

Annual Report (FY 1968) Webster Test Facility and Electrodialysis Test-Bed Plant Webster, South Dakota

United States Department of the Interior



Annual Report (FY 1968) Webster Test Facility and Electrodialysis Test-Bed Plant Webster, South Dakota

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**UNITED STATES DEPARTMENT OF THE INTERIOR • Walter J. Hickel, Secretary
Carl L. Klein, Assistant Secretary for Water Quality and Research**

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FOREWORD

This is one of a continuing series of reports designed to present accounts of progress in saline water conversion and the economics of its application. Such data are expected to contribute to the long-range development of economical processes applicable to low-cost demineralization of sea and other saline water.

Except for minor editing, the data herein are as contained in a report submitted by the contractor. The data and conclusions given in the report are essentially those of the contractor and are not necessarily endorsed by the Department of the Interior.

TABLE OF CONTENTS

	<u>Page No.</u>
I. SUMMARY	1-3
A. General	1
B. Test Bed Plant Water Production Summation	1
C. Test Bed Plant Evaluation	2
D. Pilot Plant Evaluation	2-3
II. INTRODUCTION	4-30
A. History	4
B. Water Supply	5
C. Description of Test Bed Plant	6-18
D. Technical Status Prior to Fiscal Year 1968	18-30
III. ACTIVITIES	31-50
A. General	31
B. Organization	31,32
C. Modifications	31-37
D. Operations	38-42
E. Maintenance and Repairs on Test Bed Plant	43-49
F. Safety	49-50
IV. TEST BED PLANT EVALUATION	51-97
A. General	51-53
B. Routine Data Gathering and Analysis	53-67
C. Anion Membrane Fouling	68-76
D. Acid Injection Evaluation	77-85
E. Transport Depletion Evaluation	86-97

TABLE OF CONTENTS (CONTINUED)

	<u>Page No.</u>
V. PILOT PLANT EVALUATION	98-102
A. General	98
B. Pilot Plant Evaluations	98-102
VI. WEBSTER TEST FACILITY COSTS	103-118
A. General	103
B. Investment Costs, Webster Test Facility	103
C. Capital Costs of Webster Test Bed Plant	104-106
D. Water Production Costs	107-118
VII. APPENDIX	119-128
A. Webster Engineering and Developmental Reports	119-123
B. Electrodialysis Membrane Properties	124
C. Glossary of Terms	125-127
D. Bibliography	128

LIST OF FIGURES

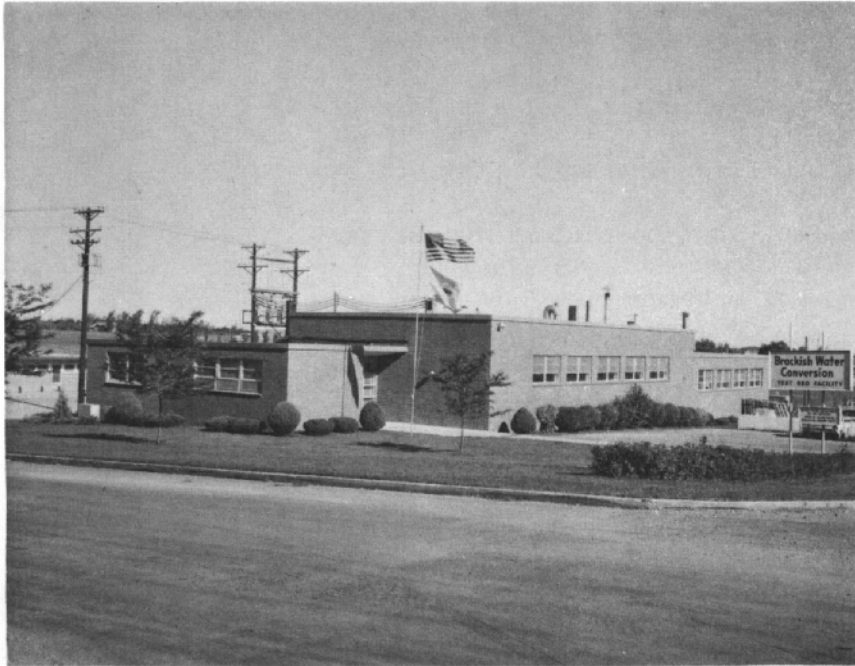
<u>Figure No.</u>		<u>Page No.</u>
1	Flow and Control Diagram - Acid Injection System	9
2	Flow Diagram - Manganese Zeolite Pretreatment	10
3	General Layout of ACI Electrodialysis Process	21
4	Electrodialysis Process Flow Diagram	22
5	Electrodialysis Process Flow Diagram	23
6	Schematic View of an ACI Test Bed Stack	24
7	ACI Feeding Frame	25
8	ACI Anode Holder and Fastening Frame	26
9	ACI Cathode Holder and "F" Frame	27
10	ACI Dilution Gasket and Separator	28
11	ACI Stack in Press	29
12	Rebuilding an ACI Stack	30
13	Personnel Organization Chart	32
14	North End of Webster Test Facility Property	34
15	Laboratory	36
16	Eroded Underdrain Strainer of Elgin Filter	44
17	Test Bed Plant Evaluation	52
18	First Stage ACI Stack Resistance	55
19	First Stage ACI Stack Resistance	56
20	Energy Consumption in First Stage	57
21	Energy Consumption in Second Stage	58
22	Energy Consumption in Third Stage	59

LIST OF FIGURES (CONTINUED)

<u>Figure No.</u>		<u>Page No.</u>
23	Energy Consumption in Fourth Stage	60
24	"Burned Out" Turnbuckle	66
25	"Burned Spots" on ACI DK-1 Membranes	67
26	First Stage ACI Stack Resistance	71
27	First Stage ACI Stack Resistance	72
28	Repeating Cell Pair Energy Consumption in Stage One	73
29	Repeating Cell Pair Energy Consumption in Stage One	74
30	Repeating Cell Pair Energy Consumption in Stage Four	75
31	Repeating Cell Pair Energy Consumption in Stage Four	76
32	Deterioration of UC Zephyr Cellulose Membranes	92
33	Deterioration of UC Zephyr Cellulose Membranes	93
34	Microscope Photo Showing UC Cellulose Membrane	94
35	Transport Depletion Stack Resistance Data	95
36	Transport Depletion Stack Current Efficiency and Energy Consumption	96
37	Pilot Plant Evaluations	101
38	Aerojet-General Reverse Osmosis Pilot Plant	102

LIST OF TABLES

<u>Table No.</u>		<u>Page No.</u>
1	Wells Available for Test Facility Use	5
2	Test Bed Plant Operation Statistics	40
3	Test Bed Plant Operation Statistics	41
4	Gross Water Production Summation	42
5	Tabulation of ACI Test Bed Plant Stack Building Data	63
6	Sample Water Analyses	82
7	Sample Water Analyses	83
8	Sample Water Analyses	84
9	Test Bed Plant Ion Removal Selectivity	85
10	Ion Removal Selectivity (Transport Depletion)	97
11	Total Webster Test Facility Investment Costs	103
12	325,000 GPD Webster Test Bed Plant Capital Costs	104-106
13	325,000 GPD Normalized Capital Costs	108-109
14	Operating Costs Using Cold Lime Softening	110-112
15	Ledger Summary	113-118



Front View of the Webster Test Facility taken from the southeast corner of the plant site. The process facility is the center portion; to the left is office, library, conference room, and visitor space; to the right is the pilot plant engineering and development facility.

SECTION I

SUMMARY

A. GENERAL

This Annual Report is prepared in compliance with Management, Operations and Maintenance Contract No. 14-01-0001-1733. This contract was awarded under the U. S. Department of the Interior, Office of Saline Water, Test Facility Program to Mason-Rust, a joint venture of Mason and Hanger - Silas Mason Co., Inc. and The Rust Engineering Company. This Annual Report presents the Test Bed Plant Maintenance and Operational history and the Experimental Program as carried out during the period 1 July 1967 through 30 June 1968.

B. TEST BED PLANT WATER PRODUCTION SUMMATION

The Test Bed Plant consists of the four stage Asahi Chemical Industry (ACI) electro dialysis process for desalination of brackish water. Well water of 1500 ppm TDS, 48°F is pretreated by potassium permanganate addition followed by manganese Zeolite filtration for iron removal. A summary of production data during this report period is as follows:

Total On-Stream Time, Days	275.0
Percent On-Stream Time	75.1
Total Downtime, Days	90.0
Total Feed Water, Gallons	118,770,500
Total Product Water, Gallons	72,189,000
Total Waste Water, Gallons	46,581,500
Average Gross Production Per Day On-Stream, Gallons	262,505.0

Of the total 90 days downtime, normal daily acid backwash of the electro dialysis stack accounted for 50.8 days; hook-up of a 400 GPM production well, 14.2 days; transport depletion plus acid-injection modification, 3.7 days; repairs to pretreatment system, 14.2 days; stack removal for disassembly, 2.5 days; and miscellaneous, 4.6 days.

C. TEST BED PLANT EVALUATION

1. Test Bed General Operation

Examination of electro dialysis performance at Webster demonstrated that the anion membrane fouling, described in the Fiscal Year 1967 Annual Report, continued to be a major problem. The fouling is thought to be caused by a poisoning of the anion exchange membrane due to "organics" with some contribution from iron, although the exact fouling agents have not been identified. Nightly acid backwashing was found to be successful towards control of iron deposits and the alkaline scale problems associated with anion membrane poisoning; however, the acid backwashing accounted for 56 percent of the plant downtime.

2. Anion Membrane Fouling of Two ACI Membranes

Test Bed Plant Operation showed that an ACI DA-2 anion permeable membrane fouls six to ten times more rapidly than a DA-1 anion permeable membrane. As the DA-1 anion permeable membrane is no longer commercially available, pilot plant tests should be started to find a replacement for the DA-2 anion permeable membrane.

3. Acid Injection Evaluation

Mason-Rust was unable to demonstrate any tangible benefit for the electro dialysis process through injection of sulfuric acid into the pretreated water for alkalinity reduction.

4. Transport Depletion Evaluation

In two separate tests, regenerated cellulose "neutral" membranes (Union Carbide Zephyr) disintegrated after five weeks when used to replace the conventional anion exchange membrane in a Test Bed stack.

D. PILOT PLANT EVALUATION

1. Reverse Osmosis

An Aerojet Model 1-5608-2 Plate and Frame Reverse Osmosis Unit was tested at 750 psi feed pressure on Webster 48°F water treated for iron and manganese removal. Initial product water flux was

23.4 gfd; after 5077 operating hours, the product flux declined to 12 gfd. The declining salt flux and disassembling of the unit illustrated the need for better water pretreatment for iron removal, and for a more rigid membrane support material to eliminate dead areas for calcium sulfate precipitation.

2. Lime Softening and Carbon Filtration Prior to Electrodialysis

Pilot plant tests performed July 1967 through December 1967 on lime softened and carbon filtered water demonstrated the suitability this process has as a pretreatment step prior to electrodialysis. Based on these tests, the increased plant amortization and chemical demand costs associated with a lime softening and carbon filtration pretreatment system is expected to be more than offset by savings due to minimization of fouling problems, plus elimination of one electrodialysis stage, plus an increased product recovery from 50 percent to 70 percent.

3. High Temperature Electrodialysis

Various commercially available membranes were tested in the ACI SV-3 Acilyzer and the Ionics Stackpack at temperatures between 48°F and 135°F using lime softened and carbon filtered Webster water. For the ACI equipment, the Asahi Glass CMV cation and AMT anion permeable membranes worked well but distorted into the flow channels when used in the Ionics Stackpack.

A paper study based on the pilot plant data dictates that the major savings in high temperature electrodialysis operation using ACI electrodialysis equipment, to 125°F, result from amortization and membrane replacement due to fewer stages required. However, if the water is to be preheated, heat exchanger and fuel costs can more than offset this; for 250,000 GPD of Webster type-water heated from 48°F to 125°F assuming Arizona Public Gas fuel costs and a 10°F heat exchanger temperature approach between the incoming feed and outgoing product water, there is little cost advantage either way.

SECTION II. INTRODUCTION

A. HISTORY

Public Law 85-8883, approved 2 September 1958, provided for the construction, operation and maintenance of not less than five demonstration plants for the production, from sea water or brackish water, of water suitable for agricultural, industrial, municipal and other beneficial consumptive uses. The electrodialysis method was selected as the process for Demonstration Plant No. 3. The Demonstration Plant was later renamed the "Webster Test Facility" at Webster, South Dakota; the process itself was renamed the "Test Bed Plant".

The Webster, South Dakota, Test Bed Plant was designed to convert brackish water to potable water at a rated capacity of 250,000 gallons per day. The Construction Contractor was Asahi Chemical Industry Company, Tokyo, Japan. The Test Bed Plant was placed on line in March 1962, following the completion of a 2,000 hour acceptance test period conducted by the Construction Contractor and accepted by the United States Department of the Interior, Office of Saline Water. The plant was modified in early 1966 to produce 325,000 gallons per day.

In March 1966, a 1,000 square foot addition to the north end of the Webster Test Facility was completed and is now used for various pretreatment pilot plants and miscellaneous experimental support equipment. Another 368 square foot addition to the south-west corner of the Test Facility was completed in October 1967 to provide office space.

In December 1967, a 400 gallon per minute well was put into service to provide additional water for test purposes.

In May 1968, a 1,200 square foot addition to the northwest corner of the Test Facility was completed. This addition houses shop equipment, parts storage, and an 80 H.P. boiler for pilot plant high temperature electrodialysis experiments.

In May 1968, a sub-contract was awarded to build a multipurpose masonry structure, 62 feet by 104 feet with a partial second floor. The structure is to house a 400 GPM Infilco lime softening and carbon filtration plant for the Test Bed Plant, pilot plant space, water analysis laboratory, and offices. The pretreatment system for the electrodialysis Test Bed Plant will be changed from Zeolite filtration to lime softening followed by carbon filtration.

B. WATER SUPPLY

Brackish water can be supplied through any one of five wells, the compositions and depths of these wells are indicated by Table 1. Well Number 6 is government-owned whereas the others are city-owned. With the possible exception of Well No. 3, all wells are drilled to the same aquifer, and their capacities are dependent upon the total amount of water pumped from this aquifer. Well No. 6, which was placed into service in December 1967, is capable of pumping 400 GPM; the capacities of the other wells are less than 200 GPM. Well Number 2 is not available for Test Facility Use.

TABLE 1

WELLS AVAILABLE FOR TEST FACILITY USE

(The Individual Wells Also Fluctuate in TDS and Ion Constituents.)

Wells Analyzed 5 June 1968.

	<u>Well 1</u>	<u>Well 3</u>	<u>Well 4</u>	<u>Well 5</u>	<u>Well 6</u>
Depth, feet	237	80	222	224	185
TDS (ppm)	1096	1336	1184	1468	1546
TDS (epm)	17.25	21.25	16.95	21.8	22.0
bicarbonate, ppm	360	436	320	335	329
pH	7.5	7.35	7.6	7.35	7.4
hardness as ppm CaCO ₃	628	614	648	1852	836
sp. resist., ohm-cm, 20°C	764	628	755	661	658
calcium, ppm	NA	118	NA	190	195
magnesium, ppm	NA	78	NA	92	85
sodium, ppm	81	78	77	93	100
potassium, ppm	6.2	8	6	9.8	8
iron, ppm	1.1	2.3	0.5	1.4	1.3
manganese, ppm	0.7	0.9	1.0	1.1	1.4
sulfate, ppm	540	750	584	796	767
chloride, ppm	11	7.5	7.5	16	18
fluoride, ppm	0.5	0.3	NA	0.5	0.4
iodide, ppm	0.71	0.07	0.67	0.72	0.41
silica, ppm	31	31	32	34	34
C.O.D., mg/liter	6	6	18	24	24

NA = not analyzed.

C. DESCRIPTION OF TEST BED PLANT

1. General

The purpose of a Test Bed Plant is to provide a full scale model for engineers to test and evaluate a method of water desalination, in this case the electro dialysis process. Information gained from a Test Bed Plant will enable water purification or desalination plants to be built and operated by communities at minimum cost. Besides defining costs associated with saline water conversion, Test Bed Plant studies reveal those areas where significant improvement can be made towards producing low-cost potable water.

The 325,000 GPD Test Bed Plant at Webster consists of (1) a water pretreatment step, (2) the electro dialysis step, and (3) post treatment. The pretreatment step is one of potassium permanganate ($KMnO_4$) oxidation of the iron and manganese in the well water to an insoluble form followed by Zeolite filtration to remove the suspended material. The pretreatment system also provides for acid injection to reduce the alkalinity. The electro dialysis step reduces the total dissolved solids content. During post treatment, the electro dialysis product water is passed through a decarbonator to increase the pH followed by chlorination. Most of the plant output is used to provide water for the City of Webster.

Design input and output for the Test Bed Plant are as follows:

Input:

506,000 GPD (335 GPM plus backwash water for the manganese Zeolite filters) of raw water containing 1500 ppm TDS, 1 ppm iron, 1 ppm manganese.

Product:

325,000 GPD (225 GPM) of product water containing 500 ppm TDS, no iron, 0.1 ppm manganese.

Waste:

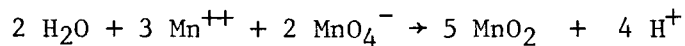
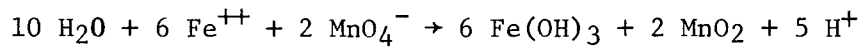
158,000 GPD (110 GPM) of waste water from the electro dialysis plant containing 3050 ppm TDS, 0.1 ppm iron, 0.2 ppm manganese.

22,800 GPD of backwash water from the manganese Zeolite filters.

2. Pretreatment by KMnO_4 Oxidation, Zeolite Filtration, and Acid Injection

During the period May 1967 through December 1967, the raw well water pretreatment system was as depicted by Figure 1. After December 1967, sulfuric acid was no longer injected into the electro dialysis feed water although the water continued to pass through the recarbonator. During May 1968, the pretreatment system was simplified as shown by Figure 2.

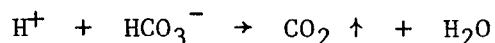
Referring to Figure 1, the incoming well water entered a 5,700 gallon holding tank (Tank 01). The O2 pump then pumped the water to a second 2,900 gallon capacity holding tank (Tank 06); a solution of potassium permanganate was pumped into the water line just after the O2 pump. In the second holding tank, the well water iron and manganese were oxidized to an insoluble form:



A second pump (Pump 02B) pumped the water to three manganese Zeolite filters where the insoluble iron and manganese flocs were removed.

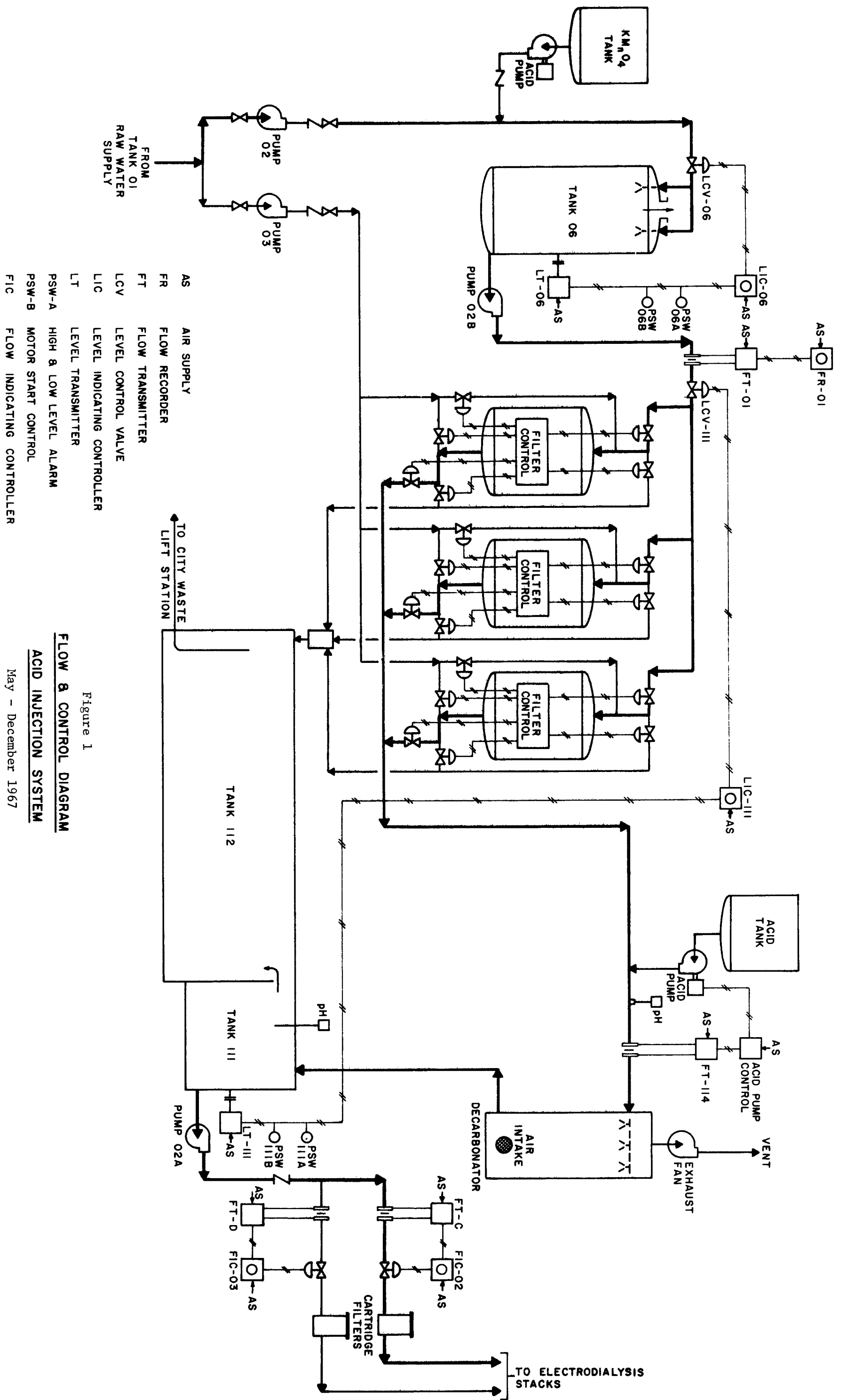
The three manganese Zeolite filters were supplied by the Elgin Filter Corporation. The filtering area of each filter is 38.5 square feet and each contains 96 cubic feet of manganese Zeolite. Each filter is automated to backwash at eight hour intervals. The backwash cycle consists of (1) backwashing the filter with untreated water at 300 GPM for 20 minutes, (2) a 20 minute rinse delay time, and (3) a 10 minute rinse period where the water passes through the filter at 160 GPM and is discarded as waste. While one filter is in backwash, the other two continue to operate.

As seen by Figure 1, sulfuric acid may be injected into the water to remove bicarbonate ion:



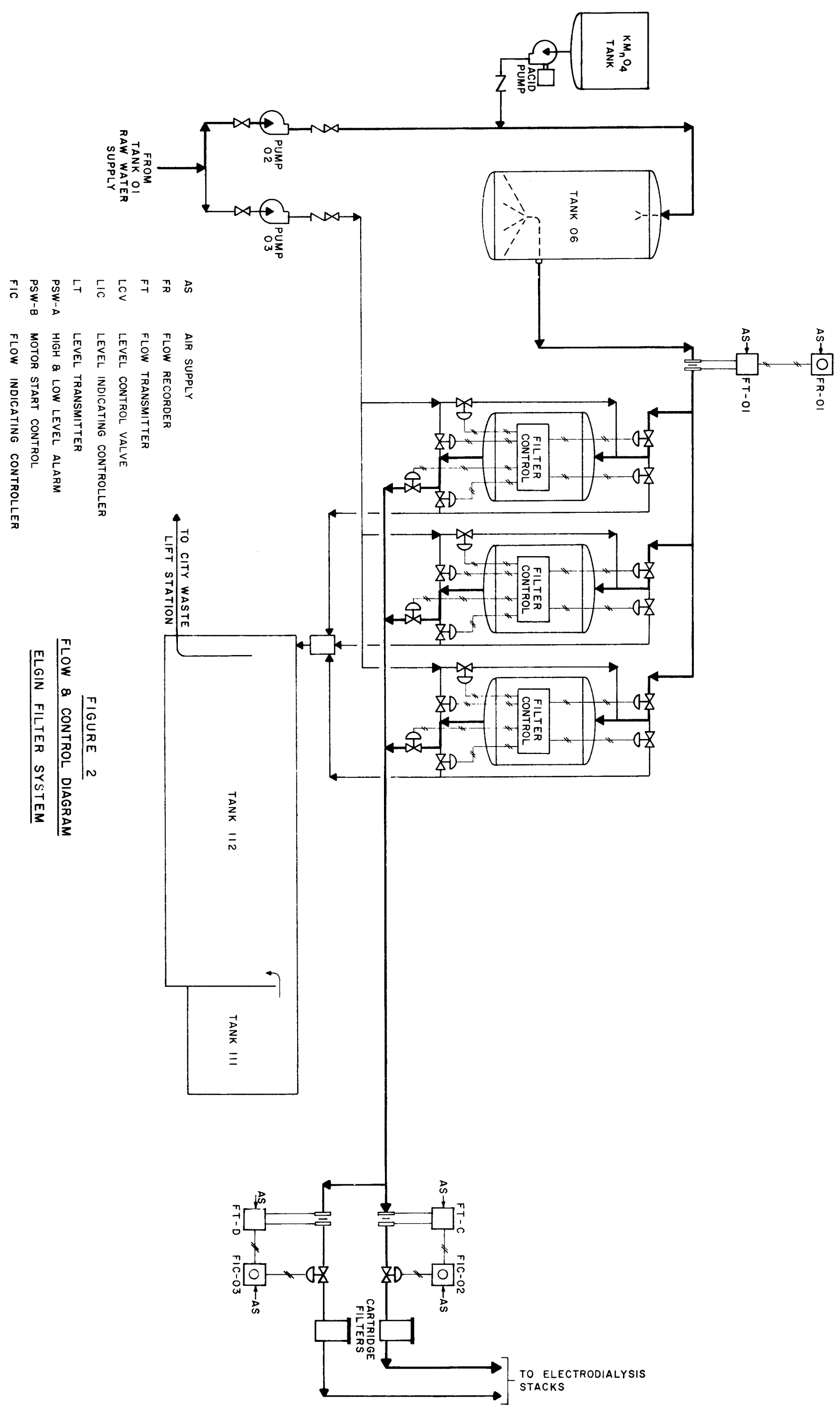
The carbon dioxide is removed in the recarbonator. The water then drops into a 500 gallon holding tank (Tank 111) from which it is pumped to the electro dialysis part of the Test Bed Plant. Waste water from backwash of the manganese Zeolite filters flows into another holding tank (Tank 112) where it is pumped with the electro dialysis waste to the City waste lift station. The pre-treated water is pumped through two banks of twenty eight 25-micron size cartridge filters just ahead of the electro dialysis Test Bed Plant.

Test Bed Plant feed water flow rates are controlled at F1C-02 (sets product water flow) and F1C-03 (sets concentrate makeup or waste flow). As the water level in Tank 111 is increased or decreased, a signal is transmitted to close or open the valve at LCV-111. A Taylor D/P Pressure Transmitter (item FT-114 on Figure 1) transmits a signal to the acid pump to inject sulfuric acid at a rate proportional to the square root of the pressure drop across an orifice prior to the de-carbonator.



- AS AIR SUPPLY
- FR FLOW RECORDER
- FT FLOW TRANSMITTER
- LCV LEVEL CONTROL VALVE
- LIC LEVEL INDICATING CONTROLLER
- LT LEVEL TRANSMITTER
- PSW-A HIGH & LOW LEVEL ALARM
- PSW-B MOTOR START CONTROL
- FIC FLOW INDICATING CONTROLLER

Figure 1
FLOW & CONTROL DIAGRAM
ACID INJECTION SYSTEM
 May - December 1967



- AS AIR SUPPLY
- FR FLOW RECORDER
- FT FLOW TRANSMITTER
- LCV LEVEL CONTROL VALVE
- LIC LEVEL INDICATING CONTROLLER
- LT LEVEL TRANSMITTER
- PSW-A HIGH & LOW LEVEL ALARM
- PSW-B MOTOR START CONTROL
- FIC FLOW INDICATING CONTROLLER

FIGURE 2
 FLOW & CONTROL DIAGRAM
 ELGIN FILTER SYSTEM

3. Electrodialysis Plant

a. Electrodialysis Theory

Electrodialysis is a unit operation in which the partial separation of the components of an ionic solution is induced by an electric current. This separation is accomplished by placing sheets of ion exchange or perm-selective membranes across the path of current flow. Two types of permselective membranes are used: one permeable to anions (e.g. bicarbonate, sulfate, chloride, etc.) and are called "anion permeable membranes" or "anion membranes"; the others are permeable to cations (e.g. sodium, calcium, etc.) and are called "cation permeable membranes" or "cation membranes".

The following concepts should be understood for the purpose of this report:

- (1) Total dissolved solids expressed in equivalent parts per million (TDS, epm).

Total dissolved solids are often expressed in equivalent parts per million (epm) rather than parts per million (ppm) because equivalents of salt transferred can be directly related to the current applied to an electro-dialysis stack. They are related as follows:

$$C = \sum_{j=1}^{m} \frac{W_j}{v_j}$$

where C = TDS in epm

W_j = ppm of cationic species j

v_j = equivalent weight of cationic species j

This expression is summed over all cationic species, $j = 1$ to m (e.g. sodium, potassium, calcium, magnesium, hydrogen ions, etc.). A similar operation can be performed for the anion species; the two results should be equal.

At Webster, quantity C is determined by passing the brackish water sample through a strong cation exchanger, where each cation is exchanged for an equivalent amount of hydrogen ions. The eluted acids are titrated with 0.05 N NaOH. After a correction for any water alkalinity, the TDS in epm is calculated.

- (2) The current efficiency is that fraction of the electrical current applied to an electro dialysis unit that results in a new transfer or separation of salt. It is calculated as follows:

$$\eta = \frac{F G (C_i - C_p)}{nI}$$

where F = Faradays constant

G = dilution flow to electro dialysis unit

I = current

n = number of dilution compartments
(Figure 6)

C_i = dilution TDS entering electro dialysis unit

C_p = dilution TDS leaving electro dialysis unit

If G is in gallons per minute (GPM), C_i and C_p in epm and I is in amperes, then:

$$\eta = \frac{6.087 G (C_i - C_p)}{nI}$$

- (3) Repeating Cell Pair Resistance

For the Test Bed Plant at Webster, the repeating cell pair stack resistance is calculated as follows:

$$r = \frac{VA}{nI}$$

where r = repeating cell pair stack resistance,
ohm-cm²/RCP

V = voltage drop across the membrane stack

A = useful membrane area, cm²

n = number of membrane pairs

I = current, amperes

The voltage drop across the membrane stack is measured from platinum probes inserted just inside the Dacron cloths of Figure 6.

(4) Current Density

The current density is calculated as follows:

$$i = \frac{I (10)^3}{A}$$

where i = current density, ma/cm²

I = current, amperes

A = useful membrane area, cm²

For practical purposes, the current density can be considered uniform across the stack area for the Test Bed Plant.

(5) Limiting Current Density

Generally, the more current applied to an electrodiolysis stack, the more salt will be removed. Even if the dilution water between the membranes is well stirred, the salt must finally reach the membrane by diffusing across a stagnant water layer immediately adjacent to the membrane. If the stack is operated above certain current density, called the limiting current density, the process of diffusion cannot supply sufficient ions across the stagnant water layer on the dilution side of the anion permeable membrane. Then, in order to maintain that current density, water splitting will occur

on the dilution side surface of the anion permeable membrane. If the dilution water has no natural buffering capacity (e.g. bicarbonate ions, etc.), the net result will be a pH drop in the dilution stream and a pH increase in the brine stream. The stagnant layer becomes the major contributor to the repeating cell pair stack resistance at the limiting current density because at this point the ionic concentration immediately adjacent to the anion membrane approaches zero. At roughly, 1.8 volts across the stagnant layer, water splitting occurs.

The limiting current density may be determined by recording the stack current at a number of different voltage levels, and noting that current at which the stack resistance begins to significantly increase with increasing current. The specific techniques for determination of limiting current density are described in a paper by Cowan (1).

(6) Transport Depletion

Transport depletion is a variation of electrodialysis where nonselective membranes are used in place of the anion permeable membrane. In normal electrodialysis, the current is carried in part by the anions and part by cations. In transport depletion, the positive ions carry virtually 100 percent of the current and these move through the cation membrane to leave a salt depleted dilution stream. Both anions and cations pass through the nonselective membrane towards the electrode of opposite charge.

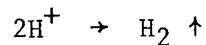
b. Process at Webster

The electrodialysis plant, illustrated by Figure 3, was manufactured by the Asahi Chemical Industry Co., Ltd., Japan, in 1961. The process flow used during 1967 - 1967 is depicted by Figures 4 and 5.

The water leaving the pretreatment plant splits into two streams: a dilution stream which eventually becomes the product water and a makeup stream to the service tank for the concentrate stream. The dilution stream is fed through the electrodialysis stages in series; the concentrate stream is fed in parallel to all stages. A portion of the concentrate stream is recycled back into the recirculating concentrate tank while the remainder is discharged as waste.

c. Sulfuric Acid Addition and Interconnected Service Tanks

Sulfuric acid (93%) is added to the electrode rinse stream service tank to acidify the electrode rinse to pH = 1.5 to 2. This is an addition to the acid added as shown by Figure 1. Acidification of the cathode stream is necessary because of the following reaction at the cathode:



Acidification of the anode stream and feeding frame streams was also done to decrease the electrical resistance in the end parts of an electrodialysis stack and to reduce the chance of alkaline scale formation in the feeding frame compartments.

