United States
Environmental Protection
Agency

Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

EPA-600/2-80-055
March 1980

Research and Development

EPA

Closed-cycle Textile Dyeing: Full-scale Hyperfiltration Demonstration (Design)

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March 1980

Closed-cycle Textile Dyeing: Full-scale Hyperfiltration Demonstration (Design)

by

Craig A. Brandon (Carre, Inc.)

LaFrance Industries
LaFrance, South Carolina 29656

Grant No. S805182
Program Element No. 1BB610

EPA Project Officer: Max Samfield

Industrial Environmental Research Laboratory
Office of Environmental Engineering and Technology
Research Triangle Park, NC 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, DC 20460

ABSTRACT

The report describes the first (design) phase of a full-scale demonstration of hyperfiltration for closed-cycle operations of a LaFrance Industries dye house. (The remaining three phases are installation, operation, and maintenance.) The decision to demonstrate the process was based on earlier projects that showed hyperfiltration to be potentially economical for recycle/reuse of energy, water, and chemicals in textile preparation dyeing, and wet finishing. On-site pilot tests of three hyperfiltration modules led to the selection of the Mott-Brandon ZOPA module. Representative wash waters from LaFrance dyeing operations were characterized as a basis for demonstration equipment design. The dye range is to be converted to counterflow with a water flow rate of 50 gpm at 82 C, with 96% of the wash water recovered as permeate for direct recycle. Reuse and/or disposal of the concentrate and dye pad residuals will require further study. Payback period, without credit for chemicals recovery, is estimated to be 5.2 years.

SUMMARY

Hyperfiltration is a pressure driven membrane separation process that has been shown to be potentially economical for recycle of energy, water, and chemicals within textile preparation, dyeing and wet finishing operations. This demonstration project follows three prior research and development projects which (1) showed reusability of membrane permeate (purified water) and the residual concentrate from hyperfiltration of a dye house wastewater, (2) general reusability of permeates for wastewater from eight selected textile plants, and (3) the potential for conservation of about 50 percent of energy used in wet processing by direct recycle of permeate at full process temperature. The three projects led to the recommendation for this full scale demonstration of hyperfiltration for closed cycle operation of a representative textile unit process - a dye range.

La France Industries was the site of the first project mentioned above and has been involved in all the subsequent projects. This dye house incorporates both batch dyeing (dye becks) and continuous dyeing (a dye range). The La France site is centrally located for the textile industry in the Southeast. The textile industry is heavily concentrated in the Southeast.

The demonstration project consists of four phases:

Phase I - Design Phase
Phase II - Equipment Installation Phase
Phase III - Demonstration Operation Phase
Phase IV - Evaluation and Reporting Phase

This report presents the results of Phase I. The textile process, a dye range, is described and the wash water is characterized (chemically) for each of several fabrics/dye classes processed. The data from on-site pilot tests are presented for three types of hyperfiltration modules. The design of the recovery system is described. The economics of the recovery system are estimated based on the quoted price for installation and projected operating costs and savings for the dynamic membrane system selected for the demonstration.

The dye range will be converted to counterflow with an expected water flow rate of 190 ℓ/min (50 gallons per minute). The principal fabrics dyed are cotton, acrylic, nylon, rayon and polyester. The dye classes used are: direct, dispersed, acid, premetalized, basic and fiber reactive. The dyes and auxiliary chemicals will result in a wash water composition of: COD (mg/ℓ) = 1200, color (ADMI) = 1750, pH of 5 to 10.5, total solids (mg/ℓ) = 1400, suspended solids (mg/ℓ) = 45, and hardness (mg/ℓ) = 30. The wash water temperature is expected to be 82°C.

The three high temperature hyperfiltration membrane module types that were pilot tested included two that utilized dynamically formed zirconium oxidepolyacrylate membranes; one with these membranes deposited on the outside of carbon tubes and one with the membranes deposited on the internal surface of sintered stainless steel tubes. The third module was a spiral-wound poly(ether)amid membrane. All these modules provided a permeate satisfactory for recycle. The degree of pretreatment, i.e., prefiltration and temperature control can be designed based on the pilot test results.

The recovery system is designed with $23m^3$ (6000 gallon) tanks for the wash water and permeate so that the membrane unit can operate continuously while the dye range undergoes periodic shutdowns between production lots. All effluent from the range, except steam condensate, is collected into the recovery system. Ninety-six percent of the wash water is recovered as permeate for direct recycle as wash water. The unused dye pad liquor goes directly to the concentrate tank and is not processed by the membranes. The control and monitoring instrumentation provide for automatic operation and complete documentation of water and energy savings achieved by recycle. The reuse and/or disposal of concentrate and dye pad residuals will be studied throughout the later phases of this project.

The economics of recovery are based on the installed cost (quoted prices) of approximately $400,000 and the operating costs estimated for the Single Pass membrane system utilizing dynamic membranes on sintered stainless steel tubing. The payback period, calculated by the Riegel Textile Corporation's standard procedure, is 3.8 years with chemical recovery and 5.2 years without chemical recovery. The annual energy savings are about 2×10^{10} Btu per year. The operating costs are estimated based on February 1979 prices for energy and materials.

The equipment selection and decision to proceed with the demonstration were made on April 6, 1979.

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ENGLISH METRIC CONVERSION TABLE*

To Convert From	To	Multiply by
Inch	Meter	2.54×10^{-2}
Feet	Meter	3.05×10^{-1}
Square inch	Square meter	6.45×10^{-4}
Square feet	Square meter	9.29×10^{-2}
Cubic feet	Cubic meter	2.83×10^{-2}
Gallon	Cubic meter	3.79×10^{-3}
Pound	Kilogram	4.54×10^{-1}
Pound per sq. inch (psi)	Atmosphere	6.80×10^{-2}
Horsepower (Hp)	Watt	7.46×10^{2}
Gallon per day	Cubic meter per day	3.79×10^{-3}
Gallon per minute (GPM)	Cubic meter per day	5.45
Gallon per sq. ft-day (GFD)	Cubic meter per sq. meter-day	4.10×10^{-2}
Gallon per minute per sq. ft.	Cubic meter per sq. meter-day	5.87×10^{1}

*The units most familiar to the projected readership of this report have been maintained.

