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UNITED STATES ATOMIC ENERGY COMMISSION

PRESSURIZED LOOP MEMBRANE DEMINERALIZER TESTS

Final Report [for] January - June 1953

By
N. W. Rosenberg

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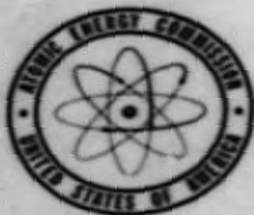
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Office of Technical Services
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Washington 25, D. C.

September 15, 1954

Ionics, Incorporated
Cambridge, Massachusetts

Technical Information Service Extension, Oak Ridge, Tenn.



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ABSTRACT

A membrane demineralizer has been tested in a pressurized loop. The loop water resistivity was maintained in the 1-2 megohm range by ionized solid transfer in the demineralizer. The size and power requirements of the unit tested, per gpm flow through the unit, were 2.3 cu ft and 100 watts. In view of the fact that present designs could reduce the size and required maintenance, further studies may be warranted.

INTRODUCTION

The operating characteristics of a membrane demineralizer designed to maintain a low solids content in a loop water have been studied in an experimental program carried out at Ionics, Incorporated, and at WAPD, under Task IIa, b, and e of WAPD Subcontract 14-316. The present report has been prepared to summarize the results of this program, which was suspended on June 30, 1953.

The goals of the program, under the wording of the contract, November 1952) were to:

- a. conduct, with a multi-membrane demineralizer loaned by Ionics, demineralization of loop water of an STR mockup installed in Pittsburgh, under atmospheric pressure,
- b. modify existing equipment to be loaned by Ionics to operate in pressure vessels, to be furnished by WAPD, for test in mockup loop under pressure, and

e. furnish reports of performance, size, weight, and power consumption of such apparatus in operations as specified above.

Earlier reports summarized operation of the membrane unit during preliminary work in Cambridge. At the request of WAPD personnel, delivery of the demineralizer to Bettis Field was delayed until January 10, 1953. A series of runs was made over the next months with modifications as shown necessary during pressurized operation. After the series of tests was completed, the unit was removed from the loop and returned to Ionics on June 30, 1953.

In the longest single run, over a period of three hundred hours, a treated water resistivity of 3-4 megohms was obtained from the electro-dialyzer unit and a loop water resistivity of 1-2 megohms was maintained.

THE MEMBRANE UNIT

An electro dialysis unit (Figure 1) consists of an alternating series of anion- and cation-selective membranes separated by solution-filled spacers with electrodes at each end of the alternating series. When an electrical current is passed through such a unit, ionized solids are transferred from alternate compartments, and the solution in one set of alternate compartments becomes more dilute and in the other set becomes more concentrated. Flow is provided through manifolds connecting alternate spacer paths.

In the particular unit provided to WAPD, a series of 15 cell pairs (each including a CR-61 cation-selective membrane, a diluting compartment, an AR-110 anion-selective membrane, and a concentrating compartment) was utilized. Each spacer was 9" x 9" in outside dimensions, defined an effective solution area of 0.12 ft², and had a thickness of 0.04". The membranes were 9" x 9" in outside dimensions and 0.03" thick. Thus the total thickness of a cell pair was 0.14" and the 15 cell pairs occupied about 2.1" in height x 81 in.² in cross section for 15 x 0.12 ft² = 1.8 ft² effective area of cell pair, or 18 ft² effective area/cu ft. (Larger present units increase this space utilization figure to 50 ft²/cu ft.) These volumes exclude the end plates and autoclave as described below.

Platinum foil electrodes and plastic end plates which compressed the entire assembly were utilized at the ends of the stack. It was found necessary to coat the entire unit with beeswax to prevent stray currents

and attendant electrical corrosion. The assembly was bolted to the head of a 12" ID autoclave through which the three streams (feed, product, and concentrate) and the two electrical leads were brought, and the unit was operated submerged in the assembled autoclave (Figure 2).

