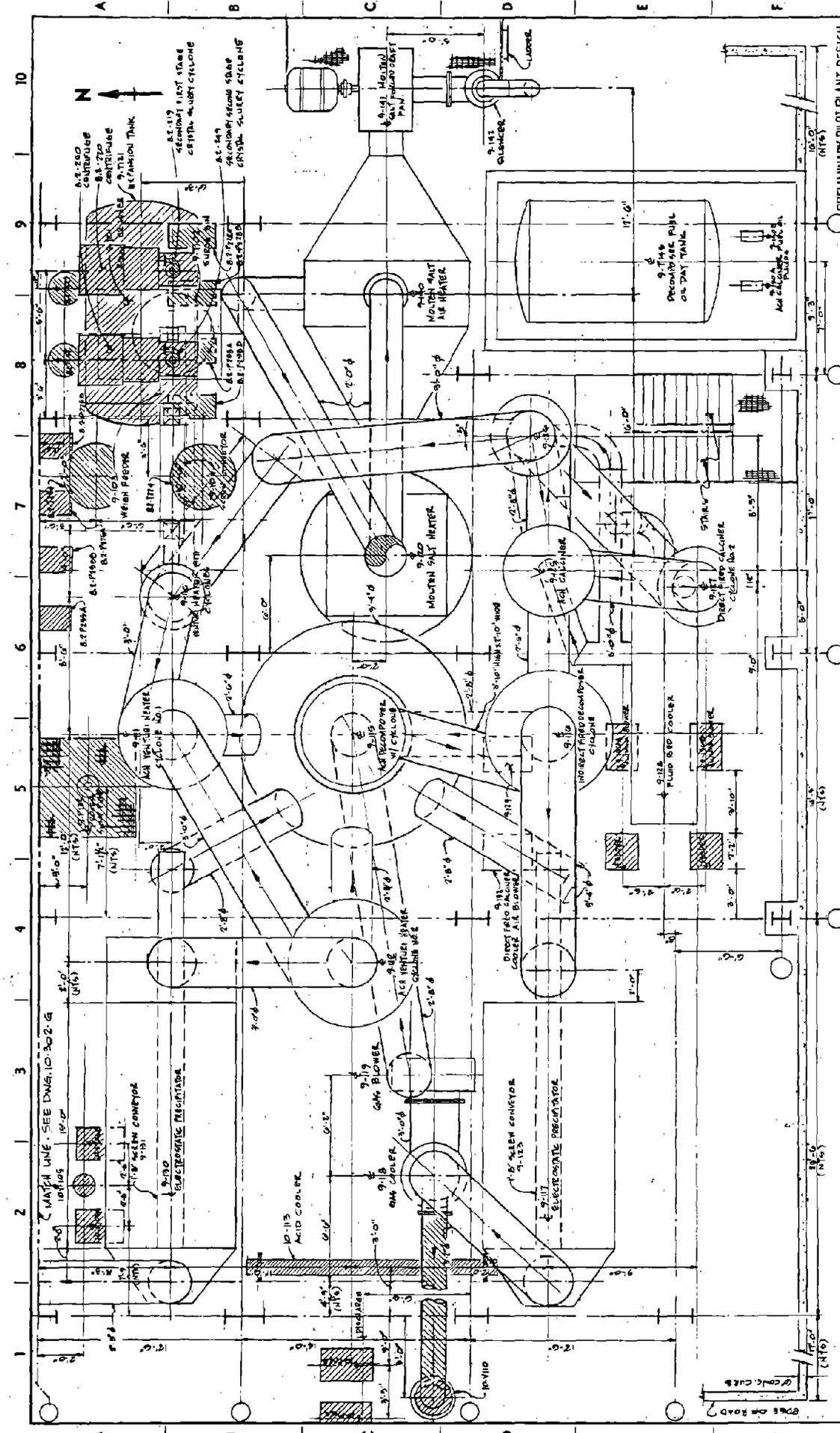
KAISER ENGINEERS

U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 22094

RECH DECOMPOSITION

PIPING & INSTRUMENT DESIGN

FDG No. 76161-003 DWG. No. 9-200-P R-0



3.2.9 Acid Recovery and Preparation - Area 10 - Dwg. Nos. 10-100-P & 10-101-P

3.2.9.1 Process Description

Summary

The Acid Preparation and Acid Recovery area receives vapors from the indirect and direct fired decomposers and produces HCl vapor in excess of 95 weight percent for the crystallizers as well as a 20 weight percent HCl liquor for centrifugation wash and dissolution. Leach acid is also prepared by blending primary crystallization mother liquor with several dilute acid streams to produce 25% leach acid.

HCl vapor generated in this area is combined with the vapors from the main ACH dissolver and the bleed stream stripper to satisfy the total HCl vapor demand at the crystallizers.

In addition to the normal operating mode, Acid Preparation and Acid Recovery will be designed to handle two abnormal operating conditions.

First, when the decomposer is out of service or the bleed stream stripper is out of service, 95 weight percent HCl vapor production will be increased in the HCl stripper to satisfy the normal HCl vapor demand at the crystallizers. Second, when the crystallizers are out of service (this requires the dissolver to be out of service also) vapors from the indirect fired decomposer can be condensed totally.

Discussion

There are four sections in Acid Preparation and Acid Recovery - 35% acid absorber, dilute acid absorber, HCl stripper, and venturi scrubber.

35% Acid Absorber

The 53 weight % HCl vapors from the indirect fired decomposer enter the top of the falling film absorber where they are condensed to produce 35 weight % HCl acid, and part of the 95 weight % HCl vapor supplied to the crystallizers. Both streams exit the bottom of the absorber at 140°F. The 35% acid stream is cooled to 105°F by the acid cooler. This column operates at atmospheric pressure with a blower boosting the outlet gas pressure to 17.7 psia, the header pressure for 95% HCl vapor.

When the crystallizers are out of service (the dissolver is also out of service), 20 weight % HCl is sent to the top of the 35% absorber from storage condensing at least 90% of the vapor as 35 weight % HCl. The excess uncondensed vapors are sent to the venturi scrubber. This abnormal operating condition is the design condition for the 35% Acid Absorber equipment and the venturi scrubber equipment.

Dilute HCl Absorber

The direct fired decomposer vapors contain inert combustion gases along with HCl and water. These vapors enter the top of the falling film

absorber along with some bottoms recycle and cleanup tails tower bottoms liquid. Approximately 74% of the HCl is absorbed in this absorber. The remaining vapors enter the bottom of the cleanup tails tower where water or very dilute acid is fed to the top. This countercurrent scrubbing of the vapors reduces the HCl content of the final vent gas to less than 5 ppm. The liquor leaving the falling film absorber is approximately 31 weight % HCl and is pumped to the 35% absorber.

HCl Stripper

When the decomposer or bleed stream stripper is out of service, this sieve tray stripper is used to generate vapors in excess of 95 weight % HCl for the main crystallizers. A by-product 20 weight % HCl is also produced and sent to the 20 weight % acid storage. This unit is designed to strip 31 or 35 weight % feed acid.

The feed is preheated with stripper bottoms and fed to the top tray of the stripper. A natural circulating reboiler with 50 psig steam on the shell side is used to generate the column vapors. The vapors from the top of the stripper enter the overhead condenser producing some 35 weight % HCl reflux to maintain a constant vapor composition of 95 weight % HCl.

Theoretically the overhead condenser heat duty is zero since vapor in equilibrium with the 35 weight % feed is in excess of 95 weight % HCl. The overhead condenser is used primarily to smooth out column upsets and to maintain 95% HCl vapor when 31 weight % feed is used.

When feeding 35% HCl the preheater will heat the feed to a temperature above the atmospheric boiling point. Therefore, some flashing will occur across the pressure control valve.

The head on the feed pump will be high enough to prohibit flashing in the preheater itself.

Venturi Scrubber

If the pressure in the HCl gas header gets too high (this can occur when the crystallizer demand is reduced quickly) the vapors are dumped automatically to the venturi scrubber. Liquor is continuously recirculated to the venturi through an external cooler. When the recirculating liquor HCl concentration is 20%, it is sent to the 20% acid storage tank. Fresh water is added to maintain a constant liquor inventory in the venturi.

When the crystallizers are out of service, 90% of the indirect fired decomposer vapors are condensed in the 35% absorber. When operating in this mode, the venturi scrubber is used to condense the remaining 10% of the vapors. (The venturi scrubber system is designed to condense 20% of the indirect fired decomposer vapors.)

Leach Acid Preparation

Leach acid is prepared by combining mother liquor from primary crystallization with the acid condensed from the flash cooler in leaching and the acid condensed in the evaporator partial condenser. This is effected by combining liquid streams entering the two 60,000 gallon leach acid storage tanks.

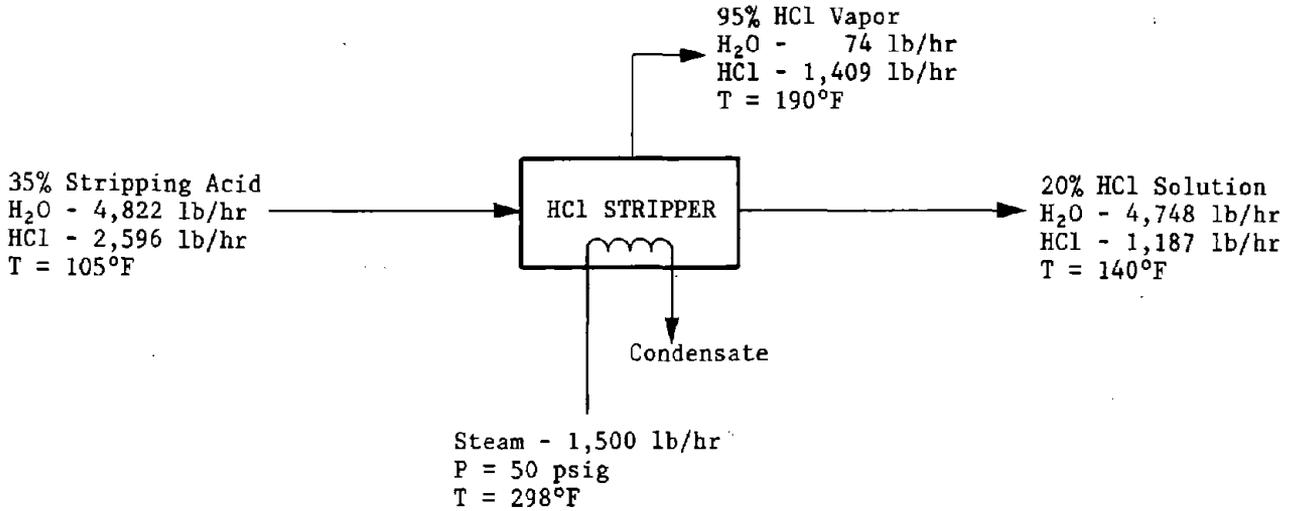
HEAT AND MATERIAL BALANCE

ACID RECOVERY - HCl STRIPPER

3.2.9.2

HEAT IN		HEAT OUT	
Stream	Btu/hr	Stream	Btu/hr
35% Stripping Acid	133,000	95% HCl Vapor	
Steam, 50 psig	1,769,000	Sensible Heat	60,000
Total	1,902,000	Desorbing Heat*	1,210,000
		20% HCl Solution	233,000
		Condensate	399,000
		Total	1,902,000

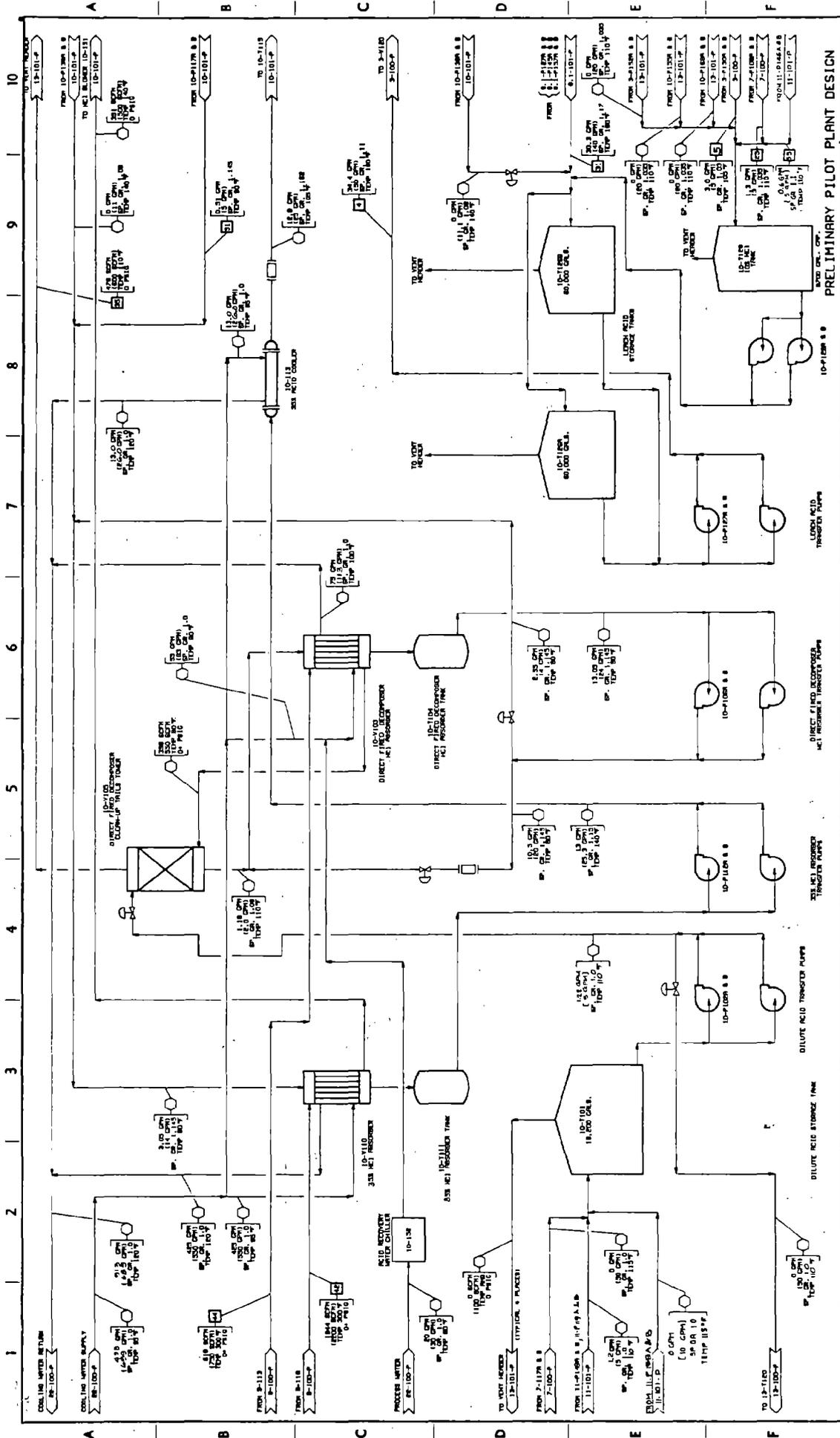
*Include heat of vaporization of 74 lb/hr H₂O



Base Temp = 77°F

Steam Base Temp = 32°F

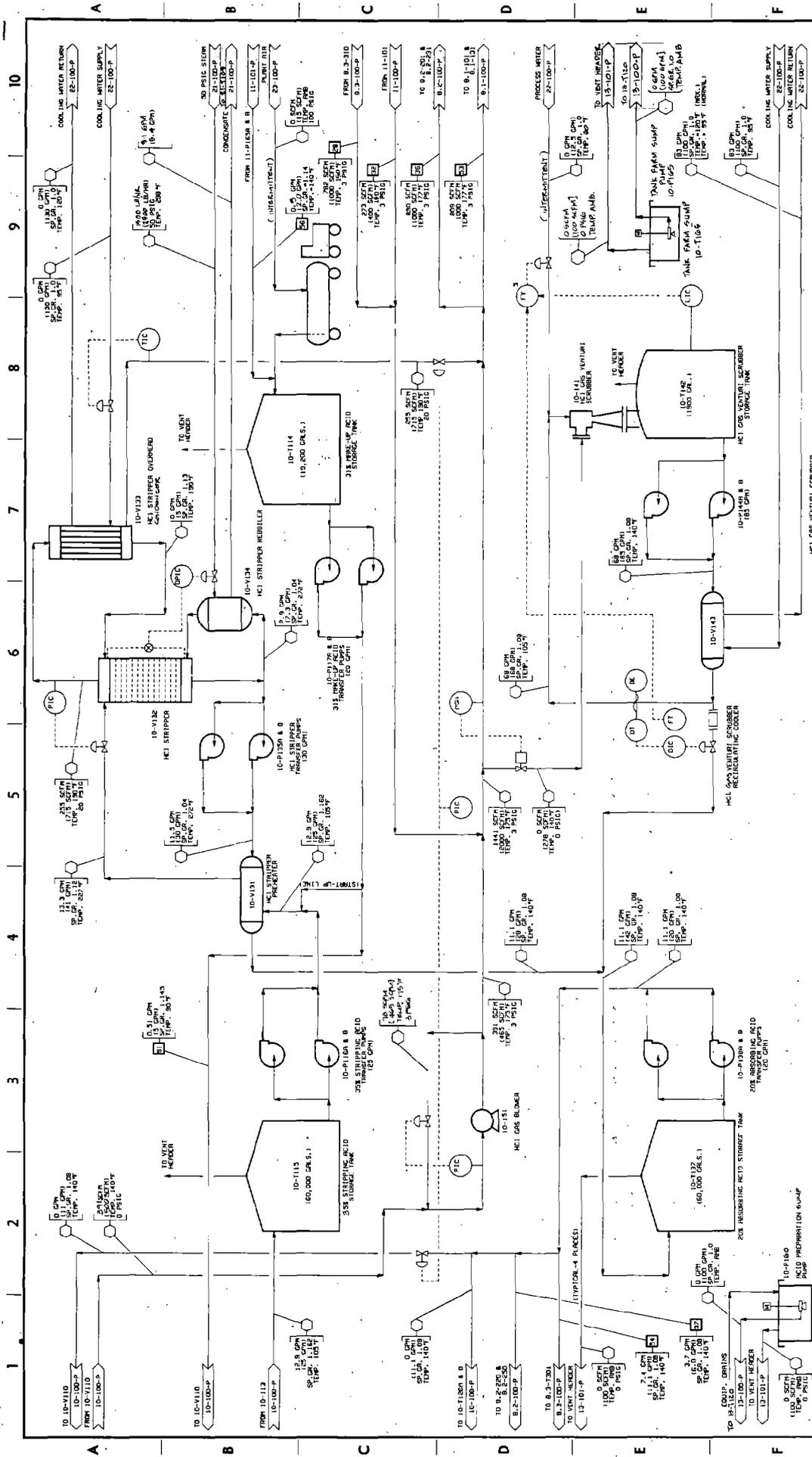
Note: Stream numbers where shown correspond to stream numbers on Dwg 50-302-G,
Block Flow Diagram



NO.	DATE	ISSUED FOR REPORT	REVISION	NOTES	CONTRACTOR APPROVAL	APPROVAL	SCALE	ASPECT	DATE
1									
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KAISER
 PRELIMINARY PILOT PLANT DESIGN
 ALUMINA RECOVERY AND PRELIMINARY PILOT PLANT DESIGN
 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 48308
 HCl RECOVERY AND PREPARATION
 PROCESS FLOW DIAGRAM
 JOB NO. 76161-003 DWG. NO. 10-100-P 11

111



PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS

U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINERAL INDUSTRIES
 ACID RECOVERY AND PREPARATION
 PROCESS FLOW DIAGRAM

NO.	DATE	SCALE	APPROVAL	DESCRIPTION
1	7-27-79	AS SHOWN	DESIGNED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
2	7-27-79	AS SHOWN	CHECKED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
3	7-27-79	AS SHOWN	APPROVED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM

PROFESSIONAL SEAL

NO.	DATE	SCALE	APPROVAL	DESCRIPTION
1	7-27-79	AS SHOWN	DESIGNED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
2	7-27-79	AS SHOWN	CHECKED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
3	7-27-79	AS SHOWN	APPROVED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM

CONSTRUCTION APPROVAL

NO.	DATE	SCALE	APPROVAL	DESCRIPTION
1	7-27-79	AS SHOWN	DESIGNED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
2	7-27-79	AS SHOWN	CHECKED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
3	7-27-79	AS SHOWN	APPROVED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM

CONSTRUCTION APPROVAL

NO.	DATE	SCALE	APPROVAL	DESCRIPTION
1	7-27-79	AS SHOWN	DESIGNED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
2	7-27-79	AS SHOWN	CHECKED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
3	7-27-79	AS SHOWN	APPROVED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM

CONSTRUCTION APPROVAL

NO.	DATE	SCALE	APPROVAL	DESCRIPTION
1	7-27-79	AS SHOWN	DESIGNED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
2	7-27-79	AS SHOWN	CHECKED BY: [Signature]	ACID RECOVERY AND PREPARATION PROCESS FLOW DIAGRAM
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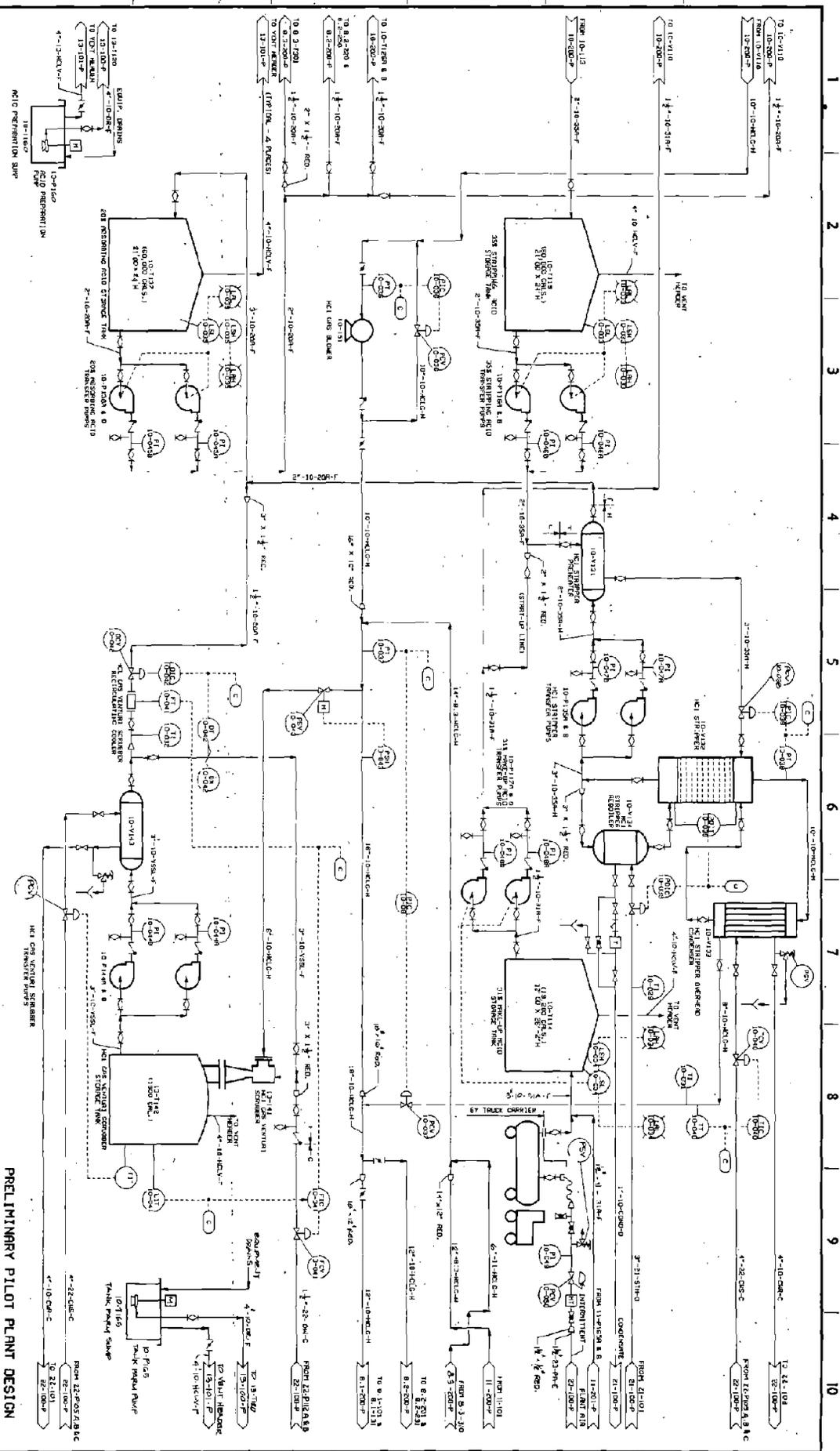
CONSTRUCTION APPROVAL

ISSUED FOR REPORT

REVISION

10-101-P

7/27/79



PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERING
 CONSULTING AND ENGINEERING FIRM
 1500 BROADWAY, SUITE 1000
 SAN FRANCISCO, CALIFORNIA 94111
 TEL. (415) 774-2000

PROJECT: ACID RECOVERY AND PREPARATION
 PIPING & INSTRUMENT DIMENSION

JOB NO. 76161-003 DWG. NO. 10-201-P

NO.	DESCRIPTION	DATE	BY	CHKD.	APP'D.
1	ISSUED FOR CONSTRUCTION	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
2	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
3	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
4	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
5	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
6	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
7	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
8	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
9	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS
10	REVISED	10/15/68	J. W. HARRIS	J. W. HARRIS	J. W. HARRIS

3.2.10 Bleed Stream Treatment - Area 11 - Dwg. Nos. 11-100-P & 11-101-P

3.2.10.1 Process Description

Summary

The Bleed Stream Treatment Section accepts aqueous streams containing dissolved $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (ACH), HCl, and all of the minor constituent acid-soluble impurities from the alumina-bearing raw material. Bleed stream treatment performs the following functions:

- Recovery of nearly all of the dissolved HCl in the bleed stream as gas containing only a small amount of H_2O .
- Recovery of a small fraction of the entering HCl as approximately 10% liquid acid.
- Recovery of a fraction of the residual dissolved ACH in the bleed stream as a crystalline product for return to the main liquor stream.
- Recovery as 20-30% hydrochloric acid of the chloride values contained in the dissolved NaCl, KCl, MgCl_2 , metal chlorides present in trace amounts, and unrecovered ACH.
- Conversion of the dissolved impurities into an anhydrous, solid, oxide-sulfate-phosphate mixture suitable for waste disposal.
- Rejection of H_2O from the primary process liquor stream.

Water is rejected in two places: evaporation and bleed stream treatment.

The cost of this does not vary regardless of where it is performed. An incentive therefore exists to operate the process taking a fairly large bleed stream from the primary crystallizer mother liquor, because doing so will hold down the concentration of soluble impurities recirculating through the first crystallization at little or no economic penalty.

Bleed Stream Treatment

Bleed liquor, upon entering this process section, is mixed with recirculating ACH crystals in the mixing tank and pumped as a slurry to the top of the HCl stripper containing the equivalent of 10 theoretical stages. The stripper operates on the same principles and in the same manner as the dissolver specified between the primary and secondary crystallizations of ACH.