ACKNOWLEDGMENTS

This study was conducted by a team and major contributions were made by a number of people. The cooperation and assistance of the La France staff members is particularly acknowledged; Mike Drummon, Perry Lockridge, Charles Smith, and many machine operators and laboratory technicians. Dr. Jim Bostic, Jr., Mr. Ted Meyer and Mr. Ernie Freeman of Riegel Corporate staff provided valuable advise.

This demonstration is an interagency program and thus benefited from the guidance of Dr. Max Samfield, U.S. Environmental Protection Agency, as Principal Project Officer; John Rossmeissel, Department of Energy, and Frank Coley, Department of the Interior, as Project Officers, and Mr. Robert Mournigham, U.S. Environmental Protection Agency, as Technical Advisor. Dr. J. S. Johnson, Jr., of the Oak Ridge National Laboratory has served as membrane technology consultant on this and all the previous related R and D projects. A special acknowledgment is given to Dr. K. C. Channabasappa of the Department of Interior whose knowledge of the membrane technolgy was instrumental in initiating the project. His untimely death was a major loss to the entire project team.

CARRE, Inc. provided overall program management and developed the conceptual design of the recovery system. The contributions of staff members at CARRE, Inc. are acknowledged. Staff members making major contributions are Drs. J. L. Gaddis and H. G. Spencer; Donald K. Todd, James Schubert and Kris Turschmid, engineers, and Roger Hunt and Don King, technicians. Dr. J. J. Porter and Mr. Grant Goodman of Texidyne, Inc. make significant contributions in providing chemical analyses and consultation. Dr. E. Harrison served as a consultant on control and instrumentation.

The detailed design and bid specifications were provided by J. E. Sirrine Company. The membrane equipment vendors made significant contributions in technical comments and advice.

Don Hill
Project Director

Craig A. Brandon
Project Coordinator

SECTION 1

INTRODUCTION

The technical feasibility of renovating textile wastewaters for recycle has been shown in a series of government sponsored research grants with the textile industry that began in 1972. The current project to demonstrate closed-cycle operation of a production dye range at La France Industries will determine the practicality of the previous feasibility research. This report summarizes the results of Phase I of the demonstration program. The wide application of hot process effluent recycle has a large potential impact on energy conservation and pollution abatement.

IMPACT OF RECYCLE

High temperature hyperfiltration applied to hot industrial process effluents will result in significant reduction in the fresh water demand and waste treatment requirements as well as achieving energy and material conservation.

The hot process water ($\sim 100^{\circ}C$) is discharged at a rate of about 2×10^{12} gallons per year* (7.6×10^{9} m^3/year). In the temperature regime to $100^{\circ}C$ (where high temperature hyperfiltration has already been evaluated) direct recycle can achieve energy conservation of 2.4×10^{15} Btu/year. For example, the estimated potential in energy and material resource conservation by recycle in the U.S. textile industry in 1978 is shown in Table 1 (1). The energy conservation for the textile industry is $\sim 15 \times 10^{6}$ barrels of oil (equivalent) per year. Other industries represent much larger potential energy savings through hot water recycle.

The reduction of the demand for fuel, industrial chemicals and treated process water has a favorable environmental impact all along the manufacturing chain from extraction through distribution of finished goods. The direct environmental impact where closed-cycle operation eliminates contamination discharges is obvious. The specific potential for reduced pollution is nearly identical with the list of potential resource savings.

BACKGROUND

Recycle is an obvious and perhaps necessary, method of conserving the available water supplies, whether from natural sources or from desalination

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* The units most familiar to the projected readership of this report have been maintained. A table of conversions is included in this report.

TABLE 1. CONSERVATION AND RESOURCE RECOVERY POTENTIAL FOR THE U.S. TEXTILE INDUSTRY (1)

	Water Discharge (10^3 kgal/d)	Dyes (10^3 lb/d)	Auxiliary (10^3 lb/d)	Salt (10^3 lb/d)	Process Thermal Energy (10^3 Btu/d)
Study Total[a]	31.4	59.80	27.50[b]	26.9	37.90
Industry Total	706[c]	2191	594	581	784[d]
Recycle Potential	635	129	489	481	352[e]
Estimated Annual Savings[f] (10^2)	79	95	24	2.6	264

[a] EPA Grant No. S802973.
[b] Exclusive of 176,000 pounds of NaOH used daily at these plants.
[c] 1972 census of manufacturers, assuming 250 days/year on stream.
[d] Industry estimate of 50 x 10^6 barrels/year; 70% in wet finishing.
[e] 15 x 10^6 barrels/year.
[f] Unit costs: water @ \$0.5 kgal; dye @ \$3/#; auxiliary chemicals @ \$0.2/#; salt @ \$44/ton; process steam @ \$4/10^6 Btu.

facilities. The engineering problem associated with the application of membrane technology to industrial effluents is different from the problems associated with desalination (2). Major differences include:

- Feed water characteristics that vary from strongly basic to strongly acid,
- Pretreatment that must not destroy the chemicals for recycle,
- High volumetric recovery to achieve direct reuse at process conditions,
- Operation at high temperature for direct recycle at process conditions, and
- Relatively low permeate quality requirements (in some cases) for direct recycle as industrial process water.

Because some of the separation results achieved with complex industrial effluents by even "reverse osmosis" membranes is really very fine filtration, the terms "hyper" filtration and "ultra" filtration are used to more accurately describe the membrane technology being demonstrated. Figure 1 gives a simplified view of the range of filtration from conventional to "hyper."

This demonstration program is the culmination of a series of cooperative research projects conducted by the present participants for the U.S. Environmental Protection Agency and the textile industry. From 1972 through 1977 (Table 2) many configurations of commercially available ultrafiltration and hyperfiltration equipment have been evaluated with both total plant composite wastewater and unit manufacturing process effluents. The research has involved both field and laboratory tests.

