United States
Environmental Protection
Agency

Industrial Environmental Research
Laboratory
Cincinnati OH 45268

EPA-600/2-80-084
May 1980

Research and Development

EPA

Evaluation of Reverse Osmosis Membranes for Treatment of Electroplating Rinsewater

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May 1980

EVALUATION OF REVERSE OSMOSIS MEMBRANES FOR TREATMENT OF ELECTROPLATING RINSEWATER

by

Kenneth J. McNulty
Peter R. Hoover
Walden Division of Abcor, Inc.
Wilmington, Massachusetts 01887
for
The American Electroplaters' Society, Inc.
Winter Park, Florida 32789

Grant No. R804311

Project Officer

Mary K. Stinson
Industrial Pollution Control Division
Industrial Environmental Research Laboratory
Edison, New Jersey 08817

INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report is a product of the above efforts. It was undertaken to demonstrate the effectiveness and economic feasibility of using reverse osmosis for closed-loop control of metal finishing rinse wastes under actual plant conditions. The reverse osmosis system concentrates the chemicals for return to the processing bath while purifying the wastewater for reuse in the rinsing operation. The results of the report are of value to R&D programs concerned with the treatment of wastewaters from various metal finishing, non-ferrous metal, steel, inorganic and other industries. Further information concerning the subject can be obtained by contacting the Metals and Inorganic Chemicals Branch of the Industrial Pollution Control Division.

David G. Stephan
Director
Industrial Environmental Research Laboratory
Cincinnati

ABSTRACT

Because of the limited pH range in which current commercially available reverse osmosis membranes can be applied, a test program was initiated to define the applicability of new membrane materials to the treatment of rinsewaters with extreme pH levels and high oxidant levels (chromic acid). Life tests were conducted with the PA-300, PBIL, NS-100, NS-200, SPPO, B-9, and CA membranes on rinsewater from copper cyanide, zinc cyanide, acid copper, and chromic acid plating baths. The PA-300 membrane exhibited superior performance for the treatment of copper cyanide, zinc cyanide, and chromic acid rinsewaters, and further development and demonstration of this membrane is recommended. The NS-200 and PBIL membranes exhibited the best performance for treatment of acid copper rinsewaters. Efforts are underway to commercialize all three of the selected membranes (PA-300, NS-200, and PBIL).

This report was submitted in fulfillment of EPA Grant Number R804311 by the American Electroplaters' Society, Inc. (AES) under the partial sponsorship of the U.S. Environmental Protection Agency. This report covers the period March 1, 1976 to October 19, 1977, and work was completed as of June 12, 1978.

CONTENTS

FIGURES

TABLES

ACKNOWLEDGMENT

The authors gratefully acknowledge the financial support of EPA (Grant No. R804311) and AES (Research Project No. 39) for this program. Technical support for this program was received from the EPA Project Officer, Ms. Mary Stinson, and from the AES Project Committee: Messrs. Jack Hyner, Joseph Conoby, Charles Levy, Herbert Rondeau, James Morse, and George Scott.

SECTION 1

INTRODUCTION

Since enactment of the Federal Water Quality Control Act and its amendments, the metal finishing industry has become increasingly concerned with techniques for wastewater treatment. A variety of new technologies have been developed for the treatment of electroplating wastewaters; however, none of these technologies appears to offer an optimum solution to all aspects of the problem. As a result, many platers are waiting for further development and demonstration of new technologies before attempting to make final decisions on the selection of wastewater treatment processes.

Wastewater treatment technologies for the electroplating industry can be broadly classified as end-of-pipe destruction processes or in-plant recovery processes. The end-of-pipe destruction processes treat a total shop effluent to remove a mixture of heavy metals. At present it is neither technically nor economically feasible to recover and recycle metals from end-of-pipe processes (1). On the other hand, in-plant recovery processes treat rinsewater from a specific plating bath (or other operation), making it possible to recover and return the heavy metals to the plating bath.

It seems reasonable to speculate that most, if not all, plating shops will require an end-of-pipe treatment process, particularly for diversified job shops where in-plant recovery of all rinsewaters is neither technically nor economically feasible. Even for less diversified shops, end-of-pipe treatment would be required for spills, contaminated plating baths, spent cleaners, etc. However, because of the inherent disadvantages of end-of-pipe treatment -- loss of valuable plating chemicals, cost of sludge disposal, and cost of treatment chemicals -- it is also reasonable to speculate that platers will use in-plant recovery processes where the economics for recovery are favorable or where recovery could reduce the load on the end-of-pipe system to the extent necessary to meet the discharge regulations for specific contaminants.

Aside from a few applications in which closed-loop recovery can be achieved by countercurrent rinsing alone, some technique must be used to separate the dissolved plating chemicals from the rinsewater. The leading techniques for making this separation are reverse osmosis (RO), evaporation, and ion exchange. This report addresses the application of reverse osmosis to the recovery of electroplating rinsewaters, particularly extreme pH rinsewaters.

SECTION 2

CONCLUSIONS

On the basis of life test results for various membranes (NS-100, NS-200, PA-300, PBIL, SPPO, B-9, and CA) with different electroplating rinsewaters (copper cyanide, zinc cyanide, acid copper, and chromic acid), it is concluded that PA-300 is the most generally applicable of the membranes tested for treatment of rinsewaters with extreme pH levels or high levels of oxidants (chromic acid). The PA-300 membrane was superior to the other membranes for treatment of copper cyanide, zinc cyanide, and chromic acid rinsewaters. However, the NS-200 and PBIL membranes proved to be better than PA-300 for the treatment of acid copper rinsewaters.

Of these three membranes, PA-300 is the closest to commercialization. A full-scale, spiral-wound module containing the PA-300 membrane has been developed and extensively tested by the Fluid Systems Division of UOP for brackish-water and seawater desalting. Although the module has not yet been officially commercialized, modules are being fabricated and supplied on a special-order basis.

The NS-200 membrane is being actively developed toward commercialization by the Fabric Research Laboratory. At present, hollow-fiber modules with an output of 380-1140 liters (100-300 gallons) per day (approximately one-tenth the output of a full-scale module) are being fabricated for testing on seawater desalination and various wastewater streams. The manufacturer anticipates commercialization within one year.

The PBIL membrane is being developed by the Walden Division of Abcor, Inc. under contract to the Office of Water Research and Technology. Membrane casting procedures have been optimized, and a program has been recently initiated to develop procedures for fabricating the PBIL membrane in a spiral-wound configuration.

SECTION 3

RECOMMENDATIONS

Additional testing and development work is required before the promising membranes identified during this program can be offered to electroplaters as a viable means of achieving closed-loop recovery of rinsewaters. Future tests should be conducted with full-scale membrane modules in order to determine the stability of both membrane and non-membrane components (such as adhesives) and to determine membrane performance under realistic hydrodynamic conditions. Future tests should also be conducted on-site under closed-loop conditions in order to expose the membranes to the same rinsewater as in actual operation. Therefore,it is recommended that a full-scale RO system, with sufficient flexibility to test various new membrane modules, be designed and operated at an electroplating facility in order to demonstrate closed-loop RO recovery of difficult-to-treat rinsewaters. For the immediate future, it is recommended that field tests be conducted with the PA-300 spiral-wound module to determine its suitability for recovery of zinc cyanide rinsewaters. It is also recommended that membrane manufacturers be encouraged to develop new membranes and improved membrane modules that will withstand the severe conditions of electroplating applications.

SECTION 4

BACKGROUND

PRINCIPLES OF REVERSE OSMOSIS

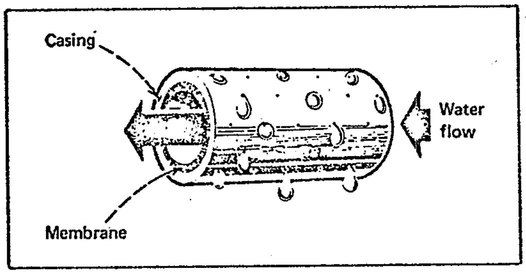
Reverse osmosis is a pressure-driven membrane separation process in which a feed stream under pressure (27.2-54.4 atm [400-800 psig]) is separated into a purified "permeate" stream and a "concentrate" stream by selective permeation of water through a semi-permeable membrane. There are three major types of commercially available membrane modules: tubular, spiral-wound and hollow-fiber. These are shown in Figure 1. Each of these modules has particular advantages and limitations. Tubular modules are not susceptible to plugging by suspended solids and can be operated at high pressures, but their space requirement (m^3 per m2 membrane surface) is relatively high and their cost is approximately five times as high as the other configurations for an equivalent rate of permeate production. Therefore, tubular modules are not recommended for plating applications.

Spiral-wound and hollow-fiber modules are essentially identical in cost for an equivalent rate of permeate production. Hollow-fiber modules have a somewhat lower space requirement per unit of permeate produced, while the spiral-wound modules are less susceptible to plugging by suspended solids.

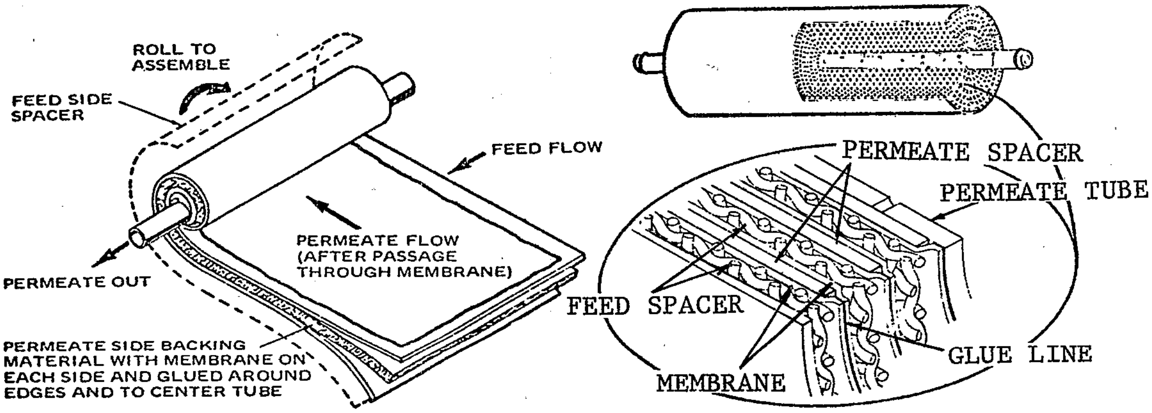
There are a number of membrane materials presently under development, but only two types are currently in commercial use. The most widely applied is cellulose acetate (or cellulose tri-acetate), which was originally developed for water desalination and has since been adopted for many industrial waste treatment applications. It is available in tubular, spiral-wound, and hollow-fiber configurations and exhibits excellent water permeation rates and high rejection of ionic species. Unfortunately, it is limited to a fairly narrow pH range (2.5-7). Operation beyond this range hydrolyzes the membrane and destroys its ability to pass water selectively.