The bleed stream-ACH crystal slurry entering the top of the stripper carries approximately half of the total ACH required to convert the bleed stream (after stripping out free HCl) to a 30% AlCl_3 solution. Descending liquid from a tray is withdrawn at the appropriate point, for

slurrying the remainder of the ACH. The ACH recovered by filtering the crystal slurries produced in the bleed stream evaporative crystallizers completes the stripping operation. The resulting slurry is returned at about 20% suspended solids, to the column on the tray next below the one from which the descending liquid was withdrawn.

Liquid from the bottom tray circulates to a thermosiphon reboiler where it is heated indirectly by steam and then recirculated back to the bottom of the column. The heated liquid, generating vapor containing sufficient heat to provide for the desorption of the free HCl in liquid entering the top of the column, also provides for the removal of the reboiler liquid product at about 260°F. This operating temperature permits the delivery of HCl gas from the top of the column at one atmosphere. The stripper will be operated near saturation with respect to AlCl_3 in order to achieve the desired separation of HCl and H_2O .

Bleed stream liquor is flow controlled to the first mixing tank along with the ACH crystals discharged from the first stage crystallizer belt filter. The effluent from this tank is fed to the stripper using level control on the mixing tank. The density of the mixing tank effluent is measured and controlled to maintain approximately a 20 weight % solids slurry by cascading to the bleed stream liquor flow controller.

Liquor is withdrawn from an intermediate stage in the stripper to a second mixing tank along with the ACH crystals discharged from the second stage crystallizer belt filter. The effluent from this tank is fed back to the next stage in the stripper using level control on the mixing tank. The liquor withdrawal rate from the stripper is on flow control. This rate is adjusted to maintain a maximum solids slurry concentration of 20 weight % in the mixing tank. Steam is fed to the reboiler to maintain the overhead column temperature which is an indirect measure of the HCl concentration in the vapor.

The liquor level in the bottom of the column is controlled by adjusting the rate of flow to the first stage crystallizer.

The materials of construction for heat transfer surfaces include titanium and glass.

The reboiler liquid product is a 30% solution of AlCl_3 containing the impurities present in the bleed stream plus small amounts of impurities included in the structure of crystals formed in the two stages of evaporative crystallization together with impurities present in mother liquor adhering to these crystals delivered by the filters. The total amount of impurities returned to the dissolver with the recycling crystals is small; most of the impurities remain dissolved in the mother liquor and are finally rejected to waste calcination.

The first stage evaporative crystallizer operates with a slight increase in the ratio of the concentration of these impurities to dissolved ACH compared with this ratio in the primary liquor. This unit therefore operates with essentially the same boiling point rise as in the final

stage of primary liquor concentration. Operation at reduced pressure is specified in order to reduce any tendency to scaling of heat transfer surfaces, to reduce difficulties with materials of construction, and to demonstrate that operation is possible at a low enough temperature to permit the eventual utilization of waste heat.

The second stage evaporative crystallizer, yielding ACH only for recycle and a mother liquor to be sent to chloride value recovery, will operate at a substantially higher dissolved impurities/ACH ratio and therefore at a slightly higher temperature when operated at the same reduced pressure as the first stage crystallizer. Crystals formed in the second stage crystallizer are washed with mother liquor from the first stage crystallizer before this liquor enters the second stage crystallizer in order to reduce the cake moisture impurity content of crystals from the second stage filter to a level approaching correspondence to that of liquor present in the first stage crystallizer.

Vapors from the two crystallizers will contain a small amount of HCl. These vapors are passed through a packed absorber irrigated countercurrently with H₂O to recover this HCl as 10% acid. The packing in the absorber is capable of satisfactory operation at the low L/V ratio corresponding to recovery as 10% liquid acid of the small fraction of HCl present.

Water vapor containing no more HCl than would produce a liquid of pH > 5 upon condensation, exits from the top of the absorber, passing to a two-stage direct contact barometric condenser supplied with H₂O recirculated from a cooling tower. Warmed H₂O from the bottom of the condenser returns to the cooling tower. Vacuum jets are provided from the top of the condenser for the removal of small amounts of inert gas.

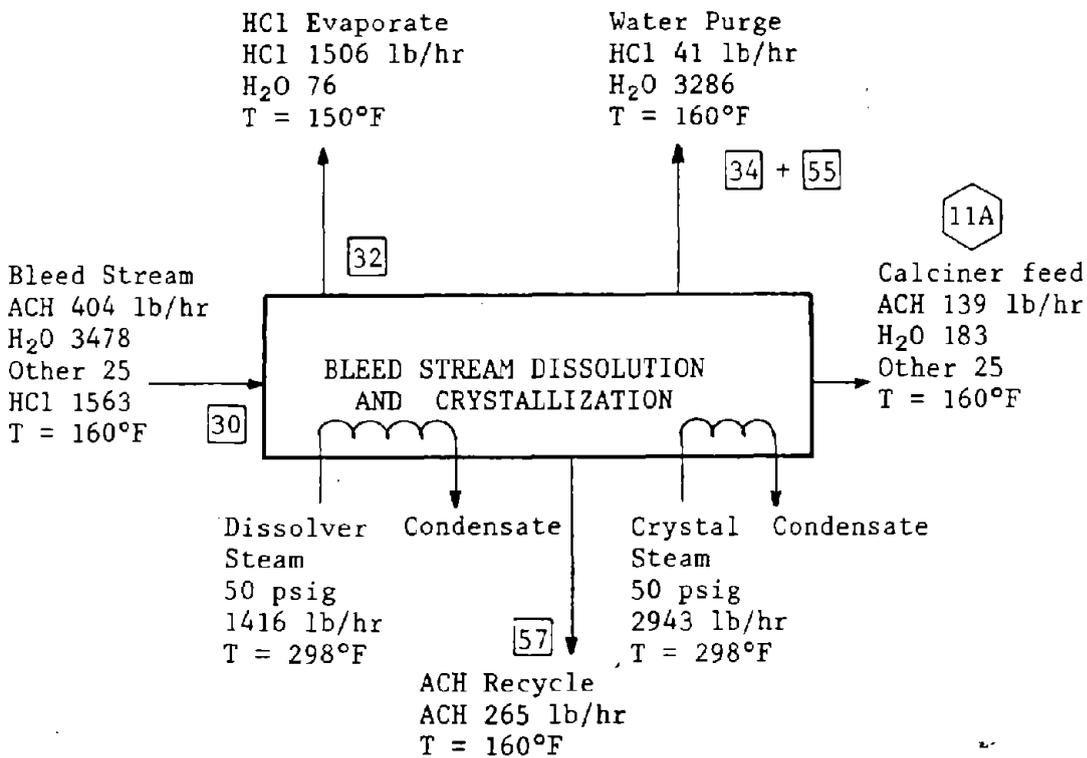
Primary control parameters for operation of the two crystallizers are liquor densities, slurry densities, pressure within the units, and temperature. Materials of construction requirements are the same as for the dissolver.

Liquid waste is contacted in a packed scrubber with fluid bed calciner exhaust gases in order to utilize waste heat contained in these gases. Moisture-laden HCl-containing gases from the packed scrubber pass to a calciner absorber for the recovery of hydrochloric acid and the rejection of the remaining H₂O vapor along with combustion product gases.

Liquid from the packed scrubber will be sprayed into a fluid bed calciner. The liquid coats existing particles in the bed causing them generally to grow in size. The H₂O content of the liquid waste is entirely evaporated. SO₂ gas is injected into the fuel oil fired calciner. The pyrohydrolysis of hydrated AlCl₃ to Al₂O₃ goes largely to completion, and the SO₂ is removed by reaction with metal chlorides. A granular solid will overflow and discharge to the calciner cooler and into portable waste disposal bins.

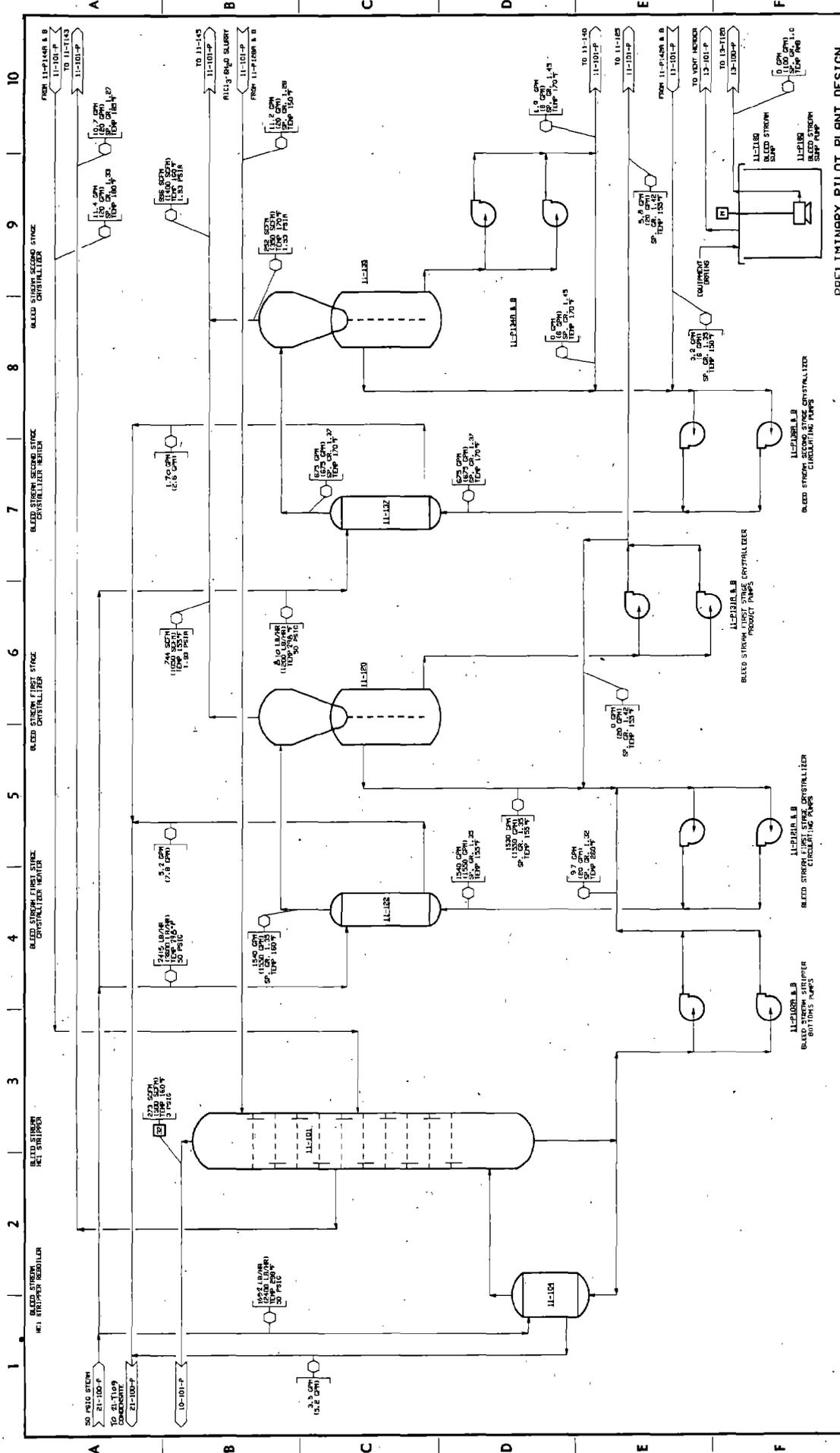
3.2.10.2 Bleed Stream Treatment Dissolution and Crystallization
Heat and Material Balance

<u>HEAT IN</u>		<u>HEAT OUT</u>	
<u>Stream</u>	<u>Btu/hr</u>	<u>Stream</u>	<u>Btu/hr</u>
[30] Bleed Stream	532,000	[32] HCl Evaporate	994,000
Dissolver Stream	1,670,000	[34] & [55] HCl/H ₂ O vapor	3,481,000
Crystallization Stream	3,470,000	Calciner Feed	25,000
		[57] ACH Recycle Condensate	6,000
			1,166,000
Total	5,672,000	Total	5,672,000



Base Temp = 32°F

- Note: 1) Stream numbers where shown correspond to stream numbers on Dwg 50-302-G Block Flow Diagram.
- 2) Not shown above, the fuel oil needed to calcine the Bleed Stream residue is 4.3 GPH (0.6 x 10⁶ BTU/T of Alumina).



NO. DATE		REVISION	ISSUED FOR	REPORT	NOTES	DESCRIPTION	COST ACCOUNT	CONSTRUCTION APPROVAL	APPROVAL	DATE	SCALE	NO./P	DATE	PRODUCTION SQA
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2														
3														
4														
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6														
7														
8														
9														
10														

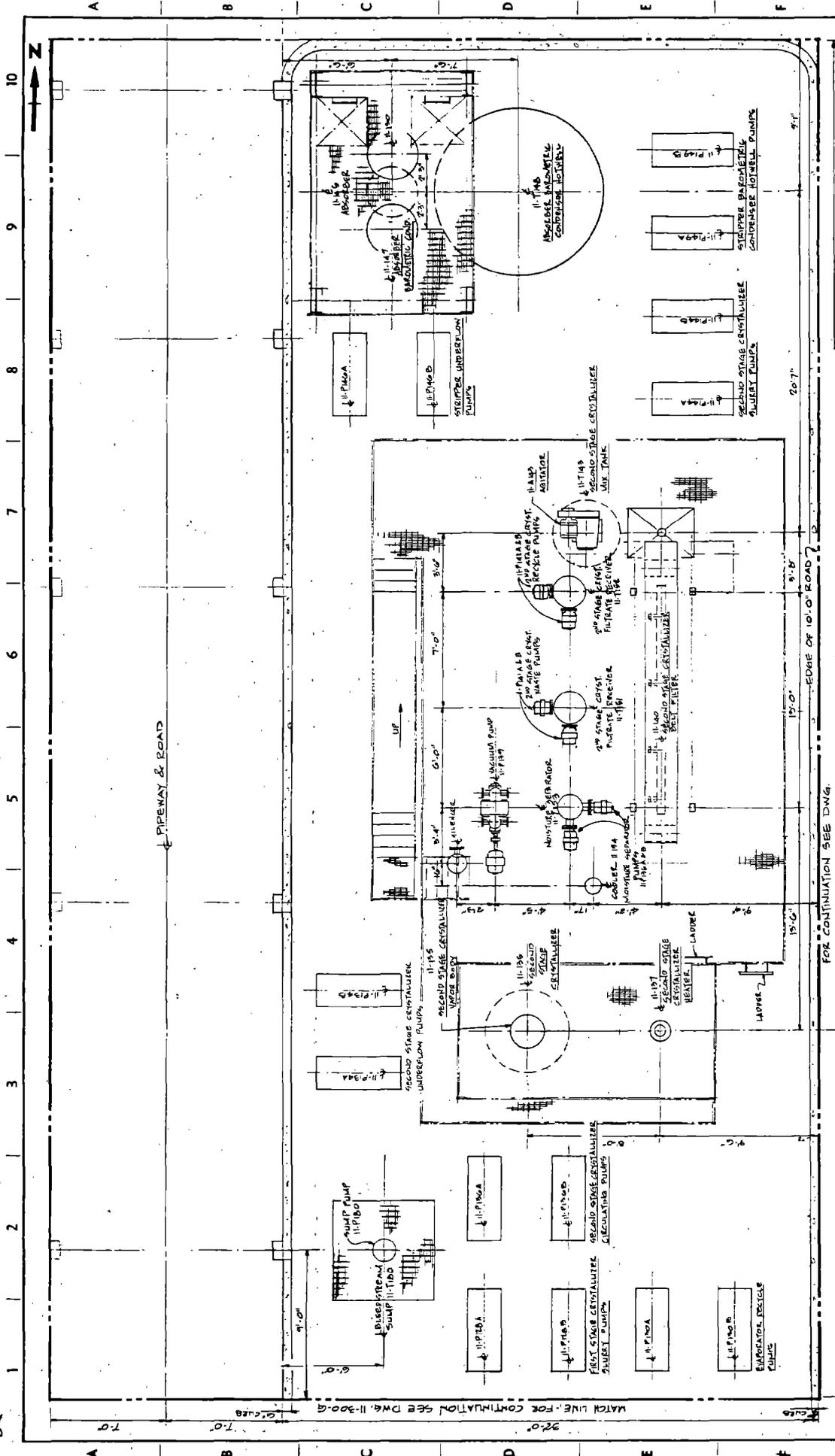
KAISER ENGINEERS
 A DIVISION OF THE UNITED STATES STEEL CORPORATION
 U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES
 CONTRACT NO. 48-01-00000-01

**BLEED STREAM TREATMENT
 PROCESS FLOW DIAGRAM**

JOB No. 76161003 DWG. No. 11-100-P

PRELIMINARY PILOT PLANT DESIGN

821



ISSUED FOR REPORT		NOTES		CONSTRUCTION APPROVAL		DATE		SCALE		DATE	
NO.	DATE	BY	REVISION	DESCRIPTION	COST	ACCOUNT	APPROVAL	DATE	SCALE	DATE	SCALE
1	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"
2	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"
3	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"
4	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"
5	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"
6	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"
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10	11-10-58	W. J. LARSEN							1/4" = 1'-0"	11-10-58	1/4" = 1'-0"

PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
ALUMINA PROCESS FACILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN
U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 34088

AREA 11
SULEID STRAUM
GENERAL ARRANGEMENT PLAN

JOB No. 76161003 DWG. No. 11-301-G

3.2.11 Alumina Storage and Load Out - Area 12

3.2.11.1 Process Description

Summary

The alumina storage and loadout section of the demonstration plant is designed to receive, store, bag and loadout product alumina having a bulk density of 30 lbs/ft³. The alumina is conveyed by two pneumatic conveyor systems to either of two 75 ton capacity alumina storage bins. Each of the storage bins can feed either a bulk truck or a portable bagging machine. The bins have a bulk load out system with a capacity of 50 tons per hour. The bagging machine is capable of bagging seven 50-lb bags per minute. The bagging and palletizing section is designed to operate 8 hours per day, 5 days per week.

Alumina Bulk Storage

Product alumina is conveyed by two pneumatic conveyors each with a capacity of 5 tons per hour. Each (3 inch schedule 40) positive pressure system is designed to convey alumina weighing 30 lbs/ft³. A rotary valve feeds the material to the pneumatic conveyor and the 310 ACFM blower, complete with required accessories, conveys the alumina to the bins. Alumina dust is filtered by a self-contained bin vent with fan and the dust is returned to the storage bin. Each alumina bin is 16 ft in diameter, 22.5 ft straight side and 60° cone bottom with a capacity of 75 tons. At the bottom discharge of each bin is a rotary valve with a capacity of 50 tons per hour. The rotary valve can discharge either to a bulk truck or to the alumina bagger. The alumina bins will store up to 6 days of alumina production from the alumina calcination area.

Alumina Bagging and Palletizing

Alumina from either of the bins feeds by gravity to the portable alumina packer. The alumina packer is a unitized bagging machine. It consists of a variable speed sealing conveyor, sealing pedestal, sealing head, bagging scale and scale pedestal complete with all motors, drives and controls for one-man operation mounted on one common frame. The unit is height adjustable. The bagging scale has a flexible connector to the bin discharge chute and is dust tight. The alumina drops into the automatic scale and is metered into the bag. Once filled, the bag drops onto a belt conveyor and is moved to the sealing section. The operator puts another bag on the automatic scale and a new weighing cycle starts. The operator then places the bag opening on the sealing head to close the bag. The bagging machine is capable of bagging up to seven 50-lb bags per minute.

A vacuum type bin dust collector will collect dust from the alumina packing area, and the bulk load out area and convey the dust back to the alumina storage bin.

The size of the bags is 26" x 17½" x 8" and will be palletized in a 5 bag pattern, 30 bags per pallet on 52" x 44" pallets. The pallets will be transported to storage by a motorized forklift truck and will be triple tiered.

The bagged alumina storage and loadout building has a covered area of 75 ft x 140 ft and can store 560 pallets of bagged alumina. This storage capacity is equivalent to about 17 days of alumina production.

3.2.12 Waste Disposal - Area 13 - Dwg. Nos. 13-100-P & 13-101-P

3.2.12.1 System Description

Summary

The waste disposal section of the demonstration plant is designed to receive, neutralize and dispose of liquid wastes and to receive, and store solid wastes. Wastewater primarily from pump seals and cleanup/wash-downs are collected in sumps located throughout the process areas. The wastewater is pumped from the sumps to various wastewater headers. pH meters are located on the headers and in the event of a major spill the water from the header can be diverted to the emergency spill tank for recycle or for treatment prior to disposal into wastewater lagoon. Wastewater from the sumps is normally discharged directly to the wastewater lagoon where the effluent is carefully monitored.

Dilute acid is bled off intermittently through another header and is metered and discharged to the effluent mixing trough where it is neutralized with calcium hydroxide.

In the event of a major spill in the plant, the effluent can be diverted into a 0.9 million gallon emergency recycle pond where it is recycled back to the effluent mixing trough and neutralized for discharge to the 3.82 million gallon wastewater lagoon.

50% solids wastes from the secondary mud washer are brought to the 543,000 ft³. lined solids impoundment pond by dump trucks. Solid wastes from the bleed stream calciner system are also brought to this pond by truck.

A 260,000 ft³ lined drainage test pond is provided to accept a 12.5% solids slurry from the secondary mud washer reslurry tank and pump. This pond is equipped with a rain runoff sump and an underdrain sump.

A secondary purpose of this section is to collect vent gases from storage tanks and equipment, and scrubbing the vent gases from this equipment with contaminated water to absorb the HCl values of the vent gases. Three 3-stage vent scrubber systems have been provided for all of the process areas. Rainwater collected in sumps is pumped and discharged directly to the wastewater lagoon. The effluent from the wastewater lagoon is carefully monitored and recorded to provide data for environmental evaluation.

Description

Wastewater from equipment drains is collected in 16 sumps and is pumped in 4 headers for treatment or discharge to the wastewater lagoon which has a capacity of 3.82 million gallons covering an area of 1.48 acres. The lagoon is lined with a 30 mil reinforced hypalon liner and is 15 ft deep. Wastewater for the leaching section and the mud separation and washing section goes into one header and may be diverted to the

emergency spill tank or to the main header to discharge. Wastewater from the clay preparation and calcination section goes into another header and into the main discharge header. Wastewaters from the utility sumps, the solvent extraction sumps and the tank farm sump go into a third header and may be diverted to the emergency spill tank. Wastewaters from the alumina building sump, the acid recovery and preparation sump, the ACH decomposer sump, the bleed stream treatment sump, the primary and secondary crystallization sumps and the evaporation sump go into the fourth header, and may be diverted into the emergency spill tank. Flows from the process area sumps are intermittent and it is assumed that at the main header to discharge the normal flow is at a rate of approximately 600 gpm.

Intermittent bleed-off from the leach hotwell and the bleed stream barometric condenser hotwell to waste disposal is provided to prevent an excess of contaminated water buildup in the cooling water system. Intermittent bleed-off from the dilute acid storage tank is also provided to prevent the tank from overflowing. While bleed-off is being done, lime slurry is metered into the effluent mixing trough to neutralize the bleed-off stream before discharging into the wastewater lagoon.

Spills diverted into the 60,000 gallon rubber-lined emergency spill tank are brought back to the process by introducing the slurry or solution to the mud thickener. Contents of the emergency spill tank may alternately be neutralized before discharge to the wastewater lagoon. The slurry or solution is pumped to a limestone pit where it is 90% neutralized. The effluent flows by gravity to the effluent mixing trough where it is fully neutralized by the addition of lime slurry.

Clarified effluent from the wastewater lagoon may be recycled to the 60,000 gallon rubber-lined wastewater tank and is reused as process water and slurry water for the solids discharge from the secondary mud washer.

A 0.9 million gallon lined emergency recycle pond is provided in case of a major spill and process upset. The pond is lined with a 30 mil reinforced hypalon liner and is 15 ft deep. By closing the gate on the main discharge line to the wastewater lagoon at the mixing trough, effluent from the system will overflow from the mixing trough into the emergency recycle pond. Wastewater in the pond is recycled upstream of the effluent mixing trough where it is neutralized. A drain line and an overflow line from the emergency pond to the wastewater lagoon are provided for rain runoff.

A 40 TPH lime unloading system and 50 ton lime storage bin is included. The lime unloading system is a vacuum system consisting of a blower, a 4" pneumatic conveying system, silencers, filter and rotary valves. It is designed to unload from bulk trucks without pressurized blower systems. The 50 ton lime storage bin has a 60° hopper bottom and is equipped with a 5 ft diameter vibratory bin bottom and a weigh feeder. Lime is fed into the lime slurry mixing tank at 500 lb/hr for two hours. It is mixed with water and agitated. The lime slurry transfer pumps meter the lime into the mixing trough during intermittent bleed-off from the

various points listed earlier. The pumps will also run when spills are pumped from the various sumps and when the emergency spill tank contents or the emergency recycle pond contents are being fed to neutralization.

The solids impoundment pond has a volume of 543,000 ft³ and an area of 1.34 acres. It has a ramp on the inside to allow dump trucks to bring in 50% solid waste from the secondary horizontal belt mud washer and the dry solid waste from the bleed stream calciner. The pond is 15 ft deep and has a 20 ft deep sump for rain runoff. One side of the sump has a 4 ft wide opening and plates can be inserted in the opening to prevent the solids from draining into the sump. The plates are inserted as required depending on the solid level of the pond.

The drainage test pond (DTP) is designed to decant water and separate out liquor, drain the residue through collector pipes and evaporate water from the residue. The DTP has a volume of 260,000 ft³, covers an area of 0.75 acre and is 15 ft deep. Residue from the secondary mud washer is slurried in a tank to a 12.5% solids consistency and is pumped into the DTP at four peripheral points and a center point. Rainwater and supernatant liquor are collected by the rain runoff sump which has a 4 ft wide opening with plate inserts which act as weirs to prevent solids from entering the sump. The DTP is lined with a 30 mil reinforced hypalon liner. At the bottom of the DTP are 8 rows of 4" perforated PVC pipe installed in 1 ft wide trenches. The perforated pipes lie on a 3" layer of batture sand. The pipe is covered with filter sand up to 1 ft above the bottom of the pond. Drainage from the sand filter bed flows into the underdrain sump through this series of collector pipes. The sump is equipped to pump the collected waste either to the wastewater lagoon or to the effluent mixing trough for neutralization prior to discharge.