PURPOSE AND SCOPE

The purpose of the demonstration proejct is to design, install, and operate a full scale commercially available membrane system to evaluate the practicality of the use of high temperature hyperfiltration in the textile industry.

Because of the background of research, the textile industry was chosen for the demonstration. Both a dye range and atmospheric beck, two types of equipment representative of the two broad categories of dyeing and wet processes of textiles, were considered. The scope of project includes the following activities divided among four phases (the phases are identified in parantheses):

- A system design based on on-site test results and including all engineering details and commercial quotations for installed cost (Phase II)
- Installation and start-up of a full scale hyperfiltration unit (Phase II)
- Evaluation of any long term effects on the manufacturing quality control and productivity of the dye range and determination of operating costs (Phase III)
- Evaluation of project results for economics of hyperfiltration (Phase IV)

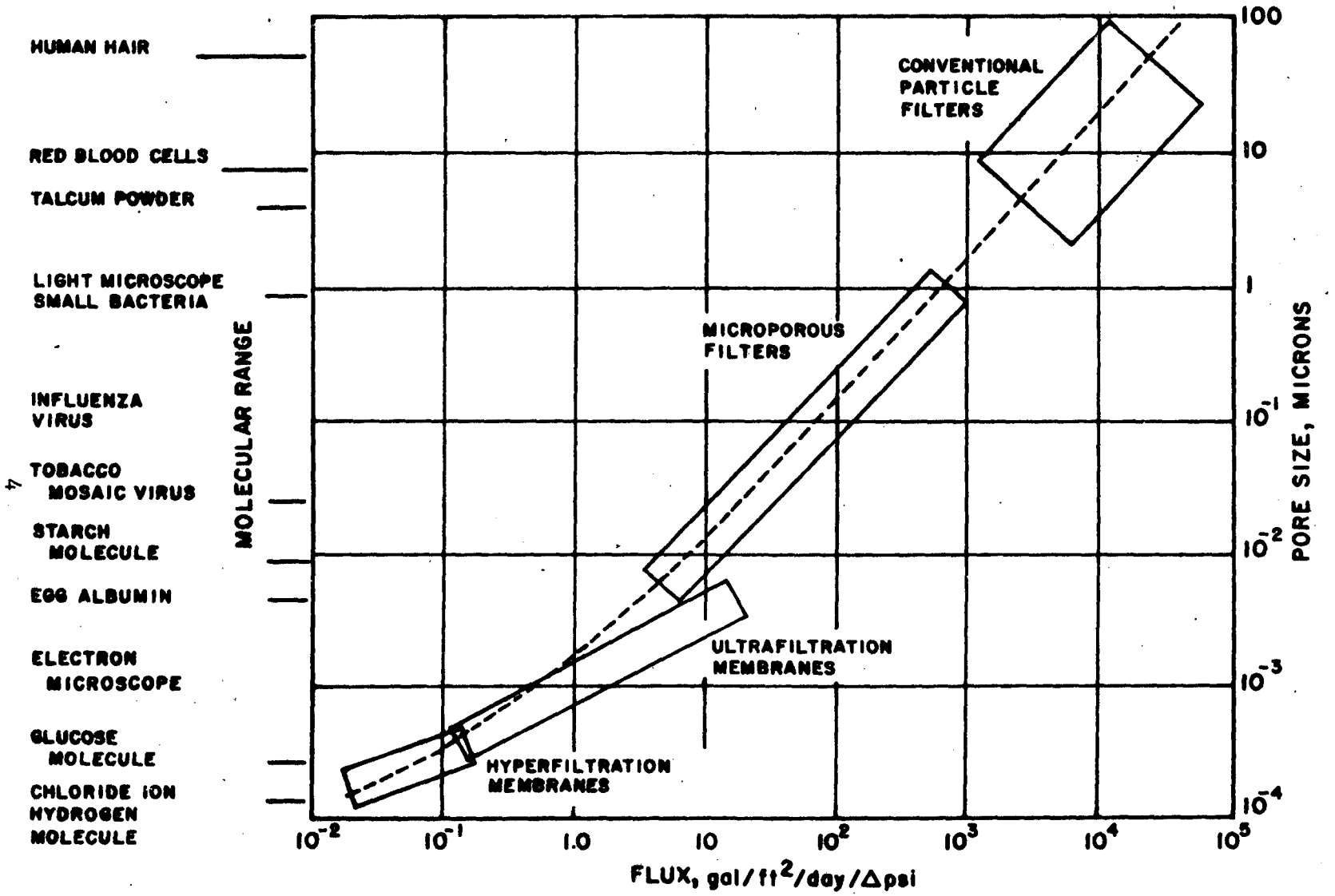

Figure 1. Molecular Range vs. Flux for Filtration Processes

TABLE 2. TEXTILE INDUSTRY EVALUATION OF HYPERFILTRATION

DATE: 1972-75 USEPA Grant No. S800929 (3)
OBJECTIVE: Complete reuse of plant wastewater at La France
GRANTEE: La France Industries, Division of Riegel Textile Corporation
PRINCIPAL INVESTIGATOR: C. A. Brandon and J. J. Porter
CONCLUSION: Both purified product and concentrate residue reused in eighteen production dyeings.

DATE: 1974-77 USEPA Grant No. S802973 (4)
OBJECTIVE: Assess application to composite wastewater at eight plant sites.
GRANTEE: South Carolina Textile Manufacturers Association
PRINCIPAL INVESTIGATOR: C. A. Brandon and J. J. Porter
CONCLUSION: Purified water was recycled in every process; chemical recovery in some cases.

DATE: 1975-78 USEPA Grant No. R803875 (5)
OBJECTIVE: Assess recycle with high temperature membranes for five selected wet finishing unit manufacturing processes.
GRANTEE: Clemson University
PRINCIPAL INVESTIGATOR: C. A. Brandon, J. L. Gaddis, and J. J. Porter
CONCLUSION: Cost effectiveness for closed-cycle operation of selected wet finishing unit processes estimated.