The other commercially available membrane is duPont's polyamide membrane which is presently available only in a hollow-fiber configuration. It also exhibits high flux and high rejection, but can be applied over a somewhat broader pH range (4-11).

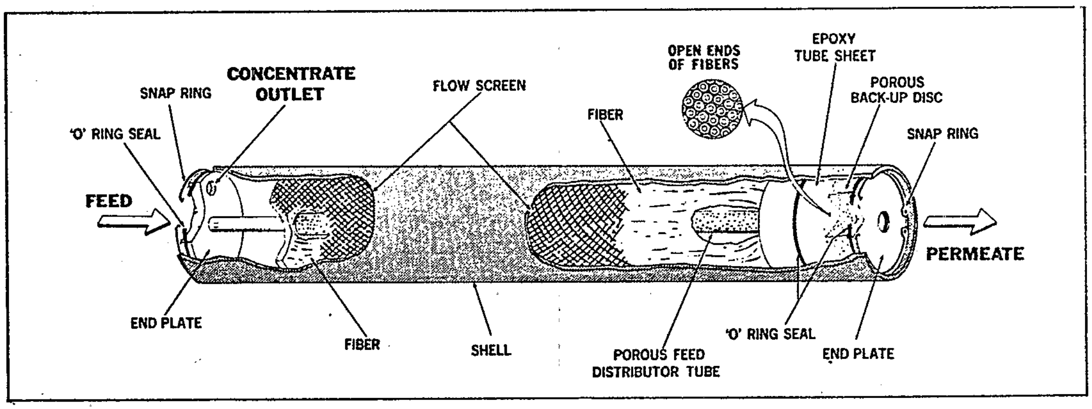
In general the cellulose acetate membrane should be used at low pH, and the polyamide membrane at high pH. In the region of pH overlap, neither membrane has an overriding advantage.



a. Tubular Membrane



b. Spiral-Wound Module



c. Hollow-Fiber Module

Figure 1. Membrane module configurations.

Membrane performance is characterized in terms of flux and rejection. Flux is the rate at which purified water permeates through the membrane per unit area of membrane surface and is given in liters per square meter per hour (l/m^2-hr). Rejection is the degree to which salts are prevented from passing through the membrane and is given by:

$$\% \text{ Rejection} = \frac{C_F - C_p}{C_F} \times 100\%$$

where: C_F = concentration in feed stream

C_p = concentration in permeate stream.

In general both flux and rejection increase with operating pressure and decrease with increasing feed concentration. Flux increases with temperature, but rejection is essentially temperature-independent. The flow rate of feed tangential to the membrane surface is also an important parameter and must be maintained at a high enough level to prevent the buildup of rejected salts at the membrane surface.

The major advantages and limitations of reverse osmosis are listed in Table 1. The objective of the research effort described below was to identify new membrane materials that would broaden the allowable pH range (Limitation #1) and would reduce the required frequency of membrane replacement (Limitation #4).

APPLICATION OF COMMERCIALLY AVAILABLE MEMBRANES TO RINSEWATER RECOVERY

Because of the potential cost advantage of RO relative to other recovery processes, EPA and AES have sponsored a number of projects aimed at developing and demonstrating RO for the treatment of electroplating rinsewaters. Work performed under EPA Grant R803753 (AES Project 32) included both in-house and field tests of commercially available membranes (2,3). The in-house tests were conducted with samples of actual plating baths diluted to various concentrations to simulate actual rinsewater. Nine different plating-bath rinsewaters were treated with the two commercially available membranes (cellulose acetate and polyamide) in full-scale RO modules.

It was concluded from these tests that RO appeared promising for the treatment of nickel baths (Watts, sulfamate, fluoborate) and copper pyrophosphate. Treatment of relatively low-pH cyanide baths also appeared feasible with the polyamide membrane. However, the commercially available membranes did not appear to have a suitable operating life for the treatment of highly oxidizing rinsewaters (chromic acid), low pH (<2) rinsewaters, and high pH (>11) rinsewaters.

Following in-house testing, various field tests were conducted to demonstrate the performance of RO under realistic conditions. The polyamide membrane in hollow-fiber configuration was successfully demonstrated for the treatment of Watts nickel rinsewaters (4). (The cellulose acetate membrane in spiral-wound configuration has also been successfully demonstrated on Watts nickel rinsewater [5]). It was concluded that either of the two commercially available membranes can be used to treat nickel rinsewaters and

TABLE 1. ADVANTAGES AND LIMITATIONS OF RO

ADVANTAGES

1. Low capital cost. The modular nature of RO units makes them particularly well-suited for small-scale installations.

2. Low energy cost. Only power for pumping is required; there is no phase change as in evaporation.

3. Low labor cost. The process is fully automated and simple to operate, requiring little operator attention.

4. Low space requirements. Since RO equipment is compact and operates continuously, it requires minimal tankage.

LIMITATIONS

1. There is a limited pH range (about 2.5 - 11) over which current commercially available membranes can operate for extended periods.

2. RO is incapable of concentrating solutions to very high concentrations. For ambient temperature baths, a small evaporator is generally required to close the loop.

3. Certain species, e.g., small non-ionized molecules, are not completely rejected by the membrane.

4. Membrane performance generally degrades with time, requiring periodic replacement of the membrane modules.

that the economics for closed-loop nickel recovery can be quite attractive. This has been proven in industrial practice: approximately 100 RO systems have been sold for the treatment of nickel rinsewaters.

In an effort to expand the application of RO to major plating baths other than nickel, two separate field demonstrations were conducted on copper cyanide rinsewaters with the polyamide membrane (6). In general, it was concluded that the polyamide membrane can be used for the recovery of relatively low-pH cyanide rinsewaters, provided that the membrane life is adequate. However, since rapid membrane deterioration was observed in one of the two field tests, the treatment of copper cyanide rinsewaters cannot be considered a proven application.

From these results as well as the known pH tolerance of the commercially available membranes, it became evident that new membranes must be developed in order to expand the applicability of RO to major plating baths other than nickel. In particular, membranes must be developed with resistance to pH extremes (<2 and >11) and with resistance to oxidants (chromic acid). The current program (EPA Grant R804311; AES Project 39) was undertaken to identify promising new membranes for electroplating applications.

SECTION 5

EXPERIMENTAL PROCEDURES

MATERIALS AND METHODS

Table 2 lists the membranes investigated during this program along with the manufacturer from which the membrane was obtained and a brief description of the membrane configuration and materials. Membranes other than those listed are under development but, for a variety of reasons, were not submitted by the manufacturer for testing during this program. In addition to the new membrane materials, the two commercially available membrane types, B-9 (polyamide) and CA (cellulose acetate) were tested in order to provide a reference level to which the new membranes could be compared. The B-9 membrane (pH range 4-11) was the reference for alkaline rinsewaters, and the CA membrane (pH range 2.5-7) was the reference for acid rinsewaters.

Table 3 lists the plating baths used to prepare the rinsewaters tested in this program. The major components and nominal composition are also listed for each plating bath. These baths were selected because of their extreme pH levels and, in the case of chromic acid, high oxidation potential. Rinsewaters were prepared from each bath of Table 3 by dilution to the appropriate concentration. Life tests were conducted at two dilutions for each bath: 5% of bath strength and 25% of bath strength. The dilutions were performed on a volumetric basis, e.g., one liter of bath per three liters of water for the 25% dilution. From actual feed analyses conducted during the life tests, the concentration ranges for the constituents of interest were determined for each dilution and are listed in Table 4.

TEST SYSTEM

A simplified flow schematic of the test system is shown in Figure 2. Feed was withdrawn from the feed tank by a centrifugal booster pump and passed through two cartridge filters in series. A high-pressure diaphragm pump was used to pressurize the feed and pass it through the membrane test cells. The pressures within the cells were controlled in the range of 27.2-54.4 atm (400-800 psi) with back-pressure regulators. An accumulator was used to dampen pressure pulsations from the high-pressure pump; a pressure relief valve and high-pressure switch were used to prevent overpressurization of the membranes; and a low-pressure switch was used to prevent the pumps from running dry. A cooling coil with automatic temperature control was used to maintain the feed at a constant temperature, and the surface of the feed tank was covered with polyethylene balls to prevent evaporation and CO_2 absorption (by alkaline solutions).