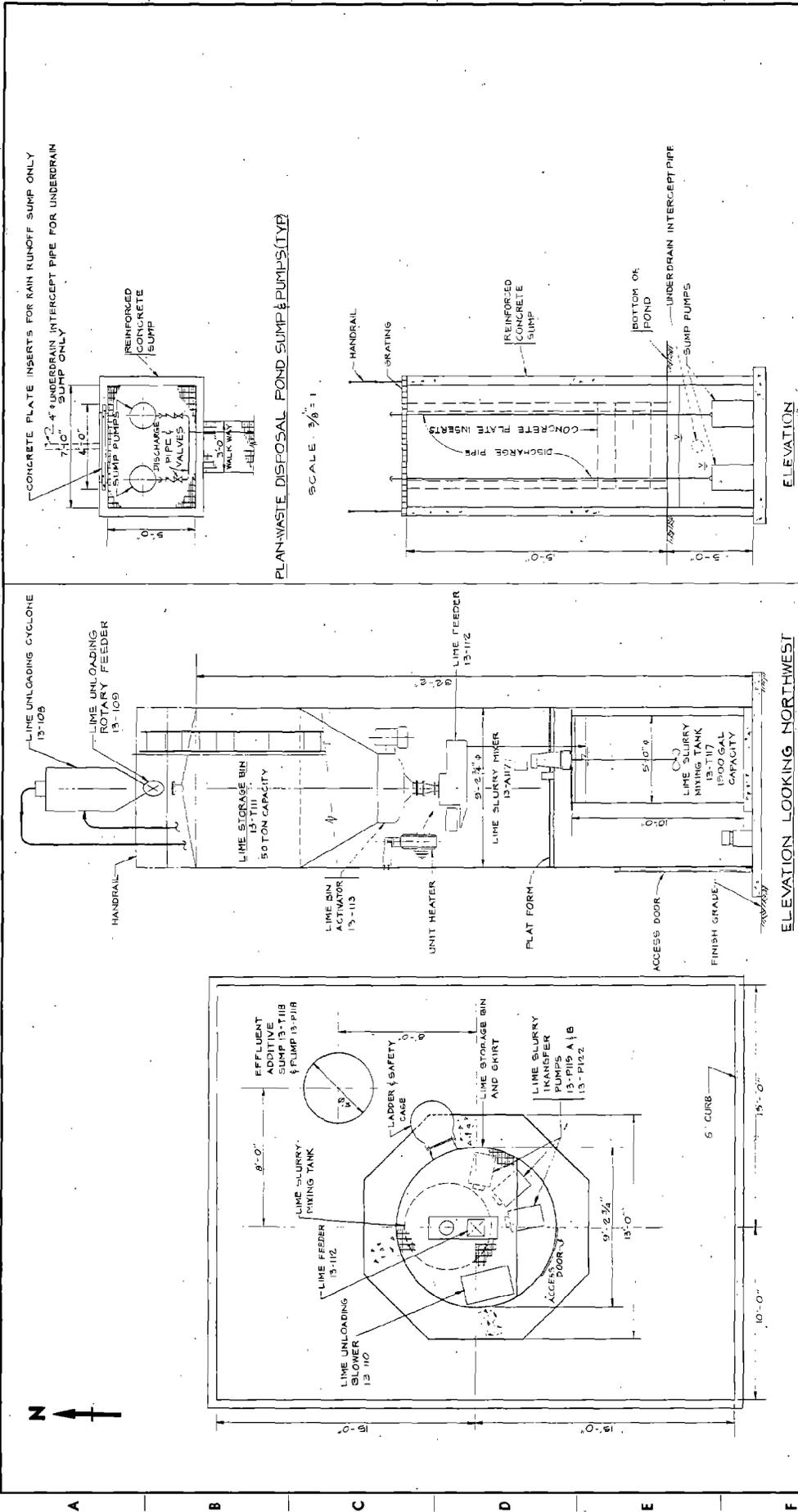
The vent scrubber system is designed to collect the vent gases from tanks and various equipment to recover HCl values in the gas streams. Each of the vent pipes is equipped with a butterfly valve to control flow of the gases. These valves have 3/4 in holes drilled through them. All tanks connected to the vent system are equipped with vacuum breakers to prevent them from collapsing. Each of the three vent scrubbers is a three stage system consisting of a two-stage venturi scrubber mounted on top of the 10% scrubber water tank. Pumps recirculate the scrubber water to the venturi and the solution is bled off to the 10% HCl storage tank as required. Makeup water comes from the 2% scrubber water tank. The gases then go to the second venturi scrubber mounted on top of the 2% scrubber water tank. Pumps recirculate 2% scrubber water to the venturi. 2% scrubber water is used as makeup to the 10% system and as scrubber water to the sieve tray fume scrubber. Gases from the 2% scrubber go through the third stage which is a sieve tray. Gases then exit the system through a blower. Makeup water enters the venturi scrubber. Each system is designed to remove 90% of the HCl from the vent gases.

The leaching scrubber system located by the mud washer tanks scrubs vent gases from equipment and tanks from the leaching section and the mud

separation and washing section. Both venturis are 10" size and the 4 tray sieve tray scrubber is 12" in diameter. The blower capacity is 550 scfm at 6" water gauge.

The acid recovery vent scrubber system is located by the secondary crystallizers and scrubs vent gases from the ACH decomposition section, the acid recovery and preparation section, the bleed stream section, the primary and secondary crystallization sections, the evaporation section, and the ACH dissolution section. Both venturis are 10" size and the 5 tray sieve tray scrubber is 12" diameter. The blower capacity is 550 scfm at 6" water gauge.

The tank farm vent scrubber system is located at the tank farm and scrubs vent gases from the tanks in the tank farm, and the tanks in the solvent extraction section. The size of the system is the same as the acid recovery vent scrubber system.



PLAN-LIME STORAGE & FEED SYSTEM
SCALE: 3/8" = 1'

ELEVATION LOOKING NORTHWEST
SCALE: N.T.S.

PLAN-WASTE DISPOSAL POND SUMP & PUMPS (TYPE)
SCALE: 3/8" = 1'

ELEVATION
SCALE: 3/8" = 1'

PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS
ALUMINA REFINERY FACILITY STUDY AND PRELIMINARY PILOT PLANT DESIGN
U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACT NO. 303094

AREA IS
GENERAL ARRANGEMENT-PLAN & ELEVATION
JOB NO. 76161003 DWG. No. 13-300-G R.O.

NO.	DATE	REVISION

DATE	DESCRIPTION	BY	CHKD.	APP'D.

FOR REFERENCE SEE DWG'S
13-100-P, 50-300-G

3.2.13 Utilities

3.2.13.1 Steam, Feedwater and Condensate - Area 21 - Dwg. No. 21-100-P

Summary

The steam, feedwater and condensate section of the demonstration plant is designed to generate and distribute process steam to the process areas requiring steam, collect condensate from the process areas where condensate is returned and to prepare feedwater for boiler use.

Equipment Description

The steam generator is a packaged water tube boiler producing a maximum of 55,000 lb/hr of steam at 100 psig when furnished with feedwater at 274°F and fired with No. 2 oil. The boiler is designed for 240 psig saturated steam and is shop assembled with the following accessories: air atomizing burner for No. 2 oil firing with an airfoil fan and motor; electric positioning combustion control system; flame safeguard system; safety vent valves, blowoff valves, feed stop and check valves; water column valves and gauge glass drain valves, and steam outlet stop/non-return valve; feedwater regulator with 3 valve by-pass, sootblower with valves and piping; water column assembly with gauge glass and trycocks; and duplex fuel oil pump set with base mounted pumps, motors with starters, duplex suction strainer with interconnecting piping; external relief valves and pressure gauges.

The continuous blowdown heat recovery system is composed of a flash tank, a heat exchanger and a flow control assembly. The low pressure condensate system includes the receiver, two pumps with motors, starters and a control panel with alarms, indicating lights and switches.

The 55,000 lb/hr packaged deaerator-heater, storage tank and pump is skid mounted, piped, wired and has a control panel with motor starters.

In addition, the boiler system has an economizer system, an instrument panel and an oxygen trim system to automatically control the boiler and fuel ratio for maximum efficiency.

At normal operating conditions, the steam produced by the boiler is 20,298 lb/hr at 100 psig. 100 psig steam requirements are 1,700 lb/hr normal and 3,010 lb/hr maximum for 3 steam ejector systems. 50 psig process steam requirements are 15,398 lb/hr normal and 28,432 lb/hr maximum. The maximum requirements include heating steam and steam usage for utility stations. Losses are assumed to be 2,000 lb/hr average based on 100 psig steam. Condensate returned from the process is expected to be 33.2 gpm normal and 61 gpm maximum. Feedwater makeup to the system is 7.4 gpm normal.

3.2.13.2 Cooling Water and Water Treatment - Area 22 - Dwg. No. 22-100-P

Summary

The cooling water and water treatment section is designed to provide 95°F water as cooling water supply and process water either as domestic water from city water, deep well water, or recycled water to the various equipment throughout the plant such as heat exchangers, barometric condensers and pumps. It is also designed to provide softened water as makeup feedwater to the boiler.

Equipment Description

The counterflow induced draft cooling tower is designed to cool 6,000 gpm of water from 115°F at a design inlet wet bulb temperature of 80°F. The design incorporates wood for structural material; HCl resistant drift eliminators, siding, stack and fan and alloy hardware. Cooling water requirements for the process areas is 2,800 gpm normal and 4,200 gpm maximum.

Process water required for the pilot plant is approximately 650 gpm normal and 1,900 gpm maximum. Process water from the city water main or from deep well pumps is stored in the 60,000 gallon process water storage tank. In addition, water recycled from the waste lagoon is stored in the 60,000 gallon wastewater recycle tank and may be used as process water.

The water softener system for boiler feedwater makeup has a capacity of 60 gpm. This automatic system consists of 2 skid mounted softener tanks and a fiberglass brine tank. The tanks are piped and valved and are supplied with a control panel for a complete operating system.

3.2.13.3 Plant and Instrument Air - Area 23 - Dwg. No. 23-100-P

Summary

The plant and instrument air section is designed to provide 100 psig compressed air for use on conveyor plows, air operated valves, unplugging of the 4 crystal slurry tanks in the crystallization section, as defoamer in the leaching section, for acid unloading, for utility areas, and for instrument air. Two 1,200 scfm packaged compressors with coolers and lube oil systems are included in this section. One air compressor will be operated as a standby unit. Two air receivers are provided, one in the utility area, and the other in the clay preparation area. Two instrument air dryer systems have been included. One is located in the utility area and the other in the ACH decomposition area.

3.2.13.4 No. 2 Fuel Oil Distribution - Area 24 - Dwg. No. 24-100-P

Summary

No. 2 Fuel Oil is received from trucks and stored in a 60,000 gallon storage tank. Fuel oil is pumped once a day or once a shift into the following day storage tanks:

1. Decomposer fuel oil day tank with a normal use rate of 1.21 gpm.
2. Bleed stream decomposer fuel oil day tank with a normal use rate of 4.3 gph.
3. Clay Calciner fuel oil day tank with a normal use rate of 1.3 gpm.
4. Boiler fuel oil day tank with a normal use rate of 2.3 gpm.

The fuel oil transfer pump will start automatically when the hand control valves located in close proximity to the day tanks are opened. The operator checks the level of the day tanks, closes the valve at the high level mark. When the valve closes, the pump automatically shuts off.

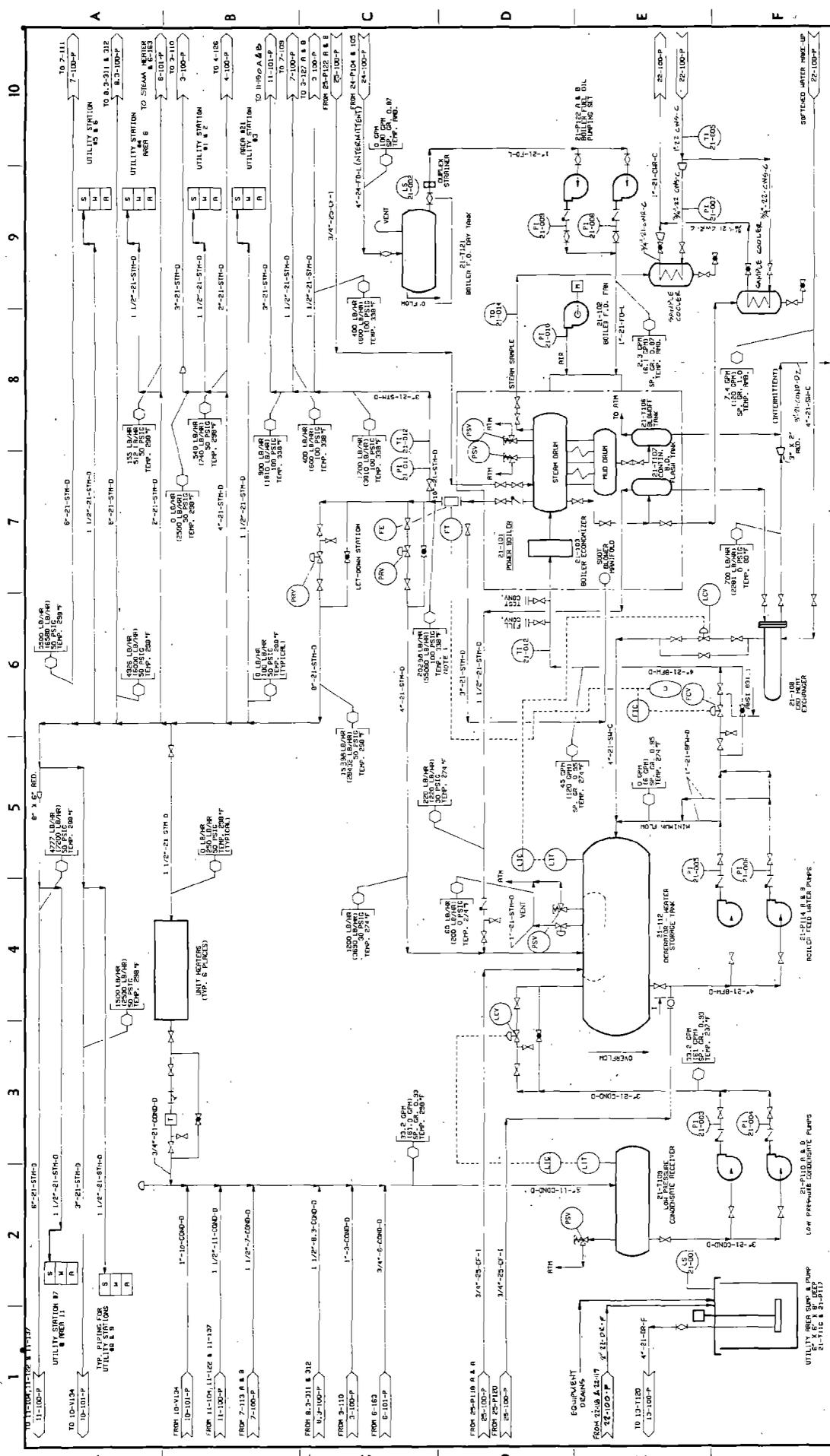
3.2.13.5 Reagents and Solvents - Area 25 - Dwg. No. 25-100-P

One-ton SO₂ tanks are received and stored. Two tanks are on the SO₂ weigh scale and SO₂ is metered out of one tank by a 3-way pressure regulator for use in the bleed steam decomposer. The weigh scale is used to determine if one of the tanks is empty so a replacement can be installed.

One-ton chlorine cylinders are received and stored. Two tanks are on the chlorine weigh scale and chlorine is metered out of one tank by a 3-way pressure regulator. The chlorine is then vaporized and sent to the chlorine ejector upstream of the polishing filters. The weigh scale is also used to determine when one of the tanks is empty.

Two types of boiler feedwater treatment chemicals are provided. One is an oxygen scavenger, the other is a corrosion inhibitor. The boiler chemical tank will be used for injecting chemicals into the boiler to prevent scale formation. The cooling tower chemical tank will be used for chemicals to control pH of the cooling water.

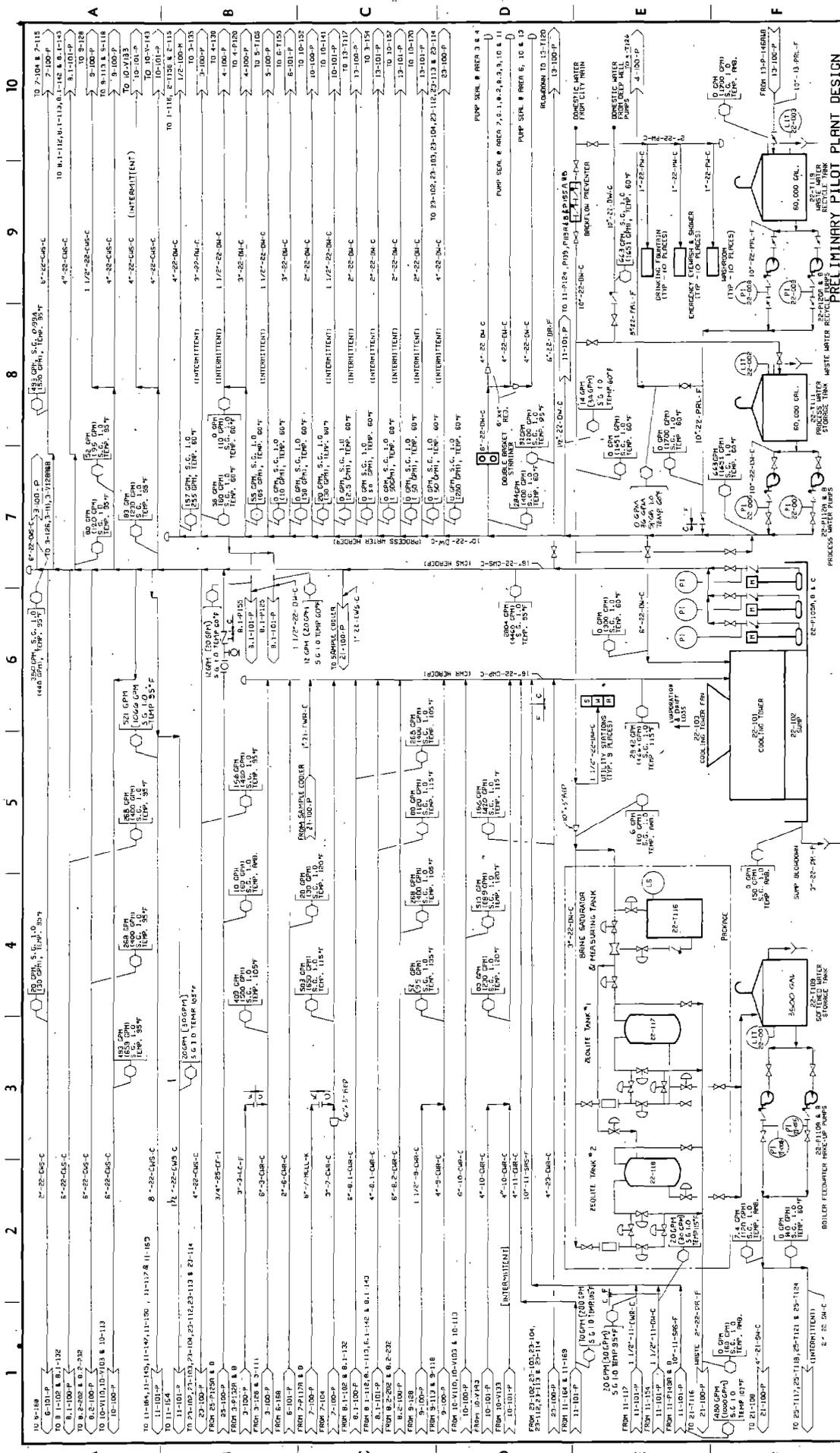
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PRELIMINARY PILOT PLANT DESIGN
KAISER ENGINEERS
 CONSULTING ENGINEERS
 U.S. DEPARTMENT OF THE INTERIOR
 BUREAU OF MINES CONTRACT NO. 14-60-01-0000
 STEAM, FEEDWATER AND CONDENSER
 JOB No. 76161.003 DWG. No. 21-100-P
 R. U.

1. INCLUDES 2000 LB/HR LOSSES.
 2. 21-110 P.A.B.
 3. LOW PRESSURE CONDENSER PUMPS
 4. 21-110 P.A.B.
 5. 21-110 P.A.B.
 6. 21-110 P.A.B.
 7. 21-110 P.A.B.
 8. 21-110 P.A.B.
 9. 21-110 P.A.B.
 10. 21-110 P.A.B.



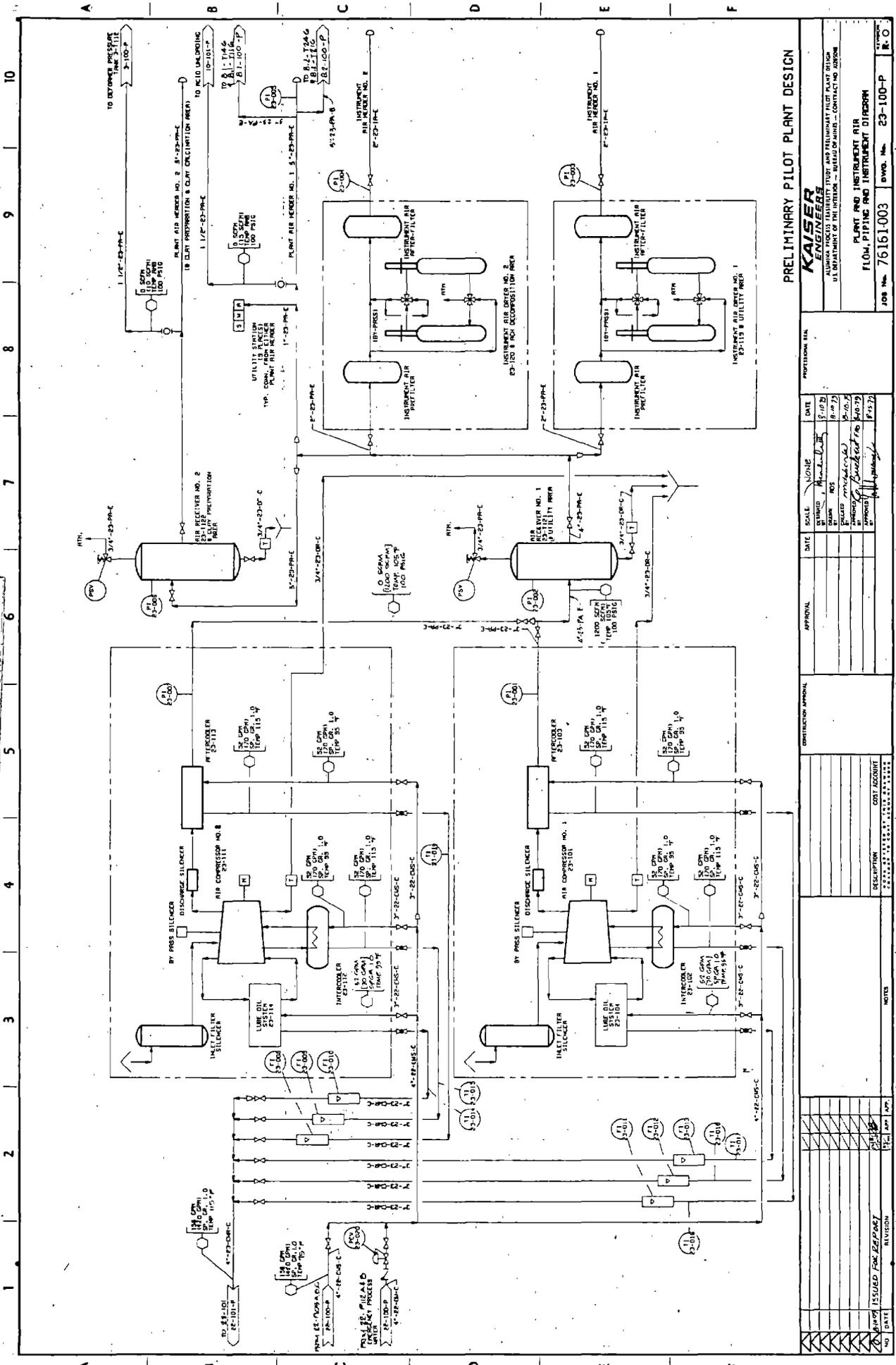
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3	10/22/88	REVISION	KAISER ENGINEERS	AP
4	11/1/88	REVISION	KAISER ENGINEERS	AP
5	11/15/88	REVISION	KAISER ENGINEERS	AP
6	11/22/88	REVISION	KAISER ENGINEERS	AP
7	12/1/88	REVISION	KAISER ENGINEERS	AP
8	12/15/88	REVISION	KAISER ENGINEERS	AP
9	12/22/88	REVISION	KAISER ENGINEERS	AP
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PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS

ALUMINA FLOWSheet MATERIAL STUDY AND PRELIMINARY PILOT PLANT DESIGN
U.S. DEPARTMENT OF THE INTERIOR - BUREAU OF MINES - CONTRACTING DIVISION

PLANT AND INSTRUMENT AIR
FLOW, PIPING AND INSTRUMENT DISBOOR

JOB No. 76161-003 DWG. No. 23-100-P

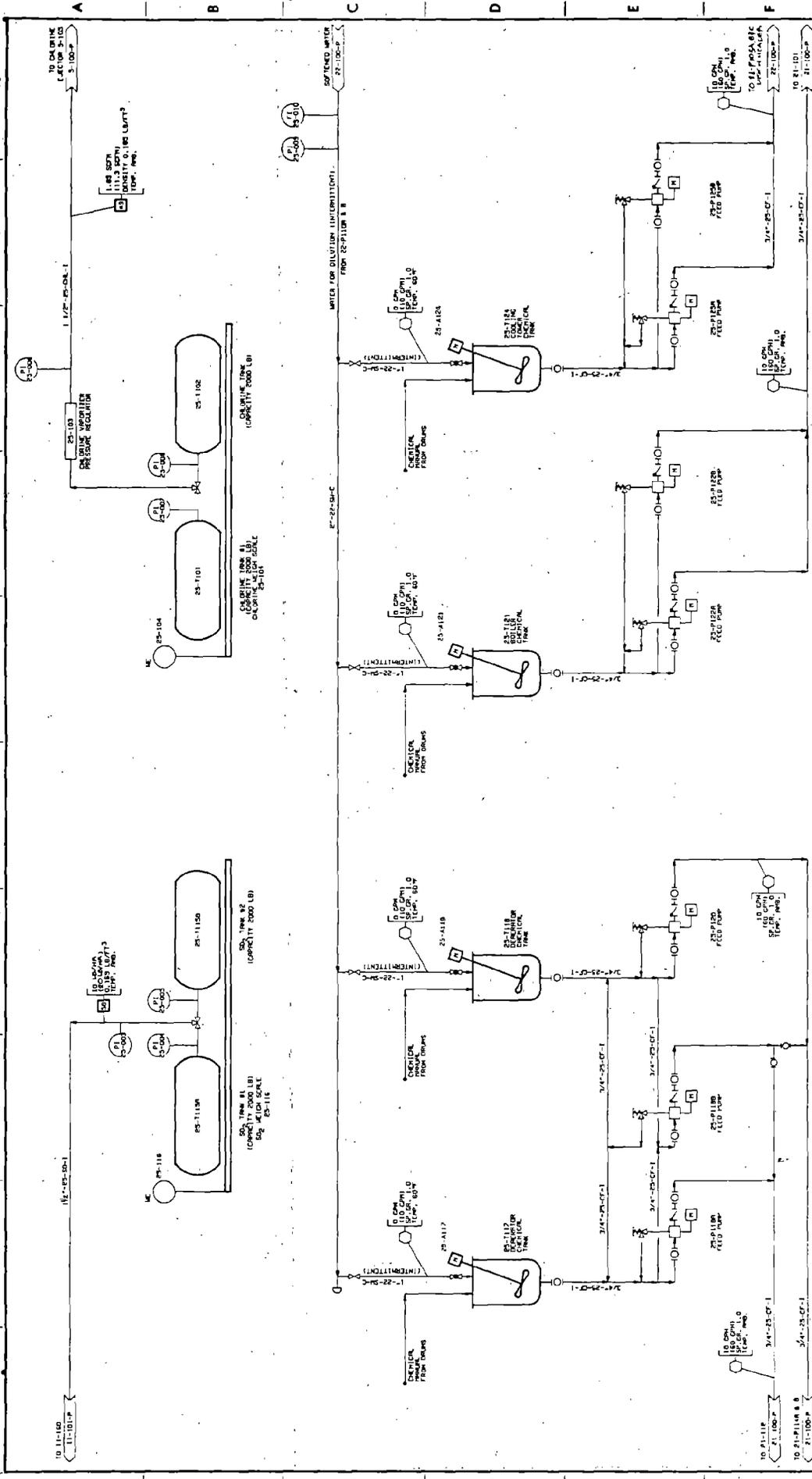
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PRELIMINARY PILOT PLANT DESIGN

KAISER ENGINEERS

ALUMINUM FLOOD TREATMENT STUDY AND PRELIMINARY PILOT PLANT DESIGN
U.S. DEPARTMENT OF COMMERCE, BUREAU OF MINING

FLOAM, PIPING AND INSTRUMENT DIAGRAM

JOB No. 76161-003 DWG. No. 25-100-P

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3.2.14 Power Distribution

Summary

Power will be received from utility at a medium voltage which has been selected at 4,160 volts for the purpose of cost evaluations. It is expected that the utility company may elect to bring a higher voltage service line to the main substation and to provide a transformer at the main substation for the 4,160 volt service of the pilot plant. A metal enclosed outdoor switchgear assembly has therefore been included in the main substation. The switchgear is rated at 4,160 volts.