DATE: 1977-81 USEPA Grant No. S805182 (6)
OBJECTIVE: Demonstration of closed-cycle operation of full scale textile processes.
GRANTEE: La France Industries, Division of Riegel Textile Corporation
PRINCIPAL INVESTIGATOR: C. A. Brandon

DATE: 1978- USEPA Grant No. R805777 (7)
OBJECTIVE: Determine feasibility of hyperfiltration membranes for toxic emission control.
GRANTEE: Clemson University
PRINCIPAL INVESTIGATOR: J. L. Gaddis and H. G. Spencer

The scope of Phase I, the design phase, included:

- characterization of the textile process,
- study of production procedure modification,
- design of the recovery system,
- on-site qualification testing of membranes, and
- estimation of the economics of recovery.

METHOD OF STUDY

This demonstration project is conducted at La France Industries, Division of Riegel Textile Corporation, with the assistance of five major subcontractors. The Oak Ridge National Laboratory loaned a hyperfiltration test trailer and provided consultation on membrane technology by Dr. J. S. Johnson, Jr. Clemson University conducted some specialty chemical analyses under the direction of Dr. H. G. Spencer. The other subcontractors and their contributions are described below.

This demonstration project was conceived of as the logical culmination of the USEPA research itemized in Table 2. Dr. C. A. Brandon developed, in cooperation with La France Industries, the grant request proposal and work plan for this demonstration. He with his associates in CARRE, Inc. provide the overall program management. They planned, conducted, and evaluated the pilot testing, developed the conceptual design of the recovery system and prepared recommendations on program continuation in Phase I.

Texidyne, Inc. performed chemical analyses and provided consultation. They assisted in the evaluation of process modifications and laboratory tests of recycle. They also performed the chemical analyses to characterize the process effluents and membrane performance during Phase I.

J. E. Sirrine Company detailed the recovery system including preparation of the equipment and installation specifications and the request for bids for equipment and installation. They also received and reviewed the quotations.

The manufacturers of the membrane equipment participated in the planning of the on-site qualification testing and in preparation of formal designs and commercial quotations of the membrane component of the recovery system, including estimates of operating and maintenance costs.

The method of study is illustrated in the list of milestones, Table 3. The duration of Phase I was September 23, 1977 to September 22, 1978.

OVERALL RESULTS - PHASE I

Two major results of the study and design activities during Phase I were:

1) The selection of the dye range, instead of the becks, for the full scale demonstration.

TABLE 3. PHASE I MILESTONES

1.	Detailed Work Plan Completed	11 November 1977
2.	Quality Assurance Plan Completed	30 November 1977
3.	Review of Membrane Process Compatability Data	15 February 1978
4.	A & E Chosen	28 February 1978
5.	Process Selected	31 May 1978
6.	Preliminary Design Drawings & Cost Estimated Completed	31 May 1978
7.	Submit Project Continuation Application	30 June 1978
8.	Recovery System Design Completed	31 July 1978
9.	Equipment & Installation Bids Received	31 August 1978
10.	Project Continuation Authorized	6 April 1979

2) The economic estimates indicated a 3.8 year payback, after taxes, for the installed recovery system.

The dye range was selected because it is the more modern dyeing equipment technology and is representative of the trend in the industry due to lower production costs. At La France, the dye range has largely replaced the becks as the standard production equipment.

The payback period was calculated based on estimated recycle savings, at current (1979) energy and chemical prices, and the quoted membrane price installed.

The decision to continue with Phase II, the equipment purchase and installation was made based on these results.

SECTION 2

CONCLUSIONS

The conclusions of Phase I, the design phase of this demonstration project, relate both to the technical feasibility and to the economic feasibility.

TECHNICAL FEASIBILITY

Technical feasibility of using hyperfiltration for closed cycle operation of a dye range involves the performance of the membrane equipment and the reuse (recycle) of the membrane permeate (the purified 95% of wash water volume) and the concentrated chemicals (in the remaining 5% of the wash water volume).

Three hyperfiltration membranes were evaluated under test conditions compatible with the membrane manufacturer recommendations and the anticipated operating conditions at La France. The test results all indicated technical feasibility for the application to dye wash water. The membrane permeates were found to be adequate for recycle as process water. The reuse and/or disposal of the concentrated chemical residue will be studied in subsequent phases of this program.

ECONOMIC FEASIBILITY

The economic feasibility of hyperfiltration is estimated based on three factors: the installed cost of the membrane system and the estimated operating costs and the savings.

The quotations accepted for the membrane recovery system and the installation of the auxiliary tanks, piping and controls totaled $485,000. It is estimated that about $85,000 is for extra features useful in a demonstration but not necessarily required for standard commercial installations. The estimated operating costs and savings yielded a 3.8 year payback period, after taxes.

SECTION 3

RECOMMENDATIONS

It is recommended, based on the above conclusions from Phase I results, that Phase II be implemented. It is recommended that the Mott-Brandon Corporation stainless steel membrane system be used in the full-scale demonstration. This system employs high temperature ZOPA dynamic membranes on the internal surface of porous sintered stainless steel tubing. Stable fluxes were achieved at all test conditions with no indication of plugging by suspended solids in wash water.

The full-scale evaluations scheduled for the latter phases of this program will determine the practical feasibility of hyperfiltration. The development of optimal membrane operation procedures, including development of cleaning and/or flux maintenance procedures may enhance the economics of applying membranes for recycle.

The development of techniques and procedures for reuse of major portions of the concentrated chemicals could contribute significantly to the economic benefit of membranes. However, the study of the disposal of the concentrate by means other than reuse is necessary to complete the final evaluation of closed cycle operation.

SECTION 4

TEXTILE PROCESS DESCRIPTION

Most of the production is done on the dye range. The range consists of a dye pad, spiral atmospheric steamer, jet washer, dip box, and four wash boxes (see Figure 2). After the dip box and each of the four wash boxes, nip rolls remove excess water. The range is fully automated to control cloth speed and process temperatures. The continuous range is designed to run in the range from 9 to 36 meters per minute depending on the fabric and process details. Cotton, acrylic, nylon, rayon, and polyester fabrics as well as their blends are processed on the range. Dyeing is done as a steady state process with both the natural and synthetic fabrics.