TABLE 2. MEMBRANE MATERIALS AND CONFIGURATIONS TESTED

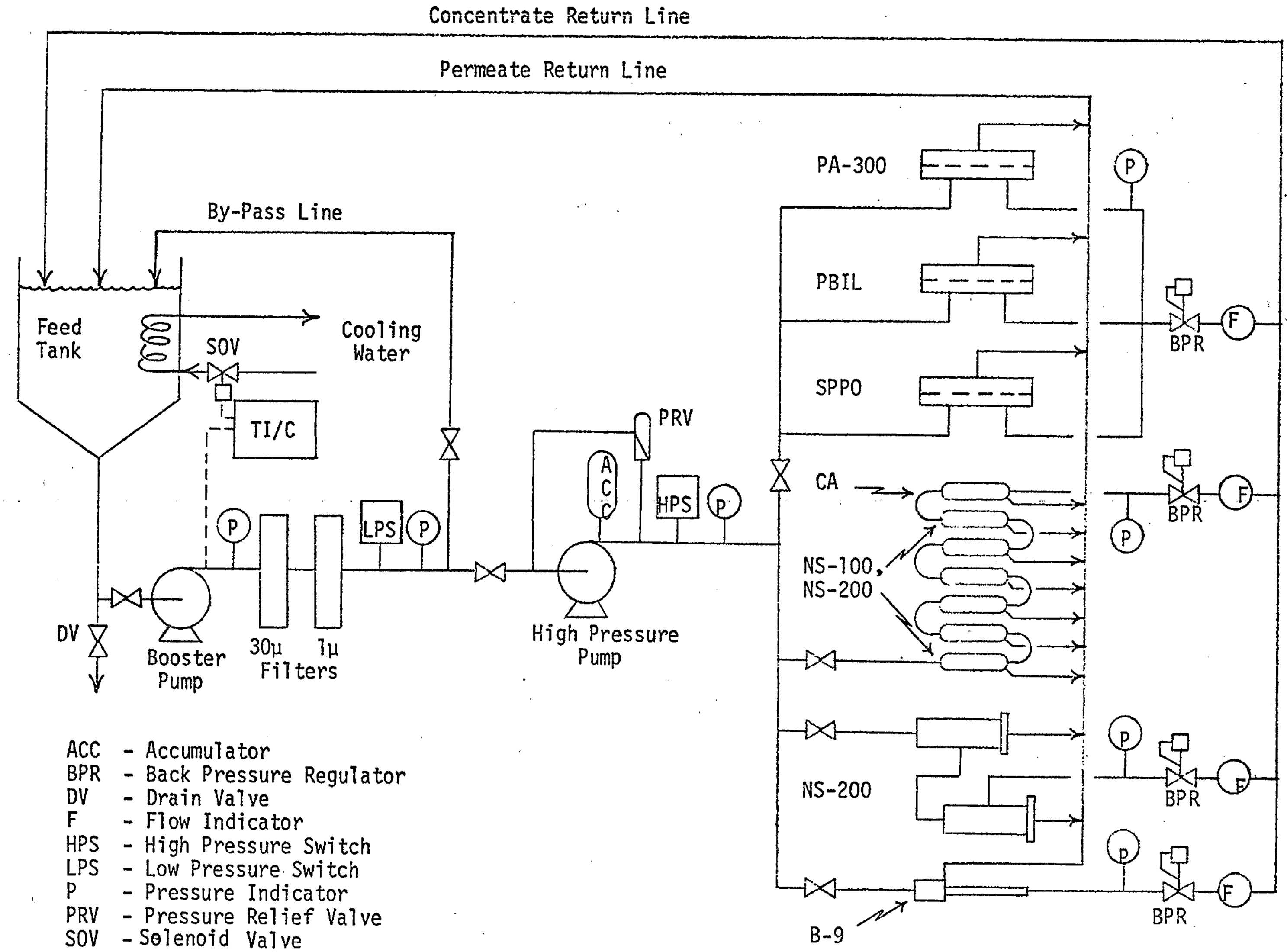
Membrane material	Source	Description
PA-300	Fluid Systems Div. of UOP, Inc. San Diego, CA	Flat-sheet composite membrane of poly (ether/amide) on polysulfone.
PBIL	Walden Div. of Abcor, Inc. Wilmington, MA	Flat-sheet asymmetric membrane of polybenzimidazolone.
NS-100	Walden Div. of Abcor, Inc. Wilmington, MA	1/2-inch tubular composite membrane of polyethyleneimine cross-linked with tolylenediisocyanate on polysulfone.
NS-200	Fabric Research Lab. Dedham, MA	0.006-inch ID hollow-fiber composite membrane of polyfurfuryl alcohol on polysulfone. Modules supplied.
NS-200	Walden Div. of Abcor, Inc. Wilmington, MA	1/2-inch tubular composite membrane of polyfurfuryl alcohol on polysulfone.
SPPO	General Electric Co. Wilmington, MA	Flat-sheet sulfonated polyphenyleneoxide.
B-9	E.I. duPont Wilmington, DE	Hollow-fine-fiber asymmetric membrane of aromatic polyamide. Mini-permeator supplied.
CA	Abcor, Inc. Wilmington, MA	1/2-inch tubular membrane of asymmetric cellulose acetate.

TABLE 3. COMPOSITION OF PLATING BATHS TESTED

Plating bath/ supplier	Component	Nominal composition
Chromic acid	Chromic acid	255 gm/l
Udylite	Sulfate	0.90 gm/l
	Catalyst (fluoride)	Unknown
Acid copper	Copper sulfate	60-90 gm/l
Lea-Ronal	Sulfuric acid	172-219 ml/l
	Brightener (Copper Gleem PC)	0.4-0.6% vol.
	Chloride	50 ppm
Copper cyanide-1	Copper as metal	47 gm/l
MacDermid	Free cyanide (as KCN)	20 gm/l
	Potassium hydroxide	15 gm/l
	Rochelle salt (Rocheltex)	6% by vol.
	Brightener (CI Bright Copper)	0.2% by vol.
	(Potassium carbonate)	(37 gm/l)
Copper cyanide-2	Copper as metal	28 gm/l
R.O. Hull	Free cyanide (as NaCN)	8.8 gm/l
	Caustic	8.8 gm/l
	Rochelle salt (Roplex)	2-4% by vol.
	Brightener	none
Zinc cyanide	Zinc as metal	18-22 gm/l
R.O. Hull	Free cyanide (as NaCN)	37-52 gm/l
	Caustic	82-97 gm/l
	Brightener (ROHCO 532)	2.3 ml/l

TABLE 4. CONCENTRATION RANGES OF CONSTITUENTS IN TEST SOLUTIONS

Plating bath	Supplier	Analysis	Concentration range 5% Bath dilution	Concentration range 25% Bath dilution
Copper cyanide-1	MacDermid	conductivity (μmhos)	12,000 - 19,000	47,000 - 56,000
		copper (mg/l)	2,063 - 2,400	11,200 - 11,400
		cyanide (mg/l)	328 - 420	1,870 - 1,920
		pH	9.9 - 12.6	12.5 - 12.8
Copper cyanide-2	R.O. Hull	conductivity (μmhos)	18,000 - 19,900	45,000 - 53,000
		copper (mg/l)	1,280 - 1,420	4,640 - 6,810
		cyanide (mg/l)	840 - 1,200	3,300 - 5,400
		pH	11.0 - 11.9	11.1 - 11.8
Zinc cyanide	R.O. Hull	conductivity (μmhos)	16,000 - 22,000	48,500 - 62,000
		zinc (mg/l)	370 - 560	1,300 - 2,500
		cyanide (mg/l)	520 - 611	2,500 - 2,730
		pH	12.0 - 12.8	>13
Acid copper	Lea-Ronal	conductivity (μmhos)	27,000 - 37,000	92,000 - 125,000
		copper (mg/l)	10 - 1,800	4,600 - 7,200
		sulfate (mg/l)	10,000 - 14,000	20,000 - 56,000
		pH	1.2	0.55 - 0.9
Chromic acid	Udylite	conductivity (μmhos)	22,000 - 30,000	75,000 - 95,000
		hexavalent chromium (mg/l)	6,050 - 7,750	24,800 - 29,500
		pH	1.3 - 1.7	1.1 - 1.2



ACC - Accumulator
BPR - Back Pressure Regulator
DV - Drain Valve
F - Flow Indicator
HPS - High Pressure Switch
LPS - Low Pressure Switch
P - Pressure Indicator
PRV - Pressure Relief Valve
SOV - Solenoid Valve
TI/C - Temperature Indicator/Controller

Figure 2. Simplified flow schematic of test system.

OPERATING PRESSURE

The test system was operated in a total recycle mode with both composite and permeate returned to the feed tank. This mode of operation permits continuous long-term operation with only a relatively small volume of feed. In general, the membranes were tested at a temperature of 25°C and at the pressures and feed flow rates listed below:

Membrane	Operating pressure, atm	Feed circulation rate, lpm
PA-300	54.4	1.1
PBIL	54.4	1.1
NS-100	40.8	1.9
NS-200 (hollow-fiber)	54.4	1.9
NS-200 (tubular)	40.8	1.9
SPPO	40.8	0.19*
B-9	27.2	0.19
CA	40.8	1.9

Membrane performance was determined by measuring the flux and rejection for each membrane as a function of operating time. The flux was determined by measuring the permeate flow rate (graduate and stopwatch technique) for each membrane, and the rejection was determined by obtaining samples of the feed and the permeate from each membrane and analyzing for various bath constituents.

Tests were conducted with the four plating bath types for a total of 1000 hours each -- 500 hours at 5% of bath strength and 500 hours at 25% of bath strength. Prior to each 500-hour life test the membranes were evaluated for flux and rejection characteristics under standard conditions using sodium chloride feed solution (see Table 5). The salt rejections of the fresh membranes selected for testing always fell within the range of 80-99%. However, several of the membranes that were previously exposed to plating bath solutions exhibited rejections of less than 80%; these membranes were used in subsequent life tests only because new replacement membranes were not readily available.

ANALYSES

The methods of analysis used are listed in Table 6.

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* Additional turbulence was promoted in the SPPO test cell by a magnetic stirrer.

TABLE 5. MEMBRANE FLUX AND CONDUCTIVITY REJECTION VALUES OBTAINED UNDER STANDARD CONDITIONS PRIOR TO EXPOSURE TO 5% AND 25% PLATING BATH DILUTIONS[a]

Membrane type	Plating bath type and percent dilution							
	CuCN,5% flux/rej	CuCN,25% flux/rej	ZnCN,5% flux/rej	ZnCN,25% flux/rej	$CuSO_4$,5% flux/rej	$CuSO_4$,25% flux/rej	H_2CrO_4,5% flux/rej	H_2CrO_4,25% flux/rej
PA-300	37.4/96.5	48.6/98.5	--	40.7/97.5	64.5/98.1	12.4 /94.1[b]	16.3/97.3	14.5/96.8[b]
PBIL	37.4/96.0	31.1/98.0	--	12.2/99.6	17.0/99.19	14.8 /98.9[b]	19.0/97.7	10.4/97.2[b]
NS-100 (Avg of 5-6)	10.8/96.0	14.3/90.6[b]	18.2 /88.3[b]	17.0/91.6[b]	20.4/95.9	15.0 /89.0[b]	11.7/84.0[b,c]	49.4/45.0[b,c]
NS-200 (Tubular)	18.5/85.1	25.3/77.4[b]	--	--	--	--	--	--
NS-200 (Hollow-fiber, avg of 2)	13.8/99.45[d]	32.5/93.7[d,e]	98.0/98.3[d]	147/61.9[b,d]	61.5/97.9[d]	32.3/93.6[b,d]	20.8/86.7[b,c,d]	--
SPPO	27.2/80.0	15.4/54.8[b]	9.7/89.7[b]	19.4/14.5[b]	--	--	--	--
B-9	2.7/98.6[d]	1.8/97.1[b,d]	1.8/93.6[d]	1.3/93.9[b,d]	--	--	--	--
CA	--	--	--	--	--	13.0 /93.8	17.3 /96.9	22.6/89.7

[a] Feed = 1,500 mg/l NaCl, feed temperature = 25^{o}C, test duration = 24 hours. Operating pressures and feed circulation rates same as those listed on page 14 of this report. Flux and rejection values are given in l/m^2-hr and percent (%), respectively, unless otherwise specified.