Some of the electrode rinse water (Figures 4 and 5), after passing through the electrodialysis stack is sent to the recirculating concentrate tank. The recirculating concentrate tank water overflows a weir to provide makeup water for the electrode rinse tank. The recirculating concentrate water pH is maintained between 3 and 5. The concentrate stream water from the first stage drops into a small weir box on the side of the waste tank before overflowing into the waste tank. Several holes on the side of the weir box allow water to flow into the electrode rinse tank (or from the electrode rinse tank to the waste tank) depending on the level of water in the electrode rinse tank.

The interconnected recirculating concentrate, electrode rinse, and waste tanks allow for unattended operation on the Test Bed as any unbalance in flow delivered to a tank will be compensated by overflow into another tank. A level control opens or closes a valve on the exit side of the waste tank to maintain a certain level of water in that tank. The major disadvantage with such a system is that it is very difficult to perform salt and flow material balances on the concentrate and electrode rinse streams.

d. Electrodialysis Stack (Cocurrent Feeding of Dilution and Concentrate Streams)

The components of an electrodialysis stack are illustrated by Figure 6; additional details are shown in Figures 7, 8, 9, and 10.

The incoming dilution water enters a manifold (not illustrated) which feeds the water to eight dilution holes at the bottom of the feeding frame (Figures 6 and 7) at the anode side of the stack. The incoming concentrate stream enters a similar manifold which feeds water to nine concentrate holes at the bottom of the feeding frame at the cathode side of the stack. The water is then channeled by way of eight dilution holes and nine concentrate holes across the bottom of the stack.

The interior of the stack contains anion and cation permeable membranes. Every other compartment has a dilution gasket slotted with eight holes at the bottom of the stack which allows dilution water to enter the 0.075 cm space between the membranes. The adjacent compartments each have a concentrate gasket slotted with nine holes at the bottom to allow concentrate water to enter. The dilution water leaves the compartment by way of nine slots at the top of the dilution gasket, and leaves the stack by way of a manifold at the top of the cathode side feeding frame. The concentrate water leaves the compartment by way of eight slots at the top of the concentrate gaskets, and leaves the stack through a manifold at the top of the anode side feeding frame.

Separate streams also bathe the anode, cathode, anode feeding frames, and cathode feeding frames. An electrode membrane separates the electrode rinse stream solutions from the feeding frame streams; it is held in place by a pair of "E" frames. A perforated PVC sheet, called an "F" frame provides additional support for the anion and cation permeable membranes.

The anion and cation permeable membranes are clamped together by a pair of fastening frames and eight turnbuckles (Figure 11) to form a part of the stack which can be moved to a different stage position or can be set on a table for rebuilding as in Figure 12. The stages are clamped together in a stack press, with each individual stack or stage separated by a wooden insulating block.

A dilution compartment consists of a slotted dilution gasket of 0.075 cm thickness and two PVC woven monofilament separators. The diameter of a monofilament is 0.018 cm. In the direction parallel to the flow there are six monofilaments per centimeter; in the direction perpendicular to the flow there are two strands of three monofilaments per centimeter.

The thickness of two separators is 0.075 cm. The slots of the dilution gasket contain numerous 1/8 inch diameter rubber disks supported on a thin rubber flap backed with a Mylar film; this flap is glued in the slot. The Japanese designer is said to have been inspired while playing a game of ping-pong, and therefore called this flap a "ping-pong slot" because of the resemblance to a ping-pong paddle (Figure 10).

A concentrate compartment consists of a slotted concentrate gasket of 0.075 cm thickness and two PVC woven monofilament separators. Unlike the dilution compartment, the concentrate gasket slot is open and part of the separators protrude into the slot to maintain the membranes 0.075 cm apart.

For test purposes, the second concentrate and dilution compartments as measured from the cathode end each contain a pressure tap located seven inches from the bottom of the stack. At present, the concentrate streams are backpressured using FVC-03C (Figures 4 and 5) such that the pressure difference between the dilution and concentrate streams at the measuring taps are zero.

Since 1965, the stack was built to 250 repeating cell pairs of anion and cation permeable membranes, with a dilution flow of 175 GPM and a concentrate flow of 100 GPM. At the suggestion of the Asahi Chemical Industry, the first five dilution and three concentrate compartments were of triple thickness (3 dilution gaskets and 6 separators per compartment); the next three dilution and two concentrate compartments from each end were of double thickness (2 dilution gaskets and 4 separators per compartment). The remaining compartments were of regular thickness. Asahi believed that the double and triple thickness end compartments would prevent massive calcium carbonate scale from building up at the ends of the stack and blocking end compartment flow. This postulate has not been proven. In addition, a thin Mylar film cut to the shape of a gasket was placed in every sixth concentrate compartment; the Mylar film was believed to protect the gaskets against electrical attack.

e. Electrodialysis Stack (Countercurrent Feeding of Dilution and Concentrate Streams)

In countercurrent flow, the dilution stream enters the bottom of the stack at the anode side and leaves at the top of the stack at the cathode side as shown in Figure 6. The concentrate stream enters the stack at the top (anode side)

and leaves at the bottom (cathode side). Both concentrate and dilution streams enter through eight holes or slots and leave through nine holes or slots.

During Fiscal Year 1968, stages one and four had a cocurrent feeding arrangement whereas the feed was fed countercurrently to stages two and three. All stages were converted to cocurrent feed in May 1968 so that the pressure between the dilution and concentrate streams could be better balanced.

f. Membranes

Except for certain experimental tests, the cation permeable membrane was the ACI DK-1 membrane. The anion permeable membrane was the ACI DA-2 or ACI DA-1 membrane. The electrode membrane was an ACI Dacron cloth. Properties of these membranes are presented in the Appendix.

D. TECHNICAL STATUS PRIOR TO FISCAL YEAR 1968

Operation of the electro dialysis Test Bed Plant during 1962 - 1965 revealed intolerable membrane losses due to calcium carbonate scale, and electro dialysis stack resistance to be as much as ten times the level predicted by theory and the original Asahi Chemical Industries pilot plant tests on synthetic water. The net result was a more costly and a poorer quality water than the ACI design calculations predicted. During late 1965 a building addition was built to house pilot plant equipment for studies to define pretreatment requirements for electro dialysis systems. The study showed that anion membrane fouling by "organics" was the primary cause of the Test Bed Plant difficulty, with iron and manganese carry-over from the existing filters and calcium sulfate precipitation being secondary and different problems. The pretreatment study and a separate study by Mason-Rust showed lime softening plus carbon filtration to be the most economical pretreatment for the ACI electro dialysis system at Webster.

Methods for measuring the effects of anion membrane fouling are described in the Fiscal Year 1967 Annual Report for the Test Facility.

The degree of fouling is also compared for pilot plant electro dialysis units operating on several different waters. The net result of fouling is an increase in stack resistance and a decrease in limiting current density as a function of time. The degree of fouling could be compared by determining the parameter γ , defined by:

$$T \equiv \frac{\left(\frac{i_{lim}}{C}\right)_t}{\left(\frac{i_{lim}}{C}\right)_{t=0}} = \frac{1}{1 + \gamma t}$$

where i_{lim} = limiting current density

C = log mean concentration difference between the dilution in and exit streams

t = total accumulative time in months logged for an anion permeable membrane

T = fractional loss in limiting current density between time t and time 0

Experiments showed that operation of an electro dialysis stack at a current density above the limiting current density resulted in calcium carbonate scale formation on the concentrate side surface of the anion permeable membranes. Therefore, the current density had to be decreased as the anion membranes fouled with resultant poorer quality water. However, a compromise was reached between lowering the current density and producing a product water with a "reasonable" amount of total dissolved solids removed. Any calcium carbonate scale that formed could be eliminated by nightly acid backwashing the dilution and concentrate compartments of the stack with water of pH = 1.8; acid backwashing the dilution compartments also removed iron and manganese floc accumulations which carried over from the pretreatment plant.

The pretreatment system was modified during Fiscal Year 1967 so that sulfuric acid may be injected into the water after passing through the manganese Zeolite filters, as depicted by Figure 1. The objective of the acid injection experiments was to determine whether the costly membrane losses due to calcium carbonate scale formation could be eliminated by reduction in water alkalinity (bicarbonate).

Plans were initiated during this report period to change the pretreatment system from manganese Zeolite filtration to one of lime softening and carbon filtration. The 1965 paper study, performed by Mason-Rust under OSW Contract No. 14-01-0001-1094, compared different pretreatment methods prior to electro dialysis; for Webster-type water, lime softening and carbon filtration seemed to be the system that would result in a least costly product water. The

study also examined segregated bed filtration, rapid sand filtration, Zeolite filtration, and diatomaceous earth filtration with and without carbon filtration. Backup pilot plant tests were started during 1965 and continued into this report period.

The lime softening and carbon filtration system was expected to result in a lower cost product water because of the following reasons: (1) by increasing plant on-stream time, thereby increasing plant production, (2) by achieving prior total dissolved solids (TDS) reduction, thereby eliminating one stage, (3) by reducing acid consumption, (4) by improving membrane lifetime, and (5) by prior hardness reduction which would allow the electro dialysis plant to operate at a higher product recovery ratio. These factors offset the disadvantage of amortization of the more expensive pretreatment system.

Based on studies performed at the Southern Research Institute and at the Denver Bureau of Reclamation, transport depletion tests were initiated at Webster using the conventional electro dialysis equipment with the anion permeable membrane replaced by a regenerated cellulose membrane. Transport depletion pilot plant tests were performed on Webster manganese Zeolite pretreated water during May - June 1967. Simulating Test Bed Plant conditions, current efficiencies of 35 percent were realized and current densities of at least twice that of the Test Bed Plant could be achieved; also, acidification of the concentrate stream was not necessary. No evidence of "fouling" of the regenerated cellulose membrane was seen during the four week tests. Plans were initiated to convert two stages of the Test Bed Plant to transport depletion in order to establish membrane lifetimes and membrane compatibility with the ACI equipment.

High temperature electro dialysis experiments (48°F to 140°F) at the pilot plant level were initiated during 1967 and continued into this report period. Electro dialysis of warm waters is less costly than cold waters because much greater current densities can be achieved on an electro dialysis stack without exceeding the limiting current density. If an economic improvement could be demonstrated, then plans were to convert two stages of the Test Bed Plant to high temperature electro dialysis for further evaluation during 1968.

The "acid injection", "transport depletion", and other tests to be carried out in the Test Bed Plant during this report period require that the plant be off line for modifications and for various experiments. Therefore, no effort was made to operate the plant as a demonstration plant where the plant would remain on line the maximum number of hours possible consistent with sound maintenance procedures in order to process water at the lowest possible cost.

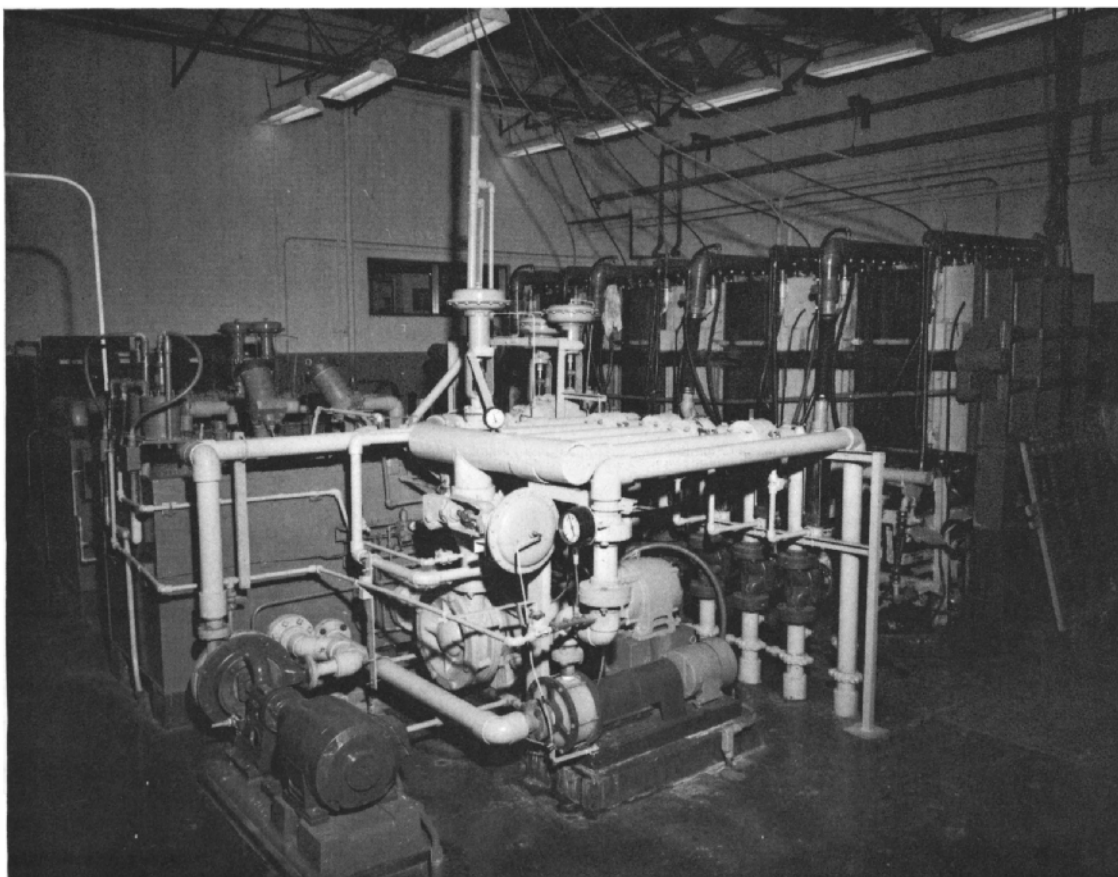


Figure 3

The general layout of the Asahi Chemical Industry Electrodesalination Process at Webster is illustrated. In the foreground are the waste pump and electrode rinse pump. At the right are the four production stages.

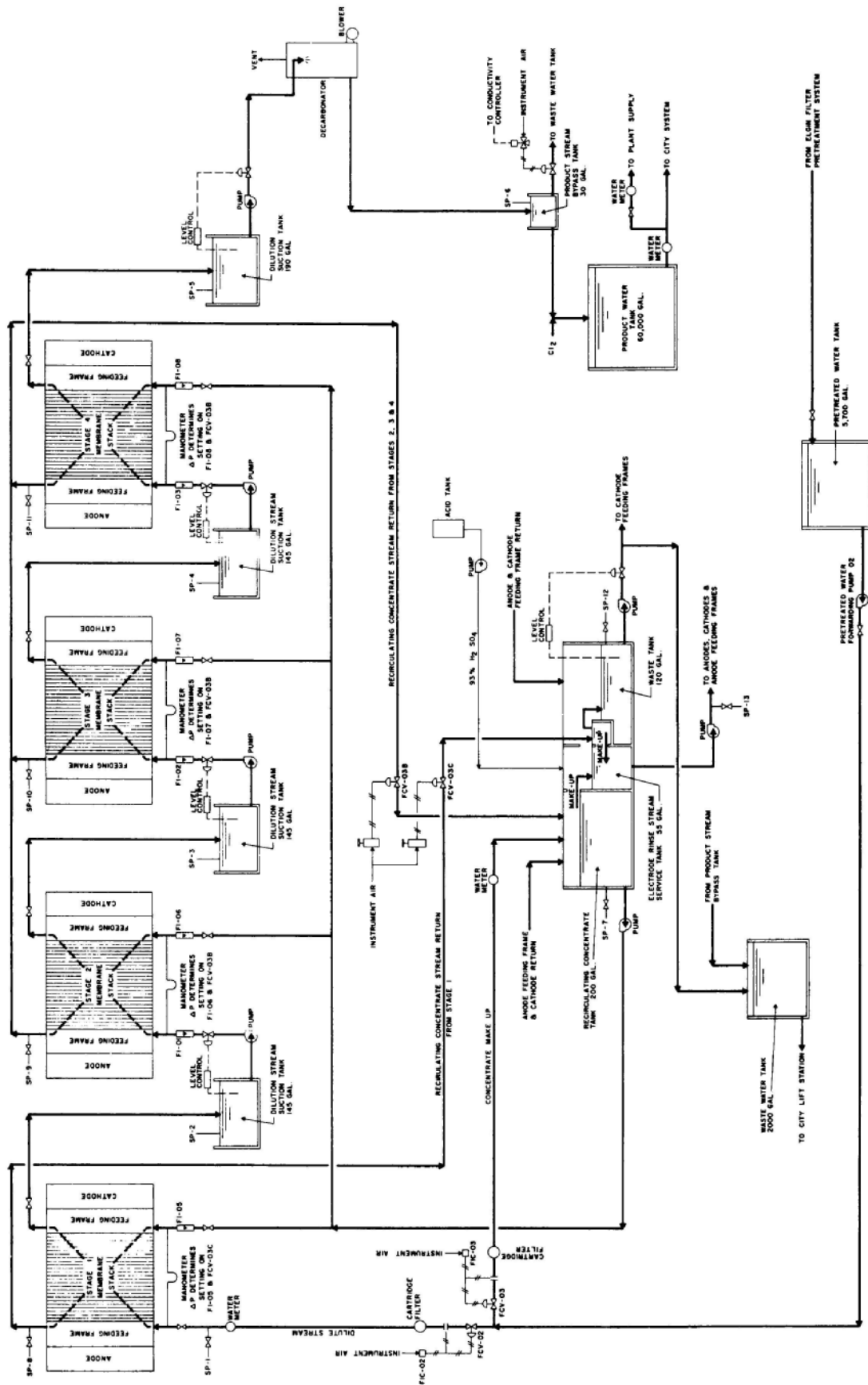


Figure 4
ELECTROLYSIS PROCESS
PRODUCT WATER FLOW
ELDM FILTER PRETREATMENT

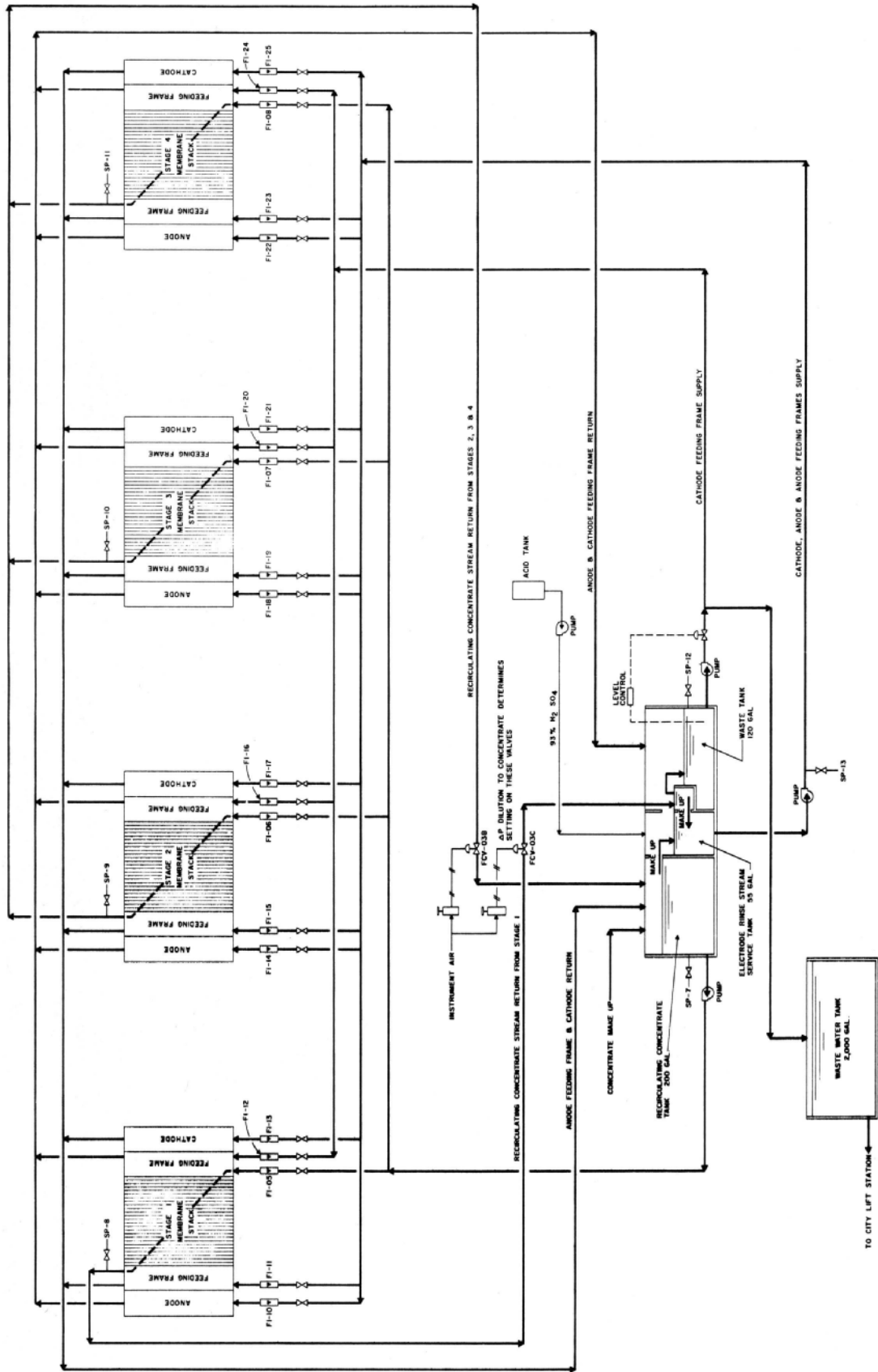


Figure 5
 ELECTROLYSIS PROCESS
 CONCENTRATE, ELECTRODE RINSE
 & FEEDING FRAME FLOW
 ELGIN FILTER PRETREATMENT

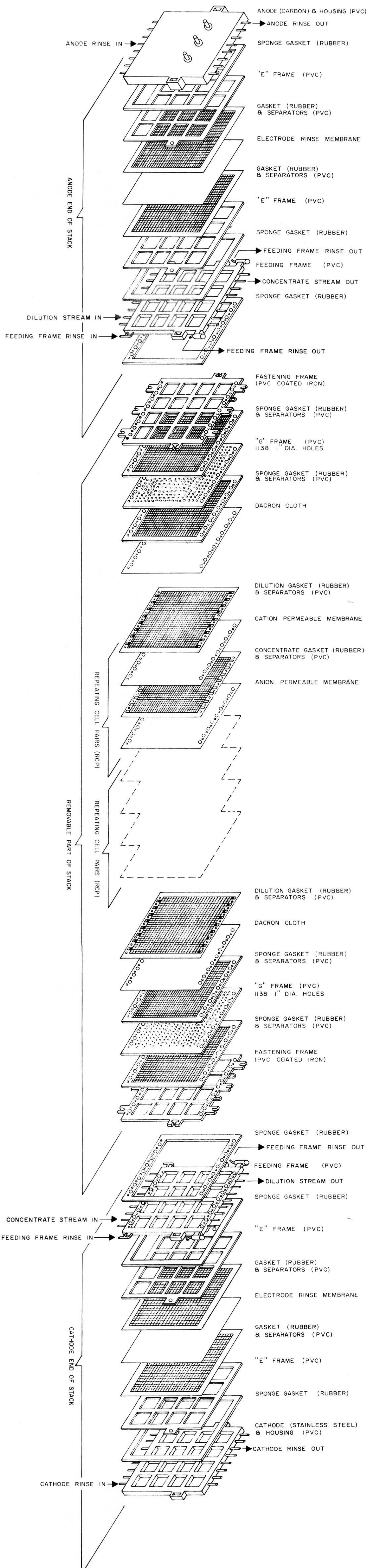


Figure 6
SCHEMATIC VIEW
OF AN
ACI TEST BED STACK

OVERALL DIMENSIONS

- ANODE: 3 CARBON BLOCKS EACH 37" x 13 1/2" x 3"
- ANODE HOUSING: 45" x 45" x 5"
- SPONGE GASKET: 45" x 45" x 1/8"
- OTHER GASKETS: 45" x 45" x 0.19"
- FEEDING FRAME: 45" x 45" x 1 3/4"
- FASTENING FRAME: 45" x 45" x 1 1/8"
- "E" FRAME: 45" x 45" x 1/8"
- "G" FRAME: 45" x 45" x 1/8"
- MEMBRANE: 44" x 44"
- USEFUL MEMBRANE AREA: 10.36 SQ. FT. (9622cm²)
- CATHODE: 37" x 40 3/4" x 1/4" 316 S. S.
- CATHODE HOUSING: 45" x 45" x 2 1/4"

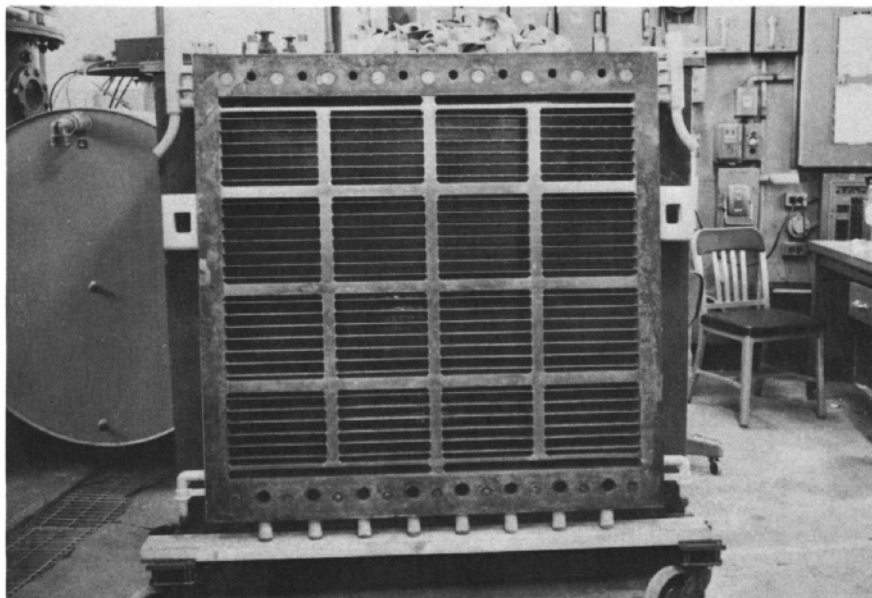
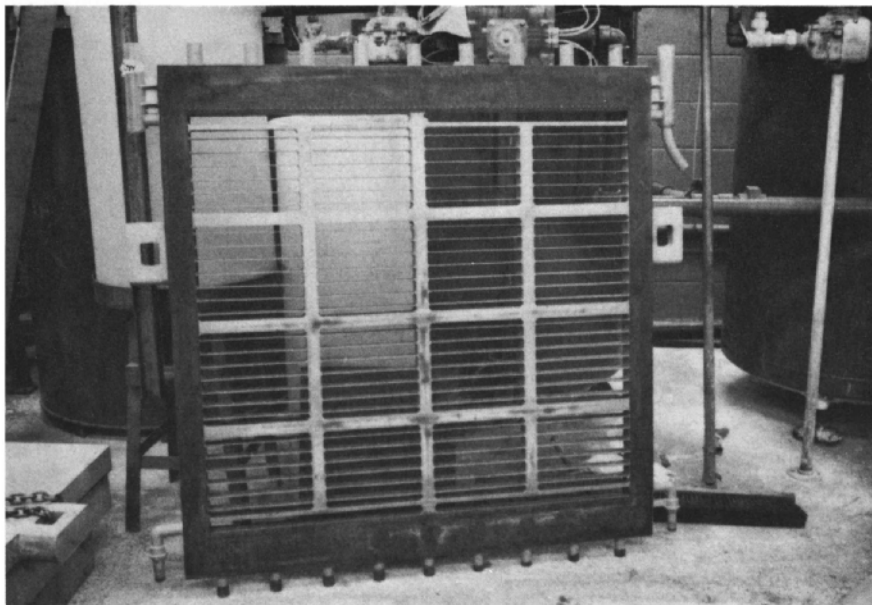


Figure 7

Asahi Chemical Industry Feeding Frames

Top Photo: Electrode rinse side of feeding frame showing a sponge gasket in place.

Bottom Photo: Stack side of feeding frame showing a sponge gasket in place.

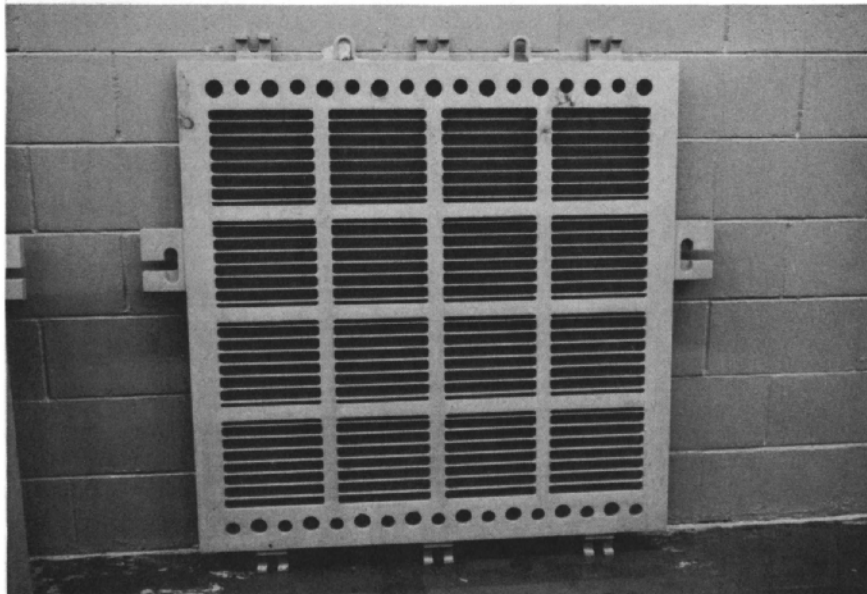
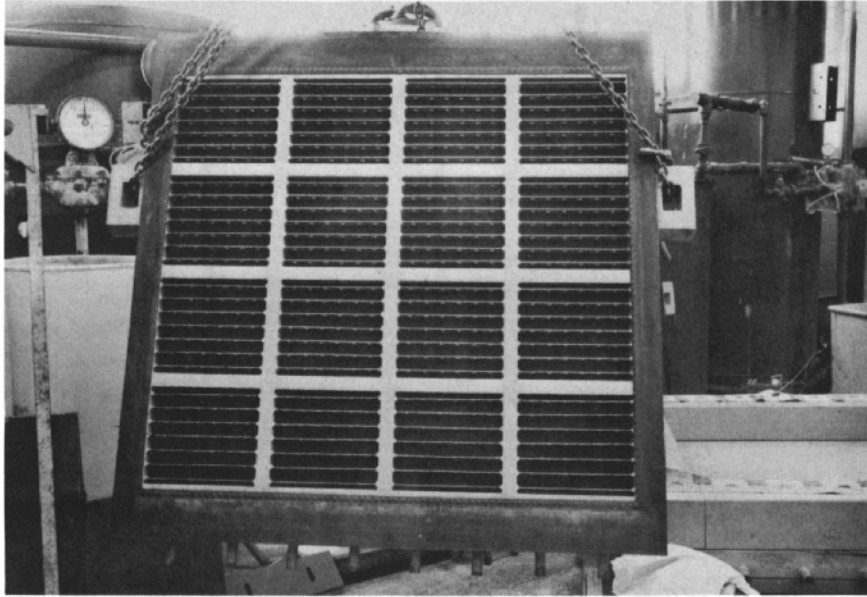


Figure 8

Asahi Chemical Industry Anode Holder and Fastening Frame

Top Photo: Anode holder with sponge gasket and carbon anodes in place. A wood insulating block which separates the stack is shown at the right.

Bottom Photo: Fastening Frame. The larger holes carry dilution water while the smaller holes manifold concentrate water.