Several classes of dyes are used: direct, dispersed, acid, pre-metalized, basic, and fiber reactive. The dye formuli include, in addition to the dyes, auxiliary chemicals. The dye formuli are mixed in the drug room and pumped to the dye pad on the range. The flow to the pad is controlled by a level control valve in the dye pad.

As the fabric leaves the pad it goes through a set of nip rollers to remove the excess dye. The fabric then goes into an atmospheric steamer. The steamer contains about 150 meters of fabric. The fabric speed is determined by the residence time required in the steamer for the fabric and dye system.

After the fabric leaves the steamer it is rinsed sequentially in a washer employing a large recirculating flow, a small dip box, and three washers. The rinse water used is plant process water. Steam injection into the washer is used to bring the rinse water to the appropriate process temperature (Table 4).

STANDARD WASHING PROCEDURE

The rinse water from the 2nd washer counterflows by gravity to the 1st washer which discharges to the drain. The rinse water from the 3rd washer is pumped to the jet washer which discharges to the drain. The rinse water from the dip box directly discharges to drain. A set of nip rollers, after the dip box and each of the wash boxes, removes the excess water in the fabric.

The 4th washer is used as a fixing pad bath approximately 90% of the time. When the 4th washer is not used as a fixing bath, the water counterflows to the 3rd washer. There is a set of nip rollers after the 4th washer also. When used as a fixing pad, the 4th washer is discharged to the drain at the end of the dye run.

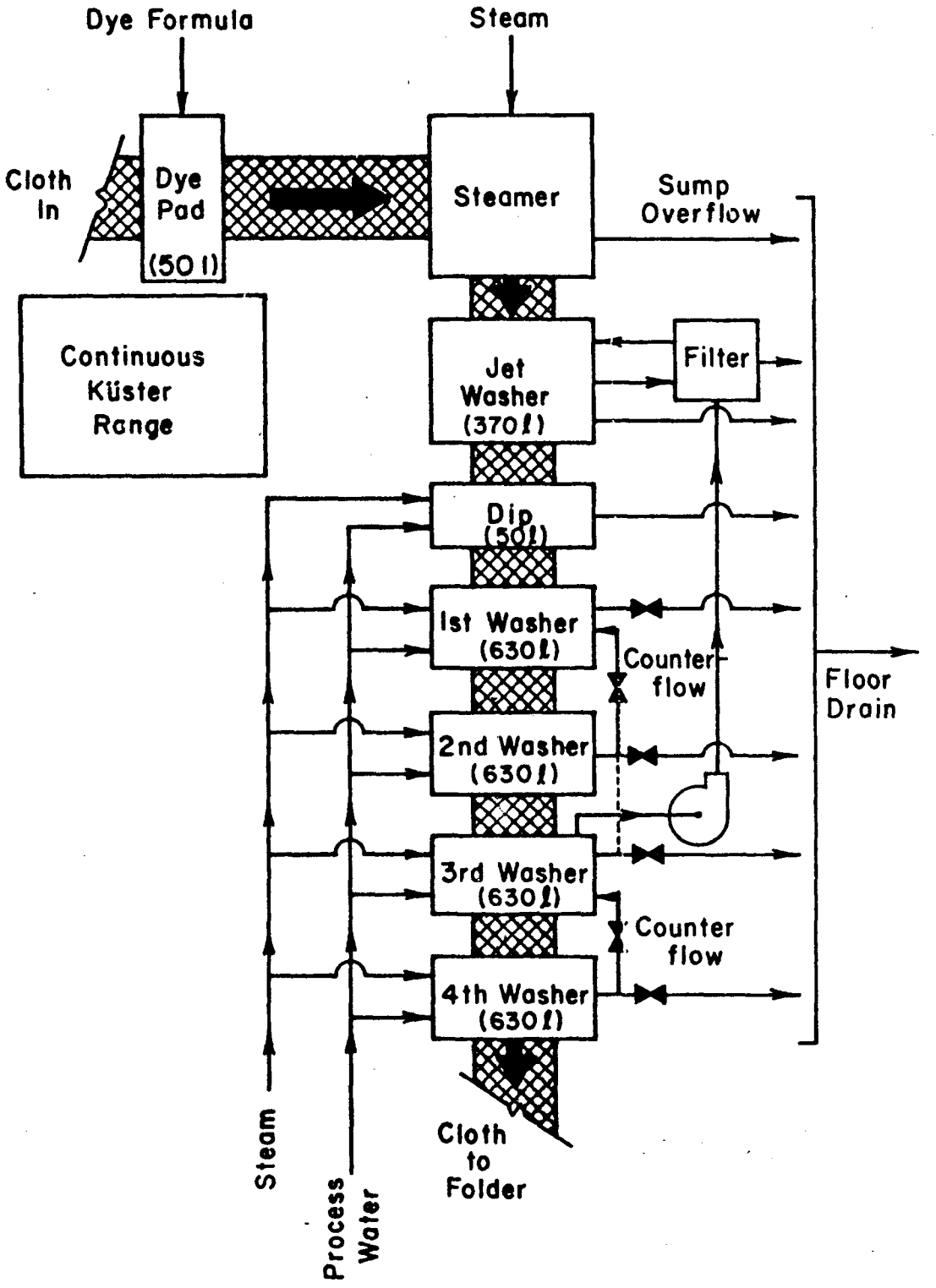

Figure 2. Continuous Dye Range Arrangement.

The physical characteristics of the wash water are indicated in Table 4. Noting from above that only the water from the jet washer, the dip box, and the 1st washer discharge to drain, the total water discharge rate is 400 ℓ/min. The 1.0 ℓ/min overflow from the steamer pump is quite pure condensate. The chemical characteristic of the individual discharges and their composite mixed effluent are tabulated in Table 5. (The expected characteristic for the modified washing procedure, described below, are also included in Table 5).