[b] Membrane previously exposed to other plating bath solution(s).

[c] Only one membrane module tested.

[d] Flux in cc/min.

[e] One of two modules previously exposed to 5% CuCN solution.

TABLE 6. CHEMICAL ANALYSES

Constituent	Method	Procedure number
Conductivity	Conductivity meter	205*
Copper	Atomic absorption	301A*
Free cyanide	Specific ion electrode	Orion Manual
Hexavalent chrome	Colorimetric	307B*
Zinc	Atomic absorption	301A*
Sulfate	Turbidimetric	427C*

*From "Standard Methods for the Examination of Water and Waste Water," APHA, 14th Ed., 1975.

SECTION 6

RESULTS AND DISCUSSION

Results are presented below for all the plating bath rinsewaters in the order in which they were tested: copper cyanide, zinc cyanide, acid copper, and chromic acid. For clarity, only those data obtained at the start, mid-point, and end of each test are shown here*; complete membrane performance data are presented in the Appendix.

COPPER CYANIDE

Copper cyanide was the first plating bath to be tested, and during the initial tests the PA-300 and PBIL membranes were not yet available. Therefore the NS-100, NS-200, SPPO, and B-9 membranes were tested with the copper cyanide-1 bath listed in Table 3. The PA-300 and PBIL membranes were tested at a later time with the copper cyanide-2 bath listed in Table 3. These baths are reasonably similar in composition, which should permit a direct comparison of the results for all the membranes.

Table 7 gives the results for tests conducted with rinsewater at 5% of bath strength. The performance parameters listed for each membrane are conductivity rejection, copper rejection, free cyanide rejection, and flux. Rejections are given as a percentage of the feed concentration rejected, and flux, for the most part, is given in liters per square meter of membrane surface per hour (l/m^2-hr). For the hollow-fiber modules (NS-200 and B-9), the productivity is reported in cc/min since: 1) the exact surface area is difficult to determine, and 2) the productivity per unit membrane area could not be usefully compared to the flux for flat-sheet membranes because of the much higher packing density (m^2/m^3) possible with the hollow-fiber configuration.

The results in Table 7 indicate that all of the membranes tested exhibited reasonably stable flux and rejection for the 500-hour life test at 5% of bath strength. The apparent drop in conductivity rejection for the NS-100, NS-200, SPPO, and B-9 membranes is believed to have resulted from the absorption of atmospheric CO_2, which gradually changed the pH of the test solution and shifted the ionic equilibria in the direction of more poorly rejected species. The copper and free cyanide rejections for

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* Actual sampling times varied slightly from the 24, 250, and 500-hour times shown.

TABLE 7. MEMBRANE PERFORMANCE DURING LIFE TEST WITH COPPER CYANIDE RINSEWATER AT 5% OF BATH STRENGTH (pH=10-13)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	97.5	97.8	97.9
	Copper rejection, %	99.1	98.0	98.9
	Free cyanide rejection, %	98.2	96.7	99.2
	Flux, l/m^2-hr	25	16	15
PBIL	Conductivity rejection, %	85.4	90.0	90.5
	Copper rejection, %	99.3	98.5	98.7
	Free cyanide rejection, %	97.2	94.3	98.8
	Flux, l/m^2-hr	22	14	14
NS-100 (avg of 6)	Conductivity rejection, %	91.1	91.3	86.4
	Copper rejection, %	96.7	96.3	96.2
	Free cyanide rejection, %	91.4	90.0	94.3
	Flux, l/m^2-hr	11	8.7	9.3
NS-200 (tubular)	Conductivity rejection, %	86.8	88.9	74.7
	Copper rejection, %	97.6	97.6	97.5
	Free cyanide rejection, %	93.1	91.8	94.8
	Flux, l/m^2-hr	20	23	25
NS-200-1 (Hollow-fiber)	Conductivity rejection, %	96.1	96.1	87.4
	Copper rejection, %	99.0	99.8	99.7
	Free cyanide rejection, %	96.7	97.0	98.6
	Flux, cc/min	51	58	54
NS-200-2 (Hollow-fiber)	Conductivity rejection, %	94.2	95.9	(epoxy pot failure)
	Copper rejection, %	99.9	99.9	
	Free cyanide rejection, %	98.9	97.9	
	Flux, cc/min	26	35	
SPPO	Conductivity rejection, %	83.1	82.2	68.2
	Copper rejection, %	95.4	91.7	94.9
	Free cyanide rejection, %	88.3	79.9	92.8
	Flux, l/m^2-hr	44	5.1	7.5
B-9	Conductivity rejection, %	97.5	96.7	90.5
	Copper rejection, %	99.9	99.3	99.4
	Free cyanide rejection, %	98.5	97.0	98.8
	Flux, cc/min	1.8	2.0	1.7

these same membranes showed no significant decline during the test. In subsequent tests, polyethylene spheres were added to the feed tank to cut down the amount of CO_2 absorption, and the pH was more carefully controlled.

In general, the PA-300, NS-200, and B-9 membranes gave the highest rejections of conductivity, copper, and free cyanide. Relative to these membranes the PBIL membrane gave equivalent rejections of copper and free cyanide but somewhat lower conductivity rejections. The NS-100 and SPPO membranes gave lower rejections, particularly for copper and free cyanide.

The results for the 500-hour life test at 25% of bath strength are given in Table 8. At this concentration the PBIL, NS-200, SPPO, and B-9 membranes showed significant degradation in performance characteristics. For the PBIL membrane the flux decreased to an extremely low level upon exposure to the 25%-of-bath rinsewaters. The rejections are also poor (<90%), probably as the result of the very low flux. Both NS-200 hollow-fiber modules seemed to perform reasonably well until after about 250 hours. After this time one of the modules failed, resulting in gross leakage between the feed and permeate sides, and the other module exhibited sharp declines in conductivity, copper, and cyanide rejections. The SPPO membrane exhibited extremely low rejections of all species, and rejections decreased with time indicating degradation of the membrane material. For the B-9 membrane the rejections of copper and cyanide declined at a moderate rate, but the flux of the membrane declined rapidly.

Only the PA-300 and NS-100 membranes showed no serious degradation in performance during the tests with the 25%-of-bath rinsewaters. Of these two membranes the PA-300 is clearly superior. The conductivity rejection was quite good, the copper and free cyanide rejections were excellent, and the flux was high. Both flux and rejection were stable throughout the test. On the other hand, the NS-100 rejections were rather poor and the flux was only moderate. It is concluded that, for the treatment of copper cyanide rinsewaters, PA-300 is the best of the membranes tested.

ZINC CYANIDE

Following the tests with copper cyanide rinsewaters at 25% of bath strength, the NS-200 hollow-fiber modules were replaced with two new modules, and a new B-9 mini-permeator was installed. The NS-100 and SPPO membranes were not changed prior to the zinc cyanide tests. The PA-300 and PBIL membranes were not obtained in time for the zinc cyanide tests at 5% of bath strength but were tested at 25% of bath strength. However, samples of PA-300 and PBIL membrane were not obtained until after the test at 25% of bath strength had been initiated, and so the total exposure time for these membranes (391 hours for the PA-300 membrane and 315 hours for the PBIL membrane) were somewhat lower than the usual 500 hours. Use of NS-200 tubular modules was discontinued for these and subsequent tests because of difficulties encountered in their fabrication.

TABLE 8. MEMBRANE PERFORMANCE DURING LIFE TEST WITH COPPER CYANIDE RINSEWATER AT 25% OF BATH STRENGTH (pH=11-13)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	96.3	97.0	98.0
	Copper rejection, %	98.9	99.0	99.3
	Free cyanide rejection, %	98.6	99.0	98.8
	Flux, l/m^2-hr	15	13	13
PBIL	Conductivity rejection, %	60.6	61.3	56.7
	Copper rejection, %	89.3	86.4	75.4
	Free cyanide rejection, %	87.2	89.6	77.0
	Flux, l/m^2-hr	1.4	1.2	1.5
NS-100 (avg of 6)	Conductivity rejection, %	75.1	76.4	78.5
	Copper rejection, %	92.3	90.0	86.7
	Free cyanide rejection, %	92.6	91.0	90.2
	Flux, l/m^2-hr	7.1	7.0	6.8
NS-200 (tubular)	Conductivity rejection, %	51.9	46.4	42.2
	Copper rejection, %	88.7	72.8	54.5
	Free cyanide rejection, %	88.5	81.9	68.8
	Flux, l/m^2-hr	29	46	49
NS-200-1 (Hollow-fiber)	Conductivity rejection, %	69.2	73.2	16.7
	Copper rejection, %	98.0	96.0	0.0
	Free cyanide rejection, %	96.8	95.6	2.6
	Flux, cc/min	27.5	33	164
NS-200-2 (Hollow-fiber)	Conductivity rejection, %	77.9	77.9	55.6
	Copper rejection, %	99.0	97.7	66.1
	Free cyanide rejection, %	98.1	96.4	55.2
	Flux, cc/min	25	30	32
SPPO	Conductivity rejection, %	31.7	25.0	24.1
	Copper rejection, %	55.8	38.6	30.4
	Free cyanide rejection, %	68.5	63.9	39.1
	Flux, l/m^2-hr	15	13	6.8
B-9	Conductivity rejection, %	75.4	79.1	79.6
	Copper rejection, %	99.1	96.8	94.2
	Free cyanide rejection, %	98.4	95.7	92.4
	Flux, cc/min	0.6	0.2	0.1

Table 9 gives the results for the test with the 5%-of-bath rinsewater. All of the membranes tested appeared to be reasonably stable at this rinsewater concentration, and the commercially available B-9 membrane appeared to have the highest overall rejections. The low rejections for the SPPO membrane were probably the result of membrane deterioration during testing with 25%-of-bath copper cyanide rinsewater.