Because certain plant processes require continuity of electric power service, an emergency generator is included at the main substation to provide 1,000 K.W. of 3 phase 60 Hz power during an outage of the utility service.

The generator is sized to handle continuity of service of agitators, thickeners solvent extraction, boiler plant and to reduced levels of area and building lighting services.

Description

Power is distributed from the main substation with 5 kV cables in underground ducts. The ducts will consist of PVC conduits directly buried in trenches. Precast concrete boxes will be used in the duct system to enable installation of cable in the ducts from the substation to the plant load centers.

Plant load center transformers are located near the change house, the control building, and the boiler house. The estimated demand loads at these load centers are 250 KVA, 1,000 KVA, and 1,500 KVA respectively. The load centers will provide 480 volt 3 phase services to building utilities and process loads.

Cooling water pumps and air compressors will be equipped with 250 horsepower motors which will be served at 4,160 volts. The demand of this service is estimated at 1,000 KVA in addition to the above 480 volt load center demands.

Process motors will be served from motor control centers located in the control building and in a building adjacent to the boiler house. Power and control circuits will be carried on cable trays from the motor control centers to the motor locations. Each motor will be served with a multiconductor cable consisting of three power conductors and control conductors as required for control at the motor location.

The utility service transformer and the load center transformers will have way grounded secondary windings. A ground grid of bare copper conductors and ground rods will be embedded under the substation area. A bare copper grounding conductor, bonded to the substation grid shall be included with the 5 kV service cables to the load centers. The

grounding system for the 480 volt service will include bare copper ground conductors carried on the cable tray system. These conductors will be bonded to ground rods at intervals along the cable tray runs. Each motor service will include a ground conductor within the cable jacket of the power conductors.

Cable tray, conduit and fittings will be treated with corrosion resistant surface coating. All motors will be TEFC or TENV and will have corrosion resistant enclosures and exposed hardware.

Hazardous areas to include the solvent and coal handling processes will be fitted with explosion proof electrical equipment and material to suit the hazard classification of the area.

4.0 PILOT PLANT OPERATION

4.1 Operating Procedures

The operating procedures section of this report briefly describes the major process control techniques used to operate the pilot plant. Each of the major operating areas of the plant is controlled by adjustment of one or more independent variables. The total plant operation is composed of the independent operation of various operating areas or groups of areas. Surge capacity located throughout the plant isolates the individual areas or groups of areas to permit partial plant operation, while some areas are shutdown, during maintenance within sub-units.

The clay preparation and calcination section operates independently of the rest of the plant. Raw clay is received by truck normally only on week-days on day shift. The clay calciner operates 24 hours per day for 5 days per week. Calcined clay storage of 600 T provides adequate inventory for week-end shutdown as well as all reasonable shutdowns for maintenance. The primary controlled variable is calcination temperature which directly sets the amount of alumina which can be extracted.

The clay leaching, mud separation and washing, and the filtration sections of the pilot plant operate as a unit. The liquor feed rate is set to leach and the clay feed rate is ratioed to the hydrochloric acid flow to provide about 5% excess acid stoichiometrically. Clay alumina content and leach acid concentration must be known to permit proper acid flow control. Residual acid is monitored at the exit from leach to assure excess acid is used. The major water addition to the process is the mud filter wash water. This water flow is preset based on clay feed rate such that approximately 2 lb of wash water are used per pound of mud solid waste.

The silica fines which do not settle in the mud thickeners are removed in sand filters prior to solvent extraction of iron. Two (2) sand filters are provided with automatic switching and back flush cleaning as differential pressure increases across the sand beds. Periodic visual monitoring of the filtrate is required to assure filtrate clarity. The filtrate can be re-filtered if required.

In order for iron to be removed in solvent extraction, all ferrous ion must be converted to ferric ion by the addition of chlorine. The redox potential of the filtrate is constantly measured and is used to adjust Cl_2 flow. Over- or under-chlorination alarms are provided. Laboratory samples are required to assure proper instrumentation operation.

Organic flow through solvent extraction is adjusted based on the iron level in the clay which will normally be about 1%. However, the equipment is designed to process as high as 3.5% Fe₂O₃ in kaolin without reduction in production rate. When lower iron clay is being processed, organic flow will be reduced to reduce pumping costs. Laboratory monitoring of raffinate iron concentration and spent organic iron loading will confirm proper operation.

In order to obtain good yields of ACH in primary crystallization, excess water must be removed in evaporation. The amount of evaporation is controlled by adjusting the temperature of the ACH liquor in the evaporator. This temperature represents the liquor boiling point and is dependent on ACH concentration.

ACH crystallization is controlled in both the primary and secondary crystallization units by continuously measuring the density of the slurry. HCl sparging gas flow is controlled to provide the desired level of supersaturation as measured by temperature rise across the HCl injection point.

Product purity is controlled primarily by the concentration of impurities in the circulating liquor stream. Increasing the bleed stream will reduce the concentration. In order to maintain liquor inventories between plant areas, when bleed stream flow changes are made, the amount of 20% HCl being sent to the ACH dissolver must be correspondingly adjusted.

ACH decomposition is controlled by adjusting the decomposer and calciner bed temperatures. The decomposer must operate hot enough to obtain at least 90% ACH to alumina conversion. The calciner bed temperature controls α - alumina and residual chloride concentrations.

4.2 Data Collection

4.2.1 Data Acquisition Computer System

Summary

The data acquisition computer system (DACS) is a computer system capable of recording, storing, calculating and alarming a broad range of analog and digital process variables. The P&ID's shown in Volume VI identify those variables selected for input to the computer. This system will have provisions for expansion to closed loop control of the process. The system will have the capability for compiling and executing FORTRAN programs in the background, i.e., the on-line data acquisition function will not be affected.

Descriptions

Process operator interfaces will include one cathode-ray tube (CRT) and a typewriter terminal located in the control room. The CRT will be used by the operators to enter commands to obtain process data through displays available to them. The typewriter will log process alarms and return to normal conditions as they occur. These operator computer interfaces are not intended to be the operators' only source of process information. They are intended to augment the information shown by the analog controllers, indicators, recorders, and alarm modules located on the main instrument panel.

The central processing unit (CPU), a system console CRT, a disc unit, a magnetic tape unit, a line printer and a typewriter terminal will be located in the computer room which is located in the administration building. The systems console will be used by the technical staff to make on-line changes in coefficients and parameters such as input signal scaling and alarm values, as well as occasional fundamental software changes in existing displays. The technical staff will also use the system to perform scientific computations, data analysis and evaluation, special report generation, and generation of any new data display programs.

The type of displays available to the operator will be process logs and trend logs. All variables will be displayed in engineering units. Any or all of these logs as well as alarm logs can be selected by the technical staff for storage on mag tape and/or printing on the high speed line printer.

The process log will present all process variables and selected calculated parameters, such as ratios. These displays are essentially shift logs stored on the computer for the past day or longer if desired. These logs would display in a table the value of the process variable on an hourly basis. With 100 variables total and assuming 10 variables/display, there would be a total of 10 displays of this type.

The snap-shot log presents one or more selected groups of data about major pieces of equipment or areas in the plant. This is current data as opposed to the process log which is historical data. Providing for process variable labels in the display there could be another 2-6 additional displays of this type.

The trend log is a display of previously stored process variables recorded at selected intervals prior to the present time. For example, the computer could retain the values of the process variables at one minute intervals for the previous half hour, or a reading of the values every five minutes for the previous eight hours. These trend logs can be thought of as analog strip chart recordings with perhaps 2-5 variables per display. Assuming two total time periods are available, 100 variables would give an additional 40-100 displays available to the operator.

In addition to these process displays the system will be used to generate management reports which will include shift or daily production/efficiency reports.

All process parameters monitored by the DACS can be displayed on the CRT to the operating personnel through a simple keyboard request. Calculated parameters such as ratios, pressure/temperature compensated flows can be displayed, together with totalized flows and production. Therefore, the operator has, at his fingertips, accurate and timely information. This same information can be displayed in the computer room on the systems console CRT and/or line printer allowing plant technical and management personnel access to operating data as if they were in the control room.

4.3 Raw Materials and Fuel Usage

In order to quantify the amount of raw materials, supplies and fuel usage in the demonstration plant, it was necessary to estimate the amount of alumina which must be produced to meet Hall cell test requirements of the various program cooperators. A survey by the Bureau of Mines indicated that 8,500 T would probably be sufficient for these tests. A further allotment of 500 T was added to provide for various governmental tests and evaluations as well as to provide for a substantial retained inventory after plant shutdown.

In order to produce the 9,000 T of alumina, the plant operating program has been divided into 6 periods of 6 months each. The first two periods will primarily be devoted to training startup, shakedown and equipment modification. The plant will be secured for safe, long-term shutdown or demolition during the last period. The estimated plant operations is shown in Figure 4.3.1.

While it is anticipated that 9,000 T of alumina will be produced that will meet all product specifications, some off-quality material will also be produced. Raw materials and fuel usage will be affected by this off-specification production. In Table 4.3.1, the anticipated total production of 10,910 T is shown along with

the anticipated production rates and on-line time. All material and fuel consumptions are based on charging the plant for 10,910 T of production.

The material balance presented in section 3.1 is the basis for determining the feed requirements for a given amount of alumina production. However, in all operating units, a certain amount of feed material is unaccountably lost. These unallocated losses occur in handling, upset condition operation, startup, and shut-down. A projection of these losses is shown in Table 4.3.2.

Certain other assumptions are required to complete a calculation of all material and fuel usages. The major assumptions are:

- 20% of the off-specification alumina is lost as ACH and must be neutralized.
- 10% of HCl losses go to the air with the balance to spills.
- 80% of neutralization is effected with limestone and the balance with slaked lime.
- Limestone is 60% consumed in neutralization.
- Slaked lime neutralization is 85% efficient.
- Separan MGL usage is based on 0.2 lb/t calcined clay fed to leach.
- Organic solvents and amine usage are based on initial fill of 32,400 gallons plus loss.
- Coal is used only for clay calcination at rate of 4.7×10^6 Btu/T Al_2O_3 plus 15% upset and plant size inefficiency losses.
- No. 2 Fuel Oil is used for all other heat energy requirements.
- Steam usage is based on a base load of 4,000 lb/hr and a variable load of 12,000 to 20,000 lb/hr depending on production rate with a total usage of 328×10^6 lb of steam.
- Decomposition fuel oil is based on 14.4×10^6 Btu/T Al_2O_3 produced plus 15% upset and plant size inefficiency losses.
- Bleed stream calcination fuel oil is based on 0.6×10^6 Btu/T Al_2O_3 produced.

The total materials, supplies and fuel usages are contained in the Operating Cost Summary reported as Table 4.7.1.

A breakdown of the fuel useage by process area is shown in Table 4.3.3. It should be noted that this fuel consumption does not represent what may be expected in a commercial plant. The pilot plant was designed with the emphasis on simplicity and flexibility. For this reason waste heat recovery was not designed into the process.

In a commercial plant however, there would be a large incentive and considerable potential for reducing the process energy consumption through the recovery of waste heat.

Candidate streams for waste heat recovery include off-gases from the ACH Decomposer, the molten salt heater, and others. The subject is discussed in more detail in section 6.

FIGURE 4.3.1

PLANT OPERATIONS SCHEDULE

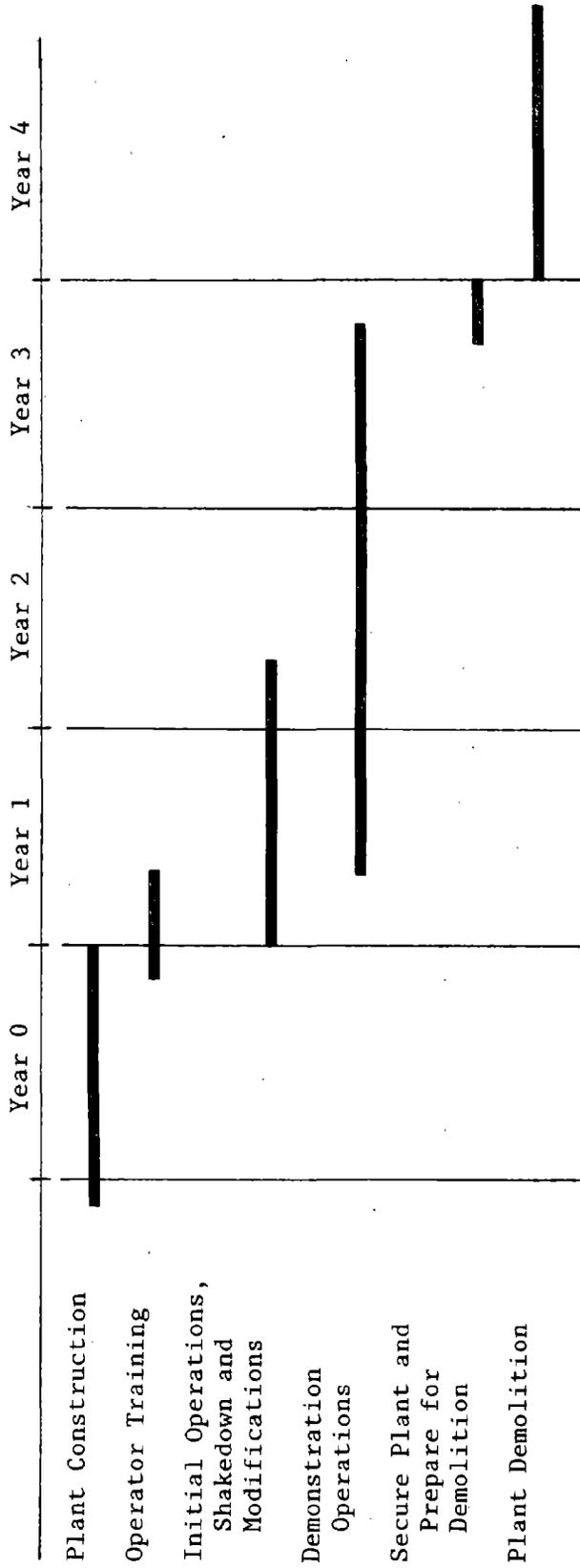


TABLE 4.3.1
PRODUCTION RATE SCHEDULE

Six Month Period	Percent of Time Feed On-Line	Percent of Maximum Feed Rate	Total Alumina (Tons)	Percent of Product On-Spec.	On Spec. Alumina Produced (Tons)
1	15	50	345	0	0
2	40	50	905	37.5	340
3	50	75	1700	70	1190
4	60	80	2200	85	1870
5	70	100	3200	95	3040
6	60*	100	<u>2560</u>	100	<u>2560</u>
			10,910 Tons		9000 Tons

*Includes shutdown to secure plant approximately 2 months before end of 6 month period.

TABLE 4.3.2
UNALLOCATED MATERIAL LOSSES

	Material Losses Percent of <u>Incoming Material</u>
Clay	20
HCl	6
Chlorine	3
Organic	5
Separan MGL	5
Coal	10
Fuel Oil	8
Lime	5 (HCl Basis)
Limestone	10 (HCl Basis)
Sulfur Dioxide	10

TABLE 4.3.3
 FUEL REQUIREMENTS FOR 25 TPD PILOT PLANT

	<u>MM BTU/T. Alumina</u>
Clay calcination	4.7
FeCl ₃ treatment with clay	0.1
Evaporation	3.9
ACH dissolution in Crystallization	5.5
ACH decomposition	12.2
ACH calcination	2.2
Acid recovery - HCL stripper	1.7
Bleed Stream	
Evaporative crystallizers	3.3
ACH dissolution	1.6
Waste calcination	.6
Vacuum jet operation	<u>1.9</u>
	37.7

4.4 Utility Requirements

The utilities which must be furnished to operate the demonstration plant are steam, compressed air, electric power and water. The plant will contain a steam boiler and an air compressor and dryer. The steam system is described in Dwg 21-100-P and contains a 55,000 lb/hr package boiler which is fired with No. 2 fuel oil. The fuel oil consumption for steam generation is discussed in section 4.3.

The plant and instrument air system are shown in Dwg 23-100-P. Two (2) 1200 CFM rotary air compressors are provided. The system is electrically operated and the electrical energy consumption discussed below includes the air energy requirement.

The pilot plant has approximately 450 electric motors with a total power rating of 4,791 horsepower. The normal operating configuration will have 3,481 horsepower of motors on-line. Based on Table 4.3.1, the plant will be on-line 45.8% of available time. The motors will draw 80% of rated horsepower during normal operation with a 90% power factor. During non-operational periods, the motors will draw 36% of normal load. An allowance of 25% of the motor current has been made for indoor and outdoor lighting, controls, air conditioning and miscellaneous uses. This represents a total electrical consumption of 47.4×10^6 kWh over the project life.

4.5 Manpower Requirements

Since the demonstration plant has been assumed to be located at a "green field" site, a full organization is required for its operation. The organization chart is outlined in Figure 4.5.1. The contractor responsible for operating the facility is assumed to have 104 employees on the site during plant operation. Sub-contracts will be issued for maintenance labor, security and janitorial service. It is assumed 62 people will be required for these services. Certain backup personnel will be required at the contractor's home office and one full time and two one-half time employees are planned. During the period of plant construction, the contractor will provide three employees to inspect, coordinate and monitor construction activities.

Within the plant operating contractor's on-site crew will be several sub-groupings. The operators will be divided into four ten-man shifts including a foreman and an assistant foreman. Additional labor is provided for bagging the product alumina (5 shifts/week) and clay calcination (15 shifts/week). A shift maintenance crew of ten R & M technicians will be allocated to provide emergency maintenance when contract maintenance personnel are not available.

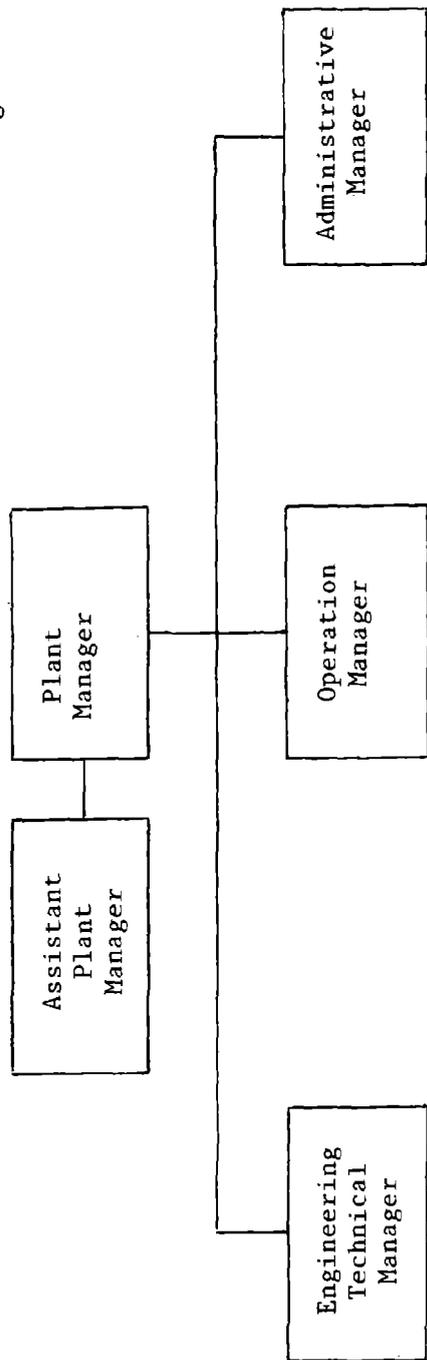
Not included in the manpower requirements are any personnel from companies participating in the pilot plant, who might be assigned to the site.

The schedule for staffing the demonstration plant by the operating contractor and his sub-contractors is shown in Figure 4.5.2. The construction supervision team will be on-site for approximately one year, beginning approximately 9 months before the first operational trials. Site management will be on-site for 3½ years beginning 3 months prior to operational trials and extending to 3 months after the plant has been shut down and secured. The post-operation period will be devoted to report writing, stores disposal and disposal of the plant equipment and buildings.

Operators, R & M, and laboratory technicians will be trained for 1-2 months prior to operational trials. Plant security and janitorial services will be provided for 3½ years.

**PILOT PLANT
ORGANIZATION CHART**

Figure 4.5.1



- 6 Chemical Engineers
- 3 Mechanical Engineers
- 1 Instrumental/Electrical Engineer
- 1 Chemist
- 1 Laboratory Superintendent
- 10 Laboratory Technicians
- 1 Draftman
- 1 Plan/Sched.
- 4 Foreman
- 4 Assistant Foremen
- 37 Operations Technicians
- 1 R & M Superintendent
- 10 R & M Technicians
- 1 Medical
- 1 Safety
- 3 Acct/Payroll
- 1 Personnel I/I.R.
- 1 Warehouse
- 7 Secretaries
- 4 Clerks
- 1 Purchasing

Contract Personnel

R & M 51 each
 Security 9 each
 Janitorial 2 each

Home Office G & A

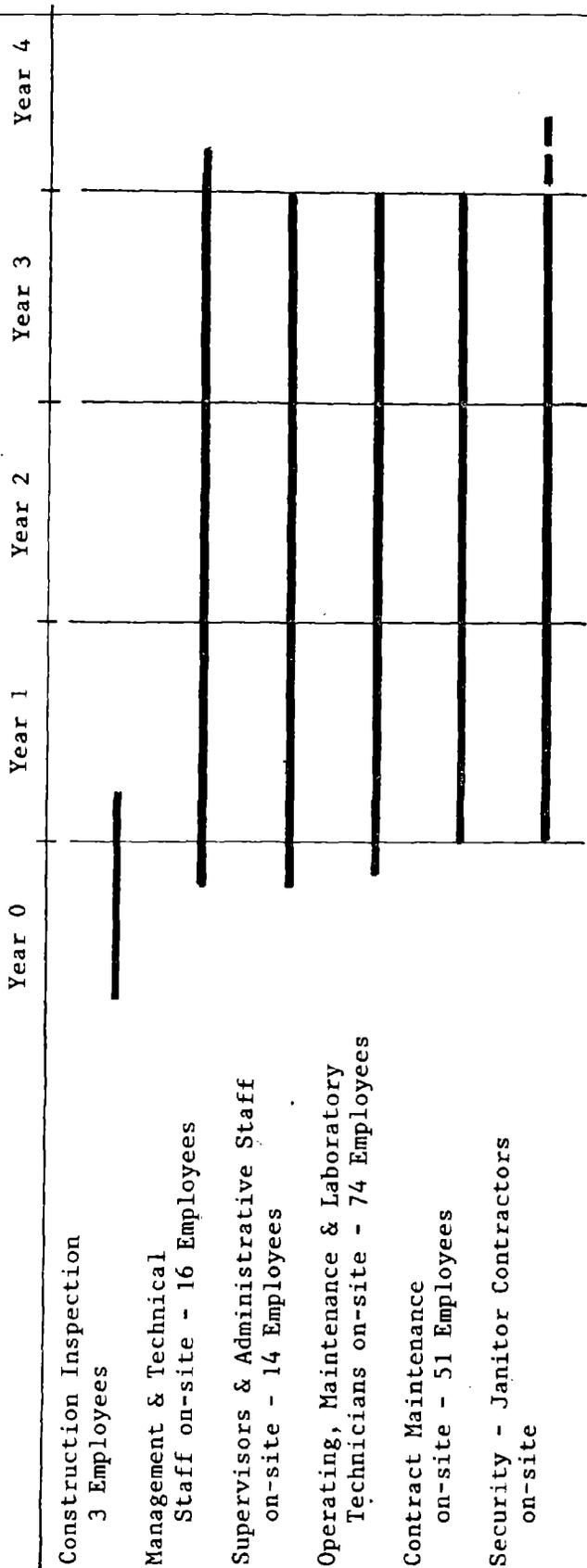
Full Time 1 each
 ½ Time 2 each

Construction Inspection

3 man-years

FIGURE 4.5.2

STAFFING SCHEDULE



4.6 Laboratory Testing

4.6.1 Sampling and Sample Analysis

In order to maintain control of the process it will be necessary to take samples of many process streams and analyze them on a regular basis.

A list of the regular daily sample load is shown in Table 4.6.2.1. This table identifies each sample by name and by stream number. The stream numbers used correlate with those shown on the block flow diagram - drawing 50-302-G, R-0.

4.6.2 Laboratory Equipment

Major Equipment

Sample Preparation Room

Chipmunk crusher
Braun pulverizer
Drying oven, mechanical convect., 37 x 25 x 9 in.

Balance and Instrument Room

Analytical balance, Mettler #H45
Sorptometer, Micromeritics Model 2200
(Surface area measurement)

Wet Acid Laboratory

Titration, recording, Sargent-Welch Model DG
Vacuum oven
Drying oven, mechanical convection, 19 x 19 x 19 in.
Drying oven, mechanical convection, 13 x 13 x 14 in.
Balance, top loading, Mettler Model P3N
(3000 g capacity)
2 pH Meters digital display, Orion Model 801A
Centrifuge, International size 2, Model K
Water bath, full visibility, Magni Whirl, Blue M, 8½ gal.
Electric muffle furnace, Lindberg with console (1010°C)
Electric muffle furnace, Lindberg with console (1200°C)
Platinum Ware (crucibles, pans, covers)

Emission Spectroscopy Laboratory

Plasma spectrometer (ICP)

Major Equipment Functions Plasma Spectrometer

In this proposal the instrumental analysis using the plasma spectrometer with inductively coupled plasma (ICP) technique, an

advanced version of emission spectroscopy, would make all of the elemental analyses on solids and liquors. Although it is a new concept, it should be mature when the demonstration plant is built. Relevant facts on this instrument are:

- Full range instrument with integral computer costs about \$170,000. Should have capability to determine elements including C, B and P (limit of detection - not yet known).
- Several manufacturers, including Jarrell-Ash and Applied Research Laboratories.
- Analysis is made on a solution. Clays and calcined alumina are solubilized via lithium tetraborate fusion. Solution samples are run directly. For example, after solution (30-60 minutes), 5 minutes required for Si, Na, Fe, Ca, Zn, Ti and V in calcined alumina.
- Advantages over emission spectroscopy are: - Simplifies work and required skill of analyst as meticulous effort required for emission spectrography is eliminated; i.e., sample preparation, manual packing of electrode, and film development and reading.