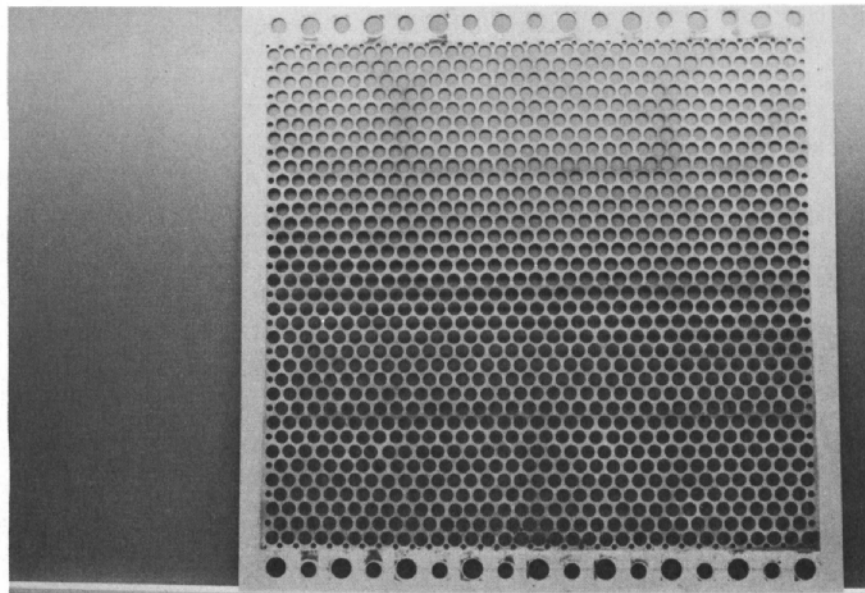
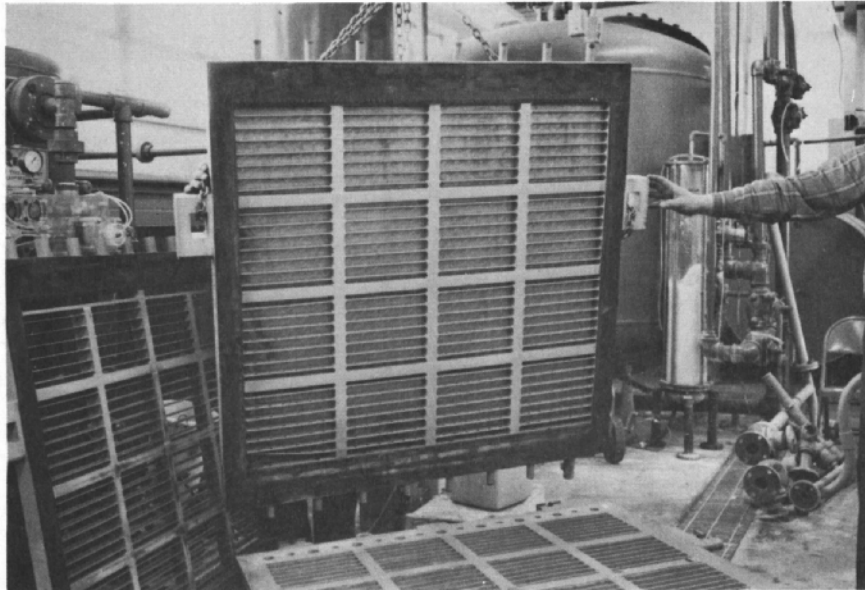


Figure 9

Asahi Chemical Industry Cathode Holder and "F" Frame

Top Photo: Cathode holder with sponge gasket and stainless steel cathode in place. The electrode side of a fastening frame with sponge gasket in place is at the left.

Bottom Photo: Perforated PVC "F" Frame, used as support for membranes in the stack.

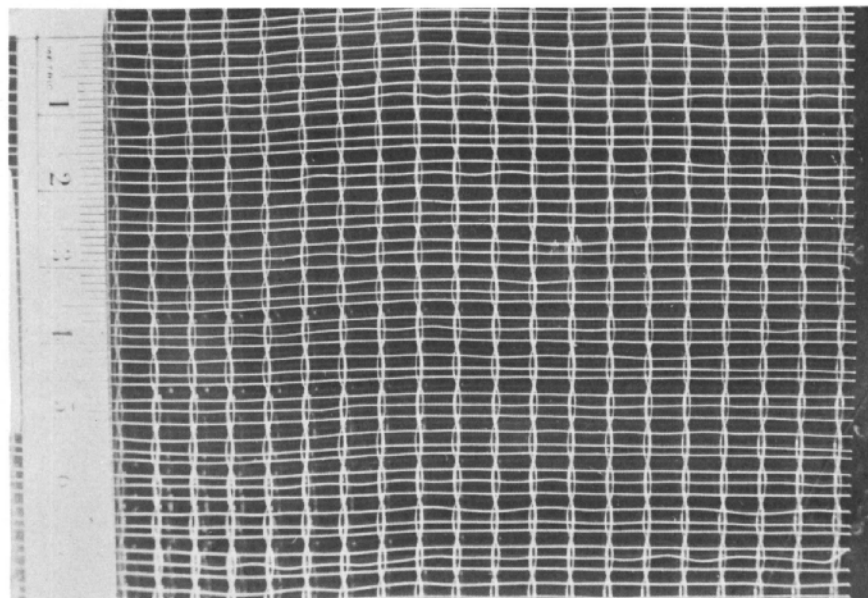
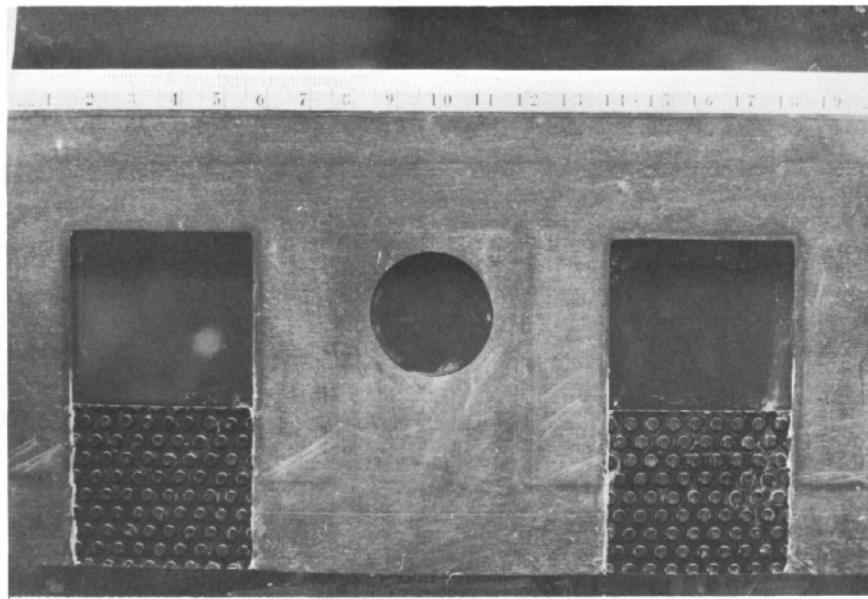


Figure 10

Portion of Asahi Chemical Industry Dilution Gasket and Separator

Top Photo: Magnification 0.58X. Portion of rubber dilution gasket showing two ping-pong slots and one concentrate hole.

Bottom Photo: Magnification 1X. PVC Separator. Water flow is from bottom to top.

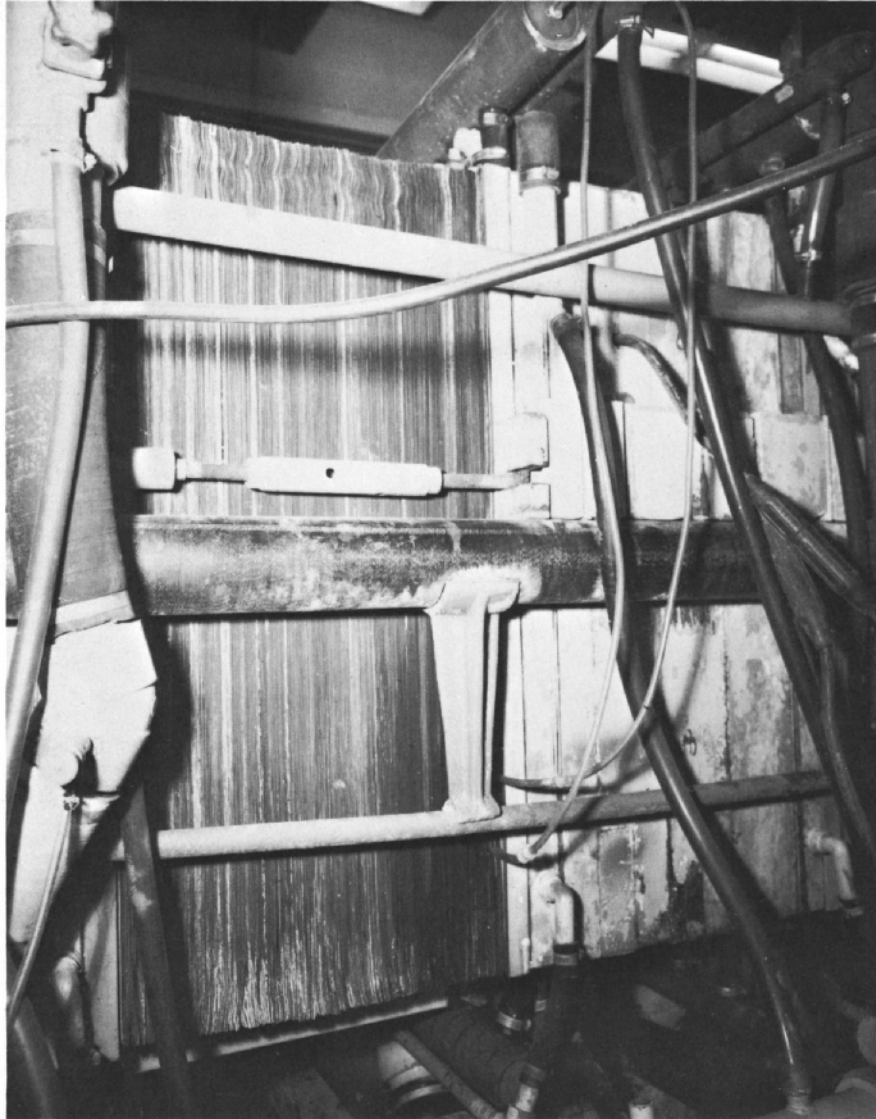


Figure 11

Asahi Chemical Industry Stack

An Asahi Chemical Industry Test Bed Stack built to 250 repeating cell pairs is shown in the electro dialysis press. A turnbuckle, which holds the stack together, is shown just above the center support rod for the entire electro dialysis press.

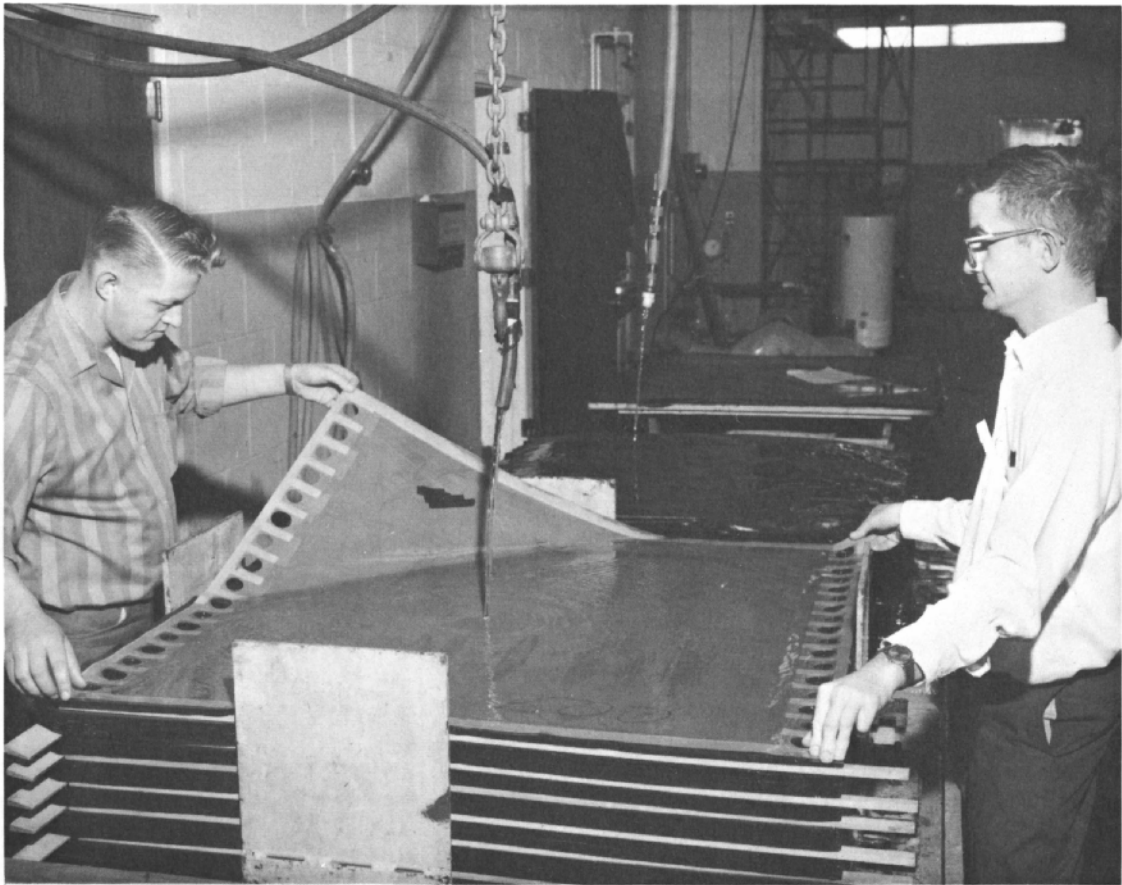


Figure 12

Two people are required to build or rebuild an Asahi Chemical Industry Test Bed Stack. A hose dribbles water on the membrane surface during rebuilding to keep them wet. PVC positioning rods keep the membranes and gaskets horizontal during building. A stack is built to 250 membrane pairs. The DA-1 anion membrane (44 inches by 44 inches, 0.23 mm thick) on top has been repaired with black electrical tape, found useful for repairing small cracks and holes. Both DA-1 and DA-2 membranes stain a darker color.

SECTION III. ACTIVITIES

A. GENERAL

This Annual Report specifically covers activities from July 1, 1967 through June 30, 1968. Prior activities are described in Office of Saline Water Research and Development Progress Reports 101, 132, 164, 241, and 296. Test Bed and Pilot Plant evaluations are discussed in Sections III and IV of this report. Economic analyses are contained in Section V.

B. ORGANIZATION

The Mason-Rust on-site technical and operating staff is depicted by Figure 13. Mr. R. A. Ackerman is Plant Manager, Dr. J. S. Nordin is Research Engineer; there is one administrative assistant, one accountant, one clerk, one chemist, four laboratory technicians, one lead plant operator, and five water plant operators. The water plant operators are also responsible for all maintenance and some pilot plant operation maintenance.

A new three year labor-management agreement was reached on 17 August 1967 between Mason-Rust and Local No. 80 of the International Union of Operating Engineers. Local No. 80 represents the water plant operating personnel at the Webster Test Facility.

During May 1968, Mr. R. D. Parikh resigned as Mason-Rust chemist; he was replaced by Mr. Donald Bogue.

C. MODIFICATIONS

1. Boiler Room Addition (Completed)

In May 1968, an addition to the northwest corner of the Test Facility was completed. Originally intended to house a 200 H.P. boiler and heat exchangers for high temperature electro-dialysis, the 1200 square feet now houses all shop equipment, a lime softening pilot plant, parts storage and an 80 H.P. surplus boiler. The boiler, which was installed in November 1967, is used for building heat and pilot plant high temperature electro-dialysis experiments.

ORGANIZATION CHART
WEBSTER TEST FACILITY

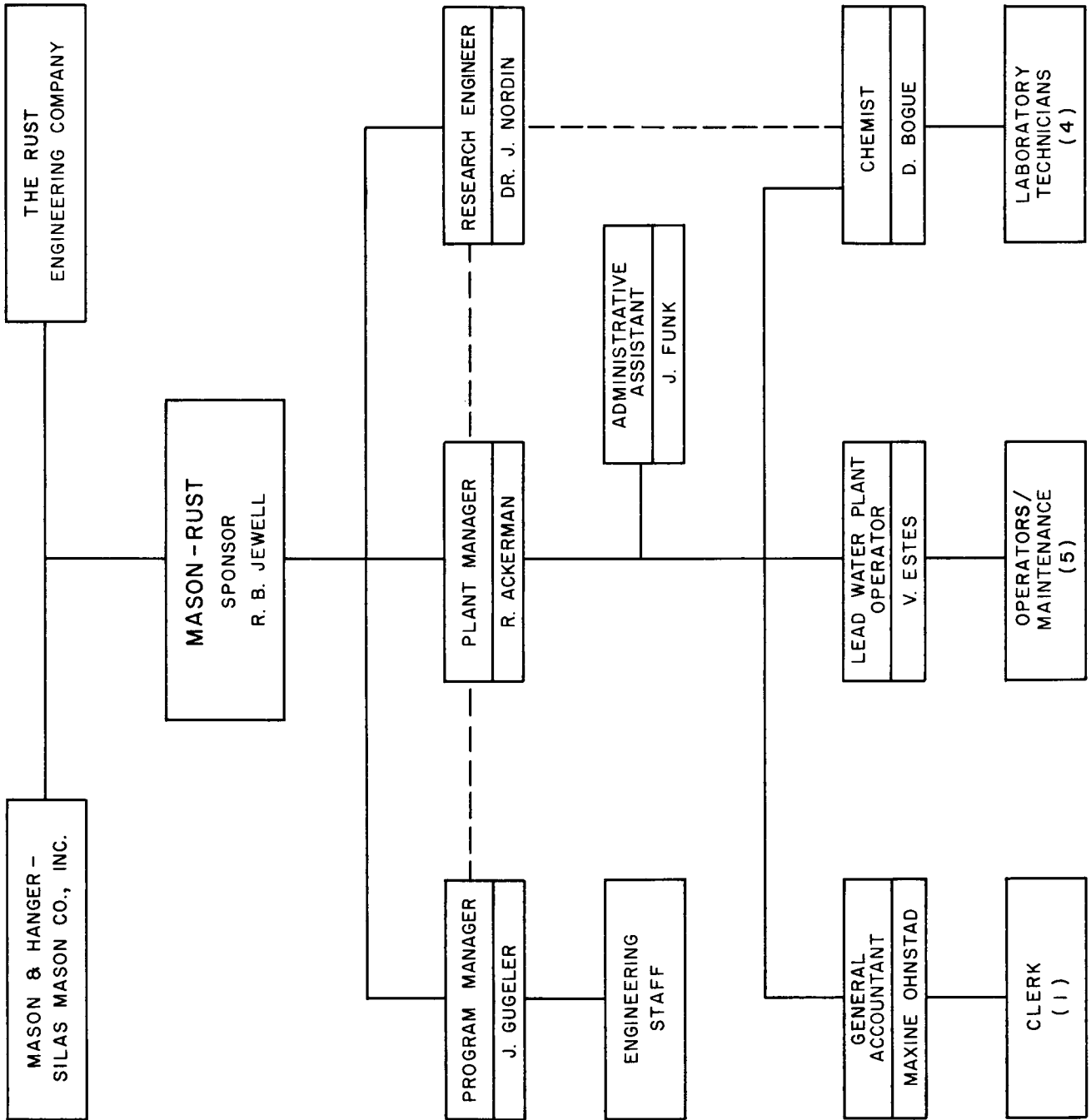


Figure 13

2. Office Room Addition (Completed)

In October 1967, an addition to the southwest corner of the Test Facility was completed. The 368 square feet is used as office for administrative, technical, and accounting personnel. The old office, 156 square feet, is now utilized as a library and conference room. The contract for the office room addition was let in July 1967 for \$14,953.00.

3. Well Number 6 Raw Water Supply (Completed)

During 1967, a contract was let to Independent Drilling Company of Aberdeen, South Dakota, to drill a test hole to granite (1,600 feet) and to drill a 400 GPM shallow production well (187 feet) at a total cost of \$40,801.00. The production well was drilled because the four city owned wells which normally supplied water to the Test Bed Facility fluctuated both in capacity and TDS resulting in a variable feed water for test purposes. Also, the city owned wells did not have sufficient capacity to supply both the City of Webster and larger capacity test units at the Test Facility.

The test hole results, as analyzed by the UOP Johnson Division of Minneapolis, Minnesota, indicated that the water would have a TDS of about 2,350 ppm, mostly sodium sulfate, about 63°F, and would yield between 100 GPM to 200 GPM. The Office of Saline Water chose not to develop the 1,600 foot well, which would cost about \$100,000.00.

The 400 GPM shallow production well was completed and was supplying water to the Test Facility by 5 December 1967. The well is 187 feet deep, and is fitted with 40 feet of 12 inch diameter No. 50 slot stainless steel screen, and a submersible 40 H.P. Jacuzzi pump.

The site of the 400 GPM production well in relation to the Webster Test Facility property line is depicted by Figure 14.

4. Multi-purpose Test Facility Addition and Lime Softening Pretreatment Plant (Started)

During September 1967, Architectural and Engineering work was started by Mason-Rust at Jacksonville, Florida, on a major building addition to the Webster Test Facility. The multi-purpose addition was to house a lime soda softening and carbon filtration plant for the Test Bed Plant, pilot plant space, a water analysis laboratory, and office space.



Figure 14

Shown above is an 8 foot by 12 foot brick structure housing Well No. 6. The well, owned by the Government, is located on the north-west corner of the Webster Test Facility plot measuring 142 feet by 144 feet. The small white structure to the far right is Well No. 3 owned by the City. The building in the rear is the local hospital. The round concrete pad in the lower left is the top of a lift station owned by the City and used to handle brine and other waste water from the Test Facility. Since the photograph was taken in November 1967, an additional 200 feet of property to the north was acquired at no cost to the Government.

During November 1967, a sub-contract was awarded to Infilco, Inc. for \$97,437.00 to provide equipment in August 1968 for a 400 gallon per minute cold-lime-soda softening plant, sand filters and activated carbon filters.

With OSW approval, a sub-contract was signed with McGrann Brothers Construction Company of Watertown, South Dakota, on 10 June 1968 for \$332,363.00 for a 62 foot by 104 foot building with a partial second floor. Approximately 2,800 square feet of floor space will be pilot plant area, and 3,000 square feet of the first floor will be used to house the 400 GPM lime soda softening plant and soda ash storage. Included also on the first floor are a small receptionist office, lobby, lavatory, and storage room. The second floor will contain 2,900 square feet of floor space including 850 square feet of laboratory space, lavatory, office area, and storage area.

Excavation for the new addition was started 10 June 1968 and completed 17 June. Building completion is expected in early 1969.

5. Miscellaneous Modifications to Test Bed Plant (Completed)

a. Air Drier

An air drier capable of drying all compressed air produced by the air compressors and used by plant instrumentation was installed in September 1967. The drier will eliminate corrosion problems which frequently occur in the plant pneumatic instrumentation. The air drier was supplied by Gas Drying, Inc. and is capable of drying 20 CFM of air.

b. Laboratory

New laboratory furnishings, purchased in September 1967, were installed in the analytical laboratory during November 1967. This included additional lighting and electrical outlets. The laboratory is capable of analyzing for most ion constituents found in brackish waters.

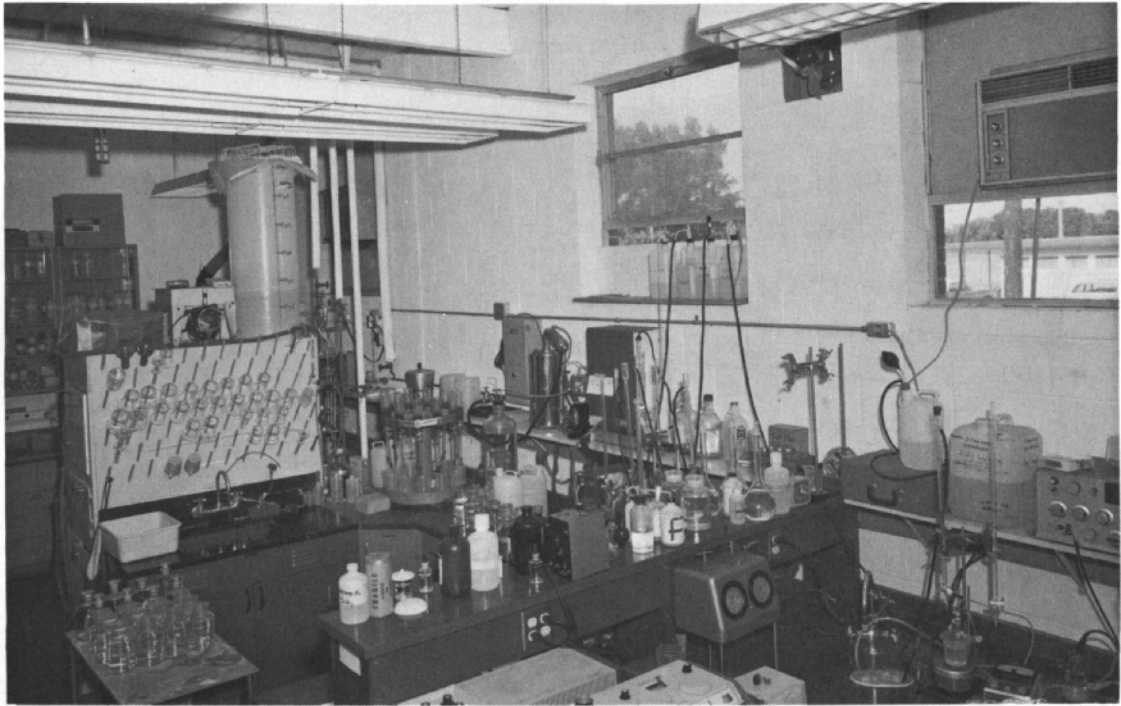


Figure 15

Figure 15 shows the west end of the furnished laboratory.

c. Electrical Revision

A new 1600 ampere electrical service was installed in March 1967 for Pilot Plant and Test Bed Plant use. High voltage rectifier primary cables and conduit were installed from the 1600 ampere service to the Ther Rectifiers used for the transport depletion studies.

d. Transport Depletion Evaluation

The major modification (and expense) to the Test Bed Plant in order to permit evaluation of the transport depletion process was installation of two 150 ampere, 1500 volt maximum capacity Ther rectifiers for the first and fourth stages. These were installed in April 1968.

The first stage was isolated from the remainder of the electro dialysis system during April 1968 so that transport depletion could be studied in the first stage. The 100 GPM concentrate make-up was fed directly to the first stage concentrate inlet manifold rather than the recirculating concentrate tank as depicted by Figures 4 and 5. The dilution flow of 175 GPM from the manganese Zeolite filters and 111 Tank (Figure 2) was fed to the first stage. The first stage dilution product was used as feed to the second stage. The first stage concentrate exit water was used as make-up water for the recirculating concentrate tank. The water from the recirculating concentrate tank was fed in parallel to the other three stages as shown in Figures 4 and 5. This arrangement permitted (1) a low TDS water in the brine stream of the first stage and (2) acidification of the concentrate streams of the remaining stage while the first stage concentrate was non-acidified. A low TDS in the concentrate stream is desirable to prevent calcium sulfate precipitation due to concentration polarization at high current density.

e. Acid Injection Evaluation

After completion of the acid injection evaluation studies in the Test Bed Plant, process piping was installed in May 1968 to bypass the acid injection equipment. This equipment included Tank 06, the PVC decarbonator, Tank 111, Pump 02A, and Pump 02B (Figure 1). The $KMnO_4$ feed was moved to a new injection point just after the 5,700 gallon storage tank. The modification was made to reduce power demand and minimize maintenance.

D. OPERATIONS

1. Test Bed Plant

The Test Bed Plant operation statistics are summarized by Tables 2, 3, and 4. The "acid backwash" referred to by Table 2 is performed each day, usually from 0100 hours to 0500 hours, and consists of flushing both concentrate and dilution compartments with pretreated water of pH = 1.8 to 2.0. The "Maintenance" described by Table 3 includes items such as instrument calibration and disinfection of water lines after shut down of the plant; there was also maintenance performed when the plant was in operation or shut down for other reasons.

The Test Bed Plant was off line for a total of 447 hours for the modifications described by Section II-C. These hours include:

Bypass acid injection system:	11 hours
Transport depletion and electrical revision:	77 hours
Well Number 6 hookup:	341 hours
Other modifications:	18 hours

The total power demand on the plant decreased about 1 KWH per 1,000 gallons of product water after the acid injection system and pumps were bypassed in May 1968 as shown by Table 2.

Webster water wells available for Test Facility use are listed by Table 1. Since 5 December 1967, Well No. 6 has been used exclusively except during backwash of the Elgin Filters. The Elgin Filters remove iron and some manganese from the raw well water after KMnO_4 injection, prior to the electro dialysis stacks.

2. Pilot Plants

Mason-Rust pilot plant operations at the Webster Test Facility during Fiscal Year 1968 are listed below:

<u>Pilot Plant</u>	<u>Study</u>	<u>Pretreatment Equipment Used To Supply Water for Pilot Plants</u>
a. Aerojet General 10,000 GPD Model 1-5608-2	Reverse Osmosis	2-stage Diatomaceous Earth Filtration
b. Ionics Stackpack	Pretreatment Study and High Temperature Electrodialysis	Lime Softening plus Carbon Filtration and Test Bed Plant Manganese Zeolite Filtered Water
c. Aqua Chem WD 6-2	Pretreatment Study and High Temperature Electrodialysis	Lime Softening plus Carbon Filtration
d. ACI SV-3 Acilyzer	Pretreatment Study, Membrane Evaluation, High Temperature Electrodialysis	Lime Softening plus Carbon Filtration, and Test Bed Plant Manganese Zeolite Filtered Water.

These studies are described in Section V and Figure 37.

TABLE 2

TEST BED PLANT OPERATION STATISTICS

Month 1967-68	Feedwater 10 ³ Gal	Product 10 ³ Gal	Percent Conversion	Product Water TDS (ppm)	KMnO ₄ lbs/10 ³ Gal	H ₂ SO ₄ * lbs/10 ³ Gal	Total Energy* KWH/10 ³ Gal	DC Demand* KWH/10 ³ Gal
July 67	10423	6293	60.4	700-800	0.0461	5.81	11.63	2.93
Aug	10484	6304	60.1	750-950	0.0450	5.92	11.26	2.98
Sept	7656	4398	57.4	900-1100	0.0451	8.64	11.49	2.82
Oct	8753	5290	60.4	475-650	0.0428	7.19	10.78	2.57
Nov	10331	6299	61.0	575-675	0.0427	6.53	10.96	2.84
Dec	10768	6445	59.9	750-825	0.0428	3.37	11.21	2.99
Jan 68	11073	6625	59.8	800-900	0.0434	3.28	10.95	3.06
Feb	7701	4542	59.0	700-750	0.0434	3.87	10.66	2.70
March	11132	6752	60.7	900-950	0.0424	3.82	10.15	2.66
April	10267	6188	60.3	700-850	0.0423	3.88	11.8	4.29
May	9851	6264	63.6	750-800	0.0409	3.91	9.6	2.92
June	10332	6789	65.7	725-775	0.0402	3.84	9.6	3.63

* Per thousand gallons of product water.

TABLE 3

TEST BED PLANT OPERATION STATISTICS

Month 1967 - 68	On Stream Hours	Acid Backwash	Change Stacks	Power Failures	Downtime Hours			Total Downtime
					Maintenance	Repairs	Modifications	
July 67	617	104	3	0	0	20	0	127
Aug	603	48	1	0	0	88 ^a	4	141
Sept	422	83	1	0	0	1	213 ^b	298
Oct	481	106	8	0	1	10	138 ^b	263
Nov	567	138	4	0	0	11	0	153
Dec	584	134	12	0	10	4	0	160
Jan 68	612	111	8	2	0	11	0	132
Feb	415	66	6	0	0	209 ^c	0	281
March	609	103	5	1	2	5	19	135
April	557	101	0	1	0	2	59	163
May	552	109	9	3	0	57 ^d	14	192
June	581	117	3	6	3	10	0	139
Total	6600	1220	60	13	16	428	447	2184

a. Most of downtime used to repair Elgin Filter underdrain strainers.

b. Off line from 22 September to 6 October to lay Well No. 6 water line under acid tank.

c. Includes 203 hours off line to repair Elgin Filter underdrain system and O2 Pump.

d. Includes 44 hours off line due to failures of O2 Pump.

TABLE 4
 GROSS WATER PRODUCTION SUMMARY
 FOR PERIOD
 1 JULY 1967 - 30 JUNE 1968

Total on Stream Time, Hours	6,600
Total on Stream Time Days	275
Percent on Stream Time	75.1
Total Downtime, Hours	2,184
Total Downtime, Days	90
Percent Downtime	24.9
Total Feedwater, 10 ³ Gallons	118,770.5
Total Product, 10 ³ Gallons	72,189.0
Total Waste, 10 ³ Gallons	46,581.5
Average Gross Production Per Day on Stream, Gallons per Day	262,505

E. MAINTENANCE AND REPAIRS ON TEST BED PLANT

1. Pretreatment System

a. Iron-Manganese Removal Filters

On 3 August 1967 and again on 12 February 1968, excessive carryover of Zeolite from the Elgin Filters necessitated plant shut down. The cause was physical erosion of the polyethylene underdrain strainers, which is depicted by Figure 16. The underdrain strainers of Elgin Filter Numbers 1 and 3 were replaced in August 1967, which caused the plant to be off line about 80 hours. The plant was off line for 203 hours in February 1968 to repair the polyethylene underdrain strainers of the Elgin Filters; in that the bases of the strainers had to be epoxy glued and patched, the amount of plant downtime was longer.

Other repairs included: (1) Repair and later replacement of the timer on the Number 1 Elgin Filter, (2) Replacement of the Cla-Valve diaphragm on the Number 1 Filter, (3) Repair sleeve and replacement of rusted nipple on water line to Number 3 Filter, and (4) Installation of sleeve between Filter Number 3 to replace cast iron pipe sections corroded during the acid injection evaluation.

b. Storage Tank and Metering Pump, $KMnO_4$ Feed

During this report period a new plunger, lantern rings, packings and diaphragm were replaced on the pump.

2. Acid Injection Pretreatment System

The Chemcon acid pump supplied with a pneumatic positioner to automatically control output failed to function as desired during bicarbonate removal test runs. The following modifications and repairs were made on the equipment to correct the malfunctions:

a. Acid Pump

1. The head of the pump was replaced due to corrosion. The original pump head was constructed of 316 stainless steel alloy while the valve bodies and seats were constructed of Carpenter 20; as the use of dissimilar alloys was thought to result in the corrosion, the pump head was replaced with one made of Carpenter 20 alloy.