Results for the life-test with 25%-of-bath zinc cyanide rinsewater are given in Table 10. Again, all of the membranes tested exhibited reasonably stable performance. However, except for the PA-300 membrane the flux and/or rejections of the membranes were too low for cost-effective recovery of zinc cyanide rinsewaters at this concentration. The low conductivity rejections (generally <50% except for the PA-300 membrane) could be the result of the high concentration of hydroxide ion (which is difficult to reject) in the zinc cyanide rinsewaters. The PA-300 membrane, by contrast, gave excellent zinc and free cyanide rejections and a conductivity rejection much higher than any of the other membranes. In addition, the flux for PA-300 was quite high and appeared to be leveling off at a stable value of about 20-22 l/m^2-hr. On the basis of these data,it is concluded that,for the treatment of zinc cyanide rinsewaters,PA-300 is the best of the membranes tested.

ACID COPPER

Following the tests with the zinc cyanide rinsewater at 25% of bath strength, new PA-300, PBIL , and NS-200 membranes were installed in the system; however, the NS-100 membranes were not changed since new samples of these membranes were not available. The SPPO membrane was replaced prior to the test at 25% of bath strength, but during initial characterization tests with a sodium chloride solution, it was discovered that the new SPPO membrane was giving very poor rejections (<60%). This membrane was therefore eliminated from the test program. The B-9 membrane, which served as a reference membrane for alkaline solutions, was replaced by a CA reference membrane for tests with acid copper and chromic acid solutions. However, the CA membrane was not installed in the test system until after the test with 5%-of-bath acid copper rinsewater.

Results are shown in Table 11 for the life test with acid copper rinsewater at 5%-of-bath strength. The PBIL membrane exhibited exceptionally high rejections for all species,including conductivity (rejection >99%), and the rejections appeared to be stable. Although the flux (~ 7 l/m^2-hr) was rather low, it is believed that membranes with a higher flux could be prepared by varying the casting procedure. (This membrane was still in the process of being optimized when the test sample of membrane was obtained.) The NS-200 membrane also exhibited quite good performance characteristics during this test. Both the flux and rejection for this membrane appear adequate for successful application to copper sulfate rinsewaters. The PA-300 membrane gave a lower level of performance, and the NS-100 performed poorly in this test. It is possible that the copper ions in the rinsewater complexed with amine groups on the NS-100 membrane surface (and to a lesser extent on the PA-300 surface),resulting in poor rejection performances.

TABLE 9. MEMBRANE PERFORMANCE DURING LIFE TEST WITH ZINC CYANIDE RINSEWATER AT 5% OF BATH STRENGTH (pH= 12-13)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
NS-100 (avg of 6)	Conductivity rejection, %	72.8	75.2	81.0
	Zinc rejection, %	--	97.6	96.8
	Free cyanide rejection, %	88.4	92.7	91.1
	Flux, l/m^2-hr	11	11	9.5
NS-200 (avg of 2)	Conductivity rejection, %	66.4	57.1	67.0
	Zinc rejection, %	--	99.7	99.2
	Free cyanide rejection, %	96.8	95.4	90.4
	Flux, cc/min	100	116	96
SPPO	Conductivity rejection, %	25.0	28.8	41.0
	Zinc rejection, %	--	73.2	75.0
	Free cyanide rejection, %	50.0	63.1	53.2
	Flux, l/m^2-hr	12	8.8	8.5
B-9	Conductivity rejection, %	74.4	81.3	83.0
	Zinc rejection, %	--	99.95	99.8
	Free cyanide rejection, %	97.3	98.5	97.7
	Flux, cc/min	1.1	1.0	0.76

TABLE 10. MEMBRANE PERFORMANCE DURING LIFE TEST WITH ZINC CYANIDE RINSEWATER AT 25% OF BATH STRENGTH (pH>13)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	86.9	86.2	86.5
	Zinc rejection, %	99.1	--	99.3
	Free cyanide rejection, %	97.4	--	96.7
	Flux, l/m^2-hr	31	25	22
PBIL	Conductivity rejection, %	35.8	42.9	52.4
	Zinc rejection, %	--	--	95.9
	Free cyanide rejection, %	--	--	90.9
	Flux, l/m^2-hr	1.9	1.9	1.5
NS-100 (avg of 6)	Conductivity rejection, %	42.3	33.3	41.6
	Zinc rejection, %	87.9	76.1	72.1
	Free cyanide rejection, %	77.5	83.2	63.1
	Flux, l/m^2-hr	5.9	36	29
NS-200 (avg of 2)	Conductivity rejection, %	35.0	8.8	31.4
	Zinc rejection, %	93.2	95.0	93.6
	Free cyanide rejection, %	80.8	88.7	82.6
	Flux, cc/min	76	82	76
SPPO	Conductivity rejection, %	33.0	0.0	33.9
	Zinc rejection, %	40.0	43.8	53.9
	Free cyanide rejection, %	46.0	42.9	27.2
	Flux, l/m^2-hr	9.3	14	3.6
B-9	Conductivity rejection, %	29.0	41.2	53.2
	Zinc rejection, %	98.1	98.0	95.2
	Free cyanide rejection, %	89.2	94.9	90.6
	Flux, cc/min	0.13	0.19	0.10

TABLE 11. MEMBRANE PERFORMANCE DURING LIFE TEST WITH ACID COPPER RINSEWATER AT 5% OF BATH STRENGTH (pH=1.2)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	79.7	89.2	82.6
	Copper rejection, %	99.7	--	--
	Sulfate rejection, %	92.9	--	94.8
	Flux, l/m^2-hr	54	39	24
PBIL	Conductivity rejection, %	99.4	99.1	99.5
	Copper rejection, %	>99.9	--	--
	Sulfate rejection, %	99.9	--	98.0
	Flux, l/m^2-hr	6.1	12	6.8
NS-100 (avg of 6)	Conductivity rejection, %	20.0	49.6	24.2
	Copper rejection, %	94.7	--	--
	Sulfate rejection, %	49.8	--	57.1
	Flux, l/m^2-hr	19	27	32
NS-200 (avg of 2)	Conductivity rejection, %	97.3	97.3	96.1
	Copper rejection, %	99.9	--	--
	Sulfate rejection, %	98.7	--	98.1
	Flux, cc/min	43	38	31

For all membranes the rejections remained reasonably stable with operating time, but significant declines in flux were observed for the PA-300 and NS-200 membranes. Results for copper rejection are incomplete because of immersion deposition of copper on various stainless steel components within the test system. Copper concentrations in the feed solution decreased to very low levels following the first analysis at 24 hours.

Results are shown in Table 12 for the life test with acid copper rinsewater at 25% of bath strength. Immersion deposition was not a problem during this test since significant deposition had already occurred in the previous test, and copper concentrations in the feed were much higher. Again the PBIL membrane exhibited extremely high rejection of all species, but the flux was low. The flux and rejections were reasonably stable for the duration of the test,with the exception of sulfate rejection. The performance of the NS-200 membrane declined relative to that shown during the test at 5% of bath strength, but is still considered adequate for successful application of this membrane. The rejection of conductivity, copper, and sulfate increased during the test, and the flux appeared to stabilize after an initial decline. One of the two NS-200 modules had to be removed from the test system after 494 hours of operation due to weld failures in its stainless steel housing; therefore,data after this point could not be obtained. The PA-300 membrane exhibited excellent copper rejections, but the sulfate and conductivity rejections were low. The PA-300 rejections generally increased with operating time, but the flux decreased substantially during the life test to only one-third of its initial value. The NS-100 membranes exhibited very poor but stable rejections and a substantial decrease in flux over the duration of the test. The CA membrane was degraded by acid hydrolysis at the low pH of the rinsewater. This deterioration was evidenced by a substantial loss in rejection with a simultaneous increase in flux.

On the basis of these life tests, it is concluded that the NS-200 and PBIL membranes exhibit the best performance characteristics for treatment of acid copper rinsewaters.

CHROMIC ACID

Following the tests with acid copper, new PA-300 and PBIL membranes were installed in the test system, while one each of the same NS-100 and NS-200 membranes were retained. For the test at 25% of bath strength, use of the NS-200 was discontinued because of severe membrane degradation.

Results of the life test at 5% of bath strength are shown in Table 13. Of the membranes tested,only the PA-300 and PBIL gave stable performance. For the NS-100, NS-200, and CA membranes, the rejections decreased and the flux levels increased with operating time. This behavior is indicative of chemical degradation of the membrane surfaces.

Both the PA-300 and the PBIL membranes gave exceptionally stable flux and rejection performances throughout the life test. Of these two membranes, PA-300 performed better in both flux and rejection.