- Level of minimum detection of elements is equivalent or better than emission spectrography.

Sorptometer

This should be a Micromeritics Model 2200 or equivalent. This instrument is used to measure the surface area of the product alumina.

Clay Digest Custom Reactor

It will be necessary to establish a standard leaching test to test the calcined clay for leachable alumina. A custom reactor will be required for this test.

4.6.3 Laboratory Personnel

Manpower requirements based on the regular sample load shown in Table 4.6.2.1, plus a sizeable "special sample load" on an irregular basis are as follows:

- 1 Chief Chemist
- 1 Emission Spectroscopist (on days)
- 1 Technician (days)
- 8 Technician-Analysts (2 per shift)

4.6.4 Laboratory Building

A layout for the laboratory section of the Administration Building is shown in Drawing 57-400-G.

Special requirements are as follows:

- The Plasma Spectrometer (P-S) laboratory must be dust-free, at 50% relative humidity and probably separately air conditioned.
- The balances and instruments should be in a separate room to minimize corrosion from the HCl. The balance bench must be free-standing and isolated from vibration.
- The small sample storage is conveniently located next to the balance room. Acid samples should be specially shelved. Laboratory supplies could be stored in it.
- The sample preparation room must be accessible from the outside without going into the main corridor. This will keep outside dust (and mud) away from the other rooms. Oversize ventilation should be provided in this room for certain dust removal.
- The wet acid lab is large enough to also provide space for bench-scale process studies. Hood vents must have scrubbers on them. All furniture and hardware must be acid resistant to avoid corrosion.

TABLE 4.6.1

SAMPLES REQUIRED FROM PILOT PLANT ON A REGULAR BAISS

Unit Operation	Material	Stream No.*	Analyses
Clay Preparation	Raw Clay	1	Moisture, Al ₂ O ₃ , SiO ₂ , Fe ₂ O ₃ , TiO ₂ , P ₂ O ₅ , CaO, MgO, Na ₂ O, K ₂ O
Clay Preparation	Calcined Clay	3	LOI, Available Al ₂ O ₃ , Leachable P ₂ O ₅ , CaO, MgO, Na ₂ O, K ₂ O
Leaching	Leach Acid	4	HCl, Al ⁺³
Leaching	Leached Slurry	6	HCl, Al ⁺³ , % Solids
Thickening	Leached Slurry	9	Settling Test
Thickening	Thickener O'Flow	13	HCl, Al ⁺³ , mg/l Solids
Thickening	Thickener U'Flow	12	HCl, Al ⁺³ , % Solids
Mud Washing	Washer O'Flow	11	HCl, Al ⁺³ , Sol. Fe, mg/l Solids
Mud Washing	Waste	15	HCl, Al ⁺³ , % Solids Soluble Fe, Ca, Mg, Na, K
Filtration	Filtrate	5A 18	Fe ⁺³ mg/l solids
Solvent Extraction	Loaded Solvent	20	gpl Fe
Solvent Extraction	Stripping Liquor	22	HCl
Evaporation	Evap. Feed	19	gpl Fe
Crystallization	Feed	26	HCl, Al ⁺³ , Fe, Ca, Mg, Na, K, P ₂ O ₅
Crystallization	Primary Crystallizer Discharge	27	% Solids, Particle Size, Solids: Al ₂ O ₃ , Impurities; Liquor: HCl, Al ⁺³ , Impurities
Crystallization	Primary Crystallizer Discharge	40	% Solids, Particle Size, Solids: Al ₂ O ₃ , Impurities; Liquor: HCl, Al ⁺³ , Impurities

TABLE 4.6.1 (Cont)

Crystallization	Primary Crystallizer Filter Cake	29	% Solids, Particle Size, Solids: Al_2O_3 , Impurities; Liquor: HCl , Al^{+3} , Impurities
ACH Dissolution	Discharge Liquor	38	Al^{+3} , HCl
Decomposition	Secondary Crystallizer Discharge	41	% Solids, Particle Size, Solids: Al_2O_3 , Impurities; Liquor: HCl , Al^{+3} , Impurities
Decomposition	ACH Decomposer Discharge	43	LOI, Cl^- , Surface Area
Decomposition	ACH Calciner Discharge	45	LOI, Cl^- , Surface Area
Chloride Recovery	$FeCl_3$ Liquor Feed	23	Fe , HCl
Chloride Recovery	Slurry	16	% Solids, Fe^{+3} , HCl
Bleed Stream Treatment	Bleed Stream	30	HCl , Al , Fe , Ca , Mg , Na , K
Bleed Stream Treatment	Waste	52	Fe_2O_3 , Al_2O_3 , CaO , MgO , Na_2O , K_2O

* Refer to Drawing 50-303-G, R-O.-Plant Material Balance.

Note: Al^{+3} and Fe^{+3} refer to soluble aluminum and soluble iron.

4.7 Operating Costs

The total cost for operating the demonstration plant to produce 9000 T of cell-grade alumina as shown in Table 4.7.1 is \$40,916,190. This cost is in mid-1979 dollars and includes contingency but excludes escalation to the date of actual operation. The method of calculating usages of materials and fuel are discussed in paragraph 4.3. Utilities requirements are discussed in section 4.4 and manpower or labor in section 4.5. Unit costs are based on vendor quotes and published prices plus freight allowance where indicated. Freight is based on a southeastern U.S. location with good truck access.

A substantial portion of the plant operating costs have not been discussed elsewhere. These are listed in the miscellaneous portion of Table 4.7.1. Generally, the individual items are self explanatory. However, several key items merit further discussion. R & M services are those maintenance requirements that are performed by others not included in the labor portion of the operating costs. These requirements could be special crane service, machining, non-destructive testing, rotary balancing, etc. The combined annual cost of all maintenance including operating contractor and R & M subcontractor labor, supplies and services is 7% of capital. While this is a relatively high rate for many types of plants, the new technology and corrosive process indicate costs of this magnitude may be experienced.

Contractor expenses represent the cost of the operating contractor incurred by personnel not assigned to the site. G & A represents supervisory and support services from the home or controlling office. Construction inspection is performed by assigned personnel on behalf of the plant owners to ensure proper plant construction in accordance with the Owner's requirements. The assigned personnel are sometimes referred to as Owner's representatives. The fee is the operating contractor's profit for administering the plant operating contract.

No salvage value has been allowed for the plant nor has site restoration been estimated due to inadequate specific site information. It is reasonable to assume these costs will be offsetting.

TABLE 4.7.1
 OPERATING COST SUMMARY

	Usage	Unit Cost (\$)	Total Cost
Raw Materials			
Kaolin Clay	47,400 T	10.	474,000
Hydrochloric Acid 31.5%	7,550 T	65.	487,500
Chlorine	170 T	200.	34,000
Sulfur Dioxide	60 T	275.	16,500
Supplies			
Limestone	4,300 T	25.	107,500
Lime, slaked	405 T	75.	30,375
Separan MGL	7,060 Lb	1.85	13,060
Kerosene	212,630 Gal.	.85	180,735
Alamine 336	287,050 Lb	1.25	358,800
Decanol	191,370 Lb	.75	143,525
Utilities			
Coal, Low Sulfur, 11,600 BTU/Lb	3,365 T	45.	151,425
Fuel Oil, No. 2	4,302,300 Gal.	.62	2,667,425
Electric Power	47,369,700 KWH	.025	1,184,250
Water	630,000 Kgal	.50	315,330
Labor			
Exempt Salary - 30 employees	102.2 man-years	35,680	3,646,500
Non-exempt Salary			
Opn, R & M, Lab Tech - 63 employees	201.6 man-years	26,800	5,402,900
Clerical Staff - 11 emp.	34.1 man-years	21,400	729,700
Sub-Contractor Labor			
R & M - 51 men	2.5% capital/yr	26,800	4,100,400
Security & Janitorial - 11 men	38.5 man-years	21,400	823,900
Miscellaneous			
R & M Supplies	3.3% capital/yr	-	5,490,000
R & M Services	0.7% capital/yr	-	1,098,000
Operating & Admin. Supplies	\$20,000/month	-	840,000
Equipment Lease	Allowance	-	150,000
Vehicle Lease	Allowance	-	50,000
Vehicle Operation	Allowance	-	75,000
Travel	Allowance	-	100,000
Telephone & Telegraph	2,000/month	-	96,000
Taxes & Insurance	1.2% capital/yr	-	1,980,000
Hiring & Relocating, 26 professionals		25,000	650,000
Operating Contractor Expenses			
Non-site G & A	7 man-years	60,000	420,000
Construction Inspection	3 man-years	60,000	180,000
Fee			2,100,000
			<u>34,096,825</u>
Contingency 20%			6,819,365
Total			<u>40,916,190</u>

5.0 CAPITAL COST OF PILOT PLANT

5.1 Basis for Estimate

The estimate prepared for this report was established under the terms of the contract. It stipulated that the Task 3 estimate should be a "Type 4 - Preliminary" as shown in Figure 5.1. This type of estimate includes the following elements:

- Capacity
- Facility Description
- Plant Layout
- Time to Prepare Estimate
- List of Equipment - Major Equipment Priced
- General Arrangements - Approved by Client
- Outline Scope (General Plant Features)
- Electrical Motor List with Horsepower
- Piping and Instrument Diagrams
- One-Line Electrical Drawings

Using these elements, the probable contingency for a "green field plant" will be 15 percent, i.e. for a facility for which historic plant costs on which to support the estimate do not exist. The confidence level for this type of estimate, however, is well established in industrial and engineering practice especially when prepared by experienced estimators having available carefully collected cost data and actual quotations and quantities for equipment and materials. The estimate accuracy increases with the availability of multi-vendor quotations, the number of drawings and specifications prepared and a well organized set of schedules for the engineering, procurement and construction of the plant facilities.

Details supporting this estimate are to be found in the several volumes comprising this report. This first volume contains the technology basis on which the pilot plant is designed and the schedules on which the estimate is based; Volume II and III contain the equipment and motor lists, the equipment specifications and selected budget type quotations used in the estimate; Volumes IV and V contain the general plant specifications for concrete, structural steel, civil work, piping materials, instrumentation and data acquisition system and electrical materials. Volume VI contains all published drawings prepared for the design and estimating of the pilot plant. A number of informal sketches prepared to assist the quantity takeoff efforts of the estimators for various materials, are not published in this report. Also, assisting the engineering and estimating efforts for this plant, is a 3/8 inch scale model of the main process areas constructed from drawings and equipment data supplied with the quotations. The model shows in some detail the process area layouts, arrangements and elevations of equipment.

FIGURE 5.1.1.1

ESTIMATE TYPES

DESCRIPTION	TYPE	ELEMENTS REQUIRED																			
		PRODUCT, CAPACITY	FACILITY DESCRIPTION	PLANT LAYOUT	TIME TO PREPARE ESTIMATE	LIST OF EQUIP. - MAJOR EQUIP. PRICED	GENERAL ARGGMTS. - APPROVED BY CLIENT	OUTLINE SCOPE (GENERAL PLANT FEATURES)	ELECTRICAL MOTOR LIST WITH HP	PIPE & INSTRUMENT DIAGRAMS	ONE LINE ELECTRICAL DRAWINGS	DRAWINGS OF PIPE SYSTEM RUNS	PRELIMINARY DESIGN DRAWINGS	DETAIL EQUIPMENT LIST - PRICED	DETAIL SCOPE OF WORK	DETAIL CONSTRUCTION DRAWINGS	DETAIL SPECIFICATIONS	SUBCONTRACT & VENDOR-FIRM L.S. QUOTES			
BID	7	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	
ENGINEERS	6	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
DEFINITIVE	5	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
PRELIMINARY	4	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
CONCEPTUAL	3	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
MAGNITUDE	2	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
RESEARCH STUDY	1	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•

The contingency for this estimate is 13 percent. It was developed based on evaluations of selected equipment quotations used in the capital cost estimate as well as quantities and unit prices used in the estimate for civil, structural, piping, electrical and instrumentation work. Thus, the contingency is a composite of amounts and factors applied to either individual items or categories of items in the estimate.

Since no factual starting date exists for the project, escalation has been excluded from the estimate. Similarly, no fee has been included for the same reason and until a complete project plan has been established. The estimate assumes that no constraints or delays are encountered during the course of the project for any reason.

5.2 Summary Capital Cost Estimate

The estimated capital cost expressed in 1979 dollars for the pilot plant and shown in Figure 5.2.1, is \$52,311,000 excluding fee and escalation. Figure 5.2.2 shows the direct construction costs summarized by subfacility accounts.

An added allowance of approximately \$1,000,000 to the total pilot plant capital cost should include the following items:

- Land and right-of-ways
- Soils investigation and testing
- Environmental and other government related permits
- Taxes
- Plant effluent services beyond plant plot limits
- Access roads to plant plot
- Electrical and utility services to plant plot
- Capital spare equipment and materials
- Office furniture and laboratory equipment
- Shop tools and equipment

Estimate Criteria

- Scope

The scope for this estimate covers design and construction (on a construction management basis) of a pilot plant, extracting 25 TPD of alumina from clay. The plant site is assumed to be level and for purposes of cost reference within a 50 mile radius of Augusta, Georgia.

- Drawings

The estimate is based on approximately 95 drawings consisting of general arrangements, plan and elevations, P&ID's and one-line diagrams contained in Volume VI.

- Specifications

Based on general specifications contained in Volume IV and V, including additional explanatory engineering and project department data.

- Estimate Format

Chart of subfacility accounts as shown at the end of this section.

- Quantities

Quantity surveys made for all process areas on a unit cost basis consistent with available information on drawings and specifications such as general arrangement, P&ID's and one-line electrical drawings with reference to applicable notes for specifications. In addition, numerous sketches and drawings marked "For Estimating Purposes Only" have been used for the estimate.

- Labor and Burden - Manhours

Labor and burden costs include all craft labor, burdens, supervisors and construction equipment operators and oilers. The typical wage rate used for the assumed plant location is \$16.03 for a composite crew of one labor hour. Construction manhour estimates are taken from historical and published information on a composite crew basis.

- Material

Cost of construction materials used by craft labor F.O.B. point of shipment. Excludes consumables, except form lumber, which is direct charged.

- Sales Taxes

Excluded

- Equipment Usage

Costs for construction equipment (excluding operator and oiler) including maintenance labor costs, fuel, oil, repair parts, operating supplies, etc., using rental rate. Move in and out, and cost of standby time is included. Excludes small construction tools costing less than \$500.00 each.

- Contracts (Construction Management Basis)

All costs for contracted work including, but not limited to, all managed contractor's labor, materials, construction equipment, capital equipment, overhead and profit.

- Equipment

The first half of 1979 cost of capital equipment F.O.B. point of shipment, excluding sales taxes according to specifications and quotations contained in Volumes II and III. All quotes prior to 1979 were escalated to present day 1979 costs. Capital equipment purchased erected in place by a contractor or vendor is included under Subcontracts.

- Freight

Freight costs were obtained by factor, applied to dollar values for classes of applicable equipment and materials, and applied to all purchased items.

- Indirect Costs

Individual estimates have been made for Process Design Engineering, Construction Management and for Project Management, Engineering and Procurement.

- Contingency

A composite contingency of 13 percent is included in this estimate, based on an analysis and applied to all costs included in this estimate.

- Escalation

All costs are present day, through mid-year 1979.

- Project and Construction Schedules

Schedules covering a span of 32 months are inclusive of design and construction. No factual starting date exists. The estimate assumes that no constraints or delays are encountered during the course of the project for any reason.

Cost Exclusions for Capital Cost Estimate

- Costs for preparing documentation and obtaining environmental and other regulatory permits and licenses from federal, state and local authorities.
- Permits, licenses and royalties if applicable. (Exception: The license fee for the ACH decomposition system)

is included as an unspecified amount in the quotation for the equipment.)

- Costs of studies, testing and other related services prior to detailed engineering.
- Interest during construction and financing costs.
- Costs of any fees for process engineering, detailed engineering, procurement and construction management services.
- Operational staff and employee housing.
- Escalation.

Additional Notes for Estimate

The following assumptions and conditions apply to this estimate:

- Standard methods for excavation and backfill work by machine.
- Site assumed to be level, and accessible. Spread footing foundation design.
- All information in this estimate is based on drawings, specifications and other data available at time of report draft completion. Changes in scope (drawings, specifications, schedules, etc.) resulting from review of the draft report are not included in this estimate.

Chart of Subfacility Accounts

<u>Description</u>	<u>Sub. No. Group</u>
● Earthwork and Foundation Preparation	- 1000
- Structural Excavation	
- Structural Backfill	
- Other	
● <u>Concrete Work</u>	- 2000
- Concrete including Forms, Rebar, and Embedded Steel	
- Other	

<u>Description</u>	<u>Sub. No. Group</u>
● <u>Metal Work</u>	- 3000
- Structural Steel	
- Miscellaneous Steel	
- Grating	
- Ducts	
● <u>Architectural</u>	- 4000
- Buildings	
- Other	
● <u>Permanent Equipment</u>	- 5000
- Purchased Equipment (except turnkey supplies)	
- Pumps, Fans, Conveyors, Agitators, Screens, Hoppers, etc.	
- Motors	
- Tanks, Vessels, etc.	
● <u>Piping</u>	- 6000
- Pipe and Fittings	
- Handling, Storage and Inventory	
- Erection	
- Supports and Pipe Racks	
- Valves	
- Plumbing	
- HVAC	
● <u>Electrical Work</u>	- 7000
- Starters, MCC's, wiring, cable trays, etc.	
- Installation	

Description

Sub. No. Group

- Other Work
- Turnkey Type Equipment
- Field Erected Tanks
- Painting and Coatings
- Insulation
- Instrumentation

- 8000

FIGURE 5.2.1
ALUMINA PILOT PLANT PROJECT
U.S. BUREAU OF MINES
SOUTHEASTERN U.S.A.

ESTIMATE SUMMARY

AREA	DIRECT CONSTRUCTION COSTS	MANHOURS	LABOR & BURDEN	EQUIPMENT USAGE	MATERIAL	SUBCONTRACT	FREIGHT	EQUIPMENT	TOTAL
0100	Clay Preparation	25,115	\$ 389,700	\$ 92,200	\$ 313,700	\$ 76,000	\$ 20,500	\$ 402,400	\$ 1,294,500
0200	Clay Calcination	15,470	223,900	38,400	154,500	1,846,000	13,900	315,200	2,591,900
0300	Leaching	14,965	239,800	38,900	143,700	-	26,900	651,600	1,100,900
0400	Mud Thickening and Washing	19,580	314,100	47,600	257,700	-	30,800	751,500	1,401,700
0500	Filtration	4,620	51,700	7,600	39,600	50,400	3,900	94,200	247,400
0600	Solvent Extraction	16,925	242,600	34,500	216,500	67,700	19,500	462,800	1,043,600
0700	Evaporation	12,950	179,100	27,400	211,200	100,800	13,900	325,200	857,600
0810	Primary Crystallization	29,750	447,800	74,300	347,700	175,100	41,900	987,900	2,074,700
0820	Secondary Crystallization	32,535	481,900	90,500	420,800	202,100	49,900	1,189,900	2,425,100
0830	Crystal Dissolving	4,880	78,200	16,000	78,100	-	9,900	241,100	423,300
0900	ACH Decomposition and Calcination	73,920	127,200	14,200	152,400	4,818,000	5,700	135,900	5,253,400
1000	Acid Recovery and Preparation	22,110	307,800	45,600	475,300	173,100	28,300	686,700	1,716,800
1100	Bleed Stream Treatment	30,515	489,000	81,800	320,600	-	64,600	1,572,000	2,528,000
1200	Alumina Storage and Handling	3,810	46,700	4,700	26,200	119,200	2,300	58,700	257,800
1400	Process Waste Disposal and Process Drain System	20,375	287,400	63,000	331,900	133,500	10,200	236,400	1,062,400
2100	Steam, Feedwater and Condensate	11,335	181,800	40,200	76,400	-	10,200	245,100	553,700
2200	Cooling Water and Water Treatment	19,955	305,500	39,800	219,400	50,400	6,000	142,800	763,900
2300	Plant and Instrument Air	4,115	66,000	9,300	33,500	-	4,900	120,700	234,400
2400	Fuel Systems	2,330	33,500	4,200	32,400	22,000	1,600	35,900	129,600
2500	Reagents Handling	3,240	52,000	6,400	24,800	-	6,600	89,100	178,900
2700	Electrical Distribution	9,305	149,200	11,900	355,000	106,000	15,100	377,000	1,014,200
2800	Domestic and Fire Water Systems	5,640	90,400	18,800	118,800	-	3,100	77,000	308,100
2900	Yard Piping	57,340	919,500	190,100	1,505,700	-	17,100	-	2,632,400
5100	Site Preparation	750	12,000	13,000	-	-	-	-	25,000
5200	Site Improvements	30,175	351,100	65,700	238,100	567,200	5,700	23,400	1,251,200
5600	Service Buildings	27,415	362,100	73,200	261,600	302,000	900	257,800	1,257,600
5700	Administration Buildings	10,710	124,500	20,900	105,400	165,200	600	15,000	431,600
5800	Yard and Road Lighting	2,420	38,800	3,100	74,400	-	-	-	116,300
5900	Communications	650	10,400	800	5,000	-	-	-	16,200
	Subtotal: DIRECT CONSTRUCTION COSTS	512,900	\$6,603,700	\$1,164,100	\$6,540,400	\$ 8,974,700	\$414,000	\$9,495,300	\$33,192,200

DISTRIBUTABLE DIRECT COSTS

Subcontractor's Overhead & Profit
Vendor/Supplier Technicians
Construction Plant

TOTAL: DISTRIBUTABLE DIRECT COSTS

TOTAL: DIRECT CONSTRUCTION COSTS

PROCESS DESIGN ENGINEERING
CONSTRUCTION MANAGEMENT
PROJECT MANAGEMENT, ENGINEERING & PROCUREMENT
CONTINGENCY
ESCALATION
PFE

TOTAL: ESTIMATE OF COST

2,861,800
142,000
724,000
3,727,800
\$36,920,000
1,412,000
2,537,000
5,412,000
6,030,000
NOT INCLUDED
NOT INCLUDED
\$52,311,000

ALUMINA PILOT PLANT PROJECT
 U.S. BUREAU OF MINES
 SOUTHEASTERN U.S.A.

ESTIMATE SUMMARY BY SUB-FACILITY ACCOUNTS

AREA	1000	2000	3000	4000	5000	6000	7000	8000	FREIGHT	TOTAL \$
0100 Clay Preparation	26,900	221,100	326,700	35,800	511,600	-	101,300	50,600	20,500	1,294,500
0200 Clay Calcination	6,700	101,000	71,000	-	432,800	31,400	71,100	1,864,000	13,900	2,591,900
0300 Leaching	2,600	18,700	14,700	-	671,200	101,000	192,300	1,100,900	26,900	1,100,900
0400 Mud Thickening and Washing	3,600	12,000	10,000	-	754,400	237,400	154,500	199,000	30,800	1,401,700
0500 Filtration	600	6,200	2,500	2,500	108,100	32,300	12,200	81,600	3,900	247,400
0600 Solvent Extraction	2,600	12,000	27,700	-	507,400	193,000	109,200	172,200	19,900	1,043,600
0700 Evaporation	3,100	20,700	27,000	-	351,800	206,200	25,900	208,700	13,900	857,600
0810 Primary Crystallization	5,600	59,100	55,900	-	1,131,000	250,400	116,700	414,100	41,900	2,074,700
0820 Secondary Crystallization	4,400	45,200	45,600	-	1,388,300	372,400	87,300	432,000	49,900	2,425,100
0830 Crystal Dissolving	700	4,600	5,300	-	277,300	77,100	15,500	32,900	9,900	423,300
0900 ACH Decomposition and Calcination	800	23,900	-	-	55,500	105,400	111,600	4,950,500	5,700	5,253,400
1000 Acid Recovery and Preparation	-	-	-	-	784,900	481,500	101,900	320,200	28,300	1,716,800
1100 Bleed Stream Treatment	2,800	35,900	62,500	-	1,718,900	223,000	123,200	297,200	64,600	2,528,000
1200 Alumina Storage and Handling	-	-	-	-	181,700	-	72,600	1,200	2,300	257,800
1300 Process Waste Disposal and Process Drain System	65,900	39,800	24,400	-	184,200	333,000	129,900	275,000	10,200	1,062,400
2100 Steam, Feedwater and Condensate	1,800	19,700	700	-	367,400	87,900	18,600	47,400	10,200	553,700
2200 Cooling Water and Water Treatment	1,500	37,600	2,000	-	190,900	393,000	62,900	70,000	6,000	763,900
2300 Plant and Instrument Air	400	7,700	-	-	130,600	34,900	24,800	31,100	4,900	234,400
2400 Fuel Systems	-	-	-	-	36,200	40,000	3,800	48,000	1,600	129,600
2500 Reagents Handling	-	3,200	-	-	42,700	6,500	61,700	98,200	1,600	178,900
2700 Electrical Distribution	-	-	-	-	-	-	999,100	-	15,100	1,014,200
2800 Domestic and Fire Water Systems	16,000	251,300	1,202,300	-	-	305,000	-	-	3,100	308,100
2900 Yard Piping	25,000	-	-	-	-	898,100	-	247,600	17,100	2,632,400
5200 Site Improvements	53,800	442,000	654,000	40,000	23,400	13,900	11,400	7,000	-	25,000
5600 Service Buildings	68,000	346,300	142,100	167,900	-	179,600	55,500	299,300	900	1,237,600
5700 Administration Buildings	8,400	85,700	38,900	126,300	-	121,300	90,400	-	600	431,600
5800 Yard and Road Lighting	-	-	-	-	-	-	116,300	-	-	116,300
5900 Communications	-	-	-	-	-	-	16,200	-	-	16,200
Subtotal: DIRECT CONSTRUCTION COSTS	299,500	1,793,700	2,710,800	372,500	9,850,200	4,724,300	2,727,100	10,300,100	414,000	33,192,200

5.3 Engineering, Procurement and Construction Schedules Summary

The schedules for the pilot plant cover all phases of the work, including engineering, procurement and construction. The schedules in this section are as follows:

- Overall Master Schedule Summary
- Principal Construction Contracts Schedule
- Principal Equipment and Materials Purchase Schedule
- Estimated Construction Manpower
- Estimated Cash Flow Requirements

5.3.1 Basis for Schedules

The following criteria have been used in developing the schedules:

- Construction Plan

In accordance with the Basis for the Estimate (refer to section 5.1 of this section), (a) the plan provides construction durations unrestricted by any site or weather conditions, and (b) the erection of the administration buildings and some of the service be started as early as possible.

- Notice to Proceed

"Notice to Proceed with Project" is that point when the owner-selected contractor may begin with detailed plant design, procurement and construction commitments. Since no factual start date exists, all schedules are expressed in project months.

- Critical and/or Governing Specifications

Critical specifications are defined as critical to project completion; governing specifications are defined as key items essential to support the proposed construction schedule. Work must start on both as soon as notice to proceed is given. Two months have been allowed as the minimum time for non-process related specifications to be prepared and requests for quotation issued. Four months have been allowed as the minimum time for other specifications to be prepared and request for quotation issued.

- Procurement Cycle

A period of three months has been provided for submittal of vendor quotations, evaluation, approval and award.

- Total Project Duration

This was determined by delivery information supplied by vendors during the study.