Under these conditions there was no more difficulty associated with operation at the high pressure of the autoclave than normally associated with operation of such a unit. The one requirement foreign to granular ion exchange demineralization was the necessity for continuous (or semi-continuous) addition of makeup water to replace the water bled from the concentrate cells of the demineralizer.

LOOP OPERATION

The electro dialysis unit was tested on a dynamic corrosion system referred to as WAPD loop M-1. The loop was made up of 1" schedule 80 stainless steel pipe and included a Westinghouse 100-A totally enclosed centrifugal pump. Normally incorporated in this loop for water purification was an ion-exchange resin container, 1" dia. x 36" long, containing 300 cc of type MB-1 resin. Also, for removal of suspended solids from the system, two perforated stainless steel micro-metallic filter discs, encased in a stainless steel body, were included in the loop. A high pressure vessel was used for purposes of oxygen additions to the loop.

The electro dialysis unit operated on WAPD loop M-1 under the following conditions:

Temperature of main loop	500°F
Pressure	1500 psi
O ₂ concentration	5-7 cc/liter
pH range	6.0-7.0
Ion exchange bed operation	continuous for first 552 hours
Main loop resistivity using ion-exchange bed	0.7-1.3 megohms
Dialyzer operation	continuous for final 304 hours
Main loop resistivity using dialyzer	0.5-3.0 megohms
Feed water condition	degassed
Loop flow rate	15.5 gpm
Resin bed flow rate	0.045 gpm
Dialyzer flow rate	0.043 gpm

The initial gas concentration of the loop was gradually reduced by the degasification of the coolant through a surge tank. This step was undertaken to eliminate the presence of inert gases, a condition which frequently interferes with chemical analyses performed during the test. The loop temperature was gradually increased at a rate of 50° per hour, while at the same time oxygen additions were made through the high pressure vessel until operating conditions were reached.

During startup, both the MB-1 resin bed and the loop filter were opened to the system. The filter operated continuously throughout the run while the resin bed purified the water for the first 552 hours of the 856 hour test. During this operation, a check of the coolant conditions before and after the resin bed was made and recorded every two hours. The influent water resistivity values ranged between 0.7 megohms and 1.5 megohms while the effluent water resistivity values remained between 15 and 25 megohms. At the end of 552 hours, the resin bed was isolated from the system and the electro dialysis unit was opened to the system replacing the resin bed as the cooling purifier. A potential of 250 v DC was placed across the electrodes of the demineralizer, resulting in an initial current of 30 ma, which decreased rapidly to a steady state value of 16-20 ma. Loop resistivity values and dialyzer product resistivity values were recorded every two hours during dialyzer operation and reported in WAPD-CP-642. A plot of these data is presented as Figure 3. This graph indicates that the loop water resistivity gradually increased from a value of 0.5 megohm at the start of the dialyzer operation to a value of 1.8 megohms at the end of the operation. Throughout this period degassed make-up water was added to the loop at a rate of 0.11 gal/hr. This high resistivity make-up water replaced the "concentrated" waste liquid which was continuously removed from the dialyzer. There was a sudden increase in loop resistivity for a 12 hour period on 6/8/54. This increase is attributed to the fact that the system temperature decreased from 500°F to 400°F during this period. The range of the pH values during the run was 6.0 to 7.0 with an average value of 6.7 for the run. The O₂ concentration, determined every two hours by the Winkler method, was maintained between 5 cc/liter and 7 cc/liter by frequent additions through the high pressure vessel. The make-up water added to the system was always degassed before addition.

DISCUSSION

The operating data indicate a three-fold reduction in conductivity was fairly consistently obtained by passage of the water through the dialyzer under the operating conditions. A flow of 0.043 gpm and a power of 5 watts through the 1.8 ft² of effective area implies an area requirement of 42 ft²/gpm = 2.3 ft³/gpm, exclusive of end plates and pressure container, and 100 watts/gpm. With the larger new unit design, the same area requirement would imply 0.9 ft²/gpm. Recent operating data obtained by Ionics in other work on higher salinity waters suggest that considerably lower area requirements would be possible — as low as one fifth of that required in the early unit utilized above — for the same fractional demineralization at the flow rate indicated. It must further be noted that more automatic operating techniques have been developed which have allowed unattended operation for a period of six months with one hour maintenance periods at weekly intervals. Although it is recognized that there are special problems associated with the application considered, a further study of the use of the technique appears warranted by the experimentation to date.