The chemical characterization of the dye range effluent involved analytical parameters selected with regard to the chemicals used in the dyeing process and to data relevent to the membrane recovery process. Analytical procedures are summarized in Appendix A. Initially each discharge point was evaluated, however, preliminary analysis indicated that the steamer effluent was essentially condensate quality, and sampling at the steamer was discontinued. The 2nd and 3rd washers were eliminated as sampling points since the effluents from these washers are counterflowed.

The process effluents were characterized during February to May 1978. A total of eleven production runs were sampled, direct dye formulations applied to three automotive fabric runs, direct dye formulations applied to three cellulosic fabric runs, fiber reactive dye formulations applied to two cellulosic runs. These process types accounted for ∿95% of the La France dyehouse production during the sampling period.

Average effluent characteristics are tabulated in Table 5 for each process effluent. The composition of the effluent streams is highly variable because of the differences in dye pad bath formulations for the wide range of shades produced with several fabrics.

The evaluation of effluent composition with time during selected runs showed that the jet washer, dip box, and wash box effluents all reached a steady concentration after 20 to 30 minutes from the start of the process run. Subsequently grab samples, after 20 to 30 minutes of run-time, were used. The fix bath samples were collected as composite samples because of the variations of the bath composition during the entire process run.

Also of potential interest for recovery is the fix bath effluent discharged at the end of each production run. The fixing chemicals are generally cationic organic polymers, some of which contain an inorganic salt such as magnesium chloride. The characteristics of the fixing bath discharge is presented in Table 6. (The complete study of fixing chemical recovery has been postponed.)

MODIFIED WASHING PROCEDURES

Studies have suggested that complete counterflow will improve the washing efficiency. Also elevated water temperature should improve the effectiveness of the washers. A series of full scale tests were conducted at 50% reduced wash water flow rate and at elevated temperatures. The fabric quality (hand and crock) improved with water temperature. However, at 82° C (180°F), the highest test temperature, dye addition was required to maintain shade. It was estimated that dye additions would range from

TABLE 4. CONTINUOUS DYE RANGE DISCHARGE RATES AND TEMPERATURES

Unit Process	Temperature (°C)	Overflow Rate (ℓ/min)	Holding Volume (ℓ)
Dye Pad	20 - 25	0.5	50
Steamer Sump	98 - 100	1.0	2500
Jet Washer	20 - 30	160	370
Dip Box	20 - 60	140	50
1st Washer	40 - 60	100	650
2nd Washer	40 - 60	35	630
3rd Washer	40 - 60	100	630
4th Washer (Fixing Bath)	20 - 40	0.0	630

TABLE 5. PRESENT AND EXPECTED CHEMICAL CHARACTERISTICS OF THE DYE RANGE EFFLUENT.

ASSAY	Average Concentrations or Flow					
	Dye Pad	Jet Washer	Dip Box	Wash Box #1	Composite Effluent	Expected Composite Effluent
Flow, ℓ/min.	-[1]	170	76	133	~380	~210
BOD, mg/ℓ	5,400	88	130	122	108	200
COD, mg/ℓ	23,900	620	620	740	660	1,200
Conductivity, mg/ℓ	1580-28,000	110-1550	190-950	130-810	140-1100[4]	200-2,000[4]
Alkalinity, mg/ℓ	4,150	160	70	40	100	180
Color, ADMI	98,800	990	370	1260	960	1,750
Hardness, mg/ℓ	-[2]	20	32	4	17	30[5]
pH	3.6-10.9	4.8-10.8	4.9-10.7	5.3-10.4	5.0-10.5[4]	5.0-10.5[4]
Phenols, mg/ℓ	0.84	0.09	-[3]	-[3]	-[3]	-[3]
TOC, mg/ℓ	6,250	180	250	150	180	325
Total Solids, mg/ℓ	20,900	630	700	600	630	1,140
Suspended Solids, mg/ℓ	1,730	17	26	35	25	45
Dissolved Solids, mg/ℓ	19,200	610	680	570	610	1,100
Chromium, mg/ℓ	5.3	0.03	0.32	-[3]	0.12	0.2
Copper, mg/ℓ	19.2	0.13	0.17	0.145	0.13	0.2
Iron, mg/ℓ	2.8	0.49	0.10	0.34	0.36	0.65
Manganese, mg/ℓ	0.2	0.08	0.08	0.05	0.07	0.1
Nickel, mg/ℓ	0.1	0.007	0.005	<0.001	0.004	0.007
Zinc, mg/ℓ	2.7	0.19	0.19	0.06	0.14	0.25
Magnesium, mg/ℓ	10.4	6.0	7.2	1.5	4.6	8.5[5]
Calcium, mg/ℓ	7.4	2.3	3.1	1.1	2.0	3.5[5]

[1]Dye pad drops depend on cloth pickup characteristics and volume of dye pad prepared. Dye pad drop will be mixed with HF concentrate and this concentration will have no influence on HF system feed quality.
[2]Sample color interferes with analytical procedure.
[3]Too few data were taken for a meaningful average.
[4]These data were estimated without averaging.
[5]These data will probably be much lower as the recirculated product will be soft water.

TABLE 6. CHARACTERISTICS OF FIX BATH EFFLUENT

Parameters	Fix Bath		
	Minimum	Maximum	Average
BOD_5, mg/ℓ	110	110	*a
COD, mg/ℓ	95	3310	770
Conductivity, μmho/cm	89	3200	1140
Alkalinity, mg/ℓ	19	32	*a
Color, ADMI	44	470	160
Hardness, mg/ℓ	63	460	250
pH	4.6	7.2	
Phenols, mg/ℓ	-	00	-
TOC, mg/ℓ	50	780	230
Total Solids, mg/ℓ	130	6980	1840
Suspended Solids, mg/ℓ	2	87	34
Dissolved Solids, mg/ℓ	120	6890	1810
Chromium, mg/ℓ	0.001	0.05	0.01
Copper, mg/ℓ	0.007	0.09	0.05
Iron, mg/ℓ	0.04	1.15	0.21
Manganese, mg/ℓ	0.06	0.46	0.20
Nickel, mg/ℓ	0.001	0.10	0.02
Zinc, mg/ℓ	0.05	210	23.0
Magnesium, mg/ℓ	1.0	1900	350
Calcium, mg/ℓ	1.93	16.0	7.6

*a Too few data were taken for a meaningful average.