5.3.2 Development of Schedules

The schedules were developed in progression, using the following criteria:

- Review of Quotations

Each quotation was reviewed and information regarding manufacturing durations, deliveries and construction durations were incorporated into the schedules.

- Preliminary Scheduling Method

Summary conceptual work schedules were prepared by area assuming no construction restraint on the start of any area.

A schedule was determined for each process area, having start-to-end durations as determined by earliest deliveries of critical and/or major equipment for that area.

As a first step, delivery and lead time were plotted for critical/major equipment and contract items in accordance with the selected quotations. Durations for placing concrete for foundations were scheduled conservatively to match the delivery dates. Subsequently, durations for other crafts such as piping, equipment setting, electrical and instrumentation were then imposed and plotted on this conceptual schedule.

Upon completion of this effort, durations for concrete work were extracted from each area bar chart and summarized on a schedule summary for concrete work. This schedule was appropriately adjusted to support an even spread for concrete labor manpower. The starting point for each area was then determined from this data.

- Principal Construction Contracts Schedule - Figure 5.3.2

A listing of major construction contract packages was established based on type of work and process areas. The required construction and lead time was developed for those packages from the data on the conceptual area bar charts and summarized. Dates were then established for bid invitations and award of contracts.

- Principal Equipment and Materials Purchase Schedule - Figure 5.3.3

The same approach was used here as for the construction contracts explained above. Delivery dates for equipment and continuous material delivery requirements were established to meet the construction package schedules. Appropriate vendor engineering and fabrication time was then added to arrive at required award dates.

- Overall Master Schedule Summary - Figure 5.3.1

The development of the above schedules results in the following summarized overall master schedule:

- Overall Project - 32 months
- Area 1 Clay Preparation - 23.5 months
- Area 2 Clay Calcination - 28.5 months
- Area 3 Leaching - 24.5 months
- Area 4 Mud Thickening & Washing - 25 months
- Area 5 Filtration - 24.5 months
- Area 6 Solvent Extraction - 23.5 months
- Area 7 Evaporation - 24.5 months
- Area 8 Crystallization - 28 months
- Area 9 ACH Decomposition & Calcination - 32 months
- Area 10 Acid Recovery & Preparation - 29 months
- Area 11 Bleed Stream Treatment - 29.5 months
- Area 12 Alumina Storage & Handling - 21 months
- Area 13 Process Waste Disposal & Process Drain System - 21.5 months
- Administration Building - 15.5 months
- Service Buildings - 19 months

5.3.3 Constraints on Schedules

Start of construction (or a critical point in the construction activity), is restrained by the supply of equipment or the start and duration of a construction contract. Major constraints for this program are as follows:

- Award of Critical Specifications
 - ACH Decomposition
 - Bleed Stream Treatment System
- Award of Governing Specifications
 - Pre-Engineered Buildings
 - Structural Steel
 - Field Erected Carbon Steel Tanks
 - Field Erected Steel Storage Bins
 - Site Preparation
 - Concrete

5.3.4 Estimated Construction Manpower - Figure 5.3.4

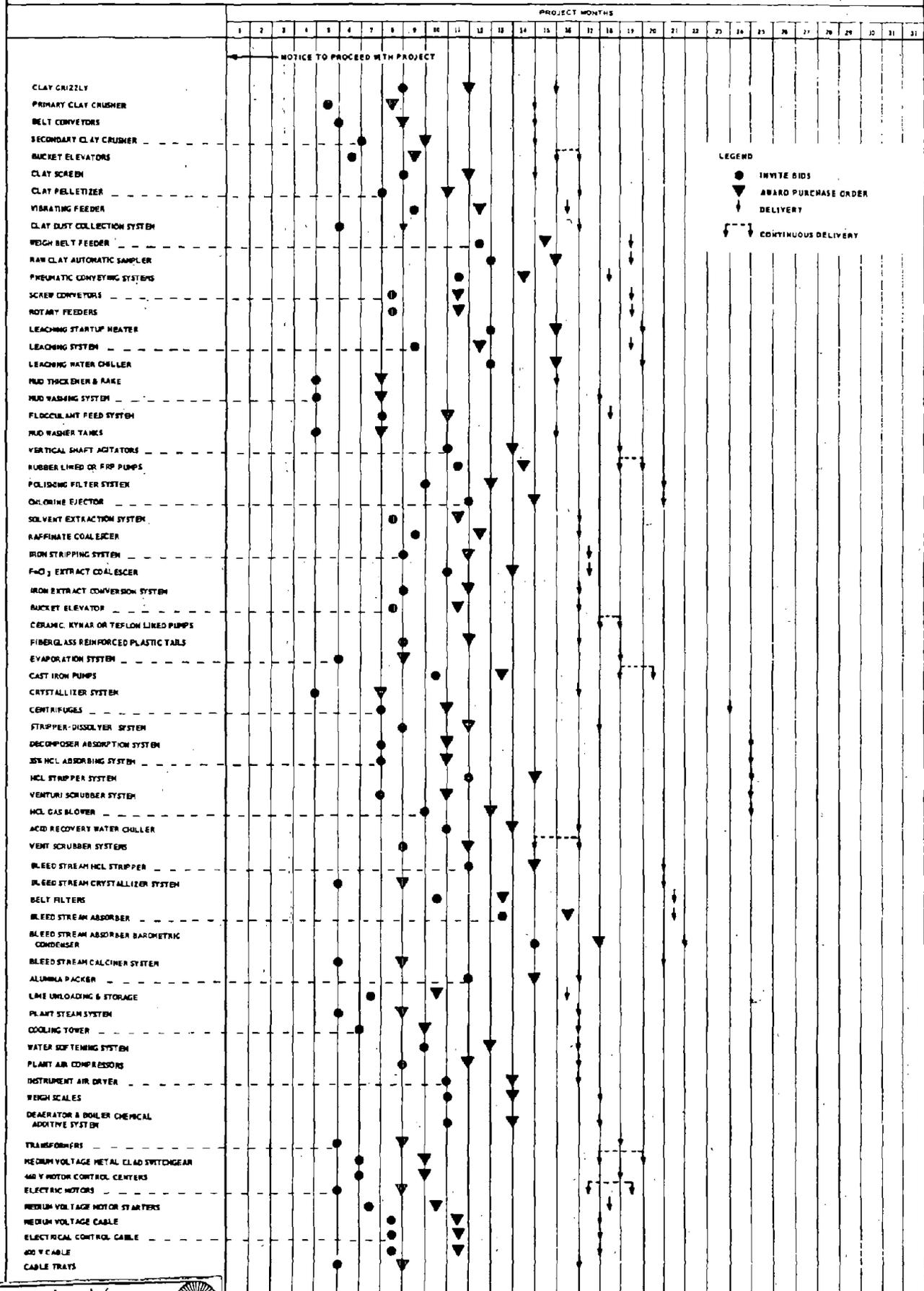
The start and duration for each of the construction activities for each area of the plant covered by the project schedule have been combined with the estimated man-hours given in the capital cost estimate to produce a manpower curve.

5.3.5 Estimated Cash Flow Requirements - Figure 5.3.5

The equipment and contractor costs have been combined with manpower costs for engineering and construction activities for the duration of the project to produce a project cash flow curve spanning a period of 12 quarters. Also included are costs for equipment, material and labor as reflected in the estimate and as shown in the Principal Equipment and Materials Purchase Schedule and the Principal Construction Contracts Schedule. The cash flow table below the curve shows quarterly expenditures excluding escalation and fee.

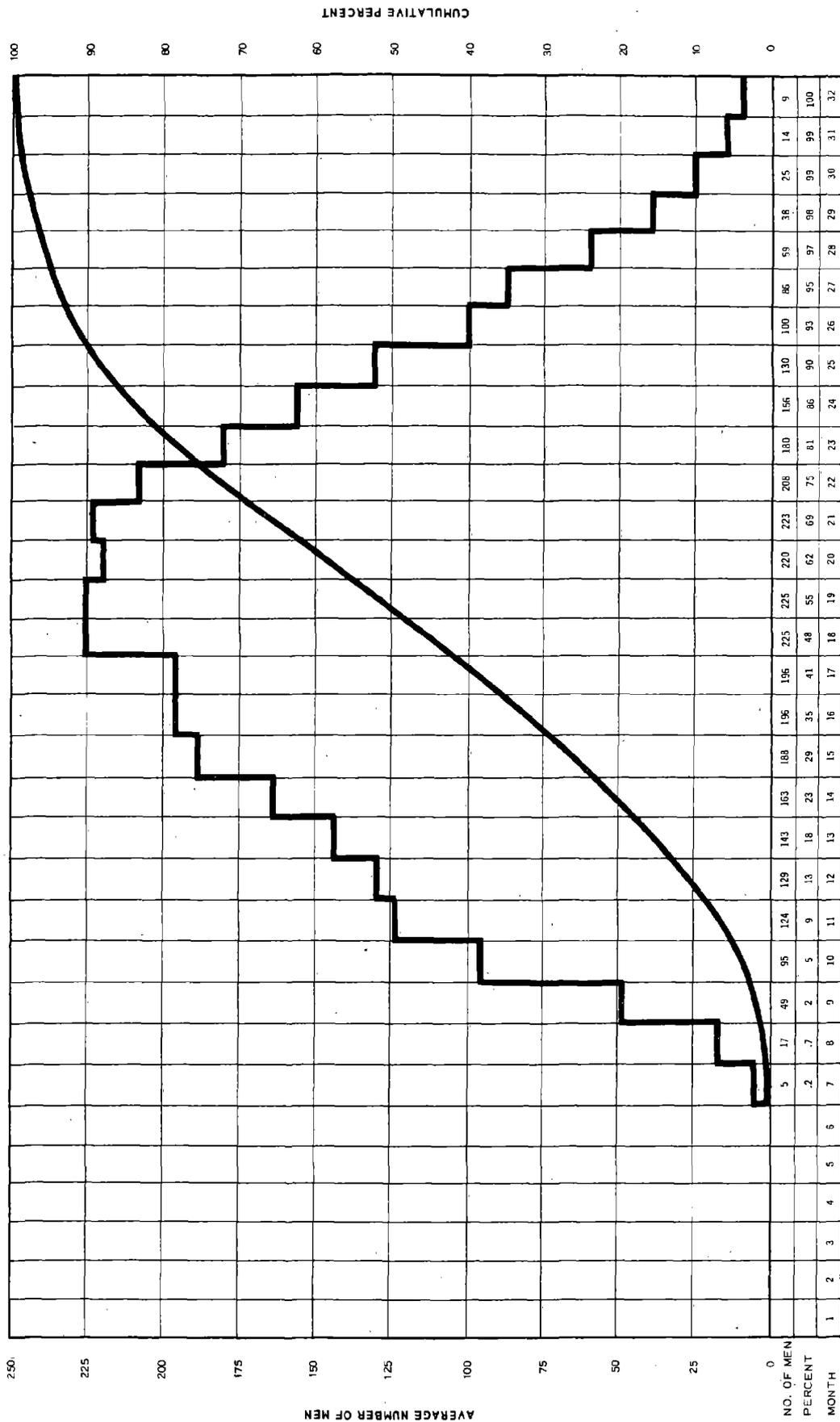
FIGURE 3.3.3

PRINCIPAL EQUIPMENT AND MATERIALS PURCHASE SCHEDULE



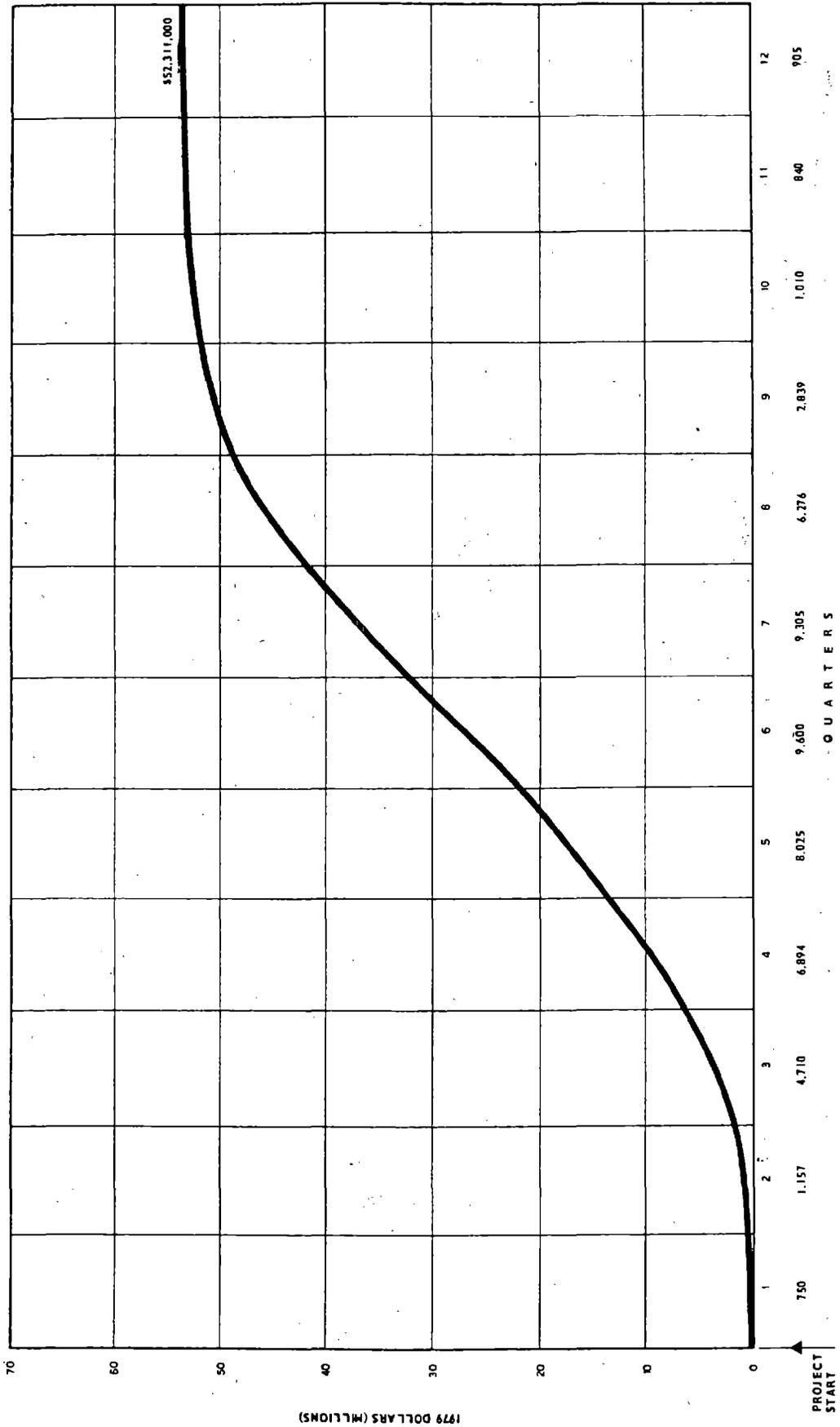
LEGEND
 ● INVITE BIDS
 ▼ AWARD PURCHASE ORDER
 ◆ DELIVERY
 ─── CONTINUOUS DELIVERY

FIGURE 5.3.4
ESTIMATED CONSTRUCTION MANPOWER



NOTE: TOTAL MANHOURS 512,400

FIGURE 5.3.5
ESTIMATED CASH FLOW REQUIREMENTS



6.0 COMPARATIVE COSTS FOR COMMERCIAL SIZE PLANTS

6.1 Summary

The purpose of this section is to provide operating and capital cost estimates for a 500,000 TPY alumina plant based on the technology proposed for the 25 TPD pilot plant in Task 3.

In order to provide continuity with previous cost estimates for commercial plants, the costs are presented in the form of a comparison with the commercial plant cost estimate included in the Task 2 report in February, 1978. It should be noted that the Task 3 costs are presented in November, 1977 dollars in order to provide a ready comparison with the previous Task 2 estimate of that date.

The Task 2 commercial plant cost estimates were based on the process information available in 1977. Since that time a considerable amount of test work has been done in the Bureau of Mines research stations or by vendors on Bureau of Mines contracts to provide additional information on the technology of the process.

Crystallization test work by the Bureau revealed that it would be necessary to redissolve and recrystallize the ACH crystals produced in the first crystallization step in order to meet the stringent specifications for P_2O_5 in product alumina of 0.001 weight percent.

Test work by Envirotech showed that the settling rates used in the Task 2 process design, which were based on early mini-plant data, were overly conservative. As a result 10 thickeners were eliminated from the plant design.

Further study of the design of the bleed stream section of the process showed advantages to a revised system which could reject water from the process as well as recover HCl in the form of HCl gas for use in crystallization.

As a result of these design changes the capital and operating cost estimates for a 500,000 TPY alumina plant as shown in Table 6.1, were increased by \$18.5 million and \$12.07 per ton of alumina, respectively, compared to the Task 2 estimate. However, it is probable that the increase in operating cost could be reduced to \$4.92 by the use of further waste heat recovery. (Refer to Waste Heat Recovery section.)

6.2 Discussion of Capital Costs

An analysis of the capital cost changes of the Task 3 estimate compared to the Task 2 estimate is shown below:

Mining Capital

- This item was unchanged.

Process Plant Capital

- Process Equipment

The purchased cost of equipment was increased by \$9.10 per annual ton of alumina by the following revisions to the equipment list:

- a. Eliminate 10 thickeners and associated equipment for mud settling and washing due to substantially higher settling rate data obtained by Eimco/Envirotech at Boulder City.
- b. Add one complete set of crystallization equipment including crystallizers, centrifuges, pumps, tanks necessary to produce ACH crystals in a secondary crystallization step.
- c. Add one set of ACH dissolving equipment including vessels, pumps and reboilers as required to dissolve ACH produced in primary crystallization.
- d. Revise bleed stream treatment to provide one ACH dissolver, one 2 stage evaporative ACH crystallizer and one fluid bed calciner with HCl recovery.

- Other Direct Capital

This item was increased by \$20.36 per annual ton of alumina which represents the increased cost to install the above equipment including erection, foundation, piping, electrical, structures, etc.

- Indirect Capital and Freight

This item was increased by \$6.72 per annual ton of alumina which represents higher engineering and freight charges for installing the additional process equipment discussed above.

Waste Disposal Capital

- This item did not change.

Working Capital

- This item was increased by \$0.77 per annual ton of alumina which represents a higher on-site inventory for coal and supplies due to higher usage rates in the revised process.

6.3 Discussion of Operating Costs

An analysis of the changes in the operating costs of the Task 3 estimate relative to the Task 2 estimate is shown below:

Ore

- Ore costs were unchanged.

Reagents

- Reagent costs were unchanged.

Utilities

- These costs were increased by \$9.75 per ton of alumina which represents the cost for the coal (\$30/T) used to generate steam used to desorb HCl gas in the dissolver after primary crystallization and in bleed stream treatment. A breakdown of the process fuel use is shown in Table 6.2. However, a definite potential exists for a considerable reduction in energy consumption through the use of waste heat recovery.

Some waste heat recovery was incorporated in the design for a 500,000 TPY alumina plant developed in Task 2 of this study. This design made provision for the recovery of 5.7 MM Btu per ton of alumina, of waste heat from the condensation of decomposer off-gases and the use of a waste heat boiler on the stack of the molten salt heater used to heat the indirect fired ACH decomposer.

The Task 3 costs shown in Table 6.1 include the same waste heat recovery provisions as in the Task 2 design. However, the potential exists for additional waste heat recovery which would reduce the process fuel usage by an additional 5.9 MM Btu per ton alumina. A preliminary investigation of these opportunities indicates that they are technically feasible. (The additional capital cost for the recovery of the 5.5 MM Btu/T of product quoted above has not been included in the Task 3 plant capital cost estimate; it being felt that the increase should be relatively small since the heat transfer systems for cooling of the various streams cited are included in the Task 3 estimate. The principal change is that instead of using cooling water to cool these streams other process streams requiring heat would be used. Some increase in heat transfer areas may be required to accommodate smaller temperature differences between heat transfer streams.) The elements of the potential waste heat recovery are summarized in Table 6.3 and are discussed below.

Waste Heat Recovery(a) ACH Decomposition Off-Gases

The off-gases from the ACH decomposer and the ACH calciner are at 600°F and 300°F respectively. These gases are primarily HCl and H₂O vapors, most of which must be condensed in the acid recovery section of the plant. In cooling and condensing these vapors to about 220°F, approximately 4.7 million Btu's per ton of alumina produced are generated. This heat can be used to provide some of the evaporation heat requirements in the process.

About 0.3 MM Btu per ton of alumina of waste heat can be recovered by cooling the off gases from about 600°F to 300°F. This heat can be used to heat the FeCl₃ -clay reactors in the solvent extraction section.

(b) Molten Salt Heater Stack Gas

A heater is provided to heat the molten salt which is used to heat the indirect fired ACH decomposer. The stack gases from this heater are expected to be about 950°F and the total heat content above 350°F is about 2.1 MM Btu per ton of alumina produced. Since this stream is at a relatively high level it should be possible, with the use of judicious engineering, to transfer this heat to the main ACH crystal dissolver which has a bottom temperature of 260°F.

(c) Crystallization

The heat of solution of HCl gas into the sparging crystallizers generates about 7.8 MM Btu's per ton of product. It is highly probable that some of this heat could be used to supply the 2.5 MM Btu/T product required by the bleed stream evaporative crystallizers. The heat transfer would be made between a circulating stream from the first stage sparging crystallizers (temperature decrease 2-5°F) and a circulating stream from the bleed stream evaporative crystallizers (temperature increase 2-5°F).

The pilot plant design uses a 160°F operating temperature for the first stage crystallizers primarily to protect the crystallizer lining material. However, there appears to be no reason why these crystallizers could not be operated at up to 200°F if a more temperature resistant crystallizer lining were used.

The task of transferring the 2.5 MM Btu/T product to the evaporative crystallizers becomes easier at higher sparging crystallizer temperatures and a temperature of 180°F was assumed in this example.

A proposal to study the effect of higher operating temperatures on the sparging crystallizer operation is made in Section 7 of this report.

(d) Leach Slurry

The slurry leaving the leach reactors must be cooled from 220°F to 140°F before the mud thickeners in order to protect the thickener lining. Currently the design incorporates a flash tank system for this purpose. However, the leach slurry could be partly cooled to 180°F by exchanging heat with the liquor feed (145°F) to the primary first stage crystal reslurry tank. A smaller flash tank and barometric condenser system could then be used to cool the leach slurry from 180°F to 140°F.

(e) Leach Tank Off Gases

The heat of reaction in the clay leaching tanks is sufficient to maintain these tanks at boiling (220°F). The off gases must be condensed

and returned to the leach tanks. With proper engineering the condensation heat load of about 1 million Btu's per ton of alumina produced could be transferred to the ACH dissolver in the crystallization section.

Labor

- These costs were increased by \$1.02 per ton of alumina.
- Operating labor increased by 2 men/shift due to the additional crystallization equipment in secondary crystallization and bleed stream treatment. R & M is labor is also increased due to the additional equipment.

Supplies

- R & M supplies increased by \$0.87 per ton of alumina (5.3%).

Other

- Taxes and insurance increased by \$0.43 per ton of alumina.

Table 6.1

CAPITAL COST COMPARISON
500,000 TPY Al₂O₃
1977 COSTS

	Task 3 HCl Gas <u>Sparging</u>	Task 2 HCl Gas <u>Sparging</u>
<u>Capital Cost Difference, \$/A.Ton*</u>		
Mining Capital Differences	-0-	Base Case
Process Plant Capital Differences		
Process Equipment	9.10	Base Case
Other Direct Capital (Foundations, Structures, Piping, Utilities, Electrical, etc.)	20.36	Base Case
Indirect Capital and Freight	6.72	Base Case
Waste Disposal Capital Difference	-0-	Base Case
Work Capital Difference	.77	Base Case
Total Capital Cost Difference, \$/A.Ton	36.95	Base Case

Note: Costs shown represent difference in capital dollars between process and base case. Positive values represent higher capital requirements. Costs are calculated based on plant sized to produce 500,000 ton/yr of alumina, and reflect differences in capital spending for each ton of alumina capacity.

*\$/A.ton: Dollars/Annual Ton of Al₂O₃

OPERATING COST COMPARISON
500,000 TPY Al₂O₃
1977 COSTS

	Task 3 HCl Gas <u>Sparging</u>	Task 2 HCl Gas <u>Sparging</u>
<u>Operating Cost Difference, \$/Ton Al₂O₃</u>		
Ore (Excluding Mining Capital)	-	Base Case
Reagents (Acids & Bases)	-	Base Case
Utilities (Oil, Coal, Power, Water)	9.75 ¹	Base Case
Labor (Operating, R & M, Supervision, Administration)	1.02	Base Case
Supplies (R & M, Operating, Processing)	.87	Base Case
Other (Taxes and Insurance)	.43	Base Case
Total Difference, \$/Ton Al ₂ O ₃	12.07 ²	Base Case

¹Could be reduced to \$2.60/T by using the waste heat recovery potential outlined in the Waste Heat Recovery section.

²Could be reduced to \$4.92/T by using the waste heat recovery potential outlined in the Waste Heat Recovery section.

Note: Costs shown represent the difference between the two processes. Positive values represent higher costs; negative values represent lower costs. Costs are calculated at 500,000 ton/yr rate and reflect differences per ton Al₂O₃ produced.

Table 6.2

TASK 3 PROCESS

FUEL REQUIREMENTS FOR 500,000 TPY ALUMINA
FROM CLAY PLANT

	<u>MM Btu/T of Alumina</u>
Clay calcination	4.7
FeCl ₃ treatment with clay	0.1
Evaporation	3.9
ACH dissolution in crystallization	5.5
ACH decomposition	12.2
ACH calcination	2.2
Bleed stream	
Evaporative crystallizers	3.3
ACH dissolution	1.6
Waste calcination	<u>0.6</u>
	34.1
Waste heat recovery*	<u>11.2</u>
	22.9

*See details in Table 6.3

Note:

This assumes use of vacuum pumps to provide vacuum wherever necessary. All fuel requirements supplied as coal, except for ACH calcination which must be oil or gas.

TABLE 6.3

WASTE HEAT RECOVERY

<u>WASTE HEAT SOURCE</u>		<u>WASTE HEAT RECIPIENT</u>		<u>Amount of Heat Transferred MM.BTU/T. PROD.</u>	<u>Method</u>
<u>Stream</u>	<u>Temp. °F</u>				
ACH decomposer	300	Main evaporators		3.9	Heat evaporator (operating temperature 160°F) by circulating liquor from acid condenser through evaporator heater (liquid in - 220°F, liquid out - 175°F)
ACH decomposer off - gases	300	Bleed stream evaporative crystallizers		0.8	Same as above.
First stage sparging crystallizers	180	Bleed stream		2.5	Heat evap. crystallizer (operating temperature - 160°F) by circulating first stage crystallizer liquor through evap. crystallizer heater. Thereby heating evap. crystallizer liquor 2° - 5°F and cooling sparging crystallizer liquor 2° - 5°F.
Molten salt heater	950	Crystallization Section dissolver		2.1	Heat reboiler on dissolver by cooling stack gas from 950°F to 350°F.
Leach reactor slurry effluent	220	Crystallization Section dissolver		0.8	Preheat liquor feed to slurry mix tank from 145°F to 200°F by cooling leach slurry from 220°F to 180°F.
Leach tank off - gases	220	Crystallization Section dissolver		1.0	Preheat slurry feed to central section of dissolver tower by condensing leach tank off-gases at 220°F.
ACH decomposer	600	FeCl ₃ - Clay reactor		0.1	Heat circulating liquor stream from FeCl ₃ - clay reactor to about 220°F.