Figure 1

Fig. 1 — Schematic Drawing of Electrodialysis Unit

- E — electrode cell
 P — product (diluting) cell
 W — waste (concentrating) cell
 C — cation-permeable (fixed-anion) membrane
 A — anion-permeable (fixed-cation) membrane

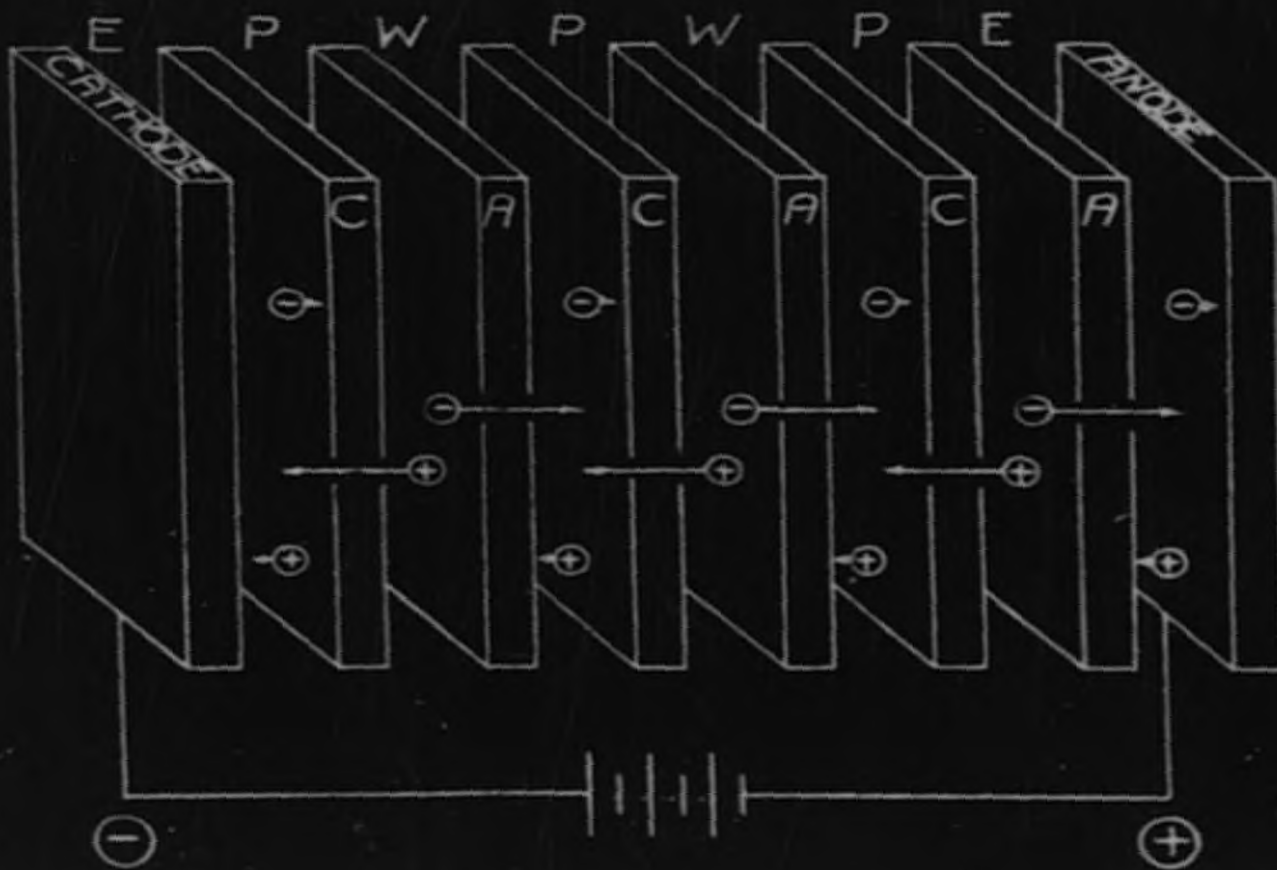


Fig. 2—Sketch of Assembled Unit Connected to Autoclave Head