0-20% depending on the dye formulation. Test results are presented in Appendix A.

The flow rates and counterflow arrangement selected for consideration in the closed cycle operation are indicated in Figure 3. The temperature for all the wash water will be 82°C. The largest portion, 76 ℓ/min of the total wash water flow, is introduced into wash box No. 1 and counterflowed. Water is also introduced, primarily as nip roller sprays, into the other wash boxes; 38 ℓ/min in wash box No. 2, and 19 ℓ/min into wash box No. 3. A significant flow, 38 ℓ/min is introduced into the dip box to prevent build-up of contaminants in the dip box. Each wash box is connected to a drain so that the water can be collected during equipment wash downs between shade changes.

Both the low flow rate and the high temperature reduce the required membrane area. With recycle, use of high temperature wash water does not result in a high energy loss. The vapor losses, of course, are larger at high temperature. However, the improved washing effectiveness with temperature elevation permits a flow reduction such that energy conservation is achieved with hot water washing, particularly if recycle is implemented.

The chemical characteristics of the wash water from the modified washer arrangement were not measured. The composition of the 190 ℓ/min completely counterflowed wash water was calculated, Table 5, assuming the same removal from the cloth as is achieved in the standard washing procedure. As indicated by the improved hand and crock quality of the fabric washed with hot water, actually more removal may be expected. The recycle system was designed for 210 ℓ/min flow (SECTION V). The calculated composition therefore may be somewhat conservative.

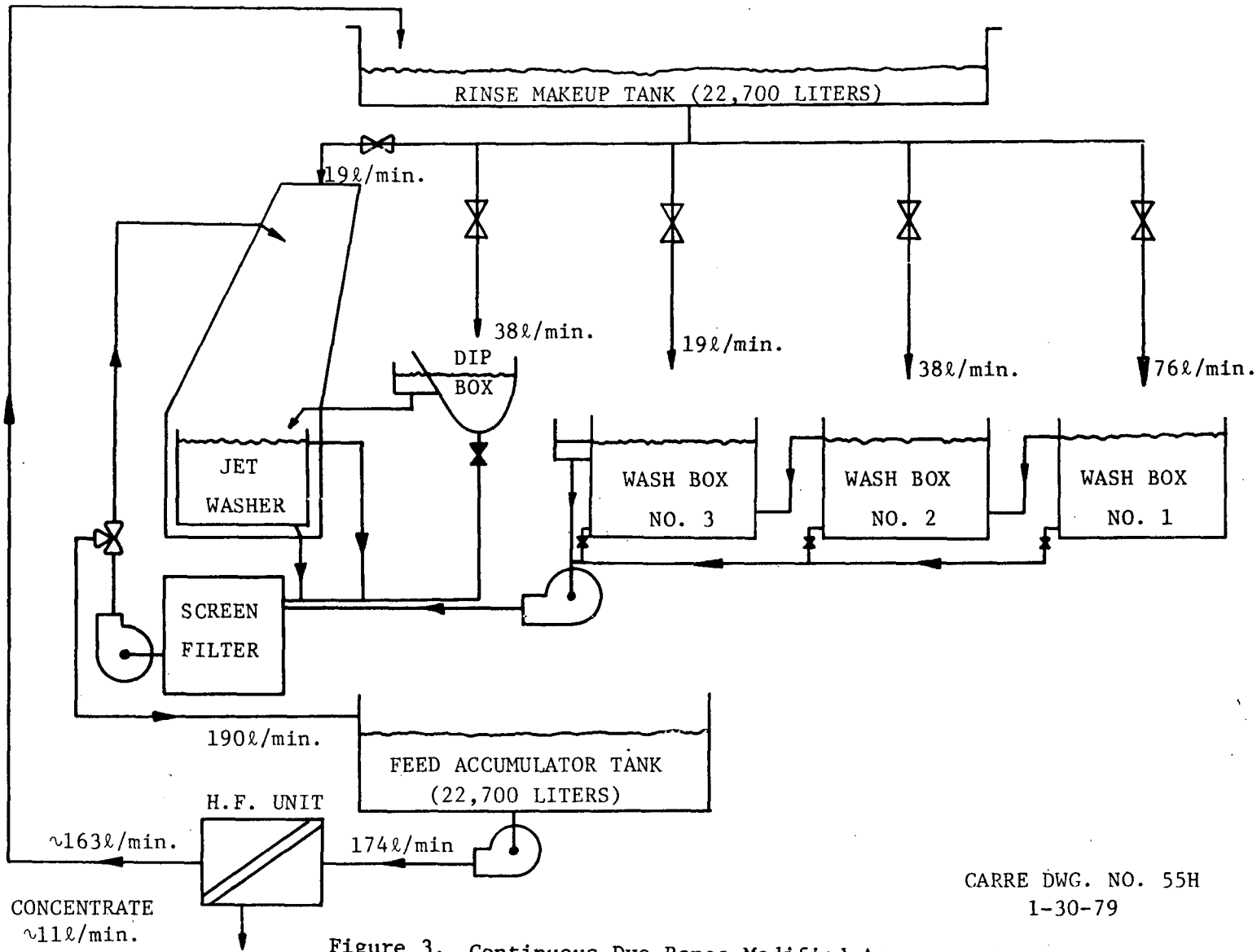

Figure 3. Continuous Dye Range Modified Arrangement.