TABLE 12. MEMBRANE PERFORMANCE DURING LIFE TEST WITH ACID COPPER RINSEWATER AT 25% OF BATH STRENGTH (pH=0.6-0.9)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	78.1	82.6	90.0
	Copper rejection, %	99.9	>99.9	>99.9
	Sulfate rejection, %	76.5	92.2	83.0
	Flux, l/m^2-hr	22	10	6.1
PBIL	Conductivity rejection, %	99.2	98.3	99.1
	Copper rejection, %	>99.9	>99.9	>99.9
	Sulfate rejection, %	99.3	98.1	94.4
	Flux, l/m^2-hr	6.6	4.2	4.8
NS-100 (avg of 5)	Conductivity rejection, %	26.7	31.5	47.2
	Copper rejection, %	98.2	98.3	96.0
	Sulfate rejection, %	1.8	60.4	60.9
	Flux, l/m^2-hr	24	14	8.0
NS-200-1	Conductivity rejection, %	86.5	84.8	91.3
	Copper rejection, %	97.8	98.5	99.0
	Sulfate rejection, %	86.1	91.2	94.9
	Flux, cc/min	25	17	18.5
NS-200-2	Conductivity rejection, %	92.1	92.2	(weld failure in housing)
	Copper rejection, %	99.58	99.74	
	Sulfate rejection, %	92.8	97.2	
	Flux, cc/min	29.5	22	
CA	Conductivity rejection, %	92.3	57.8	39.1
	Copper rejection, %	99.9	97.2	83.3
	Sulfate rejection, %	92.0	74.9	49.2
	Flux, l/m^2-hr	8.0	15	17

TABLE 13. MEMBRANE PERFORMANCE DURING LIFE TEST WITH CHROMIC ACID RINSEWATER AT 5% OF BATH STRENGTH (pH=1.3-1.7)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	97.9	97.8	97.5
	Chromium (VI) rejection, %	98.8	98.9	98.6
	Flux, l/m^2-hr	13	17	15
PBIL	Conductivity rejection, %	95.0	95.0	94.1
	Chromium (VI) rejection, %	96.8	96.6	96.3
	Flux, l/m^2-hr	6.5	12	15
NS-100	Conductivity rejection, %	43.3	23.1	22.7
	Chromium (VI) rejection, %	51.0	42.9	67.4
	Flux, l/m^2-hr	17	61	75
NS-200	Conductivity rejection, %	28.3	0.0	0.0
	Chromium (VI) rejection, %	25.8	11.4	18.0
	Flux, cc/min	17	140	150
CA	Conductivity rejection, %	96.2	88.5	31.8
	Chromium (VI) rejection, %	97.3	91.4	42.0
	Flux, l/m^2-hr	19	46	102

Results for the life test with chromic acid at 25% of bath strength are shown in Table 14. The NS-100 and CA membranes degraded quite rapidly, and the evaluation of these membranes had to be discontinued shortly after the life test was initiated. The PBIL membrane was operated for the entire 500-hour test, but the rejections of both conductivity and hexavalent chrome declined substantially. On the other hand, the PA-300 membrane exhibited very good conductivity rejection and excellent chromium rejection during the entire life test. Although the flux was low, it appeared to be reasonably stable. It is believed that, despite the low flux, PA-300 could be used economically to recover chromic acid rinsewater because of the relatively high value of the recovered chemicals.

It is concluded that, of the membranes tested, PA-300 is the only one suitable for the treatment of chromic acid rinsewater.

TABLE 14. MEMBRANE PERFORMANCE DURING LIFE TEST WITH CHROMIC ACID RINSEWATER AT 25% OF BATH STRENGTH (pH=1.1-1.2)

Membrane	Performance parameter	Level at 24 hrs	Level at 250 hrs	Level at 500 hrs
PA-300	Conductivity rejection, %	95.9	97.3	97.5
	Chromium (VI) rejection, %	97.9	99.1	98.8
	Flux, l/m^2-hr	9.2	4.2	5.3
PBIL	Conductivity rejection, %	92.2	77.7	73.3
	Chromium (VI) rejection, %	96.1	90.8	83.4
	Flux, l/m^2-hr	6.8	4.8	8.7
NS-100	Conductivity rejection, %	15.7		
	Chromium (VI) rejection, %	33.2	(test discontinued)	
	Flux, l/m^2-hr	72		
CA	Conductivity rejection, %	75.3		
	Chromium (VI) rejection, %	85.7	(test discontinued)	
	Flux, l/m^2-hr	37		

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TABLE A-1. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 517-HR LIFE TEST WITH MACDERMID COPPER CYANIDE RINSEWATER AT 5% OF BATH STRENGTH (pH= 9.9-12.6): NS-100, NS-200, SPPO, AND B-9 MEMBRANES

Membrane	Performance parameter	5 Hrs.	6 Hrs.	14 Hrs.	21 Hrs.	70 Hrs.	85 Hrs.	92 Hrs.	111 Hrs.	116 Hrs.	136 Hrs.
NS-100 (avg of 6)	Conductivity rejection, %	--	--	89.4	91.1	--	92.2	--	93.2	--	93.4
	Copper rejection, %	--	--	--	96.7	--	--	--	--	--	--
	Free cyanide rejection, %	--	--	--	91.4	--	--	--	--	--	--
	Flux, l/m2-hr	--	--	10.9	10.9	--	10.2	--	9.5	--	9.3
NS-200	Conductivity rejection, %	--	--	84.2	86.8	--	91.9	--	93.7	--	94.2
	Copper rejection, %	--	--	--	97.6	--	--	--	--	--	--
	Free cyanide rejection, %	--	--	--	93.1	--	--	--	--	--	--
	Flux, l/m2-hr	--	--	20.5	20.4	--	22.2	--	22.8	--	22.8
NS-200-1 (Hollow-fiber)	Conductivity rejection, %	96.1	96.1	--	--	97.0	--	98.3	--	98.5	--
	Copper rejection, %	--	99.0	--	--	--	--	--	--	--	--
	Free cyanide rejection, %	--	96.7	--	--	--	--	--	--	--	--
	Flux, cc/min	30	30	--	--	30	--	35	--	35.5	--
NS-200-2 (Hollow-fiber)	Conductivity rejection, %	94.1	94.2	--	--	96.6	--	98.2	--	98.6	--
	Copper rejection, %	--	99.9	--	--	--	--	--	--	--	--
	Free cyanide rejection, %	--	98.9	--	--	--	--	--	--	--	--
	Flux, cc/min	26	26	--	--	30	--	35	--	35	--
SPPO	Conductivity rejection, %	--	--	78.3	83.1	--	46.7	--	92.3	--	90.8
	Copper rejection, %	--	--	--	95.4	--	--	--	--	--	--
	Free cyanide rejection, %	--	--	--	88.3	--	--	--	--	--	--
	Flux, l/m2-hr	--	--	50.3	44.5	--	16.1	--	11.0	--	11.5
B-9	Conductivity rejection, %	--	--	96.7	97.5	--	97.2	--	98.5	--	98.8
	Copper rejection, %	--	--	--	99.9	--	--	--	--	--	--
	Free cyanide rejection, %	--	--	--	98.5	--	--	--	--	--	--
	Flux, cc/min	--	--	2.8	1.8	--	1.1	--	2.5	--	2.3

TABLE A-1. (continued)

Membrane	Performance parameter	139 Hrs.	158 Hrs.	164 Hrs.	183 Hrs.	258 Hrs.	277 Hrs.	287 Hrs.	306 Hrs.	325 Hrs.	327 Hrs.
NS-100 (avg of 6)	Conductivity rejection, %	--	94.1	--	93.8	--	92.9	--	91.3	93.0	--
	Copper rejection, %	--	--	--	--	--	--	--	96.3	--	--
	Free cyanide rejection, %	--	--	--	--	--	--	--	90.0	--	--
	Flux, $1/m^2$-hr	--	9.3	--	9.3	--	8.8	--	8.7	8.7	--
NS-200 (tubular)	Conductivity rejection, %	--	95.0	--	95.2	--	94.1	--	88.9	91.4	--
	Copper rejection, %	--	--	--	--	--	--	--	97.6	--	--
	Free cyanide rejection, %	--	--	--	--	--	--	--	91.8	--	--
	Flux, $1/m^2$-hr	--	23.3	--	24.6	--	22.8	--	22.6	22.8	--
NS-200-1 (Hollow-fiber)	Conductivity rejection, %	98.7	--	98.8	--	98.4	--	96.1	97.0	--	97.5
	Copper rejection, %	--	--	--	--	--	--	99.8	--	--	--
	Free cyanide rejection, %	--	--	--	--	--	--	97.0	--	--	--
	Flux, cc/min	35	--	34	--	33	--	34	34	--	35
NS-200-2 (Hollow-fiber)	Conductivity rejection, %	98.7	--	98.8	--	98.4	--	95.9	97.1	--	97.8
	Copper rejection, %	--	--	--	--	--	--	99.9	--	--	--
	Free cyanide rejection, %	--	--	--	--	--	--	97.9	--	--	--
	Flux, cc/min	35	--	34	--	34	--	35	33	--	36
SPPO	Conductivity rejection, %	--	75.2	--	86.3	--	81.7	--	82.2	85.5	--
	Copper rejection, %	--	--	--	--	--	--	--	91.7	--	--
	Free cyanide rejection, %	--	--	--	--	--	--	--	79.9	--	--
	Flux, $1/m^2$-hr	--	10.0	--	8.7	--	8.1	--	5.1	7.8	--
B-9	Conductivity rejection, %	--	98.2	--	98.7	--	98.2	--	96.7	97.6	--
	Copper rejection, %	--	--	--	--	--	--	--	99.3	--	--
	Free cyanide rejection, %	--	--	--	--	--	--	--	97.0	--	--
	Flux, cc/min	--	2.3	--	2.1	--	2.3	--	2.0	2.0	--

TABLE A-1. (continued)

Membrane	Performance parameter	354 Hrs.	393 Hrs.	420 Hrs.	447 Hrs.	448 Hrs.	469 Hrs.	475 Hrs.	490 Hrs.	496 Hrs.	517 Hrs.
NS-100 (avg of 6)	Conductivity rejection,%	94.0	--	90.8	88.2	--	--	84.1	--	85.3	86.4
	Copper rejection, %	--	--	--	--	--	--	--	--	--	96.2
	Free cyanide rejection, %	--	--	--	--	--	--	--	--	--	94.3
	Flux, l/m²-hr	8.8	--	9.2	9.3	--	--	9.3	--	9.0	9.3
NS-200 (tubular)	Conductivity rejection,%	93.7	--	92.3	80.6	--	--	72.6	--	72.8	74.7
	Copper rejection, %	--	--	--	--	--	--	--	--	--	97.5
	Free cyanide rejection,%	--	--	--	--	--	--	--	--	--	94.8
	Flux, l/m²-hr	23.3	--	23.3	24.3	--	--	23.3	--	23.3	24.8
NS-200-1 (Hollow-fiber)	Conductivity rejection,%	--	90.6	91.0	--	84.6	86.1	--	87.4	--	--
	Copper rejection,%	--	--	--	--	--	--	--	99.7	--	--
	Free cyanide rejection,%	--	--	--	--	--	--	--	98.6	--	--
	Flux, cc/min	--	33	34	--	32	30	--	32	--	--
NS-200-2 (Hollow-fiber)	Conductivity rejection,%	--	90.6	91.3	--	84.6	0				
	Copper rejection,%	--	--	--	--	--	--	(EPOXY POT FAILED)			
	Free cyanide rejection,%	--	--	--	--	--	--				
	Flux, cc/min	--	34	35	--	33	625				
SPPO	Conductivity rejection,%	84.1	--	75.6	71.6	--	--	60.6	--	61.1	68.2
	Copper rejection,%	--	--	--	--	--	--	--	--	--	94.9
	Free cyanide rejection,%	--	--	--	--	--	--	--	--	--	92.8
	Flux, l/m²-hr	7.8	--	7.8	7.8	--	--	8.1	--	7.8	7.5
B-9	Conductivity rejection,%	98.0	--	94.1	94.2	--	--	89.1	--	90.0	90.5
	Copper rejection,%	--	--	--	--	--	--	--	--	--	99.4
	Free cyanide rejection,%	--	--	--	--	--	--	--	--	--	98.8
	Flux, cc/min	2.2	--	1.8	1.8	--	--	1.7	--	1.8	1.7