6.4 Equipment List for 500,000 TPY Alumina from Clay Plant

Clay Preparation Area

Truck Dump Hoppers (4) - 70 ton receiving hopper of fabricated steel with vertical support legs. (1 spare)

Truck Dump Wobbler Feeders (4) - 350 ton/h. 60 in wide x 18 bar, 11 1/2 in pitch series with 2 in openings between bars. (1 spare)

Under Wobbler Feeder Belt Conveyors (4) - 350 ton/h. (1 spare)

Primary Crushers and Chutes (4) - 350 ton/h. double roll crushers, feed size - 12 in, product size - 2 in (1 spare)

Primary Crusher Belt Conveyors (4) - 350 ton/h. (1 spare)

Stockpile Distributor Belt Conveyor (1) - 1,000 ton/h

Stockpile Enclosure (1) - 100,000 ton capacity, 200 ft x 600 ft, A-frame steel building

Stockpile Reclaim Hoppers (2) - 25 ton capacity, (1 spare)

Stockpile Wobbler Feeders (2) - 270 ton/h, 60 in wide x 18 bar, 9 in pitch series with 3/4 in openings between bars. (1 spare)

Secondary Crusher Feed Belt Conveyors (2) - 300 ton/h

Secondary Crushers and Chutes (3) - 150 ton/h double roll crusher, feed size, minus 3/4 in product size. (1 spare)

Secondary Crusher Discharge Belt Conveyors (2) - 300 ton/h

Secondary Crusher Bucket Elevators (2) - 250 ton/h

Secondary Crushing Screens and Chutes (3) - 150 ton/h, plus 3/4 in oversize, minus 20 mesh undersize. (1 spare)

Disc Pelletizers (4) - 35 ton/h, 18 ft dia x 24 in deep minus 20 mesh feed size, 3/4 in product size. (1 spare)

Disc Pelletizers Recirculation Belt Conveyor (1) - 100 ton/h

Raw Clay Surge Bin Feed Belt Conveyor (1) - 30 ton/h

Raw Clay Surge Bin (1) - 1,000 ton capacity, 24 ft dia x 73 ft str. side

Raw Clay Weighfeeders (2) - 150 ton/h

Calciner Feed Belt Conveyors (2) - 150 ton/h

Equipment List (Cont)

Clay Calciner System (Fluid-Bed)(2) - 3 stage fluidized bed dryer with air heater - 19 in dia calciner - primary cooler followed by a secondary cooler for 135 ton/h feed and 95 ton/h product. Gas scrubber included.

Coal Pulverizers (4) -2 units - 5 ton/h, 2 units 2.5 ton/h at 20 mesh size

Calciner Discharge Apron Feeder (2) - 95 ton/h

Calcine Bucket Elevator (2) - 190 ton/h. (1 spare)

Calcine Vibrating Screen (3) - 100 ton/h, 20 mesh screen

Cage Mill Grinder (2) - 150 ton/h, 2-roll 60 in dia grinder, 20 mesh product size. (1 spare)

Cage Mill Discharge Belt Conveyor (2) - 200 ton/h. (1 spare)

Storage Bin Bucket Elevator (2) - 200 ton/h. (1 spare)

Calcined Clay Storage Bins (2) - 1,500 ton capacity, 30 ft dia x 88 ft str. side

Calcine Weighfeeders (4) - 200 ton/h. (2 spares)

Leach Tank Feed Conveyors (2) - 125 ton/h

Chutes and Hoppers

Dust Collection System-Bag houses with piping, blowers, and controls

Leaching, Thickening, CCD Washing, and Filtration Area

Leach Tank Screw Feeders (4) - 81 ton/h, totally sealed feeders, rubber-lined carbon steel construction. (2 spares)

Leach Tanks (8) - 16 ft dia x 26 ft str. side constructed of carbon steel shell with polymeric membrane and acid-resistant brick lining. (2 trains)

Leach Tank Condenser (4) - 1,000 ft² heat transfer area. Impervious graphite heads and tubes, carbon steel shell. (2 spares)

Leach Tank Recirculation Pumps (8) - 1,000 gpm fluoropolymer-lined cast iron construction. (4 spares)

Condensate Pumps (4) - 100 gpm, fluoropolymer-lined cast iron construction. (2 spares)

Flash Tanks (2) - 11 ft dia x 11 ft str. side constructed of carbon steel shell with polymeric membrane and acid-resistant brick lining

Equipment List (Cont)

Flash Tank Vapor Condensers (4) 5,500 ft² heat transfer area.
Impervious graphite heads and tubes, carbon steel shell.

Condensate Tanks (2) - 60,000 gal, 22 ft dia x 24 str. side. Rubber-lined carbon steel construction with steam ejectors

Leach Slurry Pumps (4) - 1,500 gpm, fluoropolymer-lined cast iron construction. (2 spares)

Dilute HCl Transfer Pumps (4) - 200 gpm, rubber-lined cast iron construction. (2 spares)

Leach Tank Agitators (8) - Fluoropolymer-lined carbon steel construction

Sand Thickener Tank (1) - 106 ft dia x 15 ft str. side. Carbon steel shell, covered and rubber-lined. Four units operating in parallel. (1 spare)

Sand Thickener Rake Mechanism (1) - two-arm rake, rubber-lined carbon steel construction. (1 spare)

Sand Thickener Underflow Pumps (2) - 200 gpm, rubber-lined cast iron construction. (1 spare)

Sand Thickener Overflow Pumps (2) - 610 gpm, rubber-lined cast iron construction (1 spare).

Sand Washer Tanks (12) - 106 ft dia x 15 ft str. side, carbon steel shell, covered and rubber-lined. (2 trains - 6 unit CCD washing)

Sand Washer Rake Mechanisms (12) - two-arm rakes rubber-lined carbon steel construction

Sand Washer Underflow Pumps (24) - 920 gpm, rubber-lined cast iron construction. (12 spares)

Sand Washer Overflow Pumps (24) - 1,030 gpm, rubber-lined cast iron construction. (12 spares)

Waste Tank (1) - 45,000 gal, 17 ft dia x 24 ft str. side, rubber-lined steel shell

Sand Slurry Pumps (2) - 2,750 gpm, rubber-lined cast iron construction. (1 spare)

Flocculant Preparation System (1) - includes hoppers, feeders, mix tank and agitator, transfer pumps, storage tank, and solution pumps

Filter Presses and Repulpers (5) - 2,900 ft² retractable shell filter. 815-gal/min feed with polypropylene leaves and all wetted parts of rubber-lined carbon steel or titanium. (2 spares)

Equipment List (Cont)

Press Cake Relay Tanks (2) - 4,600 gal, 10 ft dia x 8 ft str. side.
Rubber-lined carbon steel construction

Repulped Slurry Pumps (4) - 200 gpm, rubber-lined cast iron
construction. (2 spares)

Vibrating Screens (2) - 4 1/2 ft x 5 1/2 ft polypropylene construction

Collecting Sluice (1) - rubber-lined carbon steel construction

Spent Filter Aid Dumpers (4) - portable bins

Filtrate Surge Tanks (2) - 235,000 gal, 36 ft dia x 32 ft str. side,
rubber-lined carbon steel construction

Filtrate Pumps (4) - 2,500 gpm, rubber-lined cast iron construction.
(2 spares)

Filter Aid Preparation System (1) - includes storage tank,
conveyor, weighfeeder, slurry tanks and agitators, slurry pumps, and
liquor pumps

In-line Chlorine Blenders (2) - chlorine gas pipeline blender

Iron Removal, FeCl₃ Stripping, FeCl₃ Conversion Area

Solvent Surge Tank (1) - 65,000 gal, 22 ft dia x 24 ft str. side, FRP-
lined carbon steel construction

Solvent Surge Tank Pumps (2) - 1,000 gpm, 316 stainless steel
construction. (1 spare)

Head Tanks (8) - 2,000 gal, FRP construction

Mixer-Settlers (15) - 65 ft x 20 ft x 10 ft deep concrete tanks with
FRP lining

Mixer-Settler Covers (15) - segmented self-supporting FRP construction

Loaded Solvent Pumps (4) - 1,000 gpm, 316 stainless steel construction
(2 spares)

Mixing Pumps (15) - 316 stainless steel construction

Mixing Pump Baffles (15) - FRP construction

Raffinate Pumps (4) - 2,400 gpm, rubber-lined carbon steel construction.
(2 spares)

Raffinate Surge Tank (1) - 235,000 gal, 36 ft dia x 32 ft str. side,
FRP-lined carbon steel construction

Equipment List (Cont)

Raffinate Storage Transfer Pumps (2) - 2,400 gpm, rubber-lined carbon steel construction. (1 spare)

Loaded Solvent Surge Tank (1) - 65,000 gal, 22 ft dia x 24 ft str. side, FRP-lined carbon steel construction

Loaded Solvent Surge Transfer Pumps (2) - 1,000 gpm, 316 stainless steel construction. (1 spare)

Regenerated Solvent Pumps (4) - 1,000 gpm, 316 stainless steel construction. (2 spares)

Wash Water Tanks (2) - 15,000 gal, 13.5 ft dia x 17 ft str. side, FRP construction

Wash Water Tank Pumps (4) - 260 gpm, rubber-lined cast iron construction. (2 spares)

Spent FeCl₃ Pumps (4) - 260 gpm, rubber-lined cast iron construction. (2 spares)

FeCl₃ Liquor Surge Tank (1) - 40,000 gal, 20 ft dia x 23 ft str. side, FRP-lined carbon steel construction

FeCl₃ Liquor Surge Tank Pumps (2) - 260 gpm, rubber-lined cast iron construction. (1 spare)

Decanol Unloading Pump (1) - 400 gpm, cast iron construction

Decanol Storage Tank (1) - 5,500 gal, 10 ft dia x 12 ft str. side, carbon steel construction

Decanol Blending Pumps (2) - 250 gpm, cast iron construction. (1 spare)

Kerosene Unloading Pump (1) - 400 gpm, cast iron construction

Kerosene Storage Tank (1) - 5,500 gal, 10 ft dia x 12 ft str. side, carbon steel construction

Kerosene Blending Pumps (2) - 250 gpm, cast iron construction (1 spare)

Alamine Unloading Pump (1) - 400 gpm, cast iron construction

Alamine Storage Tank (1) - 5,500 gal, 10 ft dia x 12 ft str. side, carbon steel construction

Alamine Blending Pumps (2) - 250 gpm, cast iron construction. (1 spare)

Equipment List (Cont)

Solvent Blend Tank (1) - 4,000 gal, 7 ft dia x 15 ft str. side, carbon steel construction

Solvent Blend Tank Agitator (1) - thorough agitation. 304 stainless steel construction

12.5 Ton Load Cell (1) - 6 h cycle to fill, agitate, and empty

Solvent Make-up Pumps (2) - 40 gpm, cast iron construction. (1 spare)

Calcined Clay Conveyor (1) - pneumatic system with 28 ton/h capacity at 80 lb/ft³ with blower package, 6-in conveying line, surge hopper, binvents, and fan. Carbon steel construction

Calcined Clay Surge Bin (1) - 350 ft³ capacity, 8 ft dia x 6 ft str. side carbon steel construction

Reactor Feed Conveyor (1) - 3.5 ton/h, rubber-lined

FeCl₃ Digestion Reactors (3) - 10,000 gal, 12 ft dia x 15 ft str. side, carbon steel shell, rubber membrane, and acid brick lining

Digestion Reactor Agitators (3) - butyl rubber-lined carbon steel construction

Digestion Reactor Steam Heaters (3) - 1-in steam coils for 3.8 x 10⁶ Btu/h heat transfer service. Inconel 625 construction

Condensed Vapor Pumps (2) - 60 gpm, fluoropolymer-lined cast iron construction. (1 spare)

Digestion Slurry Surge Tank (1) - 60,000 gal, 22 ft dia x 23 ft str. side, rubber-lined carbon steel construction

Digestion Slurry Surge Tank Pumps (2) - 290 gpm, fluoropolymer-lined cast iron construction. (1 spare)

Digestion Slurry Surge Tank Agitator (1) - 100% solid suspension, rubber-lined carbon steel construction

Pregnant Liquor Evaporation, AlCl₃·6H₂O Crystallization, and Centrifuging Area

Pregnant Liquor Evaporators (5) - single effect systems in parallel operation consisting of 25 ft dia x 20 ft str. side evaporator with demister pad, vapor and circulating piping, and barometric condenser with 2-stage ejector system. Evaporation rate = 750 ton/d of H₂O. Total connected, horsepower is 700 hp and steam requirements are 1,000 lb/h at 100 psig. Constructed of rubber-lined carbon steel

Equipment List (Cont)

Feed Liquor Preheaters (2) - 2,500 ft² heat transfer area. Impervious graphite heads and tubes, carbon steel shell

Liquor Circulating Heaters (5) - 10,000 ft² heat transfer area. Impervious graphite heads and tubes, rubber-lined carbon steel shell

Liquor Circulating Pumps (10) - 15,000 gpm, rubber-lined cast iron construction. (5 spares)

Evaporator Transfer Pumps (10) - 600 gpm, rubber-lined cast iron construction. (5 spares)

Evaporator Hotwell (1) - 12 ft x 20 ft x 8 ft, FRP-lined concrete with segmented self-supporting FRP cover complete with scrubber for vent gas

Evaporator Hotwell Pumps (2) - 10,500 gpm, rubber-lined cast iron construction. (1 spare)

Evaporator Cooling Tower (1) - 11,000 gpm (10 wt % HCl) capacity from 130° F to 80° F with an evaporation rate of 240,000 lb/h single-cell cross-flow tower 66 ft x 42 ft with one 225-hp fan. Drift loss = 0.008% of circulating liquid. Tower with 5-stage scrubber to reduce HCl in vapor to 1.5 ppm. Tower constructed of FRP, spray bars and nozzles of PVC

Cooling Tower Sumps (4) - 15 ft x 18 ft x 8 ft, acid brick-lined concrete

Spray Scrubber Pumps (2) - 2,000 gpm, rubber-lined cast iron construction. (1 spare)

Cooling Tower Transfer Pumps (2) - 10,000 gpm rubber-lined cast iron construction. (1 spare)

2-Stage Crystallizers (6) - 200,000 lb/h AlCl₃ · 6H₂O crystals capacity. 36 ft dia x 26 ft str. side crystal suspension containers with central pipes, circulating piping, and coolant circulating piping all fabricated of rubber-lined carbon steel. Also included are two heat exchangers with 5,400 ft² of heat transfer surface constructed of impervious graphite heads and tubes and carbon steel shell

Coolant Circulating Pumps (24) - 4,400 gpm, rubber-lined cast iron construction. (12 spares)

1st Stage Crystallizer Product Pumps (12) - 1,360 gpm rubber-lined cast iron construction. (6 spares)

2nd Stage Crystallizer Product Pumps (12) - 1,000 gpm rubber-lined cast iron construction. (6 spares)

Emergency Storage Falling Film Absorbers (4) - 1,500 ft² heat transfer surface. Impervious graphite heads and tubes, carbon steel shell

Equipment List (Cont)

Emergency Storage Absorption Towers (4) - 11 ft² dia x 25 ft str. side with 1,425 ft³ of 2 in ceramic packing. Constructed of carbon steel shell, lined with acid-resistant brick with a polymeric membrane

Emergency Storage Tanks (6) - 230,000 gal, 35 ft dia x 32 ft str. side, rubber-lined carbon steel construction with 1 ft dia x 6 ft high vent scrubber

Emergency Storage Reboilers (4) - 400 ft² heat transfer surface. Impervious graphite heads and tubes, carbon steel shell construction

Emergency Storage Pumping Systems (6) - 3,000 gpm, rubber-lined cast iron construction

1st Stage Crystallizer Product Centrifuges (8) - 51 ton/h two-stage pusher type. Constructed of rubber-lined carbon steel housing, Hastelloy process contact parts and titanium screens. Horsepower requirements are 100 hp for basket, 60 hp for push plate

1st Stage Centrate Tanks (8) - 10,000 gal, 10 ft dia x 18 ft str. side, rubber-lined carbon steel construction

2nd Stage Crystallizer Product Centrifuges (8) - 51 ton/h two-stage pusher type. Constructed of rubber-lined carbon steel housing, Hastelloy process contact parts and titanium screens. Horsepower requirements are 100 hp for basket, 60 hp for push plate

2nd Stage Centrate Tanks (8) - 7,000 gal, 10 ft dia x 12 ft str. side, rubber-lined carbon steel construction

Hydrocyclones (16) - 24 in dia handling a flow of 460 gal/min with a sp. gr. differential of 1.7. Rubber-lined carbon steel construction

1st Stage Centrifuge Wash Acid Tanks (8) - 3,000 gal, 8 ft dia x 8 ft str. side, rubber-lined carbon steel construction

1st Stage Centrate Pumps (16) - 1,000 gpm, rubber-lined cast iron construction. (8 spares)

2nd Stage Centrate Pumps (16) - 650 gpm, rubber-lined cast iron construction. (8 spares)

Centrifuge Wash Acid Pumps (16) - 300 gpm, rubber-lined cast iron construction. (8 spares)

Crystal Bins (3) - 36,000 ft³, 36 ft dia x 36 ft str. side. Bulk density = 83 lb/ft³. Rubber-lined carbon steel construction

Crystal Weighfeeders (3) - 110 ton/h capacity, bulk density = 83 lb/ft³ rubber-lined carbon steel construction

Equipment List (Cont)

Recycle Crystal Slurry Tank (1) - 200 gal, 3 ft dia x 4 ft str. side, rubber-lined carbon steel construction

Recycle Crystal Slurry Tank Agitator (1) - rubber-lined carbon steel construction. Exposed metal to be zirconium-clad carbon steel

Recycle Crystal Slurry Pumps (2) - 20 gpm, rubber-lined cast iron construction. (1 spare)

1st Stage Centrifuge Conveyors (3) - 55 ton/h, bulk density of 83 lb/ft³. Gas-tight, rubber-lined carbon steel construction

2nd Stage Centrifuge Conveyors (3) - 55 ton/h, bulk density of 83 lb/ft³. Gas-tight, rubber-lined carbon steel construction

Crystal Conveyors to Decomposers (3) - 110 ton/h, bulk density of 83 lb/ft³. Gas-tight, rubber-lined carbon steel construction

Centrifuge Discharge Transfer Conveyors (3) - 110 ton/h, bulk density of 83 lb/ft³. Gas-tight, rubber-lined construction

Crystal Slurry Tanks (2) - 30,000 gal, 16 ft dia x 16 ft dia x 16 ft str. side, 60 cone bottom, carbon steel, acid-brick lined over polymer membrane.

Crystal Slurry Tank Agitators (2) - high temperature FRP-lined steel. Exposed metal to be zirconium-clad

Crystal Slurry Tank Pumps (4) - 2,500 gpm, polymer-lined cast iron construction. (2 spares)

Dissolving Tanks (2) - 63,000 gal, 12 ft dia x 75 ft tangent to tangent, carbon steel; acid brick-lined over polymer membrane

Dissolving Tank Agitators (2) - 10 stages, high temperature FRP-lined steel. Exposed metal to be zirconium-clad

Dissolving Tank Reboilers (4) - 4,300 ft² heat transfer surface. Constructed of impervious graphite heads and tubes; carbon steel shell

Dissolving Tank Pumps (4) - 1250 gpm, polymer-lined cast iron construction. (2 spares)

AlCl₃·6H₂O Decomposition Area

Decomposer Feed Screw Conveyors (4) - 60 ton/h of Aluminum Chloride Hexahydrate crystals at 83 lb/ft³. Gas-tight, rubber-lined carbon steel construction

Decomposer Flash Dryers (4) - 6 ft dia x 70 ft str. side, constructed of carbon steel shell, polymeric membrane, castable refractory and brick lining

Equipment List (Cont)

Decomposer Flash Dryer Cyclones (4) - 10 ft dia x 20 ft str. side, constructed of carbon steel shell, polymeric membrane, castable refractory and brick lining

Indirect Fired Fluid Bed Decomposers (2) - 21 ft dia x 30 ft str. side, constructed of carbon steel shell, polymeric membrane, castable refractory, and brick lining. Includes Inconel 625 distributor plate and heating coils (2) - 2,585 x 10 ft long heat tubes of finned 1 in diameter pipes of Inconel 625 construction

Indirect Fired Decomposer Cyclones (2) - 10 ft dia x 16 ft str. side, constructed of carbon steel shell, polymeric membrane, castable refractory, and brick lining

Indirect Fired Decomposer Gas Coolers (4) - 1,375 ft² heat transfer surface. Carbon steel shell and tube construction

Indirect Fired Decomposer Gas Blowers (3) - 18,400 SCFM capacity at 14.7 psia suction pressure and 300°F suction temperature. Discharge pressure is 20.7 psia. (1 spare)

Indirect Fired Decomposer Product Hoppers (2) - carbon steel construction

Indirect Fired Decomposer Heating Systems (3) - 200 x 10⁶ Btu/h Dowtherm "A" heating system including coal pulverizer, coal feed hoppers, feed system, combustion air blower, fluidized bed combustor with startup system, Dowtherm coils, cyclone collector, air preheater, circulating pumps, flash tanks, expansion tanks, and temperature controls

Waste Heat Boilers and Recycle Gas Heaters (3) - capacity of 85,000 lb/h of 100-psig steam supplied with 350,000 lb/h of 1,300°F waste gas

Heating System Coal Pulverizers (3) - 11.5 ton/h at 20 mesh

Heating System Ash Handling (3) - 1.1-ton/h system with blowers, piping cyclone, and ash bin

Heating System SO₂ Scrubbers (3) - 76,200 SCFM with vertical wet approach Venturi Scrubber, flooded elbows and cyclone separator capable of removing 99% of fly ash and dust and 96% of SO₂

Decomposer Flash Calciners (2) - 5 ft dia x 75 ft str. side, constructed of carbon steel shell, polymeric lining, castable refractory, and brick lining

Flash Calciner Cyclones (2) - 18 ft dia x 32 ft str. side, constructed of carbon steel shell, polymeric lining, castable refractory, and brick lining

Direct Fired Calciners (2) - 20 ft dia x 30 ft str. side, constructed of carbon steel shell, polymeric lining, castable refractory, and brick

Equipment List (Cont)

lining. Includes Inconel 625 distributor plate. 32 ton/h product capacity

Direct Fired Calciner Cyclones (2) - 21 ft dia x 38 ft str. side, constructed of carbon steel shell, polymeric lining, castable refractory, and brick lining

Direct Fired Calciner Oil Burners (2) - # 6 fuel oil burner system with air blowers, oil guns, and controls

Product Hopper and Rotary Valves (2) - 40 ton/h product capacity, product density = 60 lb/ft³. Carbon steel construction

Fluid Bed Coolers (2) - 32 ton/h product capacity. Cooling medium is both air and water through cooling coils. Product to be cooled from 1,800°F to 150°F. Heated air is used for product calciner combustion air

Alumina Conveying Systems (2) - 40 ton/h pneumatic system with blower package, 8 in conveying line, surge hopper, bin vents, and fan

Alumina Silos (2) - 7,500 ton capacity. 50 ft dia x 50 ft str. side with 60° cone bottom. Carbon steel construction

Bleed Stream Treatment

Bleed Stream HCl Stripper (1) - 6 ft dia x 40 ft tangent to tangent, carbon steel, acid brick lined over polymer membrane

Bleed Stream HCl Stripper Agitator (1) - 10 stages, high temperature FRP lined steel. Exposed metal to be zirconium clad

Bleed Stream HCl Stripper Reboiler (1) - 2,200 ft² heat transfer surface. Constructed of impervious graphite heads and tubes, carbon steel shell

Bleed Stream HCl Stripper Transfer Pumps (2) - 300 gpm polymer lined cast iron construction. (1 spare)

Bleed Stream Evaporation Systems (2) - 35 ton/h evaporation capacity 2-stage system consisting of 25 ft dia x 20 ft str. side evaporator bodies with demister pads, vapor and circulating piping and barometric condenser with 2-stage ejector system. Each 2-stage system complete with two liquor circulating heaters and 4 - 300 gpm liquor circulating pumps

Bleed Stream Evaporator Product Pumps (4) - 300 gpm polymer lined cast iron construction. (2 spares)

Bleed Stream Evaporator Centrifuges (2) - 56 ton/h two-stage pusher type. Constructed of rubber-lined carbon steel housing, Hastelloy process contact parts and titanium screens. Horsepower requirements are 100 hp for basket, 60 hp for push plate

Equipment List (Cont)

Bleed Stream 1st and 2nd Stage Evaporator Slurry Tanks (4) - 450 gal, 4 ft dia x 4 ft str. side with 60° cone bottom. High temperature FRP construction

Bleed Stream 1st and 2nd Stage Evaporator Slurry Tank Agitators (4) - High temperature FRP lined Construction. Exposed metal to be zirconium-clad

Bleed Stream 1st and 2nd Stage Evaporator Slurry Tank Pumps (8) - 50 gpm, polymer lined cast iron construction. (4 spares)

Bleed Stream 1st and 2nd Stage Evaporator Cyclones (2) - Polymer lined carbon steel construction

Bleed Stream Decomposer Feed Pumps (2) - 10 gpm polymer-lined cast iron construction. (1 spare)

Bleed Stream Evaporator Stripper (1) - 20 ft dia x 30 ft str. side rubber-lined carbon steel construction w/ceramic packing

Bleed Stream Decomposer System (1) - 3 ton/h capacity fluid bed residue calciner consisting of reactor, cyclone scrubber, cooler, absorber, exhaust fan, fluid bed cooler, burner system and pumps

Leach Acid Preparation, Wash Acid Preparation, and Dilute HCl Recovery Area

Acid Absorption Columns (4) - 13 ft dia x 48 ft high with 2,750 ft³ of 2 in ceramic packing rings in 3 sections, distributors and collectors. Constructed of carbon steel shell with acid resistant brick lining with polymeric membrane

Intermediate Cooling Heat Exchangers (4) - 500 ft² heat transfer surface. Impervious graphite heads and tubes, carbon steel shell

Intermediate Cooling Recirculation Pumps (8) - 100 gpm, fluoropolymer-lined cast steel construction. (4 spares)

Vapor Product Blowers (2) - 560 ton/d HCl gas and water vapor. Suction pressure is atmospheric and discharge pressure is 30 psig. Brake horsepower developed is 1,260 at 7,050 rpm. Housing constructed of cast steel and rotor of alloy steel. (1 spare)

Liquid Product Recirculation Pumps (8) - 2,300 gpm, fluoropolymer-lined cast steel construction

Wash Acid Falling Film Absorbers (4) - 1,500 ft² heat transfer surface. Constructed of impervious graphite heads and tubes, carbon steel shell

Equipment List (Cont)

Wash Acid Falling Film Absorber Vapor Product Blowers (2) - 19.5 ton/d HCl gas and water vapor. Suction pressure is atmospheric and discharge pressure is 30 psig. Brake horsepower developed is 600 at 10,000 rpm. Housing constructed of cast steel and rotor of alloy steel. (1 spare)

Wash Acid Falling Film Absorber Liquid Product Pumps (4) - 62 gpm, rubber-lined cast steel construction

Wash Acid Falling Film Absorber Liquid Product Heat Exchangers (4) - 400 ft² heat transfer surface. Impervious graphite heads and tubes, carbon steel shell construction

Wash Acid Absorption Towers (4) - 11 ft dia x 25 ft str. side with 1,425 ft³ of 2 in ceramic packing. Constructed of carbon steel shell lined with acid resistant brick plus a rubber membrane of fluoropolymers

Wash Acid Absorption Towers Circulating Pumps (8) - 1,250 gpm fluoropolymer-lined cast steel construction. (4 spares)

HCl Storage Tanks (4) - 230,000 gal, 35 ft dia x 32 ft str. side, rubber-lined carbon steel construction

Air Padding for HCl Unloading (2) - 1 ft dia x 8 ft str. side, packed vent scrubber of FRP construction

HCl Solution Make-up Pumps (4) - 22 gpm, rubber-lined cast steel construction. (2 spares)

Dilute Acid Gas Falling Film Absorbers (2) - 9,730 ft² heat transfer surface. Impervious graphite heads and tubes, carbon steel shell

Dilute Acid Gas Falling Film Absorber Product Pumps (4) - 166 gpm, rubber-lined cast steel construction. (2 spares)

Dilute Acid Gas Absorption Towers (2) - 7.5 ft dia x 25 ft str. side with 990 ft³ of 2 in ceramic packing rings. Constructed of carbon steel shell with fluoropolymeric lining. Teflon pad demister included

Utilities

Steam Plant and Auxiliary Systems (2) - 250,000 lb/h coal-fired boilers, 125 psig saturated steam. Complete with controls, ash handling, fly ash and SO₂ scrubber, feed water and condensate system, and coal pulverizers

Cooling Towers and Auxiliary Systems (1) - 12,000 gpm from 130°F to 90°F at 80°F ambient wet bulb temperature. Horsepower requirement is 250. Pumps and concrete basin are included

7.0 PROCESS DEVELOPMENT RECOMMENDATIONS

7.1 Introduction

Purpose

The process design for the pilot plant has been made using data provided by the Bureau of Mines from "mini-plant" tests of the process and equipment vendor's tests, published literature, and vendor expertise.