Eroded Underdrain Strainer of Elgin Filter

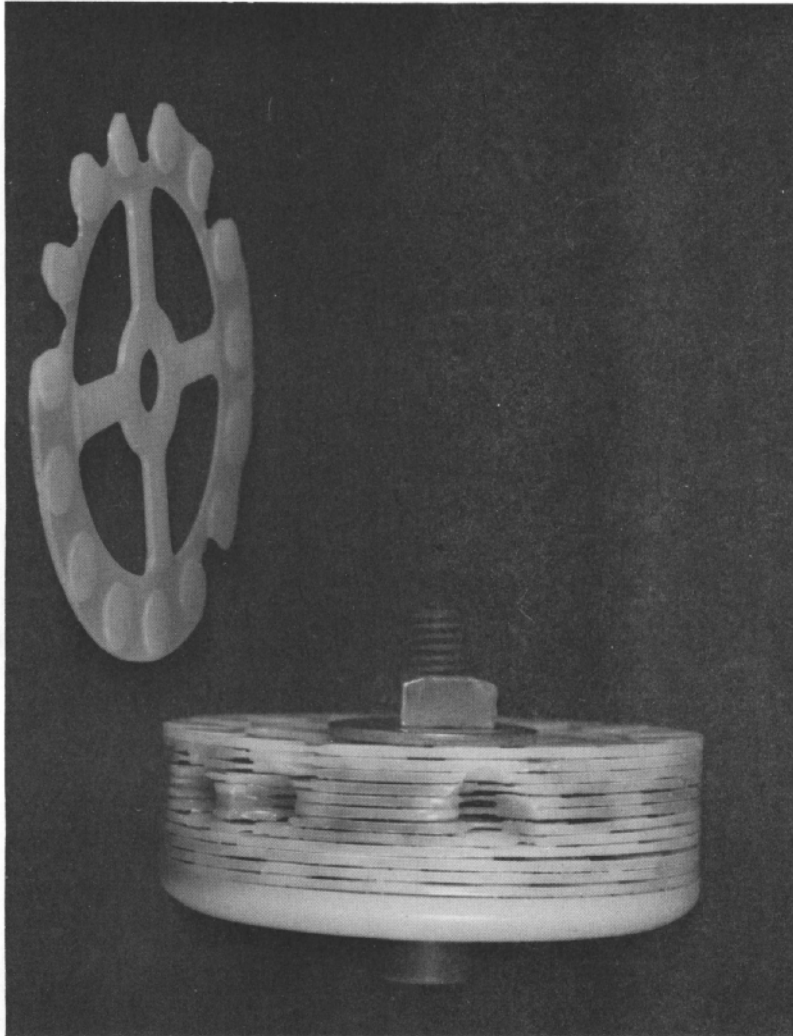


Figure 16

Shown above is a portion of an eroded polyethylene strainer of the Elgin Filter. Zeolite particles were also seen wedged between the polyethylene discs.

2. The new head was fitted with Teflon ball stops which apparently allowed for smooth operation.
3. New mounting holes were drilled through the head to allow the head to be drawn more tightly to the diaphragm housing, to eliminate leakage.

b. Pneumatic Positioner

No maintenance was required on the pneumatic positioner; however, accurate control was not possible due to the distance between the controller itself and the sensing point. Manual control gave a greater degree of accuracy during bicarbonate removal runs.

3. Electrodialysis Stacks

Maintenance and repairs on the electrodialysis stacks are described in Section IV-A.

4. Open Process and Chemical Storage Tanks

These tanks were painted with an epoxy-type paint (Prufcoat, Hooker Chemical Corporation) during this report period. The coating lasts three to four years.

5. Pump 02 - Raw Water Forwarding

During this report period, the Pump 02 solenoid valve was replaced on two occasions.

On 13 July 1967, the pump was cleaned and the bearings and couplings replaced.

On 6 October 1967, the motor on Pump 02 shorted out apparently caused by moisture accumulating in the windings during shut down periods. The motor was rewound with glass coated wire and returned to service.

On 18 December 1967, the pump was cleaned and inspected.

On 16 February 1968, it was discovered that the bronze impeller was nearly worn away caused by small rocks getting into the impeller housing. The rocks are believed to enter the lines when a City well screen failed. The screen was replaced. The impeller was rebuilt and returned to service. During this repair period, the pump received a new shaft, gaskets, bearings, and packings.

On 25 May 1968, the bearings on Pump 02 were replaced. The pump shaft was scarred, therefore, it was taken to a local blacksmith to be rebuilt and aligned.

On 30 May 1968, Pump 02 failed for the fourth time during this report period. The emergency repairs made by the local blacksmith did not last as the shaft was again scarred. The pump was shipped to a pump repair shop in Minneapolis for a complete repair cost estimate.

Close attention is being given to operational maintenance of Pump 02. If failures continue, the pump will be replaced with one of the pumps used in the acid injection evaluation.

6. Pumps 12, 14, 16 - Dilution Stream

Only routine inspection and lubrication were necessary during this report period. All pumps were cleaned and painted.

7. Pump 102 - Product Stream

Only routine maintenance was required during this report period.

8. Pump 21 - Concentrate Stream

On 30 January 1968, a solenoid valve was replaced on this pump.

On 18 June 1968, the pump received a new outboard bearing. The pump was cleaned and the glass casing checked. The casing was chipped but was not repaired as previous attempts to epoxy glue it had failed.

9. Pump 23 - Rinse Stream

Only routine maintenance was required on Pump 23 during this period.

10. Pump 25 - Waste Stream

Only routine maintenance was required on Pump 25 during this report period.

11. Pumps 107 and 108 - Clear Well

Only routine maintenance was required on Pump 107 during this report period.

The springs on the flapper check valve on Pump 108 were replaced on 27 November 1967.

On 15 April 1968, the motor on Pump 108 shorted out, apparently due to moisture accumulated during shut downs. The pump was rewound and put back into service.

12. Pumps 18 and 19 - H₂SO₄ Metering

Routine maintenance only was required on these pumps.

13. Instrumentation

a. pH Recorder and Electrodes

In accordance with an agreement between OSW and the Bureau of Reclamation, a meter technician from the Bureau substation in Watertown, South Dakota, serviced the pH recorder and conductivity recorder quarterly.

M&O Contractor maintenance required on this recorder included replacement of four drive cords, one split gear assembly, batteries, tubes, and electrodes. The meter was cleaned and oiled and the recorder calibrated on two occasions.

b. Conductivity Recorder and Cells

The conductivity recorder is serviced quarterly by the Bureau of Reclamation. M&O maintenance during the Fiscal Year 1968 included replacement of three drive cords, and new lamps. The recorder was cleaned and oiled and the probes adjusted during August 1967.

c. Level Controllers, Process Tank

No repairs or replacements were made during this report period other than the inspection of the float and tightening of the control bolts.

d. Level Controllers, Water Wells

No repairs or maintenance was made during this report period other than routine inspection.

e. Taylor Recording Equipment

In accordance with a service contract with the Taylor Instrument Company, a Taylor representative performed routine maintenance in August 1967 and June 1968 of this report period.

f. Flow Meters

The range tube assemblies were replaced on the four concentrate stream flow meters. Only cleaning of the tubes, adjustment of the packing glands and lubrication of the associated hand control valve bonnet was otherwise required on all flow meters. The concentrate flow meters were calibrated in June of this report period.

g. Annunciator and Alarms

No failures have occurred which required parts or special maintenance.

h. Water Meters, Indicating and Totalizing

No maintenance was performed during this report period other than recalibration.

i. Pressure Switches

No replacement of pressure switches or other component parts was necessary during this period.

14. Deaerators or Degasitors

Only belt replacement was required during this report period. The deaerators were cleaned and painted.

15. Chlorination Equipment

The nozzle on the chlorinator was cleaned on 6 July 1967 after erratic operation of the chlorinator was noted.

16. Submersible Sump Pump

During this report period the sump pump was cleaned and a nipple replaced. No other maintenance was required.

17. Equipment Painting

Considerable painting was done during this report period. Equipment completely cleaned and painted includes the degasitors, Elgin Filters, detention tank, all automatic valves, color coding other process valves, pumps, motors, filter press, and mixers.

18. Clean Water Tank

No maintenance was required during this report period other than routine inspection.

19. Raw Water Tank

On 4 December 1967, after the raw water line from Well No. 6 was disinfected, the raw water tank was cleaned of all silt and sand deposits. No other maintenance was necessary.

20. Waste Water Tank

The waste water tank was cleaned during this report period.

21. Mixers (4)

Maintenance required during this report period included replacement of bearings in one mixer and the cleaning and lubricating of all mixers.

22. Well No. 6 Water Line

A leaky nipple in Well No. 6 water line was replaced on 6 December 1967. No cost was involved as repairs were made under warranty.

F. SAFETY

On 17 October 1967, a Mason-Rust employee received an eight day loss time injury incurred when he sprained his ankle while performing routine duties.

Other doctor cases, no lost time, were a cut chin and cut hand.

The objective of the present Safety Program is to develop and maintain an ever increasing awareness of safety in each member of the operating staff, and foster personal participation in all phases of the program. Safety instruction and inspections are routine and will be continued in an effort to eliminate any unsafe work practices and conditions.

During this report period, guard rails were installed around the fuel oil tanks, a safety shield was built around the electrolysis stacks, several dry chemical and CO₂ fire extinguishers were added to protect added space, high voltage signs were purchased and installed, and metal build-up scaffolding was purchased for general plant use.

Rubber foot wear, safety goggles, rubber gloves and canvas gloves were provided for all operators.

Emphasis was on "Safe Handling of Hand and Power Tools".

SECTION IV. TEST BED PLANT EVALUATION

A. GENERAL

The four Test Bed Plant Programs with their time schedules are depicted by Figure 17.

Some general descriptive material on the Test Bed Plant was presented in Section II. Water production statistics, modifications, repairs, and maintenance information for Fiscal Year 1968 are presented in Section III (Activities). This section discusses special problems and experiments performed on the Test Bed Plant.

Test Bed Electrodialysis Operation is discussed under the "Routine Data Gathering and Analysis" Section, IV-B. The anion membrane fouling problem described during Fiscal Year 1967 continues to be a major problem during this report period. Nightly acid backwashing of the electrodialysis stacks is seen to almost eliminate calcium carbonate, magnesium hydroxide, and iron floc accumulations within the stack as proven by stack rebuildings at the expense of increasing plant downtime to perform the backwash. Membrane losses due to breakage are seen to increase over the previous year.

Pilot plant tests and other information said that the ACI DA-2 anion membrane might foul much more rapidly than the ACI DA-1 anion membrane. To demonstrate this, a stack was built half of DA-1 and DK-1 membranes and half of DA-2 and DK-1 membranes, and then operated from October 1967 through June 1968. Information obtained from this test is presented in Section IV-C, under "Anion Membrane Poisoning Film Buildup".

During late Fiscal Year 1967, the pretreatment system was modified to permit sulfuric acid injection into the water to remove varying levels of bicarbonate. Electrodialysis operation on such a water is discussed in Section IV-D.

Evaluation of the Transport Depletion process at Webster is discussed in Section IV-E. The ACI DK-1 membranes were used as the cation permeable membrane. Regenerated cellulose membranes supplied by Union Carbide (trade name Zephyr) were used as the "neutral" membrane.

A plan for conversion of two stages of the Test Bed Plant to high temperature electrodialysis operation was canceled at Mason-Rust's recommendation after evidence was gathered that (1) membrane "fouling" and plant corrosion problems would obscure results of

FIGURE 17

TEST BED PLANT EVALUATION

PROCESS DEVELOPMENT PROGRAMS AT WEBSTER TEST FACILITY

FISCAL YEAR 1968

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
Routine Data Gathering and Analysis	Regular water analyses of process streams. Hourly plotting of stack resistance data. Stack dismantling and building details. Production plant costs. Chemical consumption, etc.											
Anion Membrane Poisoning Film	Compare long term operation of DA-1 and DA-2 ACI anion membranes.											
Acid Injection	Inject H ₂ SO ₄ to remove bicarbonate. Study long term effects. Statistical Study of current efficiencies, stack resistances, etc. with and without acid injection, with and without pretreatment aeration. Aerator evaluation.											
Transport Depletion	Procure High Capacity Rectifiers Modify First Stage for T.D. Evaluation Conc. Use UC Zephyr Regenerated cellulose membranes, with ACI DK-1 cation membranes, in ACI equipment. Zephyr Membrane lasts only one month Conc. Stream not Acidified Studies revert back to pilot plant.											

such a study, and (2) preliminary pilot plant studies and economic considerations demonstrated that such a test would be too costly to execute during Fiscal Year 1968 - 1969 in relation to the information that would be obtained.

B. ROUTINE DATA GATHERING AND ANALYSIS

1. Objective

The objective is to critically examine electro dialysis process data in order to (a) identify problem areas, and (b) determine the contribution these problem areas have on the total cost of water production.

2. Examination of Electrodialysis Data

Much information can be learned by examination of the Test Bed Plant operation statistics presented in Tables 2, 3, and 4. One problem was that the Test Bed Plant only operated 275 days on stream, compared with the 330 days on stream figure usually used by Mason-Rust in cost analysis studies. If one prorates this 275 days on stream figure for plant modification described in Section III-C, the on stream figure is 290 days which is still low. Table 2 shows 1220 downtime hours (51 days) were due to acid backwashing of the electro dialysis stacks. Nightly acid backwashing has the effect of reducing gross annual water production. Therefore, the average yearly product water costs per 1,000 gallons must be prorated over 51 fewer days, which would increase the fixed cost contribution to product water costs by about 14 percent. There were also 11.8 days downtime to repair the under-drain strainers of the Elgin Filters; this downtime alone increased the fixed cost contribution by over 3 percent.

During July to August 1967, the frequency of acid backwashing the stacks was varied. Figure 18 depicts first stage stack resistance data for the case of nightly acid backwashing. Figure 19 presents stack resistance data when the stacks are acid backwashed twice a week. Operation is considerably smoother for the case where the stacks are acid backwashed nightly as this is a necessary maintenance procedure with the present pretreatment system. Stack acid backwashing accounts for roughly half of the total downtime, but is unavoidable. Operation during prior fiscal years has demonstrated that large membrane losses are due to alkaline scale formation when the stacks are not acid backwashed frequently.

The high product water TDS (Table 2) is caused by fouling of the anion permeable membranes. This phenomena is described in some detail in the Fiscal Year 1967 Annual Report, where it is shown that carbon filtration will remove the fouling or poisoning agent(s). Fouling of the anion permeable membranes necessitates lowering the operating current density in order to avoid excessive polarization and alkaline scale formation. The lower current density means a product water TDS of about 600 to 1000 ppm (depending on the condition of the membranes) rather than under 500 ppm as predicted by the Asahi Chemical Industry. To completely prevent excessive polarization and alkaline scale formation, the current densities would have to be lowered much more than what was done during Fiscal Year 1968 (see Table 3 of Fiscal Year 1967 Annual Report); however, the product water TDS would be in excess of 1000 ppm. Therefore, a compromise was reached between nightly acid backwashing the stacks to remove alkaline scale in the concentrate compartments and operation at higher current densities. The dilution compartments were also acid backwashed with water of pH = 1.8 to 2.0 to remove iron floc carryover from the manganese Zeolite pretreatment system.

Figures 20, 21, 22, and 23 indicate energy consumption of each of the stacks during Fiscal Year 1968. The energy is computed according to the formula using noon readings of voltage and current:

$$E_T = \frac{I V}{60 G}$$

E_T = KWH/1,000 gallons, energy consumption per 1,000 gallons of product water

I = Current, amperes

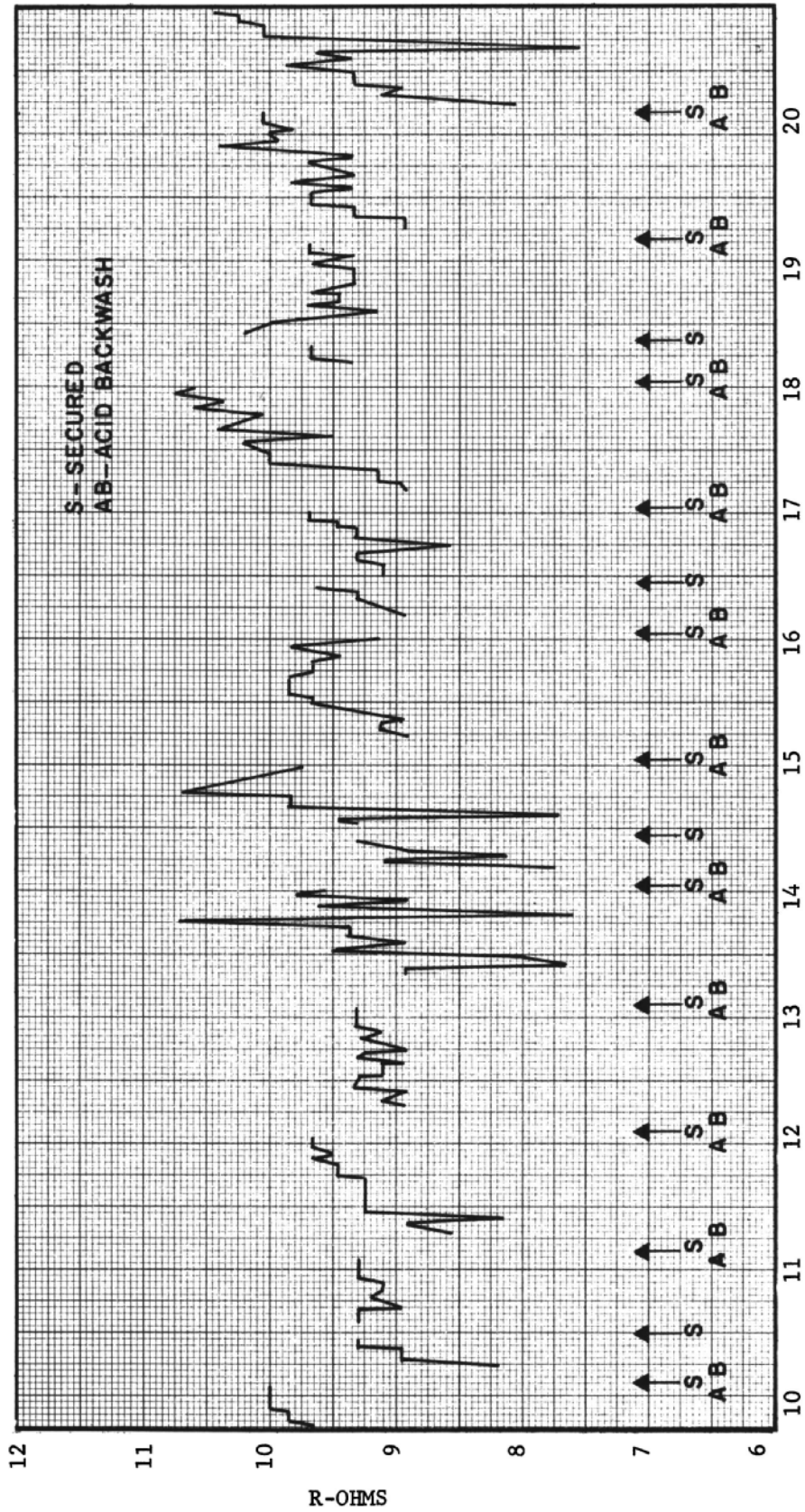
V = Total stack voltage, volts

G = Dilution flow rate, GPM: 175 GPM 1 July through 4 June, 210 GPM 5 June through 30 June

Inspection of Figures 20 through 23 show considerable fluctuations in energy consumed throughout the year, depending upon which stack (Stack A, B, C, D, or E) was operated in the position designated. The specific resistances of the process streams also varied. A detailed analysis of the individual fluctuations is beyond the scope of the report.

Figure 18

First Stage ACI Stack Resistance
175 GPM Dilution, 100 GPM Concentrate
28 Amperes, Acid Backwashed Nightly

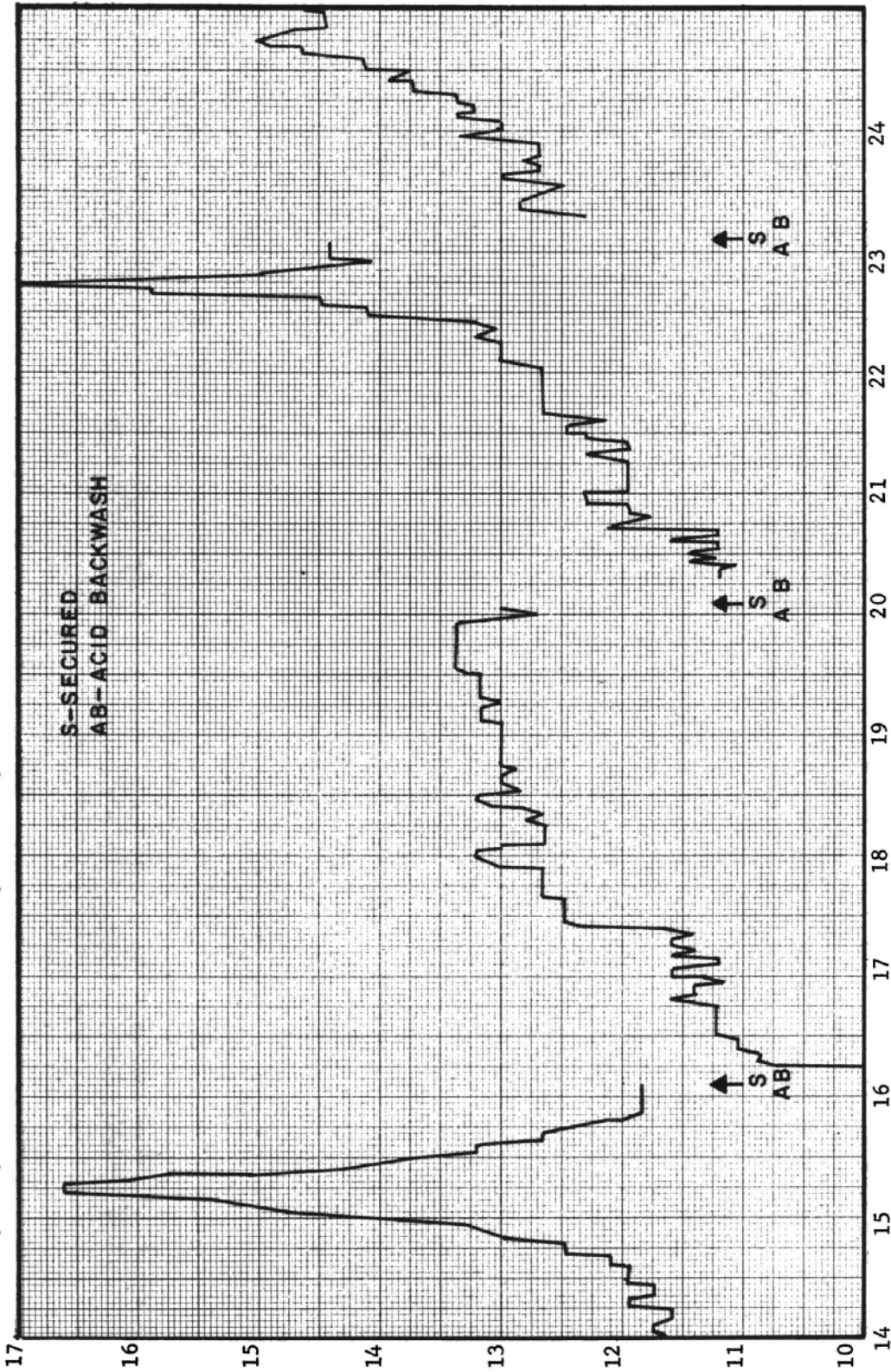


JULY 1967

Webster, South Dakota

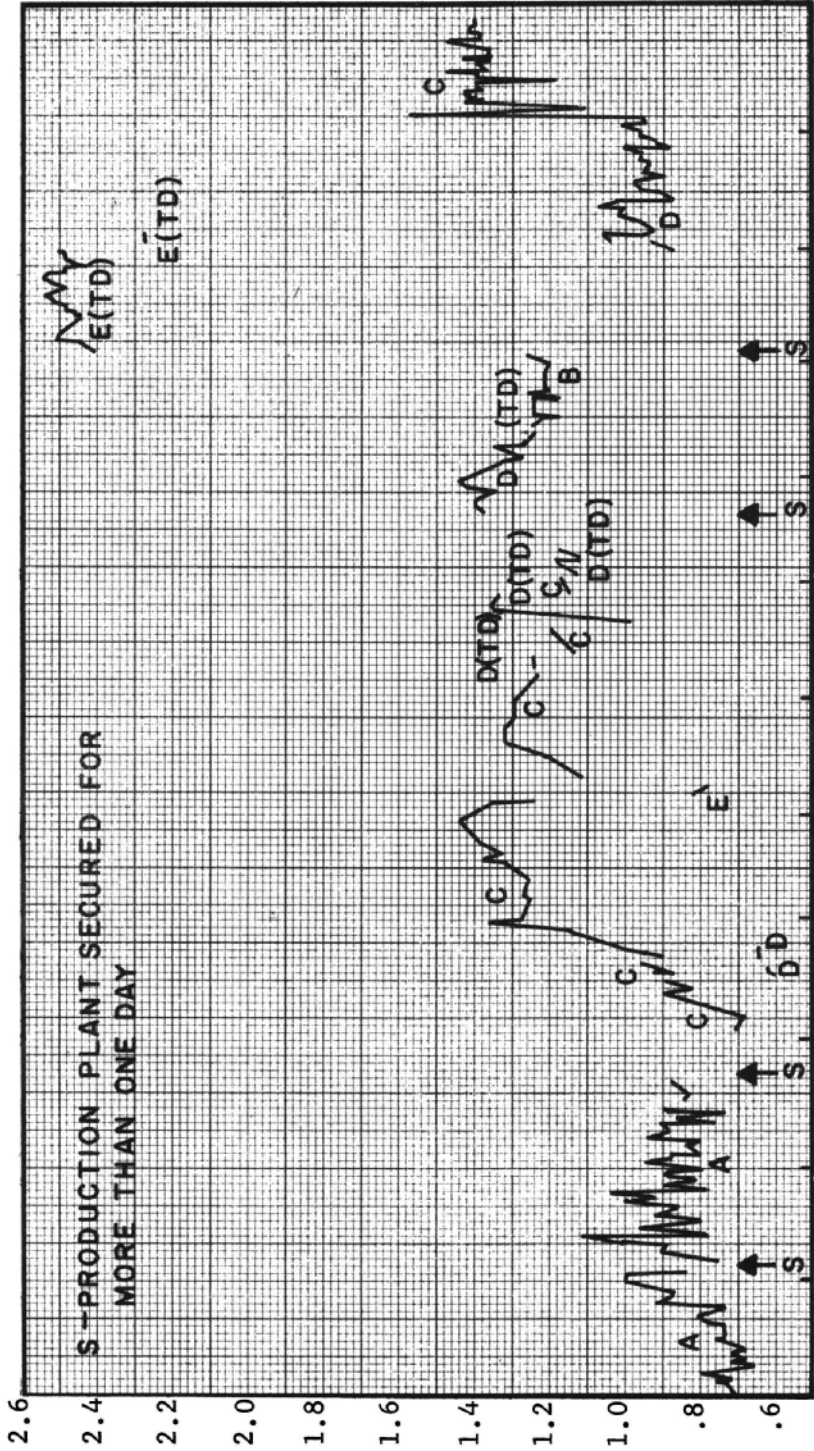
Figure 19

First Stage ACI Stack Resistance
175 GPM Dilution, 100 GPM Concentrate
28 Amperes, Acid Backwashed Every Four Days



R Ohms

Figure 20
ENERGY CONSUMPTION IN FIRST STAGE PRODUCTION STACK



$$E_T \frac{\text{KWH}}{1000 \text{ Gal.}}$$

July Aug. Sept. Oct. Nov. Dec. Jan. Feb. March Apr. May June

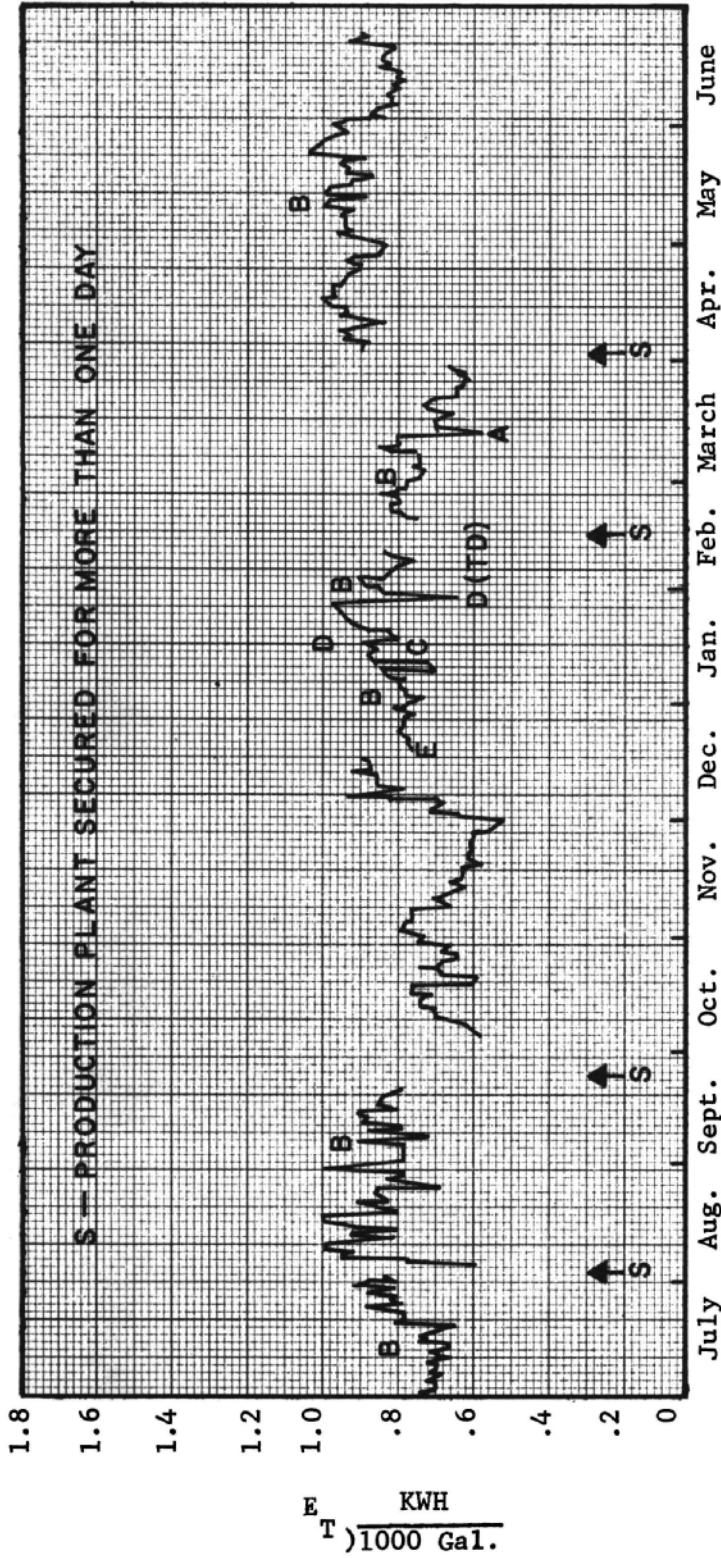
Notation:

A, B, C, D, or E: Stacks A, B, C, D, or E operated in first stage position during period indicated.

D(TD), E(TD): Stack D or E built as a transport depletion unit

Figure 21

ENERGY CONSUMPTION IN SECOND STAGE PRODUCTION STACK

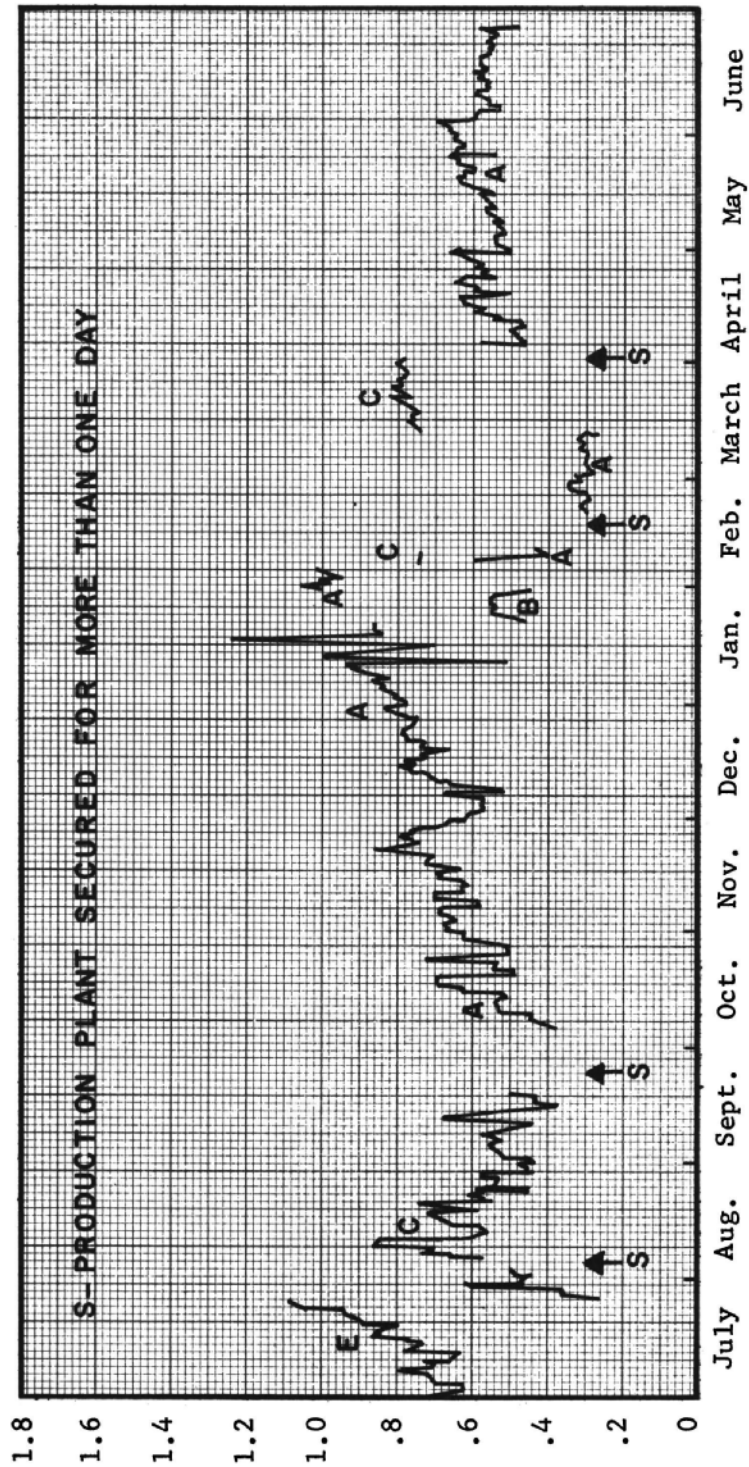


Notation:

A, B, C, D, or E: Stacks A, B, C, D, or E operated in first stage position during period indicated.