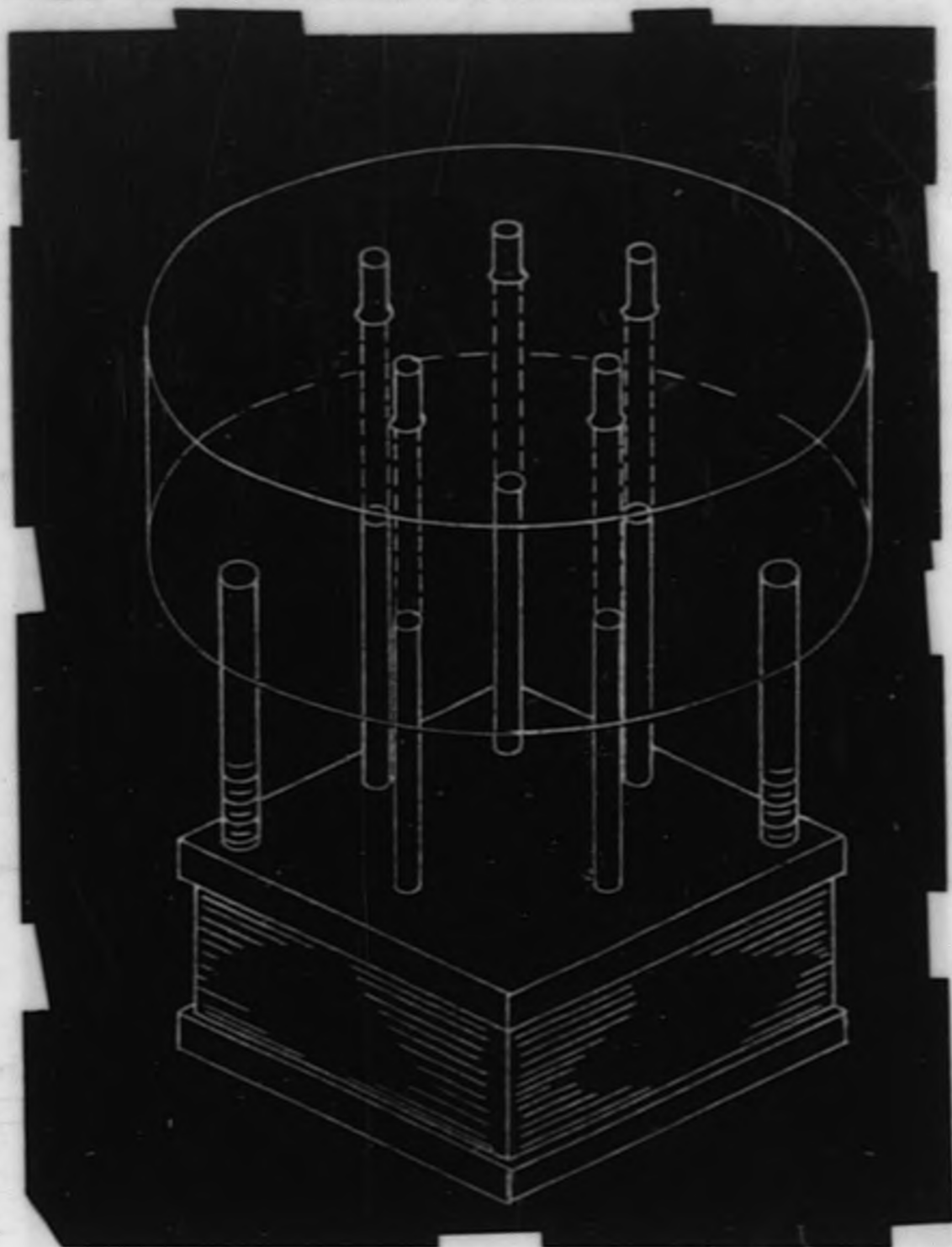
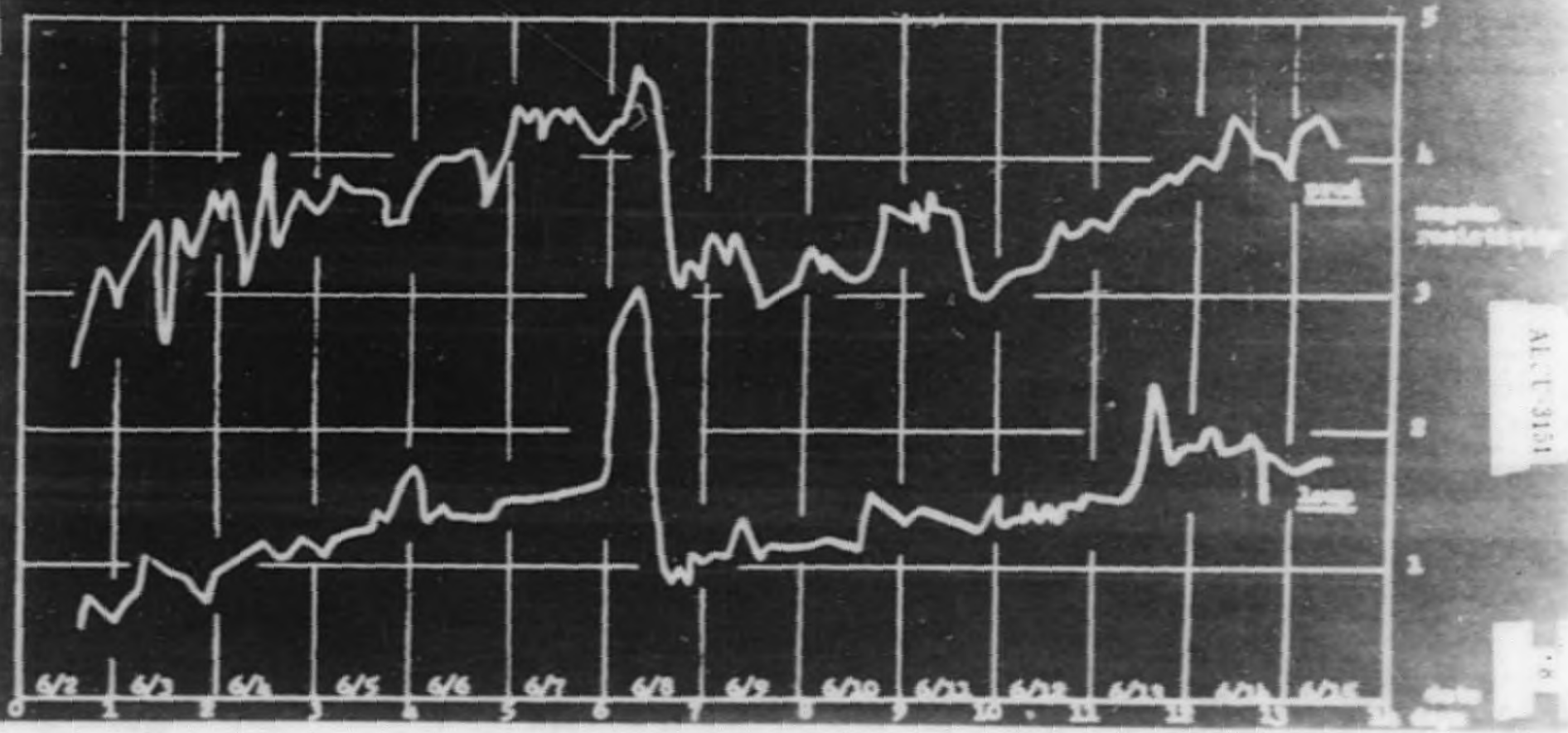


Fig. 3 — Performance of Membrane Demineralizer on Loop M-1
Solution Resistivity vs. Time



END