SECTION 5

RECOVERY SYSTEM

All the water used on the dye range, water used to wash the fabric, water used to rinse the equipment between production runs, and the residual, full strength liquor in the dye pad will be collected. The wash water and rinse water flow to a 23 m^3 accumulator tank and to be processed by the high temperature hyperfiltration unit. Approximately 95% (by volume) of the wash and rinse water will be recovered as membrane permeate. The permeate flow to 20 m^3 tank from which it will be used as needed as wash and rinse water. The remaining 5% containing the concentrated chemical residue, will be collected in a 3 m^3 tank where it will be combined with the residual full strength dye liquor. This concentrate will be used as the initial formulation for a subsequent dye shade or will be treated for ultimate disposal.

The design of the membrane recovery system for the dye range is presented in this section. Details of the actual installed system will be presented in subsequent phases of this program. The system consists of accumulator tanks, valves, pumps, meters and controls.

ACCUMULATOR TANK SIZE

Figure 4 shows conceptually the water flows involved in the recovery system. The design wash water flow rate is 210 ℓ/min during operation and approximately 2700 liters following each run for wash down of the equipment. The range is inoperative for 20 minutes or longer between consecutive production runs. The longest production run is probably 3.67 hours (80 pieces of cloth) based on current practice. Two consecutive shifts each with two 80 piece runs, separated by 20 minute clean-up periods is considered to be the heaviest probable loading on the recovery system.

The accumulator tank size was selected considering its impact on the membrane system size and capacity to permit temporary recovery unit stoppage without overflow. An accumulator tank of 23 m^3 has a holding capacity for $\sim$2 hours of wash water flow at 210 ℓ/min, thus providing two hours of spillfree operation. The tankage also allows the membrane system to be sized at 170 ℓ/min, for the two-worst-shift hypothetical case described above. The size of the membrane unit may be reduced even more if the load profile involves shorter production runs and/or longer periods between production runs.

The rinse accumulator tank is sized equal to the feed accumulator tank to prevent overflow. Normal operation has the rinse tank nearly full of clean hot water and the feed accumulator tank nearly empty.

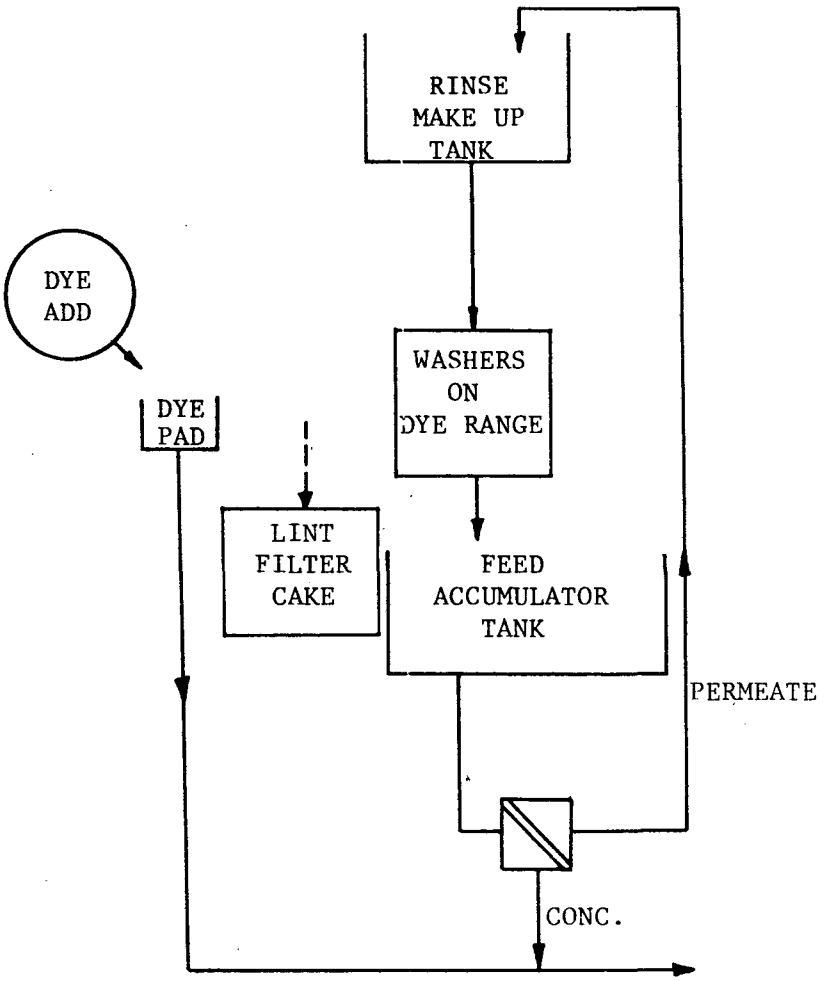

Figure 4. Conceptual Diagram of the Recovery System.

CONCENTRATE REUSE

The recovery system is configured to allow reuse of the permeate and also reuse of the concentrate. Reuse of the concentrate requires a balance of concentrate and water to the drug room. The average maximum usable flow rate of concentrate is approximately 9 ℓ/min, and the maximum usable drug room average flow is approximately 23 ℓ/min. The recovery system will require make-up to replace water drag-out by the cloth. The make-up requirement closely parallels the drug room flow, Figure 5. The result shown in Figure 5 is for one selected condition of cloth speed and water content that is reasonably typical, i.e., 20 meters/min and 1 kg per meter of cloth.

WASTE DISPOSAL

Waste from the system will include the lint cake from the 100 mesh prefilter and any non-usable concentrate. The steamer condensate overflow will be used as hot make-up water. The ultimate disposal of waste material is the subject of study in Phase II and III of this demonstration project.

PERFORMANCE REQUIREMENTS

The hyperfiltration unit is designed to treat approximately 170 ℓ/min influent at the process temperature of 82°C (or the maximum temperature for the membrane). The required color removal, to permit recycle of wash water (see Reuse Tests, Appendix C) is 97% (based on a comparison of the influent and the mixed average permeate). The concentrate flow rate from the hyperfiltration unit should be in the range of 9 to 13.5 ℓ/min to permit volume flow matching the preparation of dye formulations in the drug room.