TABLE A-2. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 508-HR LIFE TEST WITH R.O. HULL COPPER CYANIDE RINSEWATER AT 5% OF BATH STRENGTH (pH= 11.0-11.9): PA-300 AND PBIL MEBRANES

Membrane	Performance parameter	1 Hr.	29 Hrs.	100 Hrs.	200 Hrs.	274 Hrs.	349 Hrs.	434 Hrs.	508 Hrs.
PA-300	Conductivity rejection,	97.2	97.5	97.9	97.3	97.8	97.9	97.5	97.9
	Copper rejection, %	--	99.06	--	--	98.0	--	--	98.9
	Free cyanide rejection, %	--	98.2	--	--	96.7	--	--	99.17
	Flux, l/m^2-hr	26.0	25.0	17.5	13.9	15.8	15.3	13.2	15.3
PBIL	Conductivity rejection, %	86.1	85.4	86.9	86.1	90.0	89.8	90.5	90.5
	Copper rejection, %	--	99.26	--	--	98.5	--	--	98.7
	Free cyanide rejection, %	--	97.2	--	--	94.3	--	--	98.8
	Flux, l/m^2-hr	31.2	22.6	17.5	13.5	13.6	13.5	11.2	14.4

TABLE A-3. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 463-HR LIFE TEST WITH MACDERMID COPPER CYANIDE RINSEWATER AT 25% OF BATH STRENGTH (pH= 12.5-12.8): NS-100, NS-200, SPPO, AND B-9 MEMBRANES

Membrane	Performance parameter	16 Hrs.	40 Hrs.	95 Hrs.	145 Hrs.	194 Hrs.	272 Hrs.	319 Hrs.	320 Hrs.	350 Hrs.	368 Hrs.	430 Hrs.	463 Hrs.
NS-100 (avg of 6)	Conductivity rejection,%	73.0	75.1	69.9	72.4	75.2	76.4	73.2	--	--	75.6	--	78.5
	Copper rejection,%	--	92.3	--	--	--	90.0	--	--	--	--	--	86.7
	Free cyanide rejection,%	--	92.6	--	--	--	91.0	--	--	--	--	--	90.2
	Flux, l/m²-hr	7.1	7.1	6.6	6.6	7.0	7.0	6.3	--	--	7.0	--	6.8
NS-200 (tubular)	Conductivity rejection,%	52.6	51.9	44.0	48.0	59.6	46.4	43.0	--	--	47.3	--	42.2
	Copper rejection,%	--	88.7	--	--	--	72.8	--	--	--	--	--	54.5
	Free cyanide rejection,%	--	88.5	--	--	--	81.9	--	--	--	--	--	68.8
	Flux, l/m²-hr	26.1	28.9	26.7	42.6	43.8	45.7	45.7	--	--	47.7	--	49.4
NS-200-1 (Hollow-fiber)	Conductivity rejection,%	67.0	69.2	66.0	67.8	73.1	73.2	--	69.1	5.2	--	16.7	--
	Copper rejection,%	--	98.0	--	--	--	96.0	--	--	--	--	0.0	--
	Free cyanide rejection,%	--	96.8	--	--	--	95.6	--	--	--	--	2.6	--
	Flux, cc/min	27	27.5	27	34	32	33	--	33	116	--	164	--
NS-200-2 (Hollow-fiber)	Conductivity rejection,%	76.3	77.9	74.0	73.5	78.8	77.9	--	75.5	78.5	--	55.6	--
	Copper rejection,%	--	99.0	--	--	--	97.7	--	--	--	--	66.1	--
	Free cyanide rejection,%	--	98.1	--	--	--	96.4	--	--	--	--	55.2	--
	Flux, cc/min	24	25	24.5	30	29	30	--	28.5	34	--	32	--
SPPO	Conductivity rejection,%	21.6	31.7	20.0	19.4	25.0	25.0	23.4	--	--	32.7	--	24.1
	Copper rejection,%	--	55.8	--	--	--	38.6	--	--	--	--	--	30.4
	Free cyanide rejection,%	--	68.5	--	--	--	63.9	--	--	--	--	--	39.1
	Flux, l/m²-hr	76.1	14.8	13.4	15.6	14.1	12.9	11.2	--	--	9.2	--	6.8
B-9	Conductivity rejection,%	73.2	75.4	60.0	76.5	80.8	79.1	79.8	--	--	85.4	--	79.6
	Copper rejection,%	--	99.1	--	--	--	96.8	--	--	--	--	--	94.2
	Free cyanide rejection,%	--	98.4	--	--	--	95.7	--	--	--	--	--	92.4
	Flux, cc/min	0.64	0.60	0.60	0.27	0.26	0.22	0.28	--	--	0.28	--	0.13

TABLE A-4. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 555-HR LIFE TEST WITH R.O. HULL COPPER CYANIDE RINSEWATER AT 25% OF BATH STRENGTH (pH= 11.1-11.8): PA-300 AND PBIL MEMBRANES

Membrane	Performance parameter	1 Hr.	25 Hrs.	96 Hrs.	217 Hrs.	242 Hrs.	314 Hrs.	409 Hrs.	529 Hrs.
PA-300	Conductivity rejection, %	95.5	96.3	96.8	97.0	--	96.7	96.2	98.0
	Copper rejection, %	--	98.9	--	--	99.03	--	--	99.31
	Free cyanide rejection, %	--	98.6	--	--	98.96	--	--	98.8
	Flux, l/m^2-hr	15.0	15.0	13.4	12.5	--	9.7	7.3	13.4
PBIL	Conductivity rejection, %	--	60.6	62.9	61.3	--	59.0	52.8	56.7
	Copper rejection, %	--	89.3	--	--	86.4	--	--	75.4
	Free cyanide rejection, %	--	87.2	--	--	89.6	--	--	77.0
	Flux, l/m^2-hr	--	1.3	1.1	1.2	--	1.3	1.2	1.5

TABLE A-5. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 509-HR LIFE TEST WITH ZINC CYANIDE RINSEWATER AT 5% OF BATH STRENGTH (pH=12.0-12.8)

Membrane	Performance parameter	29 Hrs.	119 Hrs.	194 Hrs.	264 Hrs.	310 Hrs.	358 Hrs.	455 Hrs.	509 Hrs.
NS-100 (avg of 6)	Conductivity rejection, %	72.8	76.2	76.4	75.2	75.5	74.9	76.2	81.0
	Zinc rejection, %	--	--	--	97.6	--	--	--	96.8
	Free cyanide rejection, %	88.4	--	--	92.7	--	--	--	91.1
	Flux, l/m^2-hr	11.4	11.9	12.9	10.9	10.0	11.9	10.0	9.5
NS-200 (avg of 2)	Conductivity rejection, %	66.4	--	69.3	57.1	59.9	54.2	58.4	67.0
	Zinc rejection, %	--	--	--	99.7	--	--	--	99.2
	Free cyanide rejection, %	96.8	--	--	95.4	--	--	--	90.4
	Flux, cc/min	100	--	101	116	105	104	104	96
SPPO	Conductivity rejection, %	25.0	--	25.6	28.8	27.1	21.0	27.8	41.0
	Zinc rejection, %	--	--	--	73.2	--	--	--	75.0
	Free cyanide rejection, %	50.0	--	--	63.1	--	--	--	53.2
	Flux, l/m^2-hr	11.5	11.2	11.2	8.8	7.3	9.8	8.3	8.5
B-9	Conductivity rejection, %	74.4	84.2	81.4	81.3	84.4	78.9	78.9	83.0
	Zinc rejection, %	--	--	--	99.95	--	--	--	99.8
	Free cyanide rejection, %	97.3	--	--	98.5	--	--	--	97.7
	Flux, cc/min	1.1	1.2	1.1	1.0	1.0	1.1	0.77	0.76

TABLE A-6. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 533-HR LIFE TEST WITH ZINC CYANIDE RINSEWATER AT 25% OF BATH STRENGTH (pH>13)

Membrane	Performance parameter	31 Hrs.	100 Hrs.	145 Hrs.	218 Hrs.	287 Hrs.	384 Hrs.	533 Hrs.
PA-300	Conductivity rejection, %				86.9	85.4	86.2	86.5
	Zinc rejection, %		(TEST INITIATED AT 142 HRS)		99.1	--	--	99.3
	Free cyanide rejection, %				97.4	--	--	96.7
	Flux, $1/m^2$-hr				29.9	25.5	24.8	22.8
PBIL	Conductivity rejection, %					35.8	42.9	52.4
	Zinc rejection, %		(TEST INITIATED AT 218 HRS)			--	--	95.9
	Free cyanide rejection, %					--	--	90.9
	Flux,					1.9	1.8	1.5
NS-100 (avg of 6)	Conductivity rejection, %	42.3	--	31.6	33.3	42.4	36.9	41.6
	Zinc rejection, %	87.9	--	--	76.1	--	--	72.1
	Free cyanide rejection, %	77.5	--	--	83.2	--	--	63.1
	Flux, $1/m^2$-hr	5.9	31.9	39.2	35.1	26.3	24.6	28.7
NS-200 (avg of 2)	Conductivity rejection, %	35.0	--	24.2	8.8	20.3	19.3	31.4
	Zinc rejection, %	93.2	--	--	95.0	--	--	93.6
	Free cyanide rejection, %	80.8	--	--	88.7	--	--	82.6
	Flux, cc/min	76	97.5	89.7	82	92	90	76
SPPO	Conductivity rejection, %	33.0	--	10.6	0	17.9	25.2	33.9
	Zinc rejection,%.	40.0	--	--	43.8	--	--	53.9
	Free cyanide rejection, %	46.0	--	--	42.9	--	--	27.2
	Flux,$1/m^2$-hr	9.3	12.4	8.3	13.8	9.7	7.8	3.6
B-9	Conductivity rejection, %	29.0	--	22.4	41.2	4.0	62.2	53.2
	Zinc rejection, %	98.1	--	--	98.0	--	--	95.2
	Free cyanide rejection, %	89.2	--	--	94.9	--	--	90.6
	Flux, cc/min	--	0.13	0.19	0.19	0.90	0.12	0.10