This information is adequate for the design and successful operation of the pilot plant to provide the desired process information and sufficient alumina for subsequent cell testing.

However, as with any process at this stage of development, there remain significant opportunities for optimization of the process. This could result in considerable cost savings since the pilot plant operating costs are about one million dollars per month.

The purpose of this section of the report is to identify research and development effort which can be carried out at less than pilot plant scale, in the interim prior to completion of the pilot plant, and which might benefit the pilot plant undertaking by providing information that would:

- a) Shorten the start-up period required until the pilot plant produces alumina meeting reduction grade specifications. Development of more information about the chemical nature of the chemical systems to be dealt with in certain sections is in this category.
- b) Permit, with relatively minor modifications in the pilot plant design, improvement in the process and reduction in the capital and operating cost for manufacturing alumina commercially by it.

Summary of Recommended Effort

Feed preparation, calcination, leaching and solid-liquid separation are all closely interrelated. A fundamental premise advanced is that calcination, leaching, and solid-liquid separation will all benefit from supplying to calcination, a clayfeed with carefully controlled particle size distribution and designing the leaching process to minimize attrition.

Studies of the fundamental chemistry of the crystallization process, the temperature at which crystallization is operated, and the rate of crystallization are recommended with the goal of optimizing this process section. The pilot plant operation will have a similar goal, but much valuable information can still be gained at lesser cost in smaller existing facilities since pilot plant operating costs will be about one million dollars per month. A determination can also be made as to whether or not the installation of relatively costly high-temperature resistant polymer linings in the pilot plant crystallizers would be worthwhile.

The development of design parameters for the decomposition of ACH is recommended.

Some additional study of the bleed stream treatment section of the process is recommended. This would confirm the comparatively well understood evaporative crystallization section of the design, and verify the operability of a variation of some well-known technology to the calciner, employed as a part of this process section. These studies could minimize the learning period during the startup of the pilot plant and thereby reduce the total operating period of the pilot plant with considerable savings.

Summary of Recommendations

These recommendations are discussed at length in the pages which follow this summary.

Feed Preparation

1. Development of an economical means to produce approximately spheroidal clay feed particles of a controlled narrow particle size distribution.
2. Find a binder suitable for use in pelletizing, or by other means provide increased resistance to clay particle attrition in processing following feed preparation.

Clay Calcination

3. Study methods for minimizing fines generation.
4. Determine the optimum (minimum) operating temperature of calcination with respect to alumina activation and solids residence time.
5. Determine optimum process conditions for direct firing of the clay calciner with coal.

Leaching

6. Study methods for minimizing the generation of clay fines in the leaching step.
7. Reevaluate as may be appropriate, and particularly on the basis of work presently under way at the Bureau's research center at Albany, the methods chosen for separating and washing the waste solids.

E. Bleed Stream Treatment

8. Verify that the countercurrent slurry-vapor contacting device chosen as the dissolver, will function as designed.
9. Develop additional information relative to the purity of ACH crystals, which can be produced in the evaporative crystallization section of bleed stream treatment, as a function of dissolved impurity concentrations in the mother liquor.
10. Develop optimum operating conditions for the fluidized solids calcination of the bleed stream final mother liquor.

11. Study the employment of SO_2 in the bleed stream calciner as the preferred means of converting alkali and alkaline earth metal components of the final mother liquor to solid sulfates for disposal.

F. Crystallization

12. Elucidate, insofar as practical, the physical chemistry of the crystallization process, particularly with respect to impurities and the reasons for their inclusion in the growing crystals, on the premise that better understanding will make improved operation possible.

13. Determine the effect of higher temperature on crystallizer operation.

14. Determine more precisely how much crystal production is practical per unit of crystallizer volume per unit of time.

15. Study the use of additives as a means of optimizing the crystallization process.

16. Carry out additional experiments to make certain that small amounts of inert gas present in HCl gas, supplied to crystallization in a cyclic process, do not interfere unacceptably with crystallization.

G. Thermal Decomposition

17. Determine experimentally in a 40 vol. % HCl - 60% H_2O the percent decomposition of ACH as a function of time at a series of selected constant fluid bed temperatures.

18. Determine experimentally, using ACH that was previously 90% decomposed, the % residual chloride, surface area, and α -alumina content of the product, as a function of time at a series of higher constant temperatures in the presence of combustion gases.

19. Repeat 21 in an H_2O vapor atmosphere.

20. Determine experimentally the effect of small amounts of NaCl, KCl, CaCl_2 , and MgCl on 20 and 21.

21. Conduct additional long-term studies on materials of construction for both stages of decomposition.

7.2 Discussion

The elements of the process used in the pilot plant design are all found in the published literature. Early investigators however were handicapped by very limited chemical knowledge of the systems employed and until recent years by the virtual absence of adequate thermodynamic data relative to these systems. Materials of construction choices were very limited, and chemical engineering techniques, which are common practice today, were available only in much less developed form if at all when the earlier efforts to extract alumina via HCl were in progress. Energy was much less

expensive in relation to other process cost elements during past years than it is now or is ever likely to be in the future. None of the various processes employing HCl was ever operated on a full industrial scale.

It is therefore not surprising that early investigators were not successful in their efforts to extract alumina from non-bauxitic raw materials via hydrochloric acid at an acceptable cost. It is also not surprising--although a substantial amount of useful engineering design information pertaining to the process selected for the preliminary design is available from the literature--that some important information has been reported only in work of doubtful validity, in work limited in scope, or is not available at all. One of the very useful functions of the preliminary design and the Miniplant program has been the identification of and focusing of attention on missing or poor information, as well as the disclosure of problem areas in actual operation of sections of the process.

It is possible for skilled engineers to work around some problems, to calculate some missing information, and to make judgments as to when the extrapolation of some available information may be reasonable. The cost of constructing and operating the proposed pilot plant is, however, large enough so that a great deal of effort to develop reliable engineering design data is justified.

The U.S. Bureau of Mines through the Miniplant program and otherwise, has been attempting to develop additional engineering design information with considerable success. Sufficient information is available at this time as a result of the Miniplant effort, in the published literature, or in the case of the thermal decomposition of ACH, through purchase of a vendor's process package based on proprietary technology, to permit the design, construction, and operation of a pilot plant which would produce alumina meeting reduction-grade specifications, from clay using the design process. There do remain, however, opportunities for optimizing the final design of the pilot plant, thereby potentially reducing its cost and increasing the probability that reduction-grade alumina may one day be commercially extracted from clay. There is some information of a fundamental nature which would be very useful in operating the pilot plant and which should be developed in a bench-scale research effort. There is also some information which could be developed in the pilot plant but which can be developed at less cost in a timely manner, in smaller existing facilities. It is the purpose of this section of the Task 3 report to identify and describe this additional smaller scale development work, which can benefit the design of the pilot plant, and/or provide better insight into its operation.

FEED PREPARATION

Clay employed in the Miniplant program to date--and in all known earlier developmental efforts which progressed beyond bench scale, with the single exception of the Pechiney H⁺ Process--has all undergone some combination of drying, crushing, and screening along with a calcining operation. Calcination has always been

carried out (with the exception of a single Miniplant program test) in a rotary kiln. The rotary kiln was operated with a generally coarse feed to avoid more than minimal elutriation of solids during operation. The coarse calcine was then crushed and screened to size. This tended to produce a calcine of uneven quality, because some large pieces of raw clay escaped complete calcination while at the same time the small ones were deadburned. A substantial fraction of very fine calcine particles was always produced in this process section. The coarse fraction of the sized feed was comprised primarily of angular particles which were more subject to attrition during leaching and slurry transport than spheroidal particles were. Recent work at the Bureau's Albany Research Center suggests that the settling and filtration characteristics of the clay particles leaving pre-leach can be improved by prior treatment to produce particles which are roughly spherical and have a fairly narrow size range.

As will become evident from discussion under leaching, the settling and filtration characteristics of the clay particles should be optimized in order to render economically feasible the early rejection of acid-soluble impurities from the overall process.

It is therefore recommended, for reasons to be discussed further in subsequent sections, that a system be developed for treating the clay feeds in order to deliver the clay to the process in a size range of approximately -10 +150 mesh. It is further recommended that a search be made for a method of preparation or a binder that would impart to the particles resistance to attrition or fracture to the maximum possible extent during calcination, leaching, and solid-liquid separation. The binder must, of course, be compatible with the process as a whole and must not introduce any environmental hazard in the calciner off-gases or in disposal to the environment of acid-insoluble waste solids. Candidate binders include but are not limited to, small amounts of solutions of aluminum chloride containing dissolved alumina, sulfuric acid, or of sodium carbonate, silicate or acid sulfate.

CLAY CALCINATION

All of the older work reported in the literature on the calcination of clay was done prior to the engineering development of fluidized solids technology. On the basis of present knowledge, this technology appears ideally suited for this unit operation from the following standpoints:

1. Accomplishing the calcination at a minimum consumption of energy which is considerably less than required for rotary kiln calcination.
2. Securing the best possible control of the calcination to assure that all of the particles are calcined sufficiently to fully activate them for leaching without any becoming deadburned, in order to maximize the yield of alumina from the calcine.
3. Producing a calcine having a particle size distribution which will be optimum for both leaching and subsequent liquid-waste solids separation, and will be resistant to attrition.

As part of the Miniplant program one test of fluidized solids clay calcination has been made to date with encouraging results. Enough information is presently available, including the results of the test performed, to make possible the inclusion of fluidized solids clay calcination in the pilot plant design. However, further studies in pilot-scale equipment would permit refinement of the design and generate added confidence in it. The major areas where additional information would be helpful are:

- a) Learning how to best minimize of fines generation during calcination.
- b) Determine the optimum (minimum) operating temperature of calcination consistent with respect to alumina activation, fuel consumption, and solids residence time.
- c) Learning how to produce a calcine consistent with (1) and (2) above, that will show maximum coherence and therefore experience minimum attrition in subsequent processing steps.
- d) Verifying that the energy source can be coal, directly fired, for conditions of satisfactory operation in accordance with (b) above.

It is recommended that existing vendor expertise in fluidized solids technology and vendor facilities available on a rental basis, be utilized to develop the information listed above. It is believed that this can be accomplished at a modest cost.

LEACHING

All of the comments as well as the research recommendations in this section are made on the judgment that the most cost-effective way to dissolve the alumina content of clay is to leach calcined clay at atmospheric pressure.

Primary Leach Design

The older literature and the Bureau's Miniplant program have provided an abundance of data upon which to base the pilot plant leaching design.

The proper choice of binder for pelletizing the clay fines entering calcination could make possible the production of calcine particles resistant to degradation during leaching. It is recommended that the design of leaching agitation and slurry forwarding be studied with the goal of reducing particle attrition and improving the settling and filtering characteristics of the mud.

BLEED STREAM TREATMENT

Dissolver

Some additional development work is recommended on the ACH dissolver (bleed stream HCl stripper of flowsheet) employed as the

first step of bleed stream treatment in the pilot plant as now designed. This work would have the goal of providing further assurance that this slurry-vapor contacting device would actually handle the slurry while providing the required number of transfer units/ plates. This work can best be accomplished in vendor facilities.

Evaporative Crystallization

The evaporative crystallization of ACH is the next step in the bleed stream section. Some further study of this crystallization at Miniplant scale using simulated concentrations of impurities in the mother liquor representing a range of leaching conditions, and in which impurity concentrations in the recovered ACH are compared with those in the mother liquor, is recommended. This is because the ACH from the first stage of bleed stream evaporative crystallization is returned to the process and the purity of this ACH could affect the purity of the first primary crystallizer ACH product. This work would verify the adequacy of the design and would provide additional insight into the operation of the bleed stream treatment section which can be obtained at a lesser cost by operation at Miniplant scale than in the pilot plant.

Calcination of Second Stage Crystallizer Mother Liquor

The pilot plant design includes the recovery of chloride values, conversion of aluminum along with any dissolved iron to oxides, and the conversion of alkali and alkaline earth metals to sulfates by a stagewise oxidizing fluidized solids calcination of the mother liquor from the second stage evaporative crystallizer. Sulfur dioxide equivalent to the alkali and alkaline earth metals present in the waste stream is oxidized within the calciner bed, converting these difficultly hydrolyzable chlorides to very stable sulfates and converting the contained chloride to free HCl.

The use of sulfur dioxide for the conversion of alkali and alkaline earth metals to sulfates is less costly than the use of sulfuric acid but, perhaps more importantly, permits better control of the conversion reaction and is expected to give better assurance of calciner operating stability.

The use of sulfur dioxide in the presence of oxygen and water vapor to convert alkali metal chlorides to sulfates with recovery of hydrogen chlorides is well established commercial technology. Fluidized solids calcination/hydrolysis of iron chloride solutions is also practiced on a commercial scale; but insofar as is known, there have been no attempts to calcine/hydrolyze a solution of the composition to be discharged from the second-stage bleed stream crystallizer, with simultaneous conversion of only alkali and alkaline earth metal chlorides to sulfates. It is therefore recommended, prior to undertaking construction of the pilot plant, that simulated waste solutions be calcined at pilot scale in the

manner specified by the design. This can probably best be done on a contract basis using pilot facilities operated by a vendor skilled in the use of fluidized solids techniques for the calcination of iron chloride solutions, and/or the conversion of NaCl or HCl to the corresponding sulfate with SO₂ (modern Hargreaves Process).

CRYSTALLIZATION

Crystallization Mechanism

Impurities

The crystallization of ACH by the controlled addition of hydrogen chloride gas has been studied by the Bureau of Mines at Miniplant scale and at small pilot scale by one crystallizer vendor. Neither these studies nor limited laboratory scale studies have elucidated the mechanism by which the crystallization takes place, nor in particular the mechanism by which impurities are included in the crystal product. These phenomena are deserving of a very careful examination from a physicochemical standpoint; because so little is known about them, there exists a reasonable possibility that a better understanding would offer a means of reducing the amounts of impurities included within the crystals. It is possible that an improved crystallization technique could by itself make recrystallization of ACH unnecessary, with considerable process capital and operating cost savings. This study is recommended on a high priority basis.

Temperature

The effect of temperature on crystallizer operation is at present almost completely unknown, operating temperature selection having been determined in large part by the properties of polymers used to line the crystallization equipment. The operating temperature in the last stage of a staged crystallization is severely limited by the equilibrium partial pressure of hydrogen chloride in contact with solution, but the first stage or stages could be operated at temperatures higher than have been chosen so far. It is probable that crystal purity will be improved by operation of the crystallizer at higher temperatures due to decreased solution viscosity and increased ionic diffusivities. Such operation also would increase the possibility for reclaiming the heat of absorption of hydrogen chloride employed in the crystallization process because the heat would be rejected from the crystallizer at a more useful temperature. The effect of temperature on crystallizer operation should be studied at Miniplant scale in order that somewhat more costly equipment capable of operating at higher temperature may be provided at the time of construction, if this is determined to be worthwhile.

Crystallization Rate

Only a small number of tests have been conducted to date, either at Boulder City or in the vendor's pilot facility, with the goal of determining the effect of the rate of crystal production on product crystal particle size distribution and chemical purity. It is recommended that the effect of this variable be explored thoroughly prior to undertaking construction of the pilot plant because of possible effects on the design and because the desired information can be developed at less cost on a Mini - or pilot-plant scale.

Additives

It is known that small amounts of additives, the amount and the chemical nature of the additive often being selected in an empirical way, can influence the habit and the purity of the crystal being formed in important ways. It is recommended the efforts be made to improve the crystallization process by this means prior to construction of the pilot plant.

Effect of Inert Gases

The hydrogen chloride gas used in the crystallizers will inevitably contain a small fraction of inert gas, in part because the entire acid recovery system is expected to be operated at slightly less than atmospheric pressure. This inert gas will enter the crystallizer mixed with the hydrogen chloride but will not dissolve in the mother liquor. It is expected to rise through the crystal slurry in the form of fine bubbles to the head space in the crystallizer, from where it will be vented through a scrubber to the atmosphere. Additional testing is required, probably best performed at Miniplant scale in Boulder City, to make certain that the inert gas bubbles will not cause unacceptable crystal nucleation, inclusions of mother liquor within the crystal structure, or otherwise interfere with crystallizer operation.

THERMAL DECOMPOSITION

The proposed process requires that approximately 90% or more of the chloride content of the ACH crystals produced in crystallization be converted to hydrogen chloride by hydrolysis in the absence of combustion product gases including nitrogen, so that hydrogen chloride gas remaining after partially condensing the decomposer off-gases will be suitable for return to crystallization without further processing.

Fortunately, it is known that ACH can be decomposed to the required extent at a temperature practical for the operation of metallic heat transfer surfaces. The relatively large energy requirement is also known. Fluidized solids techniques are obviously preferred for introducing ACH to and removing decomposed product from

the decomposer bed containing an atmosphere of HCl and H₂O vapor, and for transporting the large amount of heat required to the surface of individual particles undergoing decomposition. However, very little information is available on the chemical mechanism of the decomposition or the, equilibrium partial pressures of hydrogen chloride and water vapor present in contact with the decomposing solid, as a function of temperature and as decomposition proceeds. Also unknown is the rate of decomposition as a function of temperature and whether anything other than the rate at which energy can be transferred to the particles limits the rate of decomposition as it proceeds through removal of about 90% or more of the chloride. The design of heat transfer surfaces, the choice of materials of construction of them to give satisfactory service at elevated temperatures in the presence of solids whose abrasive properties are not well known, and the presence of a mixture of water vapor with hydrogen chloride, pose many questions. The designer must cope with the evolution of a very large volume of gas during decomposition in the first stage per unit of product, and he must choose between a relatively low operating temperature with a smaller volume of gas evolved over a longer time period versus a higher operating temperature with a large volume of gas being evolved in a short time, along with more severe conditions for materials of construction. His design must give assurance that the temperature of metallic materials of construction, subject to attack by liquid, is never permitted to drop below the dew point when hydrogen chloride is present.

Specifications agreed upon for reduction grade alumina by aluminum producers, who are participants in the Miniplant program, permit a maximum of 0.1% chloride in the product alumina with a target of .05%, and limit the α -alumina content to between 10% and 25%. The product from the indirectly heated decomposition stage must therefore be subject to further heating at a much higher temperature to reduce the chloride content to the required level and develop by phase transformation, the specified α -alumina content. It is generally believed that the temperature required to achieve these two objectives is higher than what is practical for use with metallic heat transfer surfaces; therefore a direct fired fluid bed is proposed along with acceptance of the added costs for clean fuel and for the recovery of some hydrogen chloride from decomposer off-gases containing hydrogen chloride at only a small partial pressure.

Given these uncertainties in regard to the existing technology, further research and development work on the decomposition step is recommended.

First Stage Decomposer

It would be most desirable, for the first stage decomposition, to determine experimentally at 40 volume % hydrogen chloride - 60 volume % water vapor (the stoichiometric gas composition produced

by decomposition) the percent decomposition of ACH as a function of time, at a series of selected constant fluid bed temperatures. This information, along with heat transfer data already available that can be applied to this decomposition, would permit the development by knowledgeable engineers of an independent design for the first stage decomposer. The information could be obtained by experiments at pilot or possibly even bench scale.

Second Stage Decomposer

For the second stage decomposer, batch demonstration tests should be run on ACH containing only 5-10% of its original chloride, at a series of selected constant temperatures using fluidizing gas approximately in composition, to that to be obtained from combustion of fuel oil. The % residual chloride, and the corresponding alumina surface area, should be determined as a function of time for each temperature, because these critical characteristics of the product alumina are interrelated in a presently unknown manner. It is also recommended that completion of the decomposition in a steam atmosphere should be studied before all hope of completing the decomposition by means of indirect heat only is abandoned, because it is possible that the presence of an atmosphere comprising primarily of water vapor would reduce the time/temperature required to complete the decomposition enough to make totally indirectly heated decomposition practical, especially if the feed contains virtually no difficultly hydrolyzable chlorides. The use of indirect heated decomposition practical, especially if the feed contains virtually no difficultly hydrolyzable chlorides. The use of indirect heat would, of course, eliminate the clean fuel requirement for this stage of decomposition, and completion of the decomposition at a lower temperature in the absence of combustion products will allow better control over the properties of the product alumina. The absence of inert combustion products in the decomposer off-gases would also decrease the cost of recovering HCl from this stage of decomposition.

Feed Quality

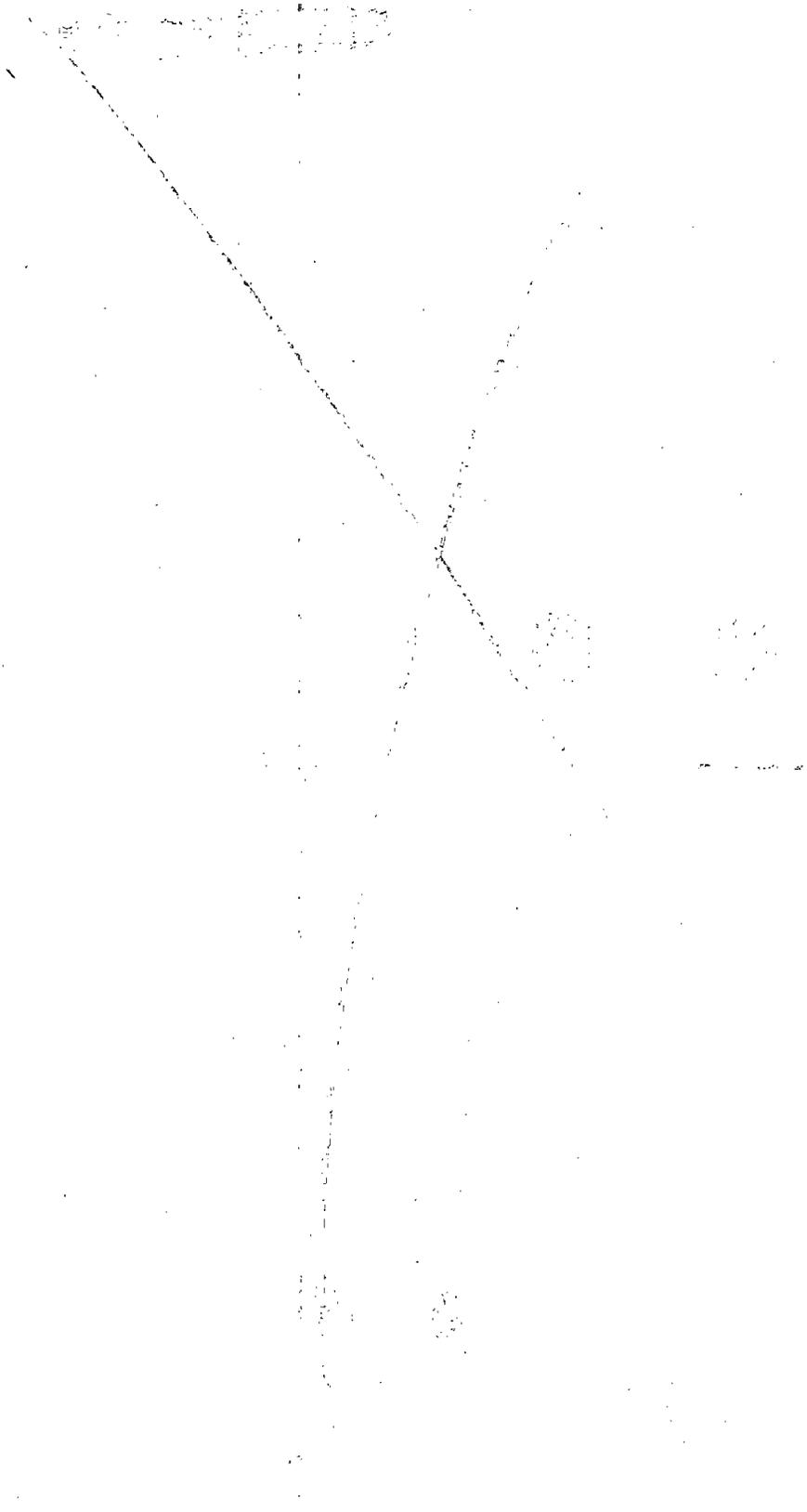
Impurities such as KCl, NaCl, and CaCl_2 are known to be difficult to pyrohydrolyze to their oxides, and either the chlorides or oxides may act as mineralizers catalyzing changes in the surface area and other properties of the Al_2O_3 . It is therefore important that feed used to study the second stage of decomposition, contain about the same amounts and kinds of impurities that will be present in the industrial crystallizer product to the second stage of decomposition. It is possible that purchased reagent grade ACH used in all or most of the decomposition studies known to have been conducted so far contains more of the difficultly hydrolyzable metal chlorides than are equivalent to the chloride specification in the product alumina, and more of these metal chlorides than will typically be in the ACH product supplied to decomposition by the process. A possibility therefore exists that the

time-temperature relationship for the second stage decomposition may yet be altered by one means or another to make possible completion of the second stage decomposition by means of indirect heat as discussed above.

Fundamental Information and Decomposer Design Development

More fundamental experimental information relating the rate of decomposition with the amount of chloride remaining in the solids, the temperature, the composition of the atmosphere surrounding the solids, the rates of phase transformation of this alumina as a function of temperature, and the effect of alumina impurities on the decomposition would be of invaluable assistance to those having responsibility for the construction of the pilot plant in understanding the decomposition process and in evaluating proposals relating to methods and equipment for accomplishing the decomposition. The development of this information is strongly recommended.

Long-term studies of candidate materials of construction for both stages of decomposition should be enlarged upon and extended simulating as best possible, the expected conditions of use.



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