D(TD), E(TD): Stack D or E built as a transport depletion unit

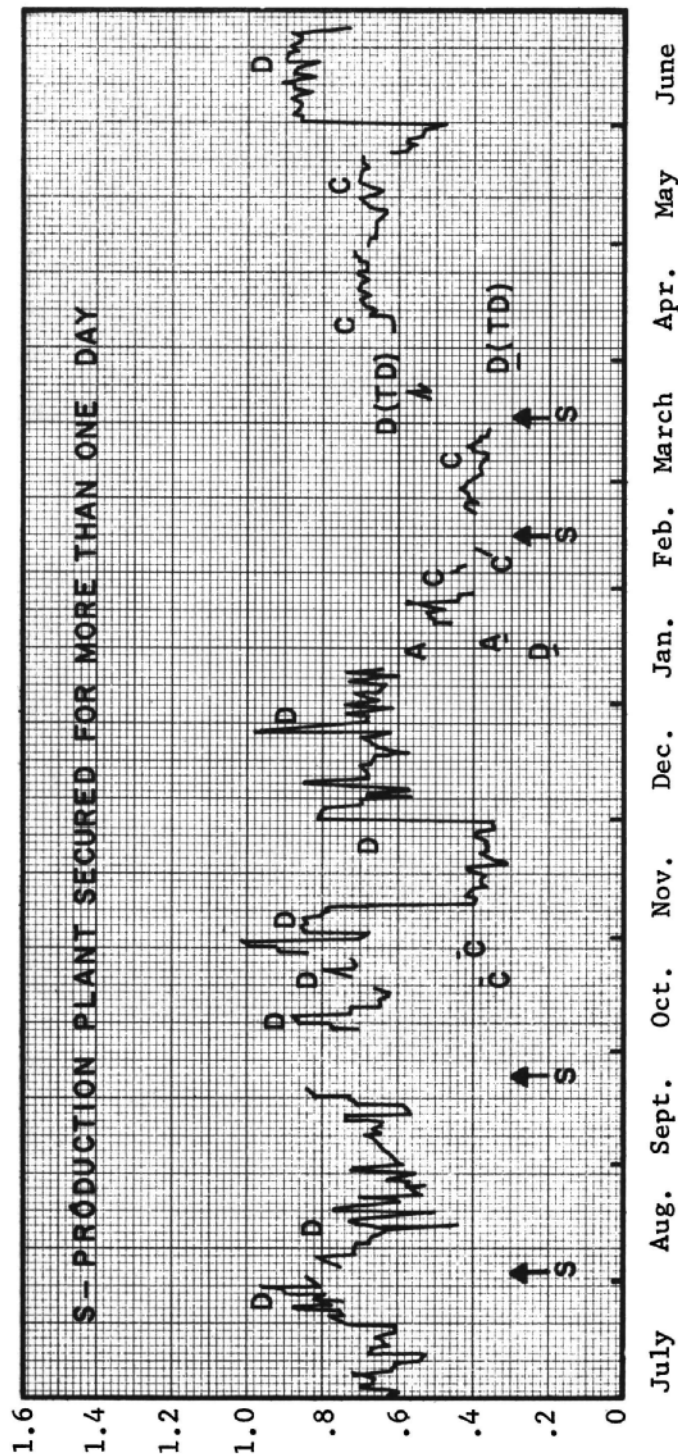
Figure 22
 ENERGY CONSUMPTION IN THIRD STAGE PRODUCTION STACK



$$\frac{E_T \text{ KWH}}{1000 \text{ Gal.}}$$

Figure 23

ENERGY CONSUMPTION IN FOURTH STAGE PRODUCTION STACK



Notation:

A, B, C, D, or E: Stacks A, B, C, D, or E operated in first stage position during period indicated.

D(TD), E(TD): Stack D or E built as a transport depletion unit

$$E_T \left(\frac{\text{KWH}}{1000 \text{ Gal.}} \right)$$

3. Electrodialysis Stack Rebuilding Data

Tabulation of ACI Test Bed Plant stack rebuilding data is presented in Table 5. Each stack is built of 250 membrane repeating cell pairs as described in Section II. A total of 17 stack rebuildings took place during Fiscal Year 1968, at a total labor expenditure of 1170 man hours. Of this total, 556 man hours or 9 stack rebuildings can be considered as experimental (430 man hours building or rebuilding transport depletion stacks and 126 man hours for two stack rebuildings for the anion membrane poisoning film study). The stacks are referred to as "A", "B", "C", "D", or "E"; these can be operated in any of the four stack positions with the fifth stack as a spare. The "stage" number presented in Table 5 is the stage the stack was removed from, which is not necessarily the same stage the rebuilt stack is reinserted.

Based on the success of the Mylar film-backed dilution gaskets installed in the first stage stack (Stack A) in January 1967, the other stacks were rebuilt during September 1967 to replace the old gaskets with the improved ones. Just before the rebuilding, distortion of the old gaskets was so great that no more than 175 GPM dilution flow could be pumped through the stages with existing pumps. No problems occurred with the Mylar film-backed gaskets in Fiscal Year 1968.

In a production plant, stacks are normally rebuilt due to high electrical resistance (which may indicate scale formation, iron floc accumulations, or mal-distribution of flow) or low current efficiencies, usually the result of broken membranes.

Stack B was rebuilt 12 to 21 December 1968 because of breakdown in the PVC insulator material of one of the turnbuckles resulting in a short. The "burned out" turnbuckle cut lengthwise for viewing is illustrated by Figure 24. Turnbuckles are used to fasten the moveable part of the stack together (Figures 6 and 11). "Burned spots" of the type illustrated by Figure 25 were seen near the dilution exit holes of a number of membranes upon disassembly of that stack.

There were heavy cation DK-1 membrane losses during the transport depletion study due to breakage along the gasket edges. The flexible regenerated cellulose "neutral" membranes tended to stretch with localized pressure differences between the dilution and concentrate streams, placing more than the usual pressure of the non-elastic and fragile DK-1 membranes, even though the pressure drop between the dilution and concentrate compartments at the measuring taps was held at zero during the test.

During Fiscal Year 1968, a total of 505 new DK-1 cation, 257 new DA-1 anion, and 137 new DA-2 anion membranes were placed into electro dialysis service, During the year, a number of membranes were discarded during stack rebuilding due to breakage or scale. During June 1968, the damaged membranes were set aside during stack buildings (including those collected prior to Fiscal Year 1968) and inventoried; of these, 402 DK-1, 169 DA-1, and 84 DA-2 were considered "unrepairable" and discarded.

The membrane consumption from stack operation in Fiscal Year 1968 was 550 DK-1 cation membranes, 280 anion membranes, and 525 regenerated cellulose "neutral" membranes.

The average rebuild time for the 17 stack rebuildings of Table 5 was 69 man hours.

4. Conclusions

- a. The necessity of acid backwashing and the associated hours downtime reflect inadequate pretreatment. Anion membrane fouling by poisoning agents also reflects inadequate pretreatment. These two factors lead to increased product water costs due to excessive power consumption, more electro dialysis stages required to achieve a desired water salinity, more maintenance, and shortened membrane lifetimes.
- b. Examination of the electro dialysis stack rebuilding data shows excessive losses due to breakage.

5. Recommendations

- a. The use of more durable anion and cation membranes, especially an anion membrane that does not foul, would significantly decrease product water costs.
- b. Until such a more durable membrane is found, or until the pretreatment of the feed water is improved, nightly acid backwashing of the electro dialysis stacks is recommended.

TABLE 5
 TABULATION OF ACI TEST BED PLANT STACK BUILDING DATA

Date	Stack and Stage	Labor	Remarks
31 July	Stack E, (Stage 3)	70 hours	191 DA-1 replaced with 191 used DA-2. A number of DK-1 membranes replaced.
12-16 Sept.	Stack A, (Stage 1)	87 hours	64 DK-1 replaced with new, 66 DA-1 replaced with new; 6 DK-1 and 3 DA-1 destroyed because of scale. Four concentrate separators replaced.
16-26 Sept.	Stack B, (Stage 2)	73½ hours	55 DK-1 replaced with new, 59 DA-1 replaced with new; 4 DK-1 and 9 DA-1 destroyed because of scale. All dilution and concentrate gaskets replaced.
26-29 Sept.	Stack C, (Stage 3)	78½ hours	144 DK-1 replaced with new and repaired DK-1. All anions replaced with 125 new DA-1 and 125 new DA-2. All gaskets replaced.
Sept.	Stack D, (Stage 4)	84 hours	27 DK-1 replaced with repaired DK-1, 115 DA-2 replaced with repaired DA-2. All gaskets replaced with Mylar film backed gaskets.
1-7 Nov.	Stack D, (Stage 4)	55 hours	46 DK-1 replaced with new, 11 DA-2 replaced with new.

TABLE 5 (Continued)

Date	Stack and Stage	Labor	Remarks
6-11 Dec.*	Stack C, (Stage 1)	69 hours	28 DK-1 replaced with new, 3 DA-1 replaced with new, one broken DA-1. Positions of DA-1 and DA-2 membranes interchanged.
12-21 Dec.	Stack B, (Stage 2)	48 hours	5 DK-1 repaired and replaced; 2 DK-1 "burned"; 3 DA-1 replaced with new; 1 anion "burned". "Burned" areas result of turnbuckle short. All anion membranes turned over.
12-14 Jan.*	Stack C, (Stage 1)	57½ hours	3 DK-1 replaced with new, 1 DA-1 and 1 DA-2 re-placed with new. All anion membranes turned over.
14-17 Jan.*	Stack D, (Stage 4)	90 hours	All DA-2 membranes replaced with Union Carbide Regenerated Cellulose membranes for transport depletion evaluation. 7 DK-1 replaced with new.
19-23 Jan.*	Stack D, (Stage 2)	51½ hours	8 DK-1 replaced with 4 new and 4 repaired. Stack dismantled to examine TD membranes.
1-5 Feb.*	Stack D, (Stage 2)	70½ hours	104 DK-1 repaired because of breakage. Of these 77 DK-1 needed cleaning because of CaSO ₄ scale. In addition, 31 DK-1 discarded because of breakage. All TD membranes intact.
7-10 Feb.	Stack A, (Stage 3)	59 hours	3 DK-1 and 10 anions discarded because of breakage. 4 other DK-1 set aside for repair. Rebuilt because of high ohmic resistance.

*Stack built or rebuilt for experimental reasons (e.g. transport depletion or anion membrane poisoning film evaluation).

TABLE 5 (Continued)

Date	Stack and Stage	Labor	Remarks
14-23 March	Stack D, (Stage 1)	100 hours	All TD membranes (250) had disintegrated and were destroyed. CaSO ₄ scale on almost all DK-1 membranes. 17 DK-1 membranes broken beyond repair and discarded. Stack rebuilt as spare stack using anions from the former spare Stack E.
24 March* 1 April	Stack E, (Spare)	87½ hours	New TD membranes used (250) for neutral membranes. 123 new DK-1 and 127 used DK-1 used for cations. Mylar-film backed gaskets used.
May*	Stack E, (Stage 1)	30½ hours	TD membranes disintegrated and discarded. Stack dismantled and not rebuilt until 25-27 June. (Spare Stack D used to replace). 25 broken DK-1 discarded.
25-27 June	Stack E	59 hours	Stack built using old and repaired DA-2 and DK-1 membranes, plus 38 new DK-1 membranes, 48 used DK-1 and 8 DA-2 discarded from building stock because of holes and unreparable cracks.

*Stack built or rebuilt for experimental reasons (e.g. transport depletion or anion membrane poisoning film evaluation).

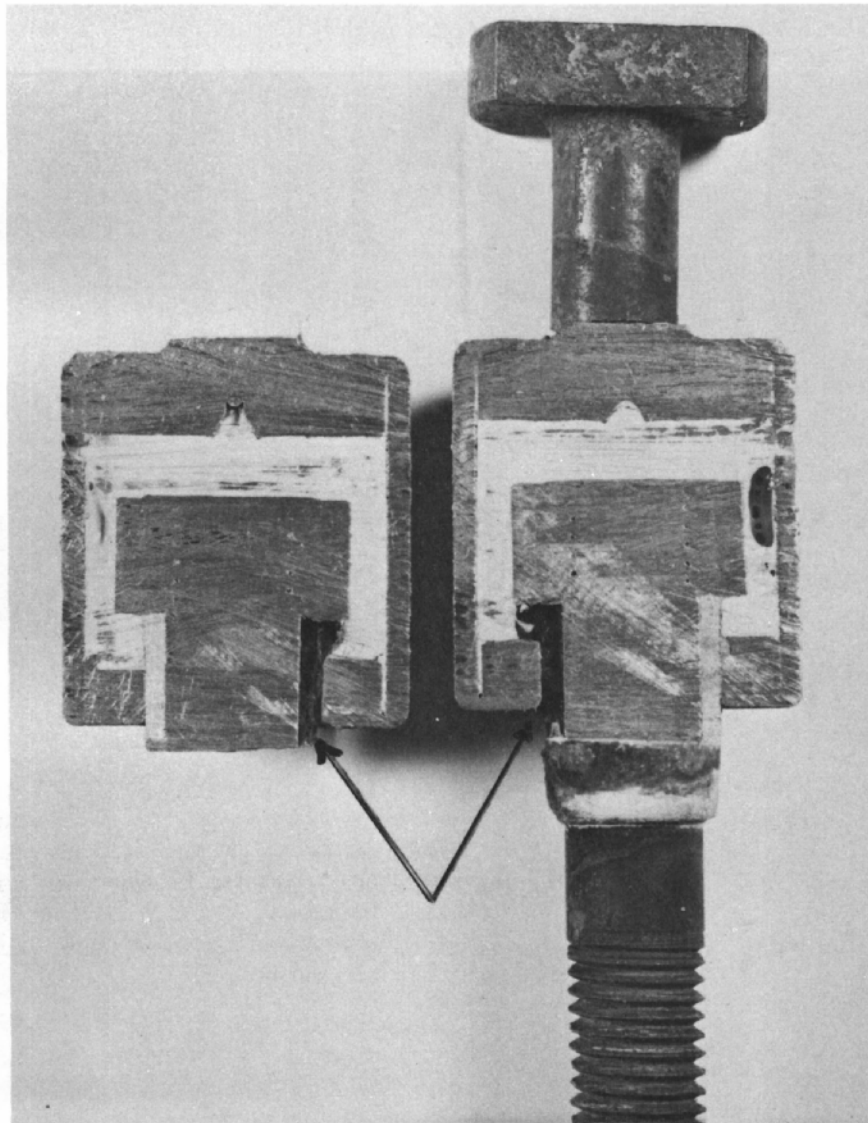


Figure 24

Shown above is a "burned out" turnbuckle insulator cut lengthwise for viewing. The "burn-out" is due to breakdown of the inert material, probably due to excessive voltage drop across the stack.

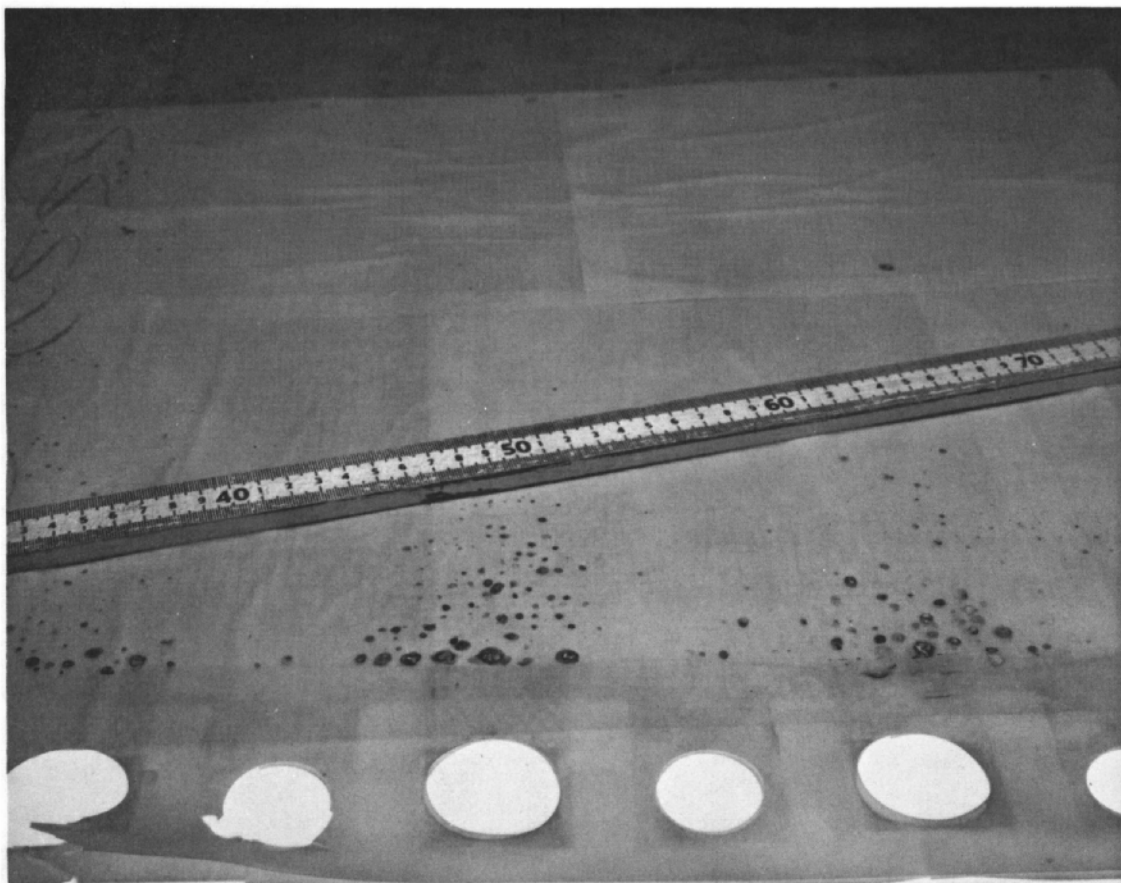


Figure 25

"Burned spots" were found on a few of the membranes upon dis-assembly of Stack B (12 - 21 December 1967) after a turnbuckle "shorted". The membrane shown is a DK-1 cation membrane; scale is seen in the center of the "burned spots". The holes at the bottom of the illustration are the manifold for the dilution and concentrate streams.

C. ANION MEMBRANE POSITIONING

1. Objective

The purpose of this study is to quantitatively describe anion membrane fouling at Webster especially with respect to "organic" fouling. The "organic" fouling of ACI DA-2 and ACI DA-1 anion membranes is compared.

2. History and Discussion

Operation of the Test Bed Plant and pilot plants suggested that the DA-2 anion membrane poisoned much more rapidly than the DA-1 membrane. However, no direct comparative test had yet been made between the two membranes. When the Test Bed Plant was placed on line in 1962, ACI DK-1 and DA-1 membranes were used; later ACI supplied DA-2 anion membranes and informed the Webster Test Facility that DA-1 membranes would no longer be available after 1967.

Anion membrane fouling manifests itself by a stack resistance rise and an apparent decrease in anion membrane limiting current density. Anion membrane poisoning due to "organics" may be distinguished from fouling due to iron floc or scale deposits by disassembly of the stack, cleaning the membranes, and noting the resistance recovery of the re-assembled stack; if the poisoning is entirely due to "organics", the stack resistance will not recover. Additional information may be found in the Fiscal Year 1967 Annual Report.

During late September 1967, Stack C was rebuilt using 125 new ACI DA-1 anion, 125 new ACI DA-2 anion membranes, and 250 new and used (better condition) ACI DK-1 cation membranes. The DA-1 and DA-2 stack halves were each probed so that the rate of increase of resistance between the two stack halves containing different anion membranes could be compared. Stack C was operated in first stage position, initially at 30 amperes (5 October 1967), which was allowed to decrease to 24 or 25 amperes by June 1968. Measurements were also taken on Stack C when it was in the fourth stage position.

Figure 26 shows the total (including electrode rinse streams) stack resistance of the combination Stack C. Initially, the stack resistance on 5 October 1967 was 6.5 ohm. From the period 6 October through 15 October 1967, there was a rapid rise in stack resistance to 11 ohms; thereafter, the resistance increased at a slower rate. By June 1968 (Figure 27),

the resistance of this stack was generally 27 to 30 ohms. The TDS of the dilution stream feed remained (within a few epm) nearly constant at 22 epm.

Figures 28 through 31 present stack energy consumption (repeating cell pair only, KWH/1,000 gallons) as a function of current for several different dates. The data show that during operation over extended periods, the electrical resistance increases, meaning increased energy consumption to achieve a given degree of desalination. Further experiments showed that the resistance buildup was due to the anion membrane only. Comparison of the DA-2 membrane and DA-1 membrane data show a much greater buildup in stack resistance for the DA-2 membrane. At 30 amperes, a first stage stack built of DA-2 membranes, which had been in service for nine months, would consume five to six times the electrical energy as a similar stack built of new DA-2 membranes; similarly, a stack built of nine month old DA-1 membranes would consume twice the electrical energy as a stack built of new DA-1 membranes.

Accompanying the increase in energy consumption, there is a decrease in anion membrane limiting current density. For the new stack, 22 epm total dissolved solids feed water, the limiting current was 40 to 45 amperes (4.0 to 4.5 ma/cm²), the same for both DA-1 and DA-2 membranes (determined by a Cowan plot of data taken on 5 October and 7 October 1967). By 4 June 1968, the DA-1 and DK-1 stack half limiting current had decreased to about 15 amperes, and the DA-2 and DK-1 stack half decreased to about 8 amperes. During normal operation of the stack at 25 to 30 amperes, there were substantial increases in free CO₂ in the dialysate and a significant increase in hydroxyl ions in the concentrate stream.

Rebuilding the stack on 6 December 1967 had only a slight effect on lowering the stack resistance, even though the membranes were washed to remove iron and scale accumulations, and the DA-1 and DA-2 membrane positions were interchanged. On 12 January 1968, the stack was again rebuilt, this time overturning the DA-1 and DA-2 membranes for the first time (so that the concentrate side now faces the dilution side); again there were only slight improvements in stack resistance and limiting current density (less than 25 percent of the values before rebuilding).

As it is impractical to operate below the anion membrane limiting current density, the stack currents are maintained at their high levels in order to achieve a reasonable product water salinity. To prevent massive alkaline scale formation on the anion

membrane concentrate side surface, the (1) concentrate stream pH is kept at a low value (pH = 3 to 4), (2) the stacks are acid backwashed (pH = 2.0) nightly, and (3) an hourly plotting of stack resistance is performed, so that the stacks can be rebuilt at the slightest hint of alkaline scale formation. During Fiscal Year 1965, these stop-gap procedures were not performed, and during this period there were massive membrane losses due to alkaline scale, especially on the DA-2 membranes.

3. Conclusions

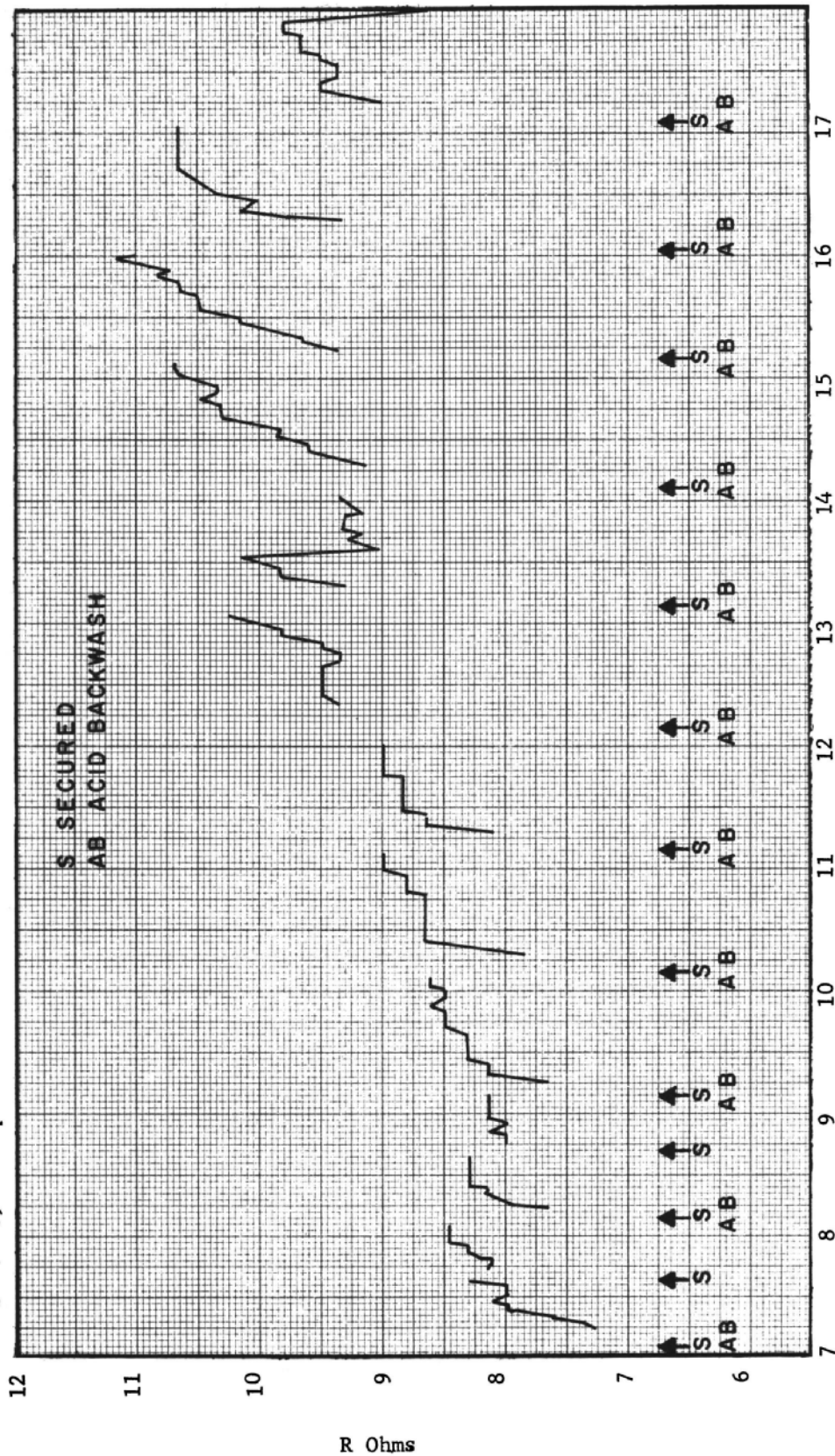
- a. Comparison of Figures 28, 29, 30, and 31 show that both DK-1, DA-1 and DK-1, DA-2 membrane combinations consume increasing amounts of electrical energy as their ages increase, comparing data taken at the same current density. The increase in electrical consumption is due to an increase in the DA-1 or DA-2 membrane resistance. The rate of resistance increase with respect to time is much greater for the DA-2, DK-1 membrane combination.
- b. There was only a slight recovery in stack resistance and apparent limiting current density (defined in Section II) upon rebuilding the stack and cleaning the membranes. The resistance data was independent of whether the DA-1 membranes were used at the anode half of the stack or at the cathode half of the stack, and independent of the DK-1 membranes and gaskets. This indicated a fouling or poisoning due to "organics" rather than from scale, iron accumulations, or poor flow distribution.
- c. Therefore, the DA-2 anion membrane fouls or poisons much more rapidly than the DA-1 anion membrane. At 30 amperes and in first stage position, this fouling rate of the DA-2 anion membrane is six to ten times that of a DA-1 anion membrane.

4. Recommendations

- a. This test should be continued into Fiscal Year 1969 in order to establish a useful membrane lifetime of the DA-2 membrane, that is, how high should membrane resistance be allowed to rise before further operation becomes impractical.
- b. Tests should be continued at the pilot plant level to look for anion membranes which are relatively resistant to fouling.
- c. No suitable membrane has yet been found to replace the ACI DA-2 anion membrane. Tests to replace this membrane should be given top priority.

Figure 26

First Stage ACI Stack Resistance
175 GPM Dilution, 100 GPM Concentrate
Stack C, 30 Amperes

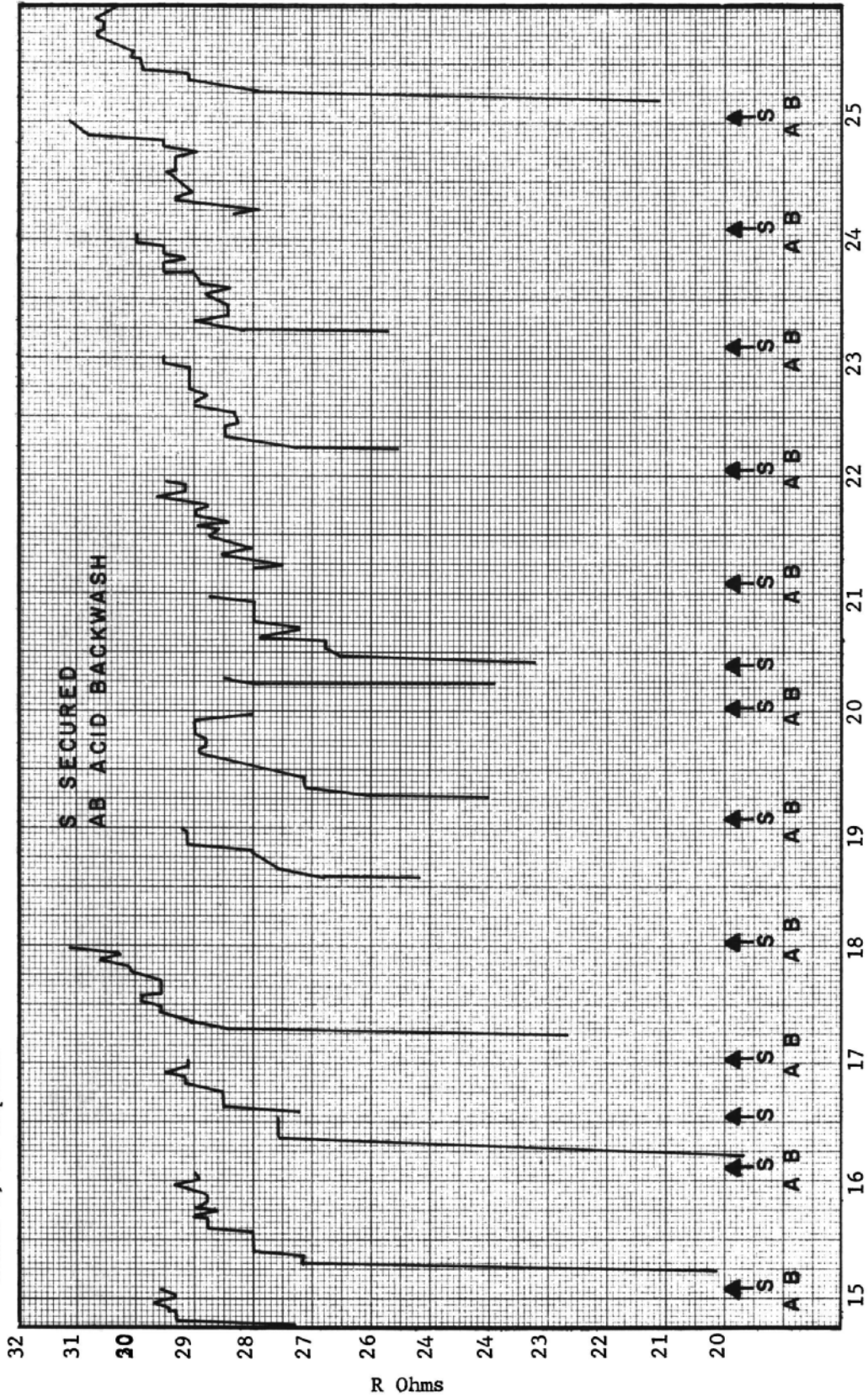


OCTOBER 1967

Webster, South Dakota

Figure 27

First Stage ACI Stack Resistance
200 GPM Dilution, 100 GPM Concentrate
Stack C, 25 Amperes



JUNE 1968

Webster, South Dakota

Figure 28

REPEATING CELL PAIR ENERGY CONSUMPTION IN STAGE ONE
DK-1 AND DA-1 MEMBRANES

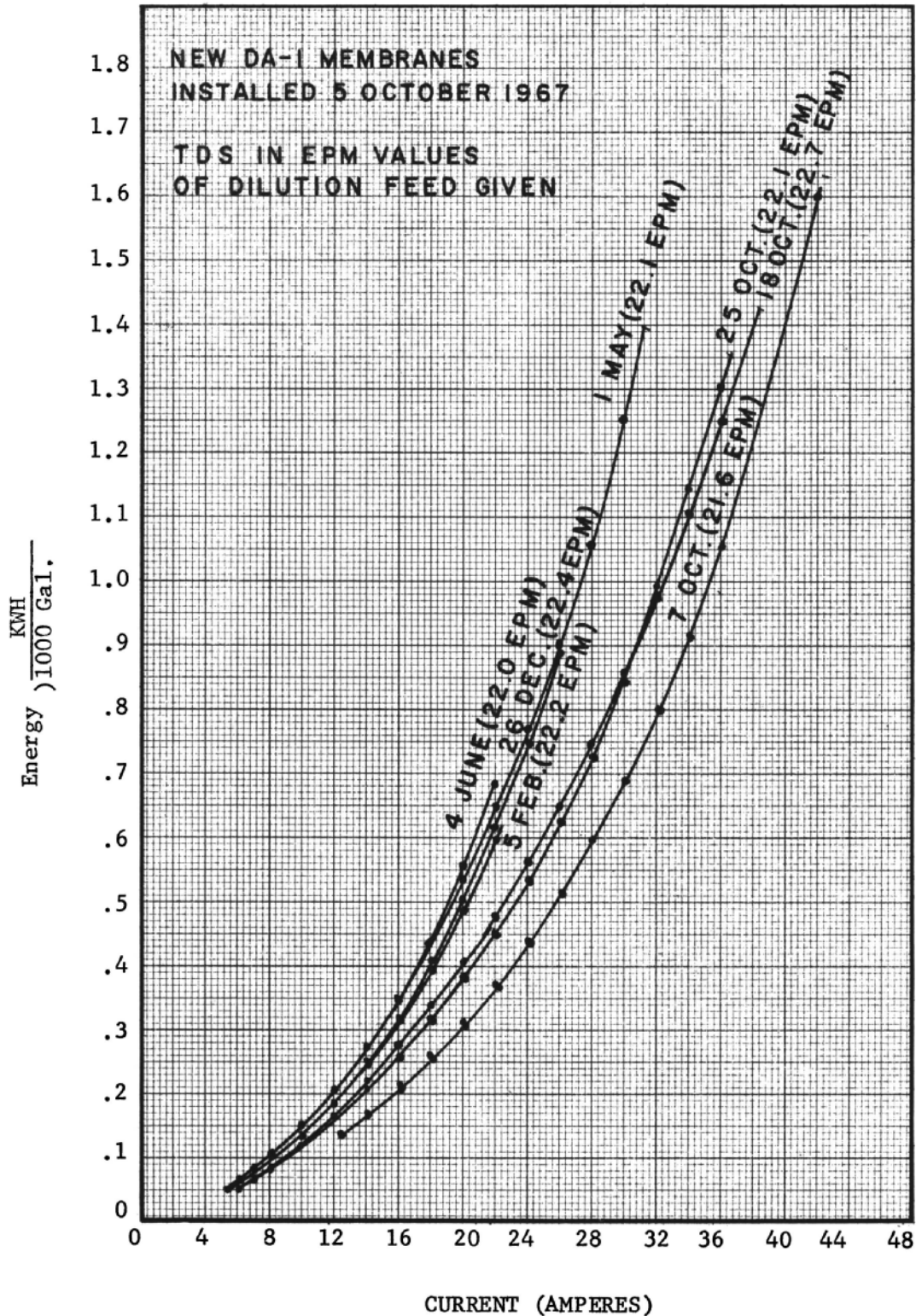


Figure 29

REPEATING CELL PAIR ENERGY CONSUMPTION IN STAGE ONE
DK-1 AND DA-2 MEMBRANES

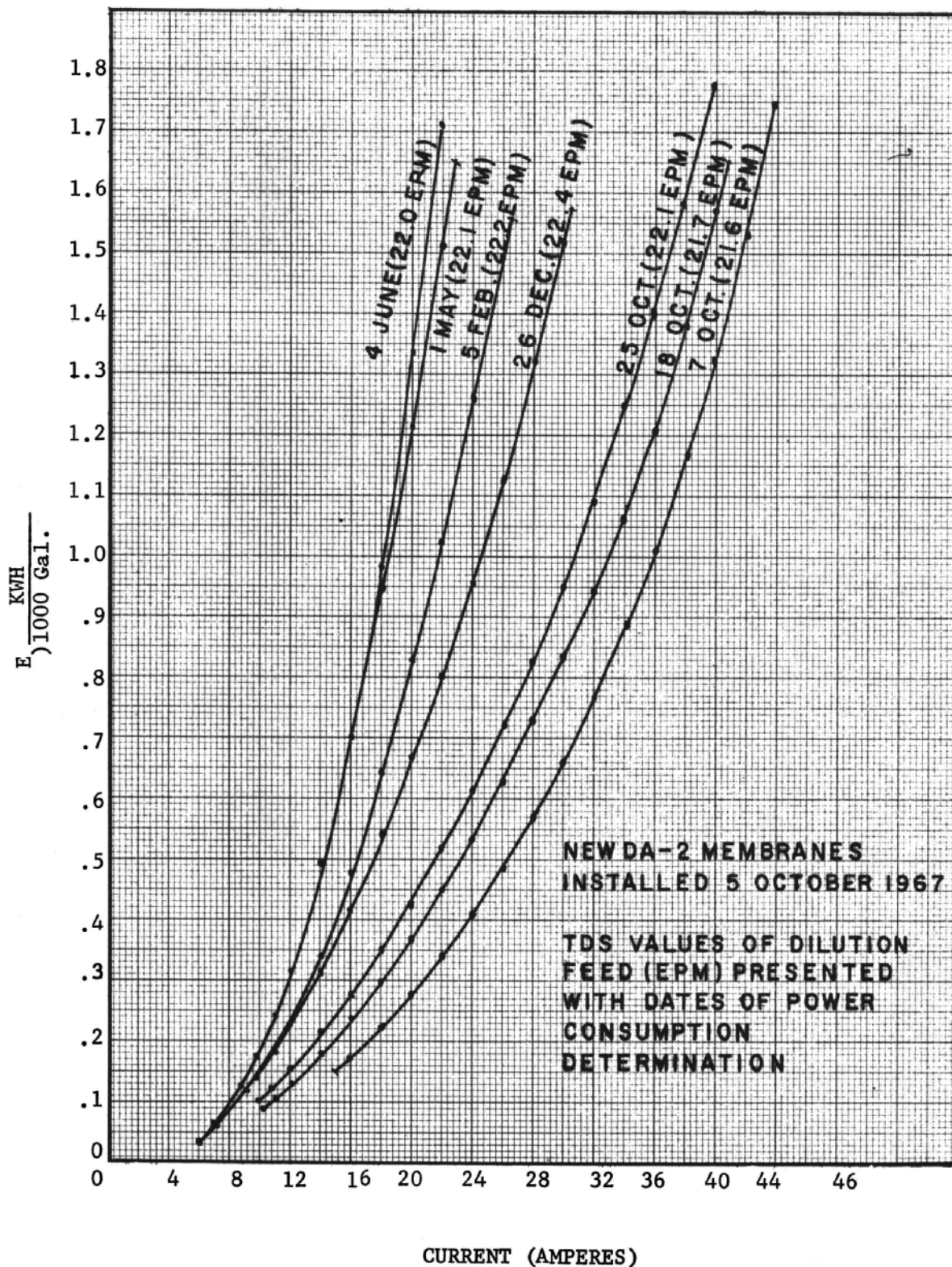


Figure 30

REPEATING CELL PAIR ENERGY CONSUMPTION IN STAGE FOUR
DK-1 AND DA-1 MEMBRANES

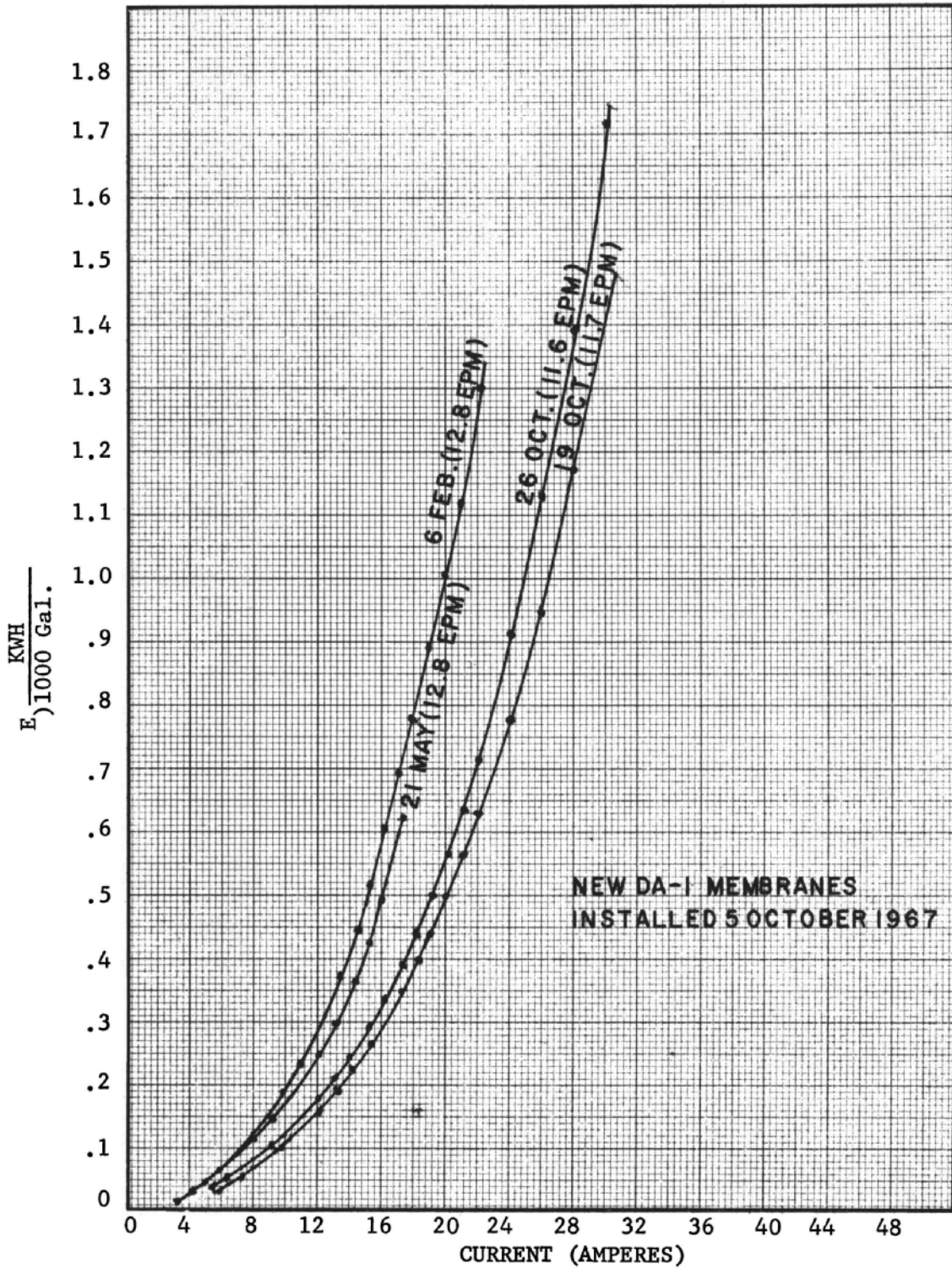
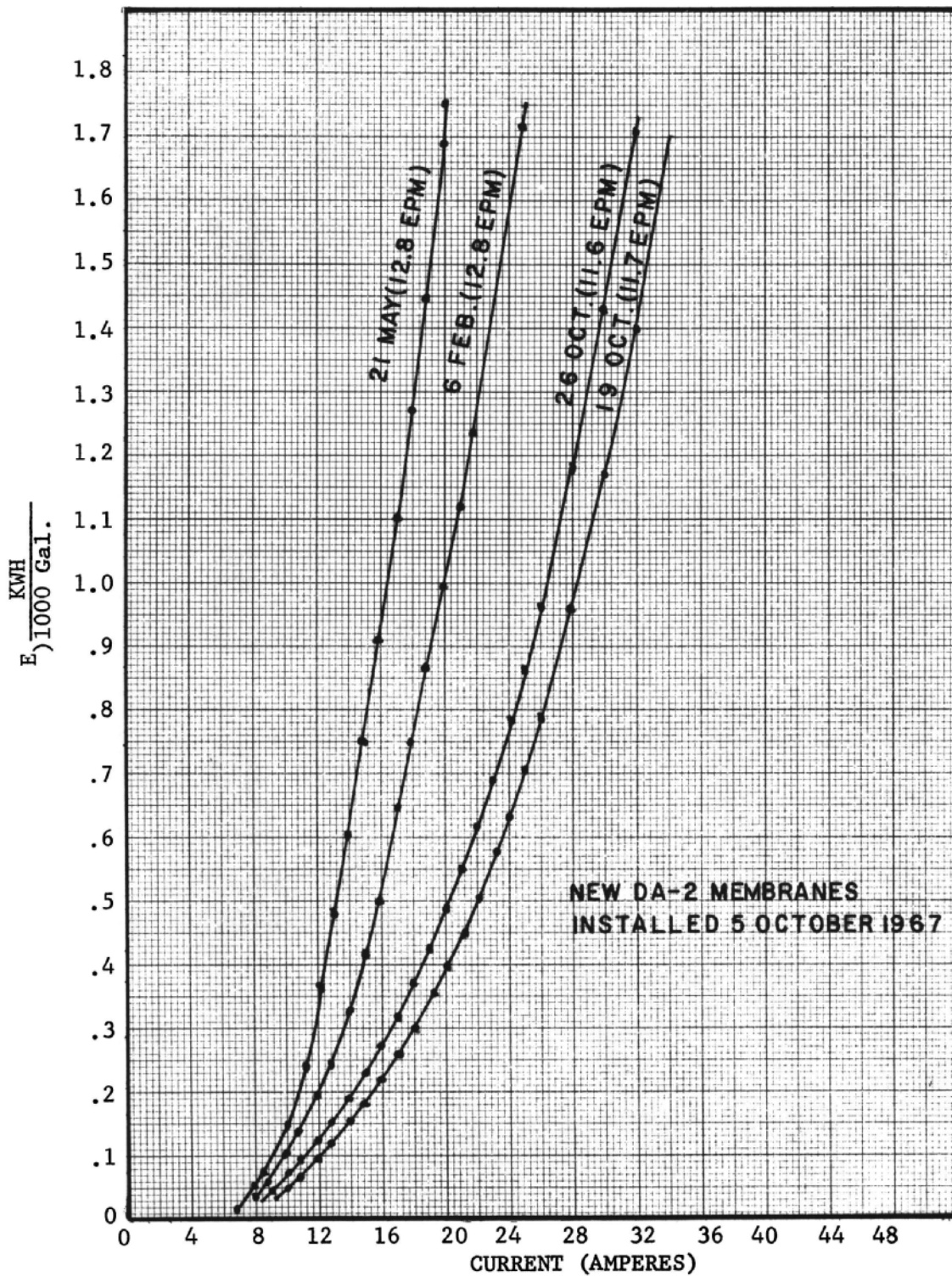


Figure 31

REPEATING CELL PAIR ENERGY CONSUMPTION IN STAGE FOUR
DK-1 AND DA-2 MEMBRANES



D. ACID INJECTION EVALUATION

1. Objective

The purpose is to define the benefits, if any, of injecting sulfuric acid into the pretreated water ahead of the electro-dialysis stacks.

2. History of Operation

During Fiscal Year 1967, the pretreatment system was modified as shown in Figure 2; the scheme allowed injection of sulfuric acid into the pretreated water after the Elgin Filters, and incorporated a detention tank (Tank 111) prior to the electro-dialysis stacks. Details are presented in the Fiscal Year 1967 Annual Report and in Section II.

The acid injection evaluation consisted of (1) comparison of electro-dialysis operation at 0 percent, 50 percent, and 87 percent bicarbonate removal levels (removal by injection of sulfuric acid with pretreatment aeration to remove 80 to 87 percent of the raw water bicarbonate) during July through September 1967, and (2) several one day experiments at various acid injection levels during the period October 1967 through mid-January 1968. In May 1968, the pretreatment system was again modified to eliminate the acid injection equipment so that the water from the Pump 02 (after $KMnO_4$ injection) was fed directly to the Elgin Filters and then to the electro-dialysis stacks.

Complete salt and ion material balances on each electro-dialysis stage were performed under the following conditions:

- (1) Acid injection to remove 80 percent of the bicarbonate, with process stream aeration; stack order 1st-C, 2nd-B, 3rd-A, 4th-D. Evaluation on 6, 7, 16, and 23 October 1967; 21 and 24 November 1967; 22 and 26 December 1967; 5 January 1968.
- (2) Acid injection to remove 70 percent of the bicarbonate, without being followed by aeration; stack order 1st-C, 2nd-B, 3rd-A, 4th-D. Evaluation on 12, 18, 25, and 31 October 1967.
- (3) Acid injection to remove 88 percent of the bicarbonate, pH of product water 4.0; stack order 1st-C, 2nd-B, 3rd-A, 4th-D. Evaluation on 24 November 1967.

- (4) No acid injection and no pretreatment aeration; stack order 1st-C, 2nd-B, 3rd-A, 4th-D. Evaluation on 6, 7, 16, 23 October 1967, 9 November 1967, 5 December 1967.
- (5) Same as (4) but stack order 1st-D, 2nd-B, 3rd-A, 4th-C. Evaluation on 19 and 26 October. The stack order was changed to study inherent differences of the particular stacks.

Air and water material balances were performed on the process stream aerator in January 1968 to determine CO₂ removal efficiency; the results agreed with design specifications, with nearly all of the CO₂ being removed.

3. Discussion of Results

Tables 6, 7, and 8 present sample water analyses at various points in the Test Bed Plant, with and without sulfuric acid injection in the pretreatment system. The location of the sampling positions is as follows (Figures 1, 3, and 4):

- raw: the well water entering the 5,700 gallon detention tank before pretreatment
- pretreated: the water (after pretreatment and aeration), just prior to entering the first stage
- 1 dil. out: the dilution water in the holding tank between the first and second stage
- product: the 4th dilution stage stream water after passing through the product water aerator and before final chlorination
- rec. conc.: the water entering the concentrate or brine compartments for each of the four stages. It is composed of (1) the concentrate water leaving the second, third, and fourth stages, (2) an amount of pretreatment water (roughly 100 GPM) equivalent to the waste water discarded, and (3) the cathode and feeding frame rinse streams leaving each of the four stages (10.5 GPM per stage, or 42.0 GPM for the four stages)

waste: the water discarded as waste, which consists of (1) the brine leaving the first stage concentrate compartments and (2) the anode rinse streams leaving each of the four stages (2.4 GPM per stage, 94 9.6 GPM for the four stages)

waste after aeration: The waste water after passing through the waste aerator

1 conc. out: the concentrate water leaving the first stage

elec. rinse: the water entering the anode and cathode rinse and anode feeding streams for all four stages. It is composed of 53 GPM circulating concentrate plus 0.06 to 0.08 GPM sulfuric acid.

The pretreated water is aerated by operating the decarbonator blower.

Inspection of Tables 7 and 8 show that there is some CO₂ formation in the dilution stream during electro dialysis. This is true regardless of whether or not sulfuric acid is injected as a part of pretreatment. The reason is concentration polarization at the anion membrane interface, especially severe for poisoned membranes, resulting in water splitting at and within the anion membrane. The hydrogen ions formed migrate to the dilution bulk stream, react with the bicarbonate, and form CO₂. The hydroxyl ions migrate to the concentrate stream. The excess acid content of the concentrate stream is always greater entering the stack than leaving the stack, indicating a transfer of OH⁻ and HCO₃⁻ to the concentrate stream (by "excess acid" is meant the epm of base that would be required to raise the water pH to 4.6). Comparison of the 12 October and 24 November 1967 data show that if sulfuric acid is injected to remove 82 percent of the raw water bicarbonate, the remaining bicarbonate in the dilution stream is sufficient to absorb hydrogen ions produced. At 88 percent bicarbonate removal, the remaining bicarbonate is sufficient to absorb the hydrogen ions, and the result is a drop in product water pH (to pH = 3.7).

Whenever the product water pH is greater than 4.5, (the bicarbonate end point) there will be a pH recovery after product water aeration (compare 4th stage dilution out data with product data) and a decrease in CO₂ content of the water. Since the pH of the waste water is generally around 3.0 or less, aeration of this stream is not absolutely necessary.

The 25 October 1967 data represent the case when the pretreated water is not aerated after pretreatment acid injection. The result is a high CO₂ content in the dilution stream. There is a decrease in CO₂ and an increase in pH after product water aeration.

Comparison of stack resistance data and limiting current density data taken during the October to December period showed no significant difference whatever if pretreatment acid injection is used, compared with usual operation, provided that the dilution pH > 4.6.

Salt material balances performed on each one of the stages (using the TDS, epm data) revealed that the concentrate brine flow as seen by the second and third stages was 130 ± 20 GPM rather than the 100 GPM indicated flow readings. This was confirmed by water meter readings. The trouble was traced to missing orifices within the second and third stage concentrate stream rotameters. Rather than change the Test Bed Plant conditions, the concentrate flow was kept at 130 GPM for stages two and three until May 1968. The concentrate stream on all stages was backpressured such that the concentrate compartment pressure equaled the dilution compartment pressure for the duration of the tests.

Table 9 presents ion removal selectivity data for the case of acid injection and pretreatment aeration. Inspection of the data shows a high degree of removal of bicarbonate ion, primarily due to anion membrane polarization and resultant hydrogen ion generation. Except for bicarbonate ion, there was no significant change in ion removal selectivity with or without acid injection.

During the period June through mid-September 1967, sulfuric acid was injected continuously in the main plant, to a level sufficient to remove roughly 70 to 80 percent of the raw water bicarbonate, roughly 0.5 pounds 60° Be sulfuric acid per minute per 275 GPM process water. Mason-Rust personnel were unable to detect any improvement in stack resistance or anion membrane poisoning tendency during the period, as compared with "normal operation". When the four stacks were rebuilt in August and September 1967, some alkaline (carbonate) scale was still found on the concentrate side surface of some of the anion membranes, about the same degree as found in stacks disassembled after operation on water containing 330 ppm bicarbonate.

The raw water bicarbonate ion content also varied (from about 5 to 6 epm) during this period. There were complaints from City residents of rusty water and odors during the June through September 1967 period.

No long term evaluations were performed at the 100 percent (or greater) bicarbonate removal level, as this would result in a product water pH less than 4, and would require addition of soda ash or caustic to the product water.

4. Conclusions

- a. Regarding the acid injection pretreatment system as a whole, Mason-Rust personnel were unable to discover any advantage to injecting sulfuric acid to remove raw water bicarbonate prior to electro dialysis (at least an advantage distinguishable in the data taken). Alkaline scale still formed in the electro dialysis stack when operated above the limiting current density, and the limiting current density was not changed upon addition of acid. The anion membrane still fouled during the acid injection. Approximately the same degree of calcium carbonate and magnesium hydroxide scale was seen on the anion membranes as after long term stack operation without acid injection.
- b. If there was any advantage to addition of the acid, membrane fouling problems including iron floc carryover from the pretreatment system and membrane breakage obscured these benefits.
- c. Corrosion problems increased, both in the plant and in the City water mains.

5. Recommendations

- a. The acid injection evaluation should be stopped and redundant pretreatment removed (Accomplished May 1968).
- b. Serious consideration should be given to possible corrosion of the City water distribution system from water processed by the planned lime softening plant, which would also reduce water alkalinity. The Test Bed Plant should be operated such that the product water pH is at least 7 with the lime softening system.

TABLE 6

SAMPLE WATER ANALYSES

Conditions: Sulfuric acid injected to remove 84 percent of raw water bicarbonate followed by pretreatment aeration.
 First stage (Stack C) 30 amperes, 285 volts, C.E. = 80 percent
 Second stage (Stack B) 22 amperes, 325 volts, C.E. = 83 percent
 Third stage (Stack A) 16 amperes, 310 volts, C.E. = 77 percent
 Fourth stage (Stack D) 10 amperes, 730 volts, C.E. = 47 percent
 12 October 1967, sample time 0830 hours, Water Temperature 48°F

	<u>Raw</u>	<u>Pretreatment</u>	<u>Product</u>	<u>Recirc. Conc.</u>	<u>Waste</u>
TDS, ppm	--	1584	588	2872	3552
TDS, epm	--	22.3	8.4	41.0	53.0
sp. resist, ohm-cm at 20°C	618	603	1383	268	262
bicarbonate, ppm	354	56	9	0	0
excess acid, epm	0	0	0	1.35	2.65
pH	7.5	6.9	6.1	3.1	2.8
free CO ₂ , ppm	43	15	7	--	--
chloride, ppm	13	13	4	26	35
sulfate, ppm	--	972	358	1720	2160
calcium, ppm	--	201	58	397	489
magnesium, ppm	--	86	32	158	209
hardness, ppm as CaCO ₃	--	856	274	1642	2084
sodium, ppm	--	65	34	98	126
potassium, ppm	--	7.9	2.8	13.7	18.2
silica, ppm	35	32	32	35	34
COD, ppm	8	8	8	--	--
iron, ppm	2.09	0.04	0.03	0.31	0.38
manganese, ppm	0.97	0.92	0.40	1.71	2.12

TABLE 7
Sample Water Analyses
19 October 1967 No Acid Injection or Pretreatment Aeration

	RAW	PRET	1 DIL OUT	2 DIL OUT	3 DIL OUT	4 DIL OUT	PROD CONC	RECIRC CONC	WASTE AERAT	WASTE AFTER AERAT	1 CONC OUT	2 CONC OUT	3 CONC OUT	4 CONC OUT	ELECT RINSE
TDS, epm	21.23	19.8	15.18	11.7	8.48	47.85	52.15	49.55	53.95	52.25	56.5				
HCO ₃ , epm	5.75	5.68	4.75	3.18	2.25	1.48	1.5	0	0	0	0	0	0	0	
Excess Acid, epm	0	0	0	0	0	0	2.75	4.7	4.7	1.05	.55	.55	.65		
free CO ₂ , ppm	33	14.3	16.5	25.3	27.5	31.9	19.8								
pH	7.6	7.9	7.8	7.6	7.2	7.2	7.6	2.9	2.7	2.6	3.35	3.65	3.5	4.4	1.7
Spec. Resist ohm-cm at 20°C	621	615	703	858	1057	1359	1326	286	247	216	302	286	292	275	75

25 October 1967 Acid Injection to Remove Most of Bicarbonate, No Pretreatment Aeration

	RAW	PRET	1 DIL OUT	2 DIL OUT	3 DIL OUT	4 DIL OUT	PROD CONC	RECIRC CONC	WASTE AERAT	WASTE AFTER AERAT	1 CONC OUT	2 CONC OUT	3 CONC OUT	4 CONC OUT	ELECT Rinse
TDS, epm	22.05	16.88	12.53	10.03	8.83	41.65	50.25	50.34	46.05	44.55	46.35				
HCO ₃ , epm	5.85	2.0	.7	.48	.28	.13	0	0	0	0	0	0	0	0	
Excess Acid, epm	0	0	0	0	0	0	1.55	2.6	2.6	1.2	1.3	1.4	.8		
free CO ₂ , ppm	31	32	37	40	47	48	12								
pH	7.2	6.4	6.2	6.0	5.6	5.0	5.8	3.1	3.0	3.0	3.2	3.2	3.2	3.45	2.0
Spec. Resist ohm-cm at 20°C	595	587	729	856	1058	1335	1126	319	260	275	294	296	307	307	166

TABLE 8

Sample Water Analyses

12 October 1967 Acid Injection to Remove 84 Percent of Bicarbonate Followed by Pretreatment Aeration

	RAW	PRET	1 DIL OUT	2 DIL OUT	3 DIL OUT	4 DIL OUT	PROD RECIR CONC	WASTE	WASTE AFTER AERAT	1 CONC OUT	2 CONC OUT	3 CONC OUT	4 CONC OUT	ELECT RINSE
TDS, epm	22.3	16.7	12.4	9.5	8.4	41.0	53.0	57.0	48.0	47.0	49.0			
HCO ₃ , epm	5.8	.925	.725	.5	.425	.1	.15	0	0	0	0	0	0	0
Excess Acid, epm	0	0	0	0	0	0	1.35	2.65	2.9	1.1	1.1	1.1	1.15	.6
free CO ₂ , ppm	43	15	18	24	27	7								
pH	7.5	6.9	6.6	6.5	6.3	5.4	6.1	3.1	2.8	2.8	3.4	3.3	3.2	3.6

! Spec. Resist
ohm-cm at 20°C
!

24 November 1967 Acid Injection to Remove 88 Percent of Bicarbonate Followed by Pretreatment Aeration

	RAW	PRET	1 DIL OUT	2 DIL OUT	3 DIL OUT	4 DIL OUT	PROD RECIR CONC	WASTE	WASTE AFTER AERAT	1 CONC OUT	2 CONC OUT	3 CONC OUT	4 CONC OUT	ELECT RINSE
TDS, epm	21.45	16.15	13.05	10.4	8.85	39.5	48.2	46.9	44.2	43.6	46.5			
HCO ₃ , ppm	6.20	.75	0	0	0	0	0	0	0	0	0	0	0	0
Excess Acid, epm	0	0	.4	.55	.55	.55	4.2	8.5	8.5	6.6	6.8	6.8	7.0	7.0
free CO ₂ , ppm														
pH	7.4	6.3	3.8	3.9	3.7	3.2	3.7	2.7	2.6	2.6	2.8	2.7	2.7	2.1
Spec. Resist ohm-cm at 20°C	590	538	538	756	868	979	962	208	193	187	209	212	215	225

!

TABLE 9

TEST BED PLANT ION REMOVAL SELECTIVITY

S = $\frac{\text{Fraction of ion removed in the four stages}}{\text{Fraction of all ions removed in the four stages}}$

No acid injection or pretreatment aeration With acid injection and pretreatment aeration

Ion	S (average)*	S (range)	S (average)**	S (range)
hardness (Ca ⁺⁺ & Mg ⁺⁺)	1.12	1.03 to 1.19	1.11	1.06 to 1.17
calcium	1.20	1.18 to 1.20	1.19	1.14 to 1.25
magnesium	1.07	0.98 to 1.13	1.01	0.89 to 1.09
manganese	***	***	1.23	0.88 to 1.54
potassium	0.73	0.49 to 0.99	0.94	0.79 to 1.04
sodium	0.60	0.57 to 0.64	0.67	0.52 to 0.77
sulfate	0.92	0.84 to 0.98	1.02	0.77 to 1.22
chloride	1.44	1.39 to 1.48	1.34	1.22 to 1.53
bicarbonate	1.32	1.22 to 1.43	1.58	1.40 to 1.98
silica	(-0.2)	(-1.0 to 0.5)	(-0.1)	(-0.2 to 0.1)

* average of data of 16 Oct., 23 Oct., 26 Oct., 5 Dec., 14 May, 14 June which range between values specified.

** average of data of 7 Sept., 12 Oct., 17 Oct., 24 Oct., 26 Dec. which range between values specified.

*** insufficient data, measurements taken on only two dates.

E. TRANSPORT DEPLETION EVALUATION

1. Objective

The purpose is to study long term performances of a 250 repeating cell pair ACI Test Bed Plsnt stack built of ACI DK-1 cation membranes and Union Carbide (UC Zephyr) regenerated cellulose "neutral" membranes.

2. History of Operation

After pilot plant tests were performed at the Denver Bureau of Reclamation, the Webster Test Facility, and at Southern Research Institute, a total of 937 Union Carbide Zephyr regenerated cellulose membranes (each 44 inch by 44 inch) were purchased for the Asahi Test Bed stacks at Webster Test Facility. Pilot plant tests at the Webster Test Facility demonstrated that (1) higher capacity rectification should be used (than the rectifiers currently in use) and (2) the concentrate stream should not be acidified (less tendency to form calcium sulfate scale) and (3) current efficiencies of about 35 percent can be expected using 22 epm manganese Zeolite Webster water. The pilot plant tests lasted one month at Webster.

Two 150 ampere, 1500 volt rectifiers, were purchased for this investigation and possible future use in high temperature electrodialysis evaluation. The transport depletion evaluation program called for isolation of one stage of the Test Bed Plant (with its own concentrate stream, rather than using the acidified recirculating concentrate water of the main plant). Use of a new "neutral" regenerated cellulose membrane instead of the usual DA-1 or DA-2 anion membrane permits operation at much greater currents without anion membrane polarization or anion membrane poisoning.

A transport depletion stack (250 DK-1, 250 UC Zephyr, ACI gaskets and separators, referred to as Stack "D") was built during early January 1968 and placed in the fourth stage position on 17 January 1968. Since the high capacity rectifiers and materials for isolation of the TD stage had not yet arrived, Stack "D" was operated as an "ordinary" stack with the concentrate stream acidified. Voltages and current efficiencies were determined on 18 January 1968 at various current levels (C.E. varied from 14 percent to 28 percent) up to 30 amperes, with repetitious cocurrent feeding of the dilution and concentrate streams. On 19 January 1968, Stack "D" was operated in the

third stage and salt material balances indicated severe leakage between the dilution and concentrate streams. Dismantling of Stack "D" revealed 33 cracked DK-1 cation membranes. These membranes were cracked along the gasket edge, which would allow intermixing of the dilution and concentrate streams. The severe cracking was believed caused by an abnormal pressure drop across the cation membranes, especially severe as the third stage was operated under counter current feeding of the dilution and concentrate streams. The cracked membranes were replaced.

The rebuilt transport depletion Stack "D" was placed in the first stage (cocurrent feeding) position on 22 January 1968, and operated in this position at 35 amperes from 24 January through 14 March 1968 (except on 6 February 1968 when comparative measurements were taken in the fourth stage position). Salt material balances performed on 30 and 31 January again suggested leakage between the dilution and concentrate streams. Stack leakage tests where the concentrate compartments were filled with water with no incoming dilution water showed water leaving the dilution exit, indicating leakage. Stack disassembly on 1 February 1968 again revealed 104 broken cation membranes at random locations in the stack. These were replaced. Calcium sulfate scale was found on some of the cation membranes. The Union Carbide Zephyr membranes appeared intact. Measurements in a special fabricated test cell showed only a negligible amount of water transported through the Zephyr membrane. The rebuilt stack was operated in the fourth stage on 6 February 1968 and in the first stage from 7 February through 14 March 1968. Current efficiencies, measured daily, average 33 percent. Stack voltages ranged from 380 to 405 volts total (RCP = 300 to 335 volts), which did not significantly increase or decrease during the test period.

On 13 March 1968, salt material balances showed severe leakage between the dilution and concentrate streams; this was confirmed by stack leakage tests. Stack disassembly showed deterioration of all the 250 regenerated cellulose neutral membranes. Deterioration was most severe at the gasket edges as shown in Figures 32 and 33. The deteriorated areas were "slimy" to the touch. Microscopic examination showed fungus-like areas (Figure 34) on the surface; the membrane under this area was structurally weak. In addition, 108 DK-1 cation membranes were broken and some calcium sulfate scale was seen on most of the cation membranes.

Samples of deteriorated membranes were sent to Union Carbide (Food Products Division, Chicago) and Aerojet-General for examination. Union Carbide found the "attacked" areas to be rich in iron but were unable to identify any microorganism that could be responsible for the deterioration. They postulated that the membrane depolymerized due to iron attack. Stack disassembly did reveal substantial iron floc accumulations adhering to all stack components. On the other hand, Aerojet-General was able to culture a number of cellulose attacking microorganisms from the membrane and indicated microbial attack as being responsible for the deterioration.

Another transport depletion stack was built from a fresh stock of membranes and designated as Stack "E". The high capacity rectifiers had been installed and isolation of the first stage was completed as discussed under Part III-C-5d. Stack "E" was operated in first stage position from 4 April through 3 May at 50 amperes, with the concentrate stream not acidified. Special experiments performed included measurements of repeating cell pair and total voltages, dilution stream current efficiencies, water analyses for different ions, and electrical heating effects as functions of current. Data was collected for 43 epm, 22 epm, and 12 epm TDS salinity feed waters. To achieve the 43 epm TDS water, sodium chloride was injected into the pretreated water for a day. To study the 12 epm TDS water, the stack was evaluated in fourth stage position for a day.

On 3 May 1968, stack leakage tests demonstrated severe bulk water flow between the dilution and concentrate compartments. The stack was dismantled on 3 May 1968. It was observed that the entire 250 regenerated cellulose membranes had disintegrated. During the period the stack was being dismantled, the regenerated cellulose membranes lost their integrity as a 44 inch by 44 inch sheet and became like "jelly". The degradation was very rapid. As of 2 May 1968, stack current efficiencies gave no evidence of trouble. Most of the cation membranes had some calcium sulfate scale on the concentrate side surface, and a number were broken at the gasket edges. There were also substantial quantities of iron floc material adhering to all stack components, which was expected as the stack had not been acid backwashed.

A membrane sample was sent to Dr. Middaul, South Dakota State University bacteriology laboratory on 3 May 1968. Microscopic examination revealed significant quantities of yeast, but fungus growths were not seen.

A small Mason-Rust fabricated pilot stack containing 10 pairs of DK-1 cation and new regenerated cellulose membranes was placed on line about 15 May 1968 using manganese Zeolite pre-treated Webster water. No current was applied to the stack. Examination of the membranes two months later showed that the regenerated cellulose membranes had deteriorated, especially at the gasket edges and under the ping-pong slots of the gasket. The deterioration was similar to that observed in the Test Bed Plant.

Another batch of regenerated cellulose membranes, which had been stored in Webster water under anaerobic conditions (i.e. "packaged") since July 1967, were examined in May 1968. There was no evidence of membrane deterioration. A characteristic hydrogen sulfide odor was noted when the stored membrane pack was opened.

Fifteen 250 ml capacity E flasks each containing 100 mls of Webster water and pieces of new regenerated cellulose membranes were sterilized to kill any possible microorganisms. Ten of the flasks also contained iron in the form of ferric hydroxide flocs from the backwash water of the manganese Zeolite filters. Five of the flasks containing iron and three of the flasks with no iron were inoculated with disintegrated cellulose membranes from the Test Bed Stack dismantled in May 1968. The flasks were incubated in the dark at 48^oF. After two months incubation, the flasks were opened. The membranes in the inoculated flasks were structurally weaker (as measured in a Mason-Rust fabricated test cell which compares the forces required to tear 0.5 inch membrane strips) than those contained in the flasks which were not inoculated with disintegrated cellulose membranes. Addition of iron (either to the inoculated or uninoculated flasks) did not appear to have any measureable effect. This evidence suggests that microbial attack rather than iron attack was responsible for the disintegration.

3. Transport Depletion Operation Data

Figures 35 and 36 and Table 10 present sample operational data of the transport depletion Stack "E" which was on line from 3 April through 3 May 1968. Figure 35 displays hourly stack resistance data for the period 15 - 26 April 1968 (other periods are similar) for the stack operating at 50 amperes. The transport depletion stack was secured for a few hours each night while the conventional electrodialysis stages were undergoing acid backwashing, but was not acid backwashed itself. Comparison of Figure 35 with Figures 18 and 19 shows that the transport depletion stack is not subject to the erratic daily resistance rises due to anion membrane polarization.

Figure 36 compares transport depletion stack energy consumption and current efficiencies for three different dilution feed water salinities. As expected, the energy consumption at a given operating current is less for the higher TDS feed water salinities since the specific resistance of these waters is less. Current efficiencies were also greater in the case of the higher TDS water. This fact was also reported by Southern Research Institute, which found that the Union Carbide Zephyr membrane became more cation selective in lower salinity waters.

Table 10 shows the ion removal selectivity obtained for transport depletion operation. The most noticeable difference between Table 10 and similar data for electrodialysis operation in Table 9 is that the iron removal selectivity for bicarbonate is much less for transport depletion; also, sodium is higher. Bicarbonate ion removal is high when ACI DA-2 anion membranes are used because of water breakdown at the anion membrane surface which consumes some of the bicarbonate ions.

4. Conclusions

- a. The Union Carbide Zephyr regenerated cellulose membranes lasted only five weeks in the Test Bed Plant. The exact cause of the cellulose membrane deterioration is inconclusive. There is more evidence that a microorganism or organisms are involved as opposed to only a simple chemical attack or physical degradation. A combination of factors, i.e., both microorganisms and iron attack in the presence of oxygen, is a strong possibility.
- b. The regenerated cellulose membranes were also incompatible with the ACI DK-1 cation membranes as indicated by repeated cation membrane breakages in spite of all efforts to control pressures (at least for the flow rates used).
- c. Transport depletion stack operational data agreed with the earlier pilot plant tests at Webster.

5. Recommendations

- a. The study shows the need for development of more rigid membranes for transport depletion resistant to microbial attack.
- b. The regenerated cellulose membrane tested was slightly cation selective and not strictly "neutral". The development of neutral or slightly anion selective membranes,

especially for low salinity waters, will allow higher current efficiencies. A reasonable salt cut could be obtained at a low current efficiency by operation of the stack at a higher current density, but power consumption is more and calcium sulfate precipitation on the cation membrane surface from concentration polarization is greater.

- c. The further development of pilot plant tests is recommended before trial in the Test Bed Plant for future programs. For example, membranes should be tested for 2,000 hours in pilot plants before use in the Test Bed Plant.

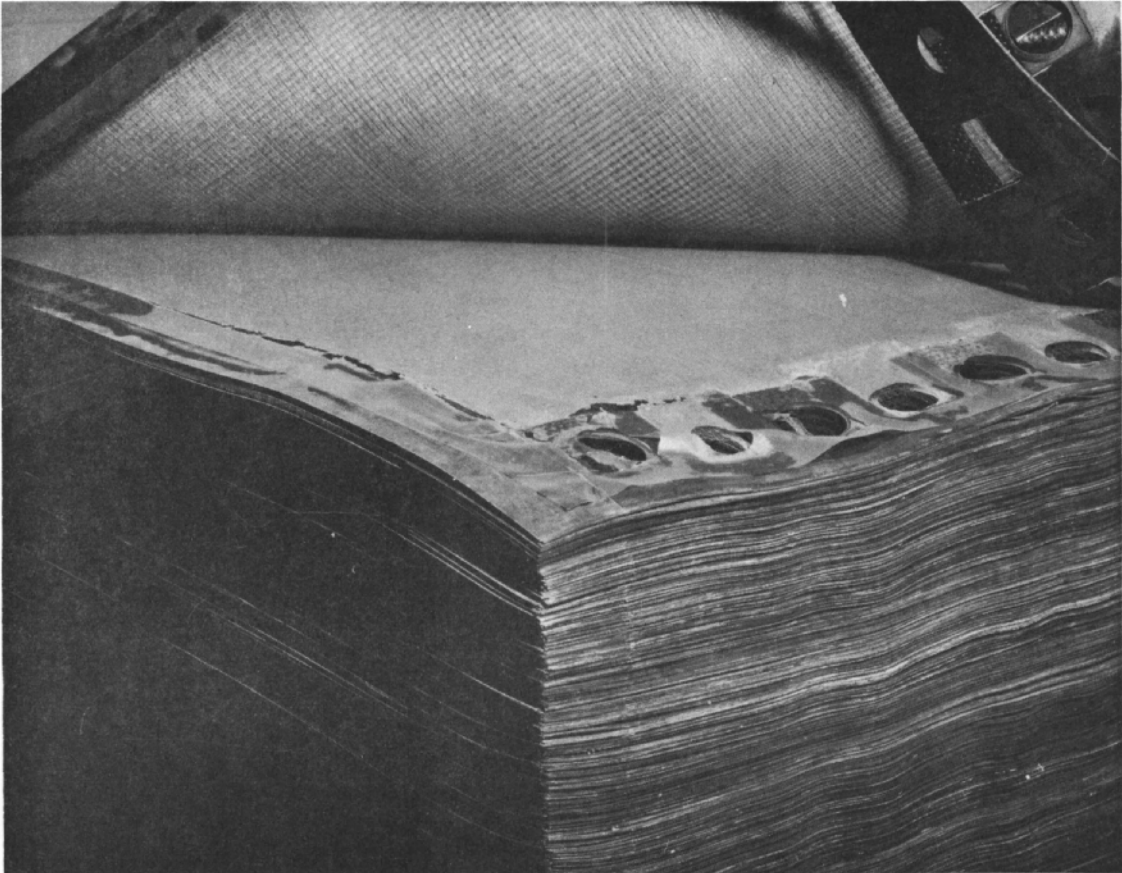


Figure 32

Deterioration of the Union Carbide Zephyr regenerated cellulose membranes is evident, especially at the edges of the rubber gaskets, after 1 1/2 months operation of the stacks as a transport depletion unit. A dilution compartment is shown. March 1968.



Figure 33

The deterioration of this Union Carbide Zephyr membrane is especially severe where the membrane is in contact with the slotted rubber gasket. All 250 Zephyr membranes were similarly affected, after 1 1/2 months operation in a TDS stack. March 1968.

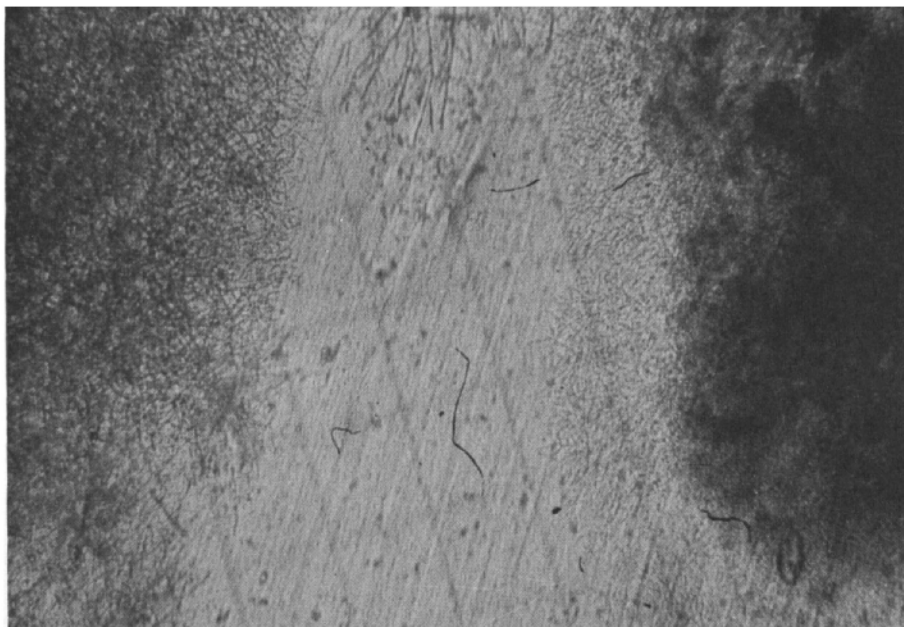


Figure 34

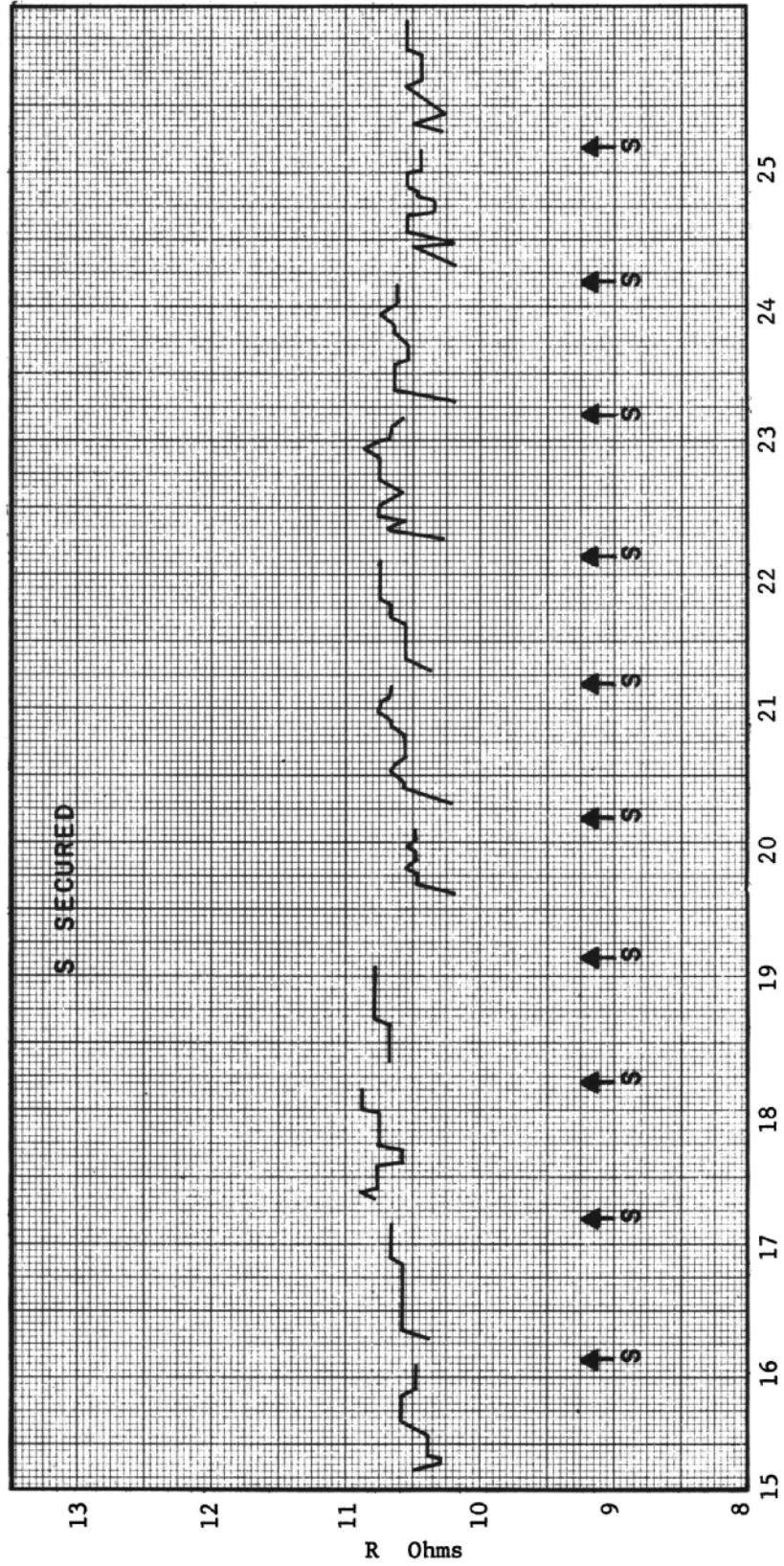
This microscope photo shows deteriorated areas of a Union Carbide membrane surface, left and right ends of photo, concentrate side, after 1 1/2 months of TD operation.

Scale: 1 cm = 0.123 mm (81X)

Figure 35

TRANSPORT DEPLETION STACK RESISTANCE DATA

First Stage ACI Stack Resistance
175 GPM Dilution, 100 GPM Concentrate
Stack "E", 50 Amperes



APRIL 1968
Webster, South Dakota

TRANSPORT DEPLETION STACK CURRENT EFFICIENCY AND ENERGY CONSUMPTION
FOR THREE DIFFERENT SALINITY FEED WATERS

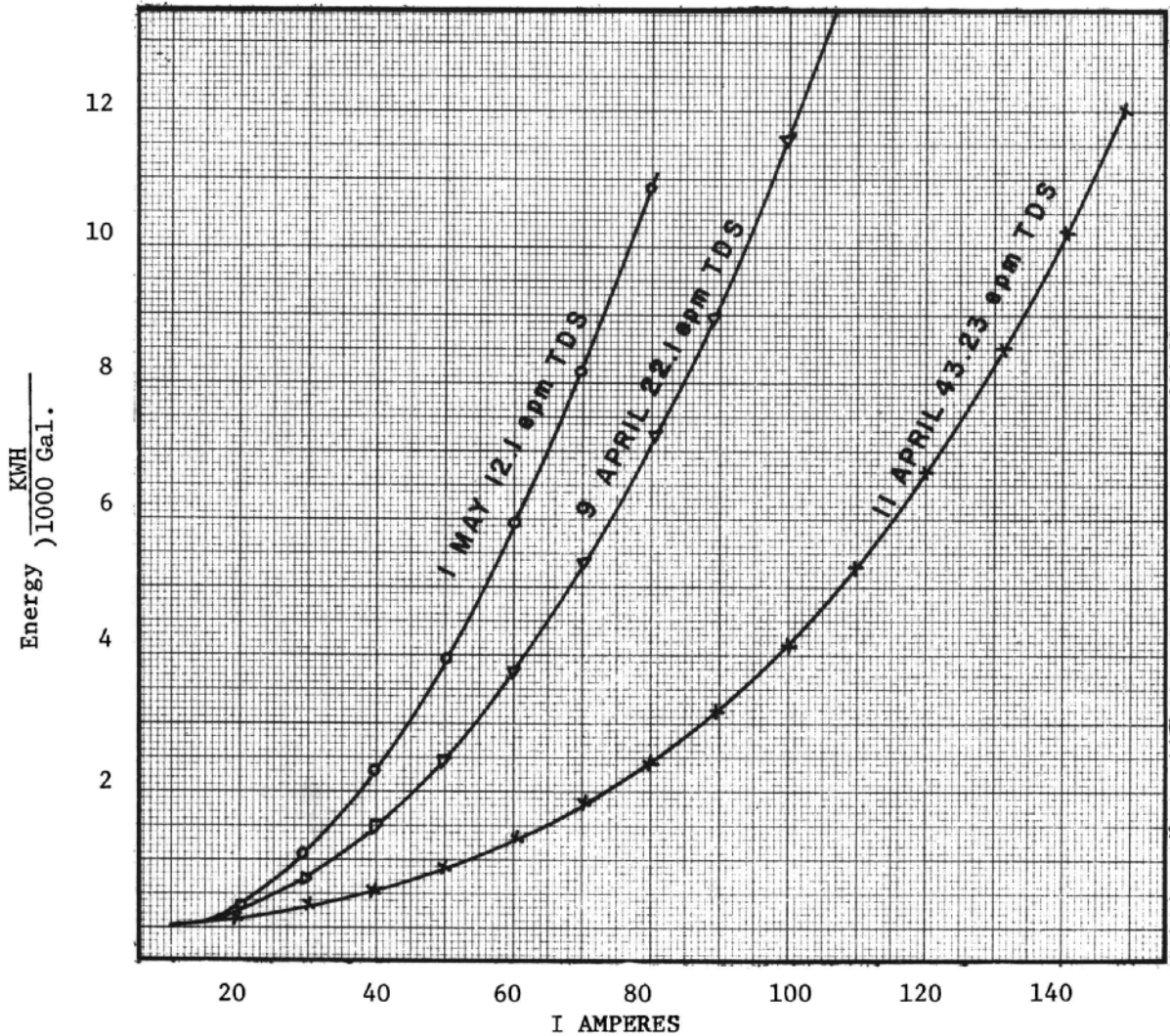
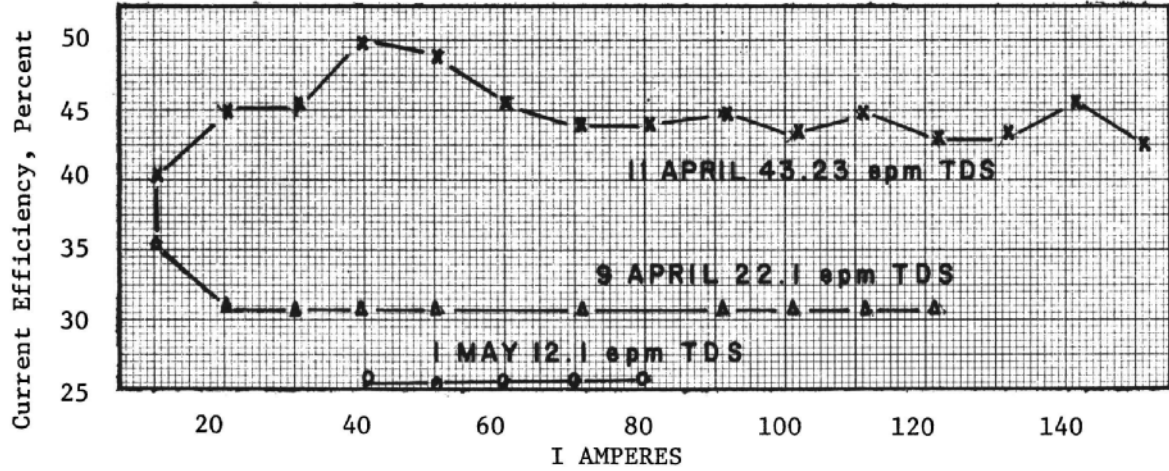


TABLE 10

ION REMOVAL SELECTIVITY

$$\text{Ion removal Selectivity} = \frac{\text{Fraction of Ion Removed in Stack "E"}}{\text{Fraction of all ions removed in Stack "E"}}$$

Transport Depletion stack at 120 amperes, 22 epm feed

	<u>Average*</u>	<u>Range</u>
bicarbonate	0.91	0.77 to 1.03
sulfate	0.98	0.88 to 1.12
chloride	1.44	1.25 to 1.93
calcium	0.97	0.84 to 1.28
magnesium	1.01	0.31 to 1.75
sodium	1.05	0.85 to 1.18
potassium	0.84	0.69 to 0.93
manganese	1.01	0.64 to 1.25
silica	0.07	0.08 to 0.34

* Average of six values taken during April, which range within the values specified.

SECTION V. PILOT PLANT EVALUATION

A. GENERAL

Pilot plant evaluation performed at the Webster Test Facility during the period 1 July 1967 through 30 June 1968 included (1) operation of the 10,000 GPD Aerojet-General Reverse Osmosis Unit, (2) evaluation of lime softening as a pretreatment procedure prior to electro dialysis, (3) high temperature electro dialysis studies, and (4) membrane evaluation including operation of an Ionac experimental anion membrane. The time schedule for these evaluations is presented in Figure 37. In addition, the DuPont Company used the Webster Facilities in September 1967 to test their reverse osmosis pilot plant on Webster water. In August 1967, Elgin, Inc. field tested their Sul-Bi-Sul Ion Exchange Process.

As the data and technical evaluations are published elsewhere, this section will not attempt to discuss the details. The data and evaluations are submitted to the Office of Saline Water in the form of Webster Engineering and Developmental Reports (WEDR). The reports submitted this report period are abstracted in the Appendix.

B. PILOT PLANT EVALUATIONS

1. Aerojet-General 10,000 GPD Pilot Plant

The Aerojet-General Corporation Model 1-5603-2 10,000 GPD Plate and Frame reverse osmosis pilot plant was operated continuously on diatomaceous earth pretreated (to remove iron and manganese) Webster water during the period 1 February through 4 September 1967. Mason-Rust operation of the unit during the period 7 July through 4 September 1967 is described in Webster Engineering and Developmental Report No. 30, which has been reissued as a part of OSW Research and Development Progress Report No. 427. After operation at Webster, the unit was shipped to the Denver Bureau of Reclamation. Figure 38 depicts membranes and other module components upon disassembly after testing at Webster.

2. Lime Softening Pretreatment Prior to Electro dialysis

During the period July 1967 through December 1967, Mason-Rust personnel operated the Asahi Chemical Industry SV-3 Acilyzer on lime softened, carbon filtered Webster water. Included in this operation is a study of the conditions of calcium

sulfate scaling on the cation membrane interface. The results were submitted to the Office of Saline Water in Webster Engineering and Developmental Report No. 32.

3. High Temperature Electrodialysis

Operation of an Ionics Stackpack pilot plant on different candidate high temperature electrodialysis membranes was accomplished at temperatures ranging from 50°F to 140°F on lime softened Webster water. The results have been reported to the Office of Saline Water as Webster Engineering and Developmental Reports No. 31 and 33. Report No. 31 has been re-issued as Office of Saline Water Research and Development Progress Report No. 342.

Different membranes were compared in the Asahi Chemical Industry SV-3 Acilyzer at temperatures of 50°F through 140°F. The study included economics of high temperature electrodialysis up to 10,000,000 GPD production plants. The results are reported as Webster Engineering and Developmental Report No. 34.

During June 1968, and continuing into Fiscal Year 1969, a study was performed on the Aqua-Chem WD 6-2 electrodialysis unit using Asahi Glass membranes.

All high temperature work was performed using lime softened and carbon filtered Webster Well No. 6 water.

4. Membrane Evaluation

a. Union Carbide Zephyr Membranes

Support laboratory evaluations were performed in conjunction with use of the Union Carbide Zephyr regenerated cellulose membranes in the production plant.

- b. A study of Ionics Anion Membrane fouling on Webster manganese Zeolite filtered water (iron and manganese removal), 48°F, was begun in May 1968 and will be continued into Fiscal Year 1969. The Ionics Stackpack was utilized. Included in the study were a comparison of methods of regenerating poisoned anion membranes.

c. Ionac Modified Anion - Neutral Membranes

A program was started in May 1968 of testing at Webster heterogeneous "anion-neutral" membranes developed by Ionac under OSW Contract No. 01-0001-1258. The goal of the contract was to develop slightly anion selective membranes relatively resistant to "organic" fouling to be used in electrodialysis or transport depletion. The tests continued into Fiscal Year 1969.

FIGURE 37

PILOT PLANT EVALUATIONS

AT WEBSTER TEST FACILITY

PROCESS DEVELOPMENT PROGRAMS

FISCAL YEAR 1968

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
Reverse Osmosis	<p>Aerojet-General 10,000 GPD</p>											
Lime Softening Pretreatment with carbon filtration	<p>ACI Acilyzer, Aqua-Chem WD6-2, Ionics Stackpack</p> <p>CaSO₄ Scaling Study on DK-1 Membranes in ACI Acilyzer</p>											
High Temperature Electrolysis	<p>Preliminary Experiments</p> <p>Ionics: Stackpack 48°F to 140°F</p> <p>ACI Acilyzer</p> <p>Various Membranes 48°F to 140°F</p> <p>Stackpack Asahi Glass</p> <p>10K GPD</p> <p>Aqua-Chem</p> <p>Asahi Glass to 140°F</p> <p>Various Laboratory Tests</p>											
Membrane Evaluation	<p>Various Membranes ambient conditions</p>											

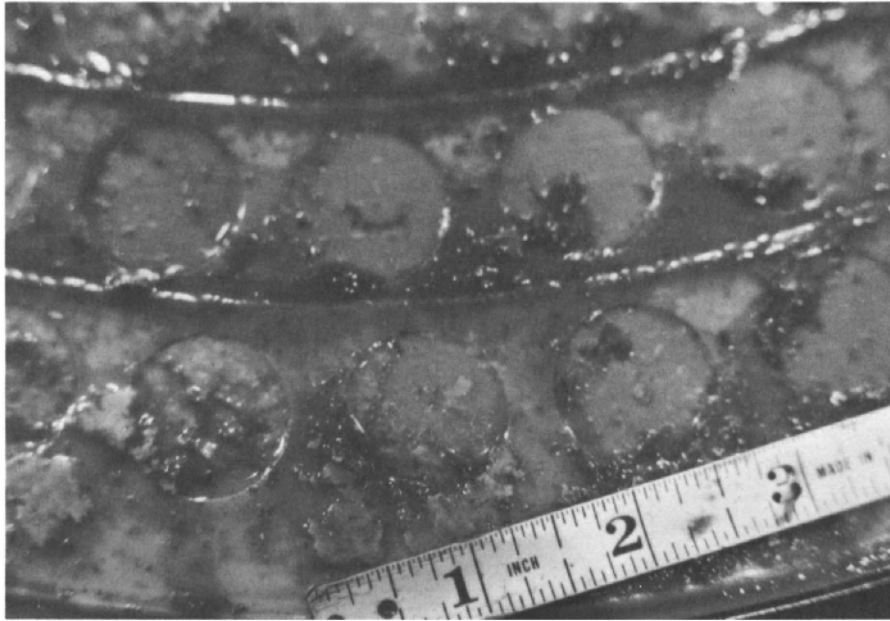


Figure 38

The Aerojet-General Reverse Osmosis pilot plant was dismantled at the Denver Bureau of Reclamation after 9 months of operation on Webster diatomaceous earth pretreated water. Top photo shows brownish (black in photo) iron flocs loosely clinging to spiral baffle and cellulose acetate membrane. Areas of CaSO_4 scale formation are seen in the photo at the outermost (5th, 6th, and 7th) spiral baffle ridge. Bottom photo shows compression of membrane into network grooves of support plate. The dark 1/2 inch strip under numeral "10" is the Mylar tape used to splice membranes. The cellulose acetate membranes were unstained.

SECTION VI. WEBSTER TEST FACILITY COSTS

A. GENERAL

This section of the report includes a summary and listing of the capital investment at the Webster Test Facility (Tables 11 and 12), ledger summaries for the report period (Table 15), and a set of normalized Test Bed Plant costs (Table 13).

B. INVESTMENT COSTS, WEBSTER TEST FACILITY

Table No. 11 lists the fixed assets at the Webster Test Facility as of 30 June 1968. The total capital investment as of that date is \$895,632.39. This figure represents the total capital investment of the Webster Test Facility which included items of experimental or research nature used to carry out various technical programs and support services for pilot plants.

TABLE 11
TOTAL WEBSTER TEST FACILITY INVESTMENT COSTS
WEBSTER, SOUTH DAKOTA
AS OF 30 JUNE 1968

ACCOUNT NO.

100.1	Lands	4,824.83
100.3	Roads & Walks	1,674.00
100.4	Buildings	173,287.87
100.5	Process Plant	551,183.27
100.6	Other Structures	682.00
100.7	Grounds & Other Improvements	762.00
100.8	Experimental Plant Facilities	28,677.00
100.9	Plant Not in Operation	4,019.00
100.15	Scientific Exhibits	1,275.00
100.16	Moveable Equipment	19,423.22
100.17	Pretreatment Research Equipment	97,462.10
100.18	Reverse Osmosis	<u>12,362.10</u>
		<u>\$895,632.39</u>

C. CAPITAL COSTS OF WEBSTER TEST BED PLANT

Beginning 1 July 1964, Webster Test Facility capital investment and depreciation was changed to comply with OSW instructions received in May 1964. The change reflected the updating and improvement of a uniform reporting method.

The important modifications were the deletion of these items which were classified either as not in operation or totally experimental from the depreciable plant and also altering unit equipment life to standard values. The resultant depreciable plant capital cost was thereby reduced to \$667,949.94. This figure includes all investment items purchased during Fiscal Year 1968. Table 12 presents Capital Cost information for the 325,000 GPM Webster Test Bed Plant.

Other action, started in June 1967, to survey all unused equipment and property will further reduce capital costs.

TABLE 12

325,000 GPD WEBSTER TEST BED PLANT CAPITAL COSTS

I. Principal Items of Equipment (P.I.E.)

1. Special Equipment (Shop and Field Assembled)

	<u>Amount</u>	<u>% P.I.E.</u>
a. First stage and all associated equipment	41,499.00	13.1
b. Second stage and all associated equipment	32,649.00	10.3
c. Third stage and all associated equipment	33,016.00	10.5
d. Fourth stage and all associated equipment	38,973.00	12.4
e. Iron Removal and Auxiliary equipment	44,638.00	14.2
	-----	-----
Subtotal	190,725.00	60.5

TABLE 12 (Cont'd)

325,000 GPD WEBSTER TEST BED PLANT CAPITAL COSTS

2.	<u>Standard Engineering Equipment</u> <u>(includes all necessary standby</u> <u>units; includes spare parts)</u>		
a.	Process Plant Foundation	22,005.00	7.0
b.	Interstage pumps & drives	1,384.00	0.4
c.	Air Compressor, Air Dryer & Auxiliaries	2,761.00	0.8
d.	Rectification Equipment	22,200.00	7.0
e.	Product & Waste Water Conditioning Equipment	41,379.90	13.1
f.	Spare membranes & components	12,624.70	4.0
g.	Small tools and laboratory equipment	6,234.27	2.0
h.	Furniture and Office equipment	9,028.95	2.9
i.	Inventory spares	240.00	0.1
j.	Miscellaneous	6,878.25	2.2
		<hr/>	<hr/>
	Subtotal	124,736.07	39.5
	TOTAL P.I.E.	<u>\$315,461.07</u>	<u>100.0</u>

TABLE 12 (Cont'd)

325,000 GPD WEBSTER TEST BED PLANT CAPITAL COSTS

I. Process Facilities

3. Raw feed buffer chamber	4,199.00
4. Waste detention chamber	4,000.00
5. Site development (includes grading, paving landscaping, etc.)	2,258.00
6. Insulation	0
7. Electrical	18,034.98
8. Piping	1,700.00
9. Instruments	24,131.33
10. Building	173,287.87
11. Fresh water storage & forwarding facilities	<u>19,246.00</u>
TOTAL PROCESS FACILITIES	\$247,857.18

II. Other Plant Costs

13. Contingencies	0
14. Engineering	38,732.82
15. Interest on investment during construction	12,041.00
16. Startup expense	49,033.04
17. Cost of site	<u>4,824.83</u>
TOTAL OTHER CAPITAL COSTS	<u>\$104,631.69</u>
<u>TOTAL PLANT COSTS</u>	<u><u>\$667,949.94</u></u>

D. WATER PRODUCTION COSTS

The ledger summary for this report period is presented in Table 15.

The Test Bed Plant was not operated as a demonstration plant with the object of gathering electro dialysis cost data because of the experimental work described in Section IV. For example, of the 2184 hours downtime during the year, 447 hours were the result of equipment modification and experimental work which would not normally occur in a production plant. A total of 1220 hours were used for acid backwash of the stacks. There were additional labor expenditures, membrane losses, and equipment purchases associated with the transport depletion evaluation that would not normally occur in a production plant.

One can adjust Table 12 to arrive at a set of normalized plant capital costs operating on Webster water by elimination of experimental equipment. The net result is Table 13, which reflects a \$461,558 capital investment rather than the \$667,950 investment of Table 12.

Table 14 represents the results of a paper study for a water production plant using the electro dialysis process with lime softening and carbon filtration pretreatment, Webster-type water. Costs are seen to vary from \$1.12/1,000 gallons for the 250,000 GPD plant to \$0.52/1,000 gallons for the 10 million GPD plant.

TABLE 13

325,000 GPD NORMALIZED CAPITAL COSTS
FOR AN ELECTRODIALYSIS PLANT OPERATING
ON WEBSTER WATER

I. Principal Item of Equipment (P.I.E.)

1. Special Equipment

	<u>Amount</u>	<u>%P.I.E.</u>
a. Stacks and Membranes	106,099.00	42.27
b. Rectifiers	22,200.00	8.84
c. Decarbonator	3,094.00	1.23
d. Iron and Manganese Removal System	21,990.00	8.76
	<hr/>	<hr/>
Subtotal	153,383.00	61.10

2. Standard Engineering Equipment

a. Pumps, Process Piping and Equipment	34,164.00	13.61
b. Chlorinator	1,439.00	0.57
c. Instrumentation and Controls	16,829.00	6.70
d. Moveable Equipment	2,557.00	1.02
e. Standby Equipment and Spare Parts	23,476.00	9.35
f. Waste Water Disposal Piping	1,700.00	0.68
g. Chain Hoist	384.00	0.15
h. Process Metalwork	621.00	0.25
i. Process Wiring	16,500.00	6.57
	<hr/>	<hr/>
Subtotal	97,670.00	38.90
TOTAL P.I.E.	251,053.00	100.00

TABLE 13 (Cont'd)

II. Other Plant Costs

3. Site	\$ 4,824.00
4. Building	79,404.00
5. Water Storage	23,260.00
6. Engineering	43,233.00
7. Start-up Expense	49,033.00
8. Interest on Construction Money	<u>10,751.00</u>
TOTAL OTHER PLANT COSTS	\$210,505.00
<u>TOTAL PLANT COSTS</u>	<u>\$461,558.00</u>

TABLE 14

OPERATING COSTS FOR NORMALIZED ELECTRODIALYSIS PLANT
USING COLD LIME SOFTENING AND ACTIVATED CARBON FILTRATION PRETREATMENT

Basis: One Stream Day and 330 Operating Days per Year

		Yearly Cost, \$				¢/1000 Gal. Plant Size			
		<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
I. <u>DIRECT OPERATING COSTS</u>									
A. <u>Energy</u>									
1. Electrical Energy (a)									
a.	Pumps, Lights & Auxiliaries	3885.	6961.	12902.	114965.	4.71	4.22	3.91	3.48
b.	Electrodialysis	1690.	3380.	6760.	67600.	2.05	2.05	2.05	2.05
2. Fuel		416.	559.	776.	2772.	0.50	0.34	0.24	0.08
B. <u>Chemicals & Supplies</u>									
1. Sulfuric Acid		653.	1307.	2614.	26136.	0.79	0.79	0.79	0.79
2. Filter Cartridges (b)		2743.	5486.	10972.	109720.	3.32	3.32	3.32	3.32
3. Chlorine		126.	251.	502.	5020.	0.15	0.15	0.15	0.15
4. Purifloc (c)		2695.	5391.	10781.	107811.	3.27	3.27	3.27	3.27
5. Lime, Industrial		3165.	6336.	12672.	126720.	3.84	3.84	3.84	3.84
6. Activated Carbon (d)		4320.	8640.	17280.	172800.	5.24	5.24	5.24	5.24
7. Propane		679.	1358.	2717.	27166.	0.82	0.82	0.82	0.82
C. <u>Operating Labor</u> (e)		6600.	9900.	9900.	19800.	8.00	6.00	3.00	0.60
D. <u>Maintenance Labor</u> (e)		7199.	11097.	12294.	43740.	8.73	6.72	3.73	1.33

TABLE 14 (Continued)

	Yearly Cost, \$				¢/1000 Gal.			
	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
<u>I. DIRECT OPERATING COSTS (Cont'd)</u>								
<u>E. Maintenance Materials</u>	2689.	3954.	6079.	37256.	3.26	2.40	1.84	1.13
<u>F. Membrane Replacement (3 yr.) (f)</u>	10000.	20000.	35000.	300000.	12.12	12.12	10.61	9.09
TOTAL DIRECT COSTS	46860.	84620.	141249.	1161506.	56.79	51.28	42.80	35.19
<u>II. INDIRECT OPERATING COSTS</u>								
<u>A. Payroll Extras</u>	2760.	4199.	4439.	12708.	3.35	2.54	1.34	0.39
<u>B. General & Administrative Expense</u>	4968.	7559.	7990.	22874.	6.02	4.58	2.42	0.69
<u>C. Depreciation & Interest (g)</u>								
1. Building	788.	1055.	1455.	4945.	0.95	0.64	0.44	0.15
2. Equipment	24984.	36966.	57197.	359752.	30.28	22.40	17.33	10.90
3. Land	120.	179.	271.	1502.	0.15	0.11	0.08	0.05
<u>D. Taxes & Insurance</u>	10756.	15816.	24310.	149023.	13.04	9.58	7.37	4.52
<u>E. Interest on Working Capital</u>	867.	1408.	2208.	15683.	1.05	0.85	0.67	0.48
TOTAL INDIRECT COSTS	45243.	67182.	97870.	566487.	56.79	40.70	29.65	17.18
TOTAL OPERATING COSTS	92103.	151802.	239119.	1727993.	111.63	91.98	72.45	52.37

TABLE 14 (Continued)

Footnotes:

- (a) Electric energy rate: 10 mils/kwh
- (b) Filter cartridge life: 50,000 gallons
- (c) Cost studies indicate that if Alum were used instead of Purifloc, cents/1000 gallons costs would be 1.12 instead of 3.84; however verifying Pilot Plant experiments with Alum had not been run when the report was written.
- (d) Carbon lifetime 6 months (3 gal/min., 3 foot depth)
- (e) Operating and Maintenance Labor rate \$2.50/hour. Operating labor 8 hrs/day at 250,000 GPD, 12 hrs/day at 1 million GPD, 24 hrs/day at 10 million GPD. Maintenance labor same as operating labor. Each stack disassembled once every nine months.
- (f) Membranes: 250 pairs/stage, ACI DA-2 with DK-1, \$20/membrane at 250,000 and 500,000 GPD, \$15/membrane at 10 million GPD. Three stages in series for each 250,000 GPD module.
- (g) Depreciation and Interest Charges:
 - Building - 45 year life with straight line depreciation.
 - Equipment - 20 year life with straight line depreciation.
 - Land - 4½ percent interest on initial cost.

TABLE 15
LEDGER SUMMARY

ACCUMULATIVE TOTALS TO END OF REPORT PERIOD

ACCOUNT NUMBER	LABOR		MATERIAL	OTHER	TOTAL
	HOURS	MONEY			
<u>OPERATING EXPENSE ACCOUNTS</u>					
500.00 Production Expense					
.01 Supervision & Engineering	816½	3295.49			3295.49
.06 Labor	5572 3/4	16031.05			16031.05
.11 Operating materials			364.25		364.25
.13 Contract services & mat'l.					
.21 Sulfuric Acid (Pretreat)			3053.37		3053.37
.22 Permanganate (Pretreat)			1009.61		1009.61
.26 Chlorine (Product)			49.28		49.28
.32 Sulfuric Acid (Process)			3053.41		3053.41
.37 Expendible Items			437.49		437.49
.82 Deprec. Expense, Membranes and other Components				9235.44	9235.44
TOTAL	6389¼	19326.54	7967.41	9235.44	36529.39
520.00 Utilities					
.41 Boiler Fuel			1432.46		1432.46
.43 Electricity				11854.41	11854.41
TOTAL			1432.46	11854.41	13286.87
530.00 Laboratory					
.06 Labor	16½	46.77			46.77
.11 Operating supplies			1762.18		1762.18
.36 Chemicals			837.50		837.50
TOTAL	16½	46.77	2599.68		2646.45
540.00 Test Facility					
.01 Supervision & Engineering	2888½	16840.37			16840.37
.06 Labor	11186½	36601.25			36601.25
.11 Material & supplies			11499.75		11499.75
TOTAL	14075	53441.62	11499.75		64941.37
601.00 Maintenance					
General Maintenance					
.01 Supervision & Engineering	202½	822.34			822.34
.06 Labor (Gen'l Plant)	942¼	2650.80			2650.80
.12 Maintenance material			643.41		643.41
.82 Other Expense				28.50	28.50
TOTAL	1144 3/4	3473.14	643.41	28.50	4145.05

LEDGER SUMMARY

ACCUMULATIVE TOTALS TO END OF REPORT PERIOD

ACCOUNT NUMBER	LABOR		MATERIAL	OTHER	TOTAL
	HOURS	MONEY			
Maintenance					
606.00 Building Maintenance					
.01 Supervision & Engineering					
.06 Labor	1816	3769.58			3769.58
.12 Maintenance material			2429.60		2429.60
.82 Other Expense				25.00	25.00
TOTAL	1816	3769.58	2429.60	25.00	6224.18
607.00 Site Improvements					
.01 Supervision & Engineering					
.06 Labor	212¼	593.46			593.46
.12 Maintenance materials			1147.21		1147.21
.82 Other Expense				37.50	37.50
TOTAL	212¼	593.46	1147.21	37.50	1778.17
Process Equipment					
611.00 Electrodialyzer, Stage #1					
.06 Labor	146	430.54			430.54
611.01 Electrodialyzer, Stage #2					
.06 Labor	121½	365.58			365.58
611.02 Electrodialyzer, Stage #3					
.06 Labor	205	631.71			631.71
611.03 Electrodialyzer, Stage #4					
.06 Labor	603½	1828.39			1828.39
611.04 Electrodialyzer, Stage #5					
.06 Labor	342½	1016.95			1016.95
TOTAL	1418½	4273.17			4273.17
615.00 Tanks and vessels					
.01.06 Labor - Tanks 04A, B & C	227½	708.25			708.25
.12 Materials " " " "			155.90		155.90
TOTAL	227½	708.25	155.90		864.15

LEDGER SUMMARY

ACCUMULATIVE TOTALS TO END OF REPORT PERIOD

ACCOUNT NUMBER	LABOR		MATERIAL	OTHER	TOTAL
	HOURS	MONEY			
616.00 Gas Removal Equipment					
.06 Labor-Degasitors, Aerators	1	3.46			3.46
.12 Material " "			7.20		7.20
TOTAL	1	3.46	7.20		10.66
617.00 Other Process Equipment					
.01.06 Labor - Air Compressors	19½	53.28			53.28
.12 Material " "			381.70		381.70
.03.06 Labor-Chlorination Equip.	5	13.08			13.08
.04.06 Labor-Air Dryer	10½	27.60			27.60
.05.12 Material-Hoist, Electrical			65.82		65.82
TOTAL	35	93.96	447.52		541.48
618.00 Process Pumps & Drivers					
.01.06 Labor-Pump KMnO ₄ Feed	10½	42.73			42.73
.12 Material " " "			1.27		1.27
.02.06 Labor-Pump 02	79½	236.74			236.74
.12 Material " "			483.96		483.96
.03.06 Labor-Pump 12	3½	8.93			8.93
.04.06 Labor-Pump 14	3½	8.93			8.93
.05.06 Labor-Pump 16	3	8.93			8.93
.07.06 Labor-Pump 21	13	37.69			37.69
.12 Material " "			20.17		20.17
.10.06 Labor-Sump Pump (Tunnel)	3½	8.81			8.81
.13.06 Labor-Pump 108	14½	44.27			44.27
.12 Material " "			185.00		185.00
.14.06 Labor-Motors	1½	3.83			3.83
TOTAL	132½	400.86	690.40		1091.26

LEDGER SUMMARY

ACCUMULATIVE TOTALS TO END OF REPORT PERIOD

ACCOUNT NUMBER	LABOR		MATERIAL	OTHER	TOTAL
	HOURS	MONEY			
619.00 Instrumentation & Controls					
.06 Labor - pH Recorder	35	106.05			106.05
.12 Material " "			21.26		21.26
.01.06 Labor-Conductivity Cells	5½	14.24			14.24
.12 Material " "			4.05		4.05
.05.06 Labor-Pressure Transmitters	26½	73.80			73.80
.06.06 Labor-Flow Meters	10½	27.45			27.45
.12 Material " "			145.91		145.91
.09.06 Labor-Voltmeters					
Material " "			2.50		2.50
.10.12 Material - Ammeters			17.32		17.32
TOTAL	77½	221.54	191.04		412.58
620.00 Accessary Elec. Equipment					
.06 Labor-Rectifiers	11½	30.07			30.07
.12 Material-Rectifiers			62.40		62.40
.02.06 Labor-Miscellaneous	72	208.88			208.88
.12 Material-Miscellaneous			519.24		519.24
TOTAL	83½	238.95	581.64		820.59
621.00 Tanks					
.06 Labor-Raw Water Tanks	6	19.18			19.18
TOTAL	6	19.18			19.18
622.00 Circulating Water Lines					
.05.06 Labor-Acid	38½	111.96			111.96
.12 Material-Acid			71.40		71.40
.06.06 Miscellaneous	128	360.43			360.43
.12 Material-Misc.			987.45		987.45
TOTAL	166½	472.39	1058.85		1531.24

LEDGER SUMMARY

ACCUMULATIVE TOTALS TO END OF REPORT PERIOD

ACCOUNT NUMBER	LABOR		MATERIAL	OTHER	TOTAL
	HOURS	MONEY			
633.00 Feedwater Treatment Equip.					
.06 Labor-Filters - Dil.	2½	6.49			6.49
.01.06 Labor-Filters - Conc.	6	18.50			18.50
TOTAL	8½	24.99			24.99
General Plant Maintenance					
693.00.12 Materials - Shop			2184.60		2184.60
694.00.12 Materials - Tools			2177.06		2177.06
TOTAL			4361.66		4361.66
699.00 Other Equipment					
.06 Labor - Heating System	28½	76.84			76.84
.12 Material " "			674.66		674.66
.01.06 Labor - Painting	341½	924.09			924.09
.12 Material - Painting			510.59		510.59
.02.06 Labor - Fastening Frames	112	330.61			330.61
TOTAL	482	1331.54	1185.25		2516.79
<u>INDIRECT EXPENSE & OTHER ACCOUNTS</u>					
701.00 General & Adm. Expense					
.01 Supervision & Engineering	841½	2889.97			2889.97
.02 Office Services	4140½	12628.75			12628.75
.03 Report Preparation	2752	9075.76			9075.76
.07 Recruiting, Training, etc.	2200	5020.74		443.73	5464.47
.11 Office Supplies			5306.76		5306.76
.51 Equipment Rent				308.00	308.00
.52 Building Rent				285.00	285.00
.53 Professional Services				1445.80	1445.80
.54 Travel	591	2298.40		4696.99	6995.39
.55 Insurance - Other				1504.00	1504.00
.56 Communications	16	41.62	1365.74	4202.02	5609.38
.57 Public Relations				263.26	263.26
.58 Safety	42½	134.18			134.18

LEDGER SUMMARY

ACCUMULATIVE TOTALS TO END OF REPORT PERIOD

ACCOUNT NUMBER	LABOR		MATERIAL	OTHER	TOTAL
	HOURS	MONEY			
701.00 General & Adm. Expense					
.59 Safety Supplies			491.58		491.58
.64 Outside Services Employed				1662.33	1662.33
.65 Outside Services-Jacksonville				19401.28	19401.28
.82 Other Adm. Expense			83.25	950.75	1034.00
TOTAL	10583¼	32089.42	7247.33	35163.16	74499.91
702.00 M&O Contractor Expense					
.61 Contractor Fixed Fee				30000.00	30000.00
.62 Contractor Overhead (G&A)				2419.51	2419.51
TOTAL				32419.51	32419.51
710.00 Depreciation Expense					
.71 Plant in Service				43534.93	43534.93
.73 Amortization Expense				68972.37	68972.37
TOTAL				102507.30	102507.30
740.00 Disposal of Plant					
TOTAL				7834.77	7834.77
910.00 Plant Jobs (Job Orders)					
.82 Other (Burden absorbed in Cost of Job Order)				(109.06)	(109.06)
TOTAL				(109.06)	(109.06)
GRAND TOTALS	36875½	120528.82	43646.31	198996.53	363171.66

A. WEBSTER ENGINEERING AND DEVELOPMENTAL REPORTS

During Fiscal Year 1968, six "Webster Engineering and Developmental Reports" were submitted by Webster personnel to the Office of Saline Water. These reports contain technical information too extensive to be conveniently handled by this Annual Report. Most of these reports are scheduled to be published as Office of Saline Water Research and Development Progress Reports. An abstract of each report is given below.

1. HISTORY OF 1601 FOOT DEEP TEST HOLE AND SHALLOW PRODUCTION WELL NO. 6 AT WEBSTER, SOUTH DAKOTA. (MASON-RUST - WEDR NO. 19, MARCH 1968).

During the summer of 1967, Independent Drilling Company of Aberdeen, South Dakota drilled a test hole to granite 1601 feet below the surface and drilled a 187 foot deep, 400 GPM production well at the Webster Test Facility.

- a. The 1601 foot deep test hole investigation consisted of (1) soil samples gathered and analyzed at five foot intervals, (2) gamma ray, sonic, and electric logs for purpose of locating water bearing strata, (3) water analyses at 1370 to 1595 feet. Based on these results as analyzed by UOP-Johnson Company and the opinion of Mason-Rust, a 100 to 200 GPM maximum capacity gravel-packed deep production well (Na₂SO₄ type water, about 2350 ppm, 63^oF at surface) could be constructed at an estimated cost of \$101,000.00
- b. The 400 GPM shallow production well (Well No. 6) was fitted with 40 feet of 12 inch diameter, No. 50 slot stainless steel screen and a submersible 40 H.P. pump. The water obtained from this well is similar in composition to that of Well No. 5 (City owned).

2. REVERSE OSMOSIS USING WEBSTER PRETREATED WATER. PART II: MASON-RUST OPERATION OF AEROJET MODEL 1-5608-2 REVERSE OSMOSIS UNIT DURING PERIOD 30 JUNE TO 4 SEPTEMBER 1967. (MASON-RUST - WEDR NO. 30, SEPTEMBER 1967).

This report covers Mason-Rust operation of the Aerojet Model 1-5608-2 10,000 GPM Plate and Frame Reverse Osmosis pilot plant at Webster, South Dakota during the period 30 June 1967 to 4 September 1967. During this time, a total of 1579.8 on stream hours and 104.9 downtime hours was logged against the

unit. Of the total downtime, the unit was off line 84 hours while awaiting replacement of Gardner Denver pump packing parts to be shipped from the supplier; scheduled downtime accounted for an additional eight hours. A total of 362,820 gallons of product water was produced; the unit consumed an average of 33.9 KWH electrical energy per 1,000 gallons of product water. Diatomaceous earth filtration was used as raw water pretreatment to remove iron and manganese. The final average water flux on 4 September 1967 (750 psi operating pressure) was 12 gfd. Product water TDS was 150 ppm (feed 1690 ppm) at run termination, with the following approximate order of ion removal selectivity: sulfate > calcium > magnesium > bicarbonate, silica and chloride > sodium.

Since insertion of the membrane module on 2 February 1967, a total of 1,037,364 gallons of product water, 5077 operating hours, and about 185 downtime hours were logged against the unit. The product water flux at 750 psi on 2 February 1967 was 23.4 gfd.

Perhaps the greatest difficulty encountered was the lack of a constant quality feed water necessary for proper technical evaluation of the unit. Even when a single well (Webster Well No. 3) was used as feed water, the total dissolved solids content and ion composition varied hourly according to the demands of the water production plant on the other wells.

Approximate relationships between the calcium sulfate solubility products and ionic strengths of Webster water at pH 4.5 are presented in Appendix B. of WEDR No. 30. Precipitation of insoluble salts such as CaSO_4 places an upper limit on the product feed recovery ratio of the Reverse Osmosis unit.

3. INVESTIGATION OF HIGH TEMPERATURE, WEBSTER, SOUTH DAKOTA WATER DESALINATION USING THE IONICS STACKPACK. (MASON-RUST - WEDR NO. 31, JANUARY 1968.)

Operation of the Ionics Stackpack was compared at temperatures of 50°F, 60°F, 80°F, 110°F, and 140°F using lime softened and carbon filtered Webster, South Dakota well water. The major benefit of operation at 140°F as opposed to operation at 68°F or 50°F is in decreased energy requirements, principally because of a decrease in Stackpack electrical resistance with increasing temperature. For example, at 50°F and $I = 1.5$ amperes (TDS reduction from about 1290 ppm to 760 ppm) repeating cell pair (RCP) energy expenditure is 1.55 KW-hr/1,000

gallons; at 140°F and I = 1.5 amperes, the RCP energy expenditure is 0.56 KW-hr/1,000 gallons. Energy expenditures for a variety of operating currents and temperatures are presented in graphical and tabular form in this report.

The major contribution to the RCP resistance is from the dilution stream rather than the membranes, regardless of temperature. Graphs and tables specifying the individual area resistances (ohm-cm²) contributed by anion and cation membranes, dilution and concentrate streams as functions of temperature and operating current are presented in this report.

No special problems or limitations occurred as a result of high temperature electrodialysis (up to 140°F), provided polyethylene spacers are used below 110°F and block polymer spacers are used in the 110°F to 140°F range, and the flow channel linear velocity does not exceed 50 cm/sec.. Prolonged use of polyethylene spacers at 140°F will cause deformation of the spacer straps and low limiting current values.

Within the temperature range studied, the anion membrane limiting current density data was found to obey the following empirical relationship:

$$\frac{i_{lim}}{C} = 0.067e^{0.016T - 0.58v}$$

where i_{lim} = limiting current density (ma/cm²), C = dilution normality (epm), v = dilution linear velocity (cm/sec) and T = temperature, in degrees Centigrade.

4. ELECTRODIALYSIS OF LIME SOFTENED, CARBON FILTERED, WEBSTER, SOUTH DAKOTA WELL WATER. (MASON-RUST - WEDR NO. 32, MAY 1968).

This report describes the electrodialysis pilot plant (Ionics Stackpack, Aqua-Chem WD 6-2, and ACI Acilyzer) experiments for July to December 1967 using lime softened, carbon filtered, Webster, South Dakota well water (48°F to 55°F) as feed. The ACI Acilyzer data are used to predict production plant performance, including product water costs for 250,000 GPD to 10,000,000 GPD plants in Section VI and Appendix B of WEDR No. 32.

The Ionics Stackpack (dilution $v = 35$ cm/sec. flow velocity) appears to tolerate small amounts of lime turbidity in the feed, while the Aqua-Chem and ACI Acilyzer (both dilution $v = 5.73$ cm/sec.) will rapidly scale on the same feed. Sand filtration and pH control ($\text{pH} < 7.5$) are necessary to eliminate trace turbidity; pH control, carbon filtration, and cartridge filtration only are insufficient to remove lime turbidity.

Acilyzer pilot plant experiments demonstrated no great improvement (within the accuracy of the experiments) with respect to calcium sulfate scaling of the cation membranes when a current polarity reversal cycle of 10 second forward, 1 second reverse was applied, as opposed to no reversal.

Water production costs for ACI equipment on Webster type water at 250,000 GPD product (raw water 1690 ppm total dissolved solids; product water 490 ppm TDS, 48°F) are calculated to be \$1.30/1,000 gallons, assuming 330 days on-stream per year. Three stages would be needed, 250 membrane pairs per stage; current 30, 21, and 15 amperes; feed water 360,000 GPD; carbon filter lifetime 6 months; membrane lifetime 3 years, labor rate \$2.50/hour. The \$1.30/gallon includes \$0.49 for amortization; chemicals \$0.22; membrane replacement \$0.12; labor \$0.26; energy \$0.07; taxes, overhead, and interest on working capital \$0.14. Water costs for other capacity plants, and various carbon filter lifetimes are presented in Section VI if WEDR No. 32.

5. INVESTIGATION OF HIGH TEMPERATURE ELECTRODIALYSIS USING ASAHI GLASS MEMBRANES IN THE IONICS STACKPACK (MASON-RUST WEDR NO. 33, MAY 1968).

This report compares operation of the Ionics Stackpack using Asahi Glass membranes and lime softened Webster, South Dakota well water at temperatures of 50°F , 68°F , 80°F , 110°F and 140°F .

The Asahi Glass anion AMT membranes tended to work their way into the Stackpack spacer flow channels during electrodialysis, particularly at elevated temperatures. The AMT membranes did not distort at 50°F during a day of electrodialysis operation. At 68°F and at a velocity of 50 cm/sec, they distorted after two hours of operation. Distortion at 80°F and 110°F is less severe than distortion at 140°F .

The limiting current densities of Asahi Glass AMT membranes at 50°F are of the same magnitude as the Ionics Anion membrane. For higher temperature, the limiting current densities are not repeatable, because of anion membrane distortion.

Since the membranes distort, the energy requirement benefit through high temperature electro dialysis is inconclusive.

6. INVESTIGATION OF HIGH TEMPERATURE ELECTRODIALYSIS USING ASAHI CHEMICAL INDUSTRY EQUIPMENT (MASON-RUST - WEDR NO. 34, JUNE 1968).

This report compares operation of the ACI SV-3 Acilyzer on various membrane cell pairs using lime softened and carbon filtered Webster, South Dakota well water at temperatures of 48°F, 68°F, 80°F, 110°F, and 140°F. From Acilyzer data, economic (projected water costs) of high temperature electro dialysis saline water conversion are described, for ACI production stacks from 250,000 GPD to 10,000,000 GPD plant sizes.

Of the three cation-anion membrane pair combinations tested (Japan Organo C100 and A100; Asahi Chemical Company DK-1 and DA02; and Asahi Glass CMV and AMT), only the Asahi Glass CMV and AMT combination was found suitable for electro dialysis operation at 140°F. All were suitable for operation between 48°F and 100°F. Above 110°F, the DA-2 membrane softened significantly and the A100 membrane developed pinhole leaks.

Limiting current density data and membrane resistance data are presented as function of temperature; for each of the membranes studied.

B. ELECTRODIALYSIS MEMBRANE PROPERTIES

Membrane	Type	Manufactured By:	Thickness (Wet) Millimeters	Description
DK-1	cation	Asahi Chemical Industry	0.23	Homogeneous, unreinforced, styrene divinyl benzene polymer.
DA-1	anion	Asahi Chemical Industry	0.23	Homogeneous, unreinforced; styrene divinyl benzene polymer.
DA-2	anion	Asahi Chemical Industry	0.23	Homogeneous, unreinforced; vinyl pyridine polymer.
C100	cation	Japan Organo	0.15	Heterogeneous, reinforced with polyethylene resin fiber
A100	anion	Japan Organo	0.15	Heterogeneous, reinforced with polyester resin fiber.
CMV	cation	Asahi Glass	0.32	Homogeneous, reinforced with heat treated PVC; Styrol and Butadiene polymer.
AMT	anion	Asahi Glass	0.20	Homogeneous, reinforced with Tetron; Styrol and Butadiene polymer.
Zephyr A	"neutral"	Union Carbide	0.13	Homogeneous, regenerated cellulose membrane manufactured by Union Carbide for meat packaging.

C. GLOSSARY OF TERMS

Anion Membrane:

Membranes containing fixed positive charges which repel cations (sodium, calcium, magnesium, etc.) and pass anions (chloride, sulfate, bicarbonate, etc.). Also called anion permeable membrane or anion transfer membrane.

Blowdown:

The amount of brine water of a desalination unit which is discarded as waste.

Brackish Water:

Water which ranges from 500 ppm total dissolved solids to the dissolved content of sea water.

Cation Membrane:

Membrane containing fixed negative charges which repel anions and pass cations. Also called cation permeable membrane or cation transfer membrane.

C. O. D.:

Chemical Oxygen Demand.

Current Efficiency:

Ratio of the equivalents of salts actually transferred to the Faradays of electricity passed through the membranes.

Electrodialysis:

The transport of ions through membranes in which the driving force is an electrical field applied across the membranes.

epm:

Equivalent parts per million, and is defined as a unit chemical equivalent weight per million unit weight of solution. It can be calculated by dividing ppm by the chemical equivalent weight. For ions,

$$(\text{epm of ion } i) = \frac{(\text{ppm of ion } i)}{|(\text{ion charge})|(\text{ion molecular weight})}$$

Faraday:

The amount of electricity necessary to free or transfer one gram equivalent weight of an element or ion. This amount is 96494 coulombs per equivalent.

Fouling of Membranes:

Either a deposition of iron, manganese, organics, or scale in the pores of a membrane or an irreversible neutralization of the fixed charges of the membrane by iron, manganese, or organics. At Webster, anion membrane fouling due to "organics" is believed to result in an irreversible neutralization of membrane fixed positive charges.

Limiting Current Density:

The maximum permissible current density before the effects of polarization becomes significant to result in water breakdown at the membrane surface.

Normality:

Defined as the number of gram molecular weight of the dissolved substance divided by the gram equivalent weight per liter of solution

$$\text{Normality} = \text{epm} (10)^{-3}$$

ppm:

Parts per million and defined as a measure of proportion by weight per million weight of solution

ppm as CaCO₃:

Parts per million of an ion or compound expressed in parts per million as equivalent calcium carbonate.

$$[\text{ppm ion as CaCO}_3] = \frac{50 (\text{ppm ion}) |\text{ion charge}|}{(\text{ion molecular weight})}$$

Reverse Osmosis:

A method of separation of salt ions from solution using a membrane permeable to the solvent and relatively impermeable to the solute (salt ions); pressure applied to the solution or brine side in excess of the osmotic pressure supplies the driving force to affect this separation.

Scale:

A deposit of minerals or solids on a surface such as a membrane.

Transport Depletion:

A form of electrodialysis where the anion membrane is either eliminated or replaced with a near "neutral" or non-selective membrane.

D. BIBLIOGRAPHY

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