TABLE A-7. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 550-HR LIFE TEST WITH ACID COPPER RINSEWATER AT 5% OF BATH STRENGTH (pH=1.2)

Membrane	Performance parameter	1 Hr.	20 Hrs.	94 Hrs.	242 Hrs.	311 Hrs.	453 Hrs.	550 Hrs.
PA-300	Conductivity rejection, %	75.6	79.7	81.5	89.2	84.6	83.4	82.6
	Copper rejection, %	--	99.73	--	--	--	--	--
	Sulfate rejection, %	--	92.9	--	--	--	--	94.8
	Flux, l/m^2-hr	40.7	54.3	37.4	38.7	47.2	38.0	23.4
PBIL	Conductivity rejection, %	99.51	99.41	97.1	99.11	98.2	97.8	99.52
	Copper rejection, %	--	>99.96	--	--	--	--	--
	Sulfate rejection, %	--	99.85	--	--	--	--	98.0
	Flux, l/m^2-hr	6.1	--	9.2	12.2	11.2	11.5	6.8
NS-100 (avg of 6)	Conductivity rejection, %	27.3	20.0	28.4	49.6	32.4	29.2	24.2
	Copper rejection, %	--	94.7	--	--	--	--	--
	Sulfate rejection, %	--	49.8	--	--	--	--	57.1
	Flux, l/m^2-hr	20.0	19.4	23.1	26.5	31.2	29.7	32.1
NS-200 (avg of 2)	Conductivity rejection, %	98.0	97.3	96.9	97.3	96.7	95.9	96.1
	Copper rejection, %	--	99.88	--	--	--	--	--
	Sulfate rejection, %	--	98.7	--	--	--	--	98.1
	Flux, cc/min	32.5	43.2	37.0	38.0	42.5	42.5	30.9

TABLE A-8. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 610-HR LIFE TEST WITH ACID COPPER RINSEWATER AT 25% OF BATH STRENGTH (pH=0.55-0.9)

Membrane	Performance parameter	1 Hr.	20 Hrs.	246 Hrs.	323 Hrs.	563 Hrs.	610 Hrs.
PA-300	Conductivity rejection, %	73.1	78.1	81.6	82.6	90.0	90.0
	Copper rejection, %	--	99.87	--	99.97	--	>99.99
	Sulfate rejection, %	--	76.5	--	92.2	--	83.0
	Flux, l/m^2-hr	25.3	21.2	8.9	10.4	6.8	6.0
PBIL	Conductivity rejection, %	98.1	99.22	99.04	98.3	99.30	99.09
	Copper rejection, %	--	>99.99	--	99.97	--	99.96
	Sulfate rejection, %	--	99.32	--	98.1	--	94.4
	Flux, l/m^2-hr	6.6	--	3.5	4.2	4.9	4.7
NS-100 (avg of 5)	Conductivity rejection, %	30.7	26.7	29.4	31.5	42.8	47.2
	Copper rejection, %	--	98.2	--	98.3	--	96.0
	Sulfate rejection, %	--	1.8	--	60.4	--	60.9
	Flux, l/m^2-hr	28.0	24.4	20.5	14.3	10.3	8.0
NS-200-1	Conductivity rejection, %	86.0	86.5	88.4	84.8	90.0	91.3
	Copper rejection, %	--	97.8	--	98.5	--	99.03
	Sulfaterejection, %	--	86.1	--	91.2	--	94.9
	Flux, cc/min	24.4	24.9	18.6	16.8	18.0	18.5
NS-200-2	Conductivity rejection, %	92.6	92.1	93.8	92.2	--	--
	Copper rejection, %	--	99.58	--	99.74	--	--
	Sulfate rejection, %	--	92.8	--	97.2	--	--
	Flux, cc/min	29.0	29.5	22.5	22.0	--	--
CA	Conductivity rejection, %	88.2	92.3	74.4	57.8	37.0	39.1
	Copper rejection, %	--	99.90	--	97.2	--	83.3
	Sulfate rejection, %	--	92.0	--	74.9	--	49.2
	Flux, l/m^2-hr	15.1	8.0	13.2	14.7	22.6	16.7

TABLE A-9. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 530-HOUR LIFE TEST WITH CHROMIC ACID RINSEWATER AT 5% OF BATH STRENGTH (pH=1.3-1.7)

Membrane	Performance parameter	5 Hrs.	31 Hrs.	99 Hrs.	196 Hrs.	243 Hrs.	387 Hrs.	506 Hrs.
PA-300	Conductivity rejection, %	93.2	97.9	98.1	97.9	97.8	97.9	97.5
	Chromium (VI) rejection, %	--	98.8	--	--	98.9	--	98.6
	Flux, l/m^2-hr	--	13.1	16.3	17.7	17.3	9.2	15.1
PBIL	Conductivity rejection, %	92.4	95.0	94.3	95.7	95.0	94.5	94.1
	Chromium (VI) rejection, %	--	96.8	--	--	96.6	--	96.3
	Flux, l/m^2-hr	--	6.4	9.1	11.5	12.2	8.5	14.6
NS-100	Conductivity rejection, %	68.0	43.3	42.7	21.4	23.1	20.4	22.7
	Chromium (VI) rejection, %	--	51.0	--	--	42.9	--	67.4
	Flux, l/m^2-hr	--	16.3	54.8	62.0	62.0	67.6	75.0
NS-200	Conductivity rejection, %	72.0	28.3	0.0	0.0	0.0	0.0	0.0
	Chromium (VI) rejection, %	--	25.8	--	--	11.4	--	18.0
	Flux, cc/min	--	17.0	54.3	125	140	150	150
CA	Conductivity rejection, %	96.0	96.2	93.1	88.9	88.5	57.6	31.8
	Chromium (VI) rejection, %	--	97.3	--	--	91.4	--	42.0
	Flux, l/m^2-hr	--	18.0	44.5	33.8	45.2	90.2	101.5

TABLE A-10. COMPLETE MEMBRANE PERFORMANCE DATA OBTAINED DURING 563-HOUR LIFE TEST WITH CHROMIC ACID RINSEWATER AT 25% OF BATH STRENGTH (pH=1.1-1.2)

Membrane	Performance parameter	1 Hr.	25 Hrs.	121 Hrs.	202 Hrs.	298 Hrs.	374 Hrs.	470 Hrs.	562 Hrs.
PA-300	Conductivity rejection, %	97.3	95.9	97.5	98.0	97.3	98.6	97.2	97.5
	Chromium (VI) rejection, %	--	97.9	--	--	99.12	--	--	98.8
	Flux, l/m^2-hr	21.6	9.1	5.7	4.4	4.3	4.3	8.5	5.3
PBIL	Conductivity rejection, %	92.8	92.2	90.5	90.7	77.7	82.2	77.6	73.3
	Chromium (VI) rejection, %	--	96.1	--	--	90.8	--	--	83.4
	Flux, l/m^2-hr	26.9	6.8	6.8	4.7	4.7	4.9	10.1	8.6
NS-100	Conductivity rejection, %	38.8	15.7	(TEST DISCONTINUED)					
	Chromium (VI) rejection, %	--	33.2						
	Flux, l/m^2-hr	33.8	71.8						
CA	Conductivity rejection, %	85.9	75.3	(TEST DISCONTINUED)					
	Chromium (VI) rejection, %	--	85.7						
	Flux, l/m^2-hr	13.1	36.7						

TECHNICAL REPORT DATA
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1. REPORT NO. EPA-600/2-80-084	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE EVALUATION OF REVERSE OSMOSIS MEMBRANES FOR TREATMENT OF ELECTROPLATING RINSEWATER		5. REPORT DATE May 1980 issuing date 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Kenneth J. McNulty and Peter R. Hoover Walden Division of Abcor, Inc., Wilmington, MA 01887		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS The American Electroplaters' Society 1201 Louisiana Avenue Winter Park, Florida 32789		10. PROGRAM ELEMENT NO. 1BB610 11. CONTRACT/GRANT NO. R804311
12. SPONSORING AGENCY NAME AND ADDRESS Industrial Environmental Research Laboratory - Cinn, OH Office of Research and Development U.S. Environmental Protection Agency Cincinnati, OH 45268		13. TYPE OF REPORT AND PERIOD COVERED Final 3/1/76 - 10/19/77 14. SPONSORING AGENCY CODE EPA/600/12

15. SUPPLEMENTARY NOTES

Project Officer: Mary Stinson (201) 321-6683

16. ABSTRACT

Because of the limited pH range over which current commercially available reverse osmosis membranes can be applied, a test program was initiated to define the applicability of new membrane materials to the treatment of rinsewaters with extreme pH levels and high oxidant levels (chromic acid). Life tests were conducted with the PA-300, PBIL, NS-100, NS-200, SPPO, B-9, and CA membranes on rinsewaters from copper cyanide, zinc cyanide, acid copper, and chromic acid plating baths. The PA-300 membrane exhibited suprior performance for the treatment of copper cyanide, zinc cyanide, and chromic acid rinsewaters, and further development and demonstration of this membrane is recommended. The NS-200 and PBIL membranes exhibited the best performance for treatment of acid copper rinsewaters. Efforts are underway to commercialize all three of the selected membranes (PA-300, NS-200, and PBIL).

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Electroplating Waste Treatment Reverse Osmosis	Reverse Osmosis Membranes: PA-300 PBIL NS-100 NS-200 SPPO	13B
18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC	19. SECURITY CLASS *(This Report)* UNCLASSIFIED 20. SECURITY CLASS *(This page)* UNCLASSIFIED	21. NO. OF PAGES 51 22. PRICE

United States
Environmental Protection
Agency

Center for Environmental Research
Information
Cincinnati OH 45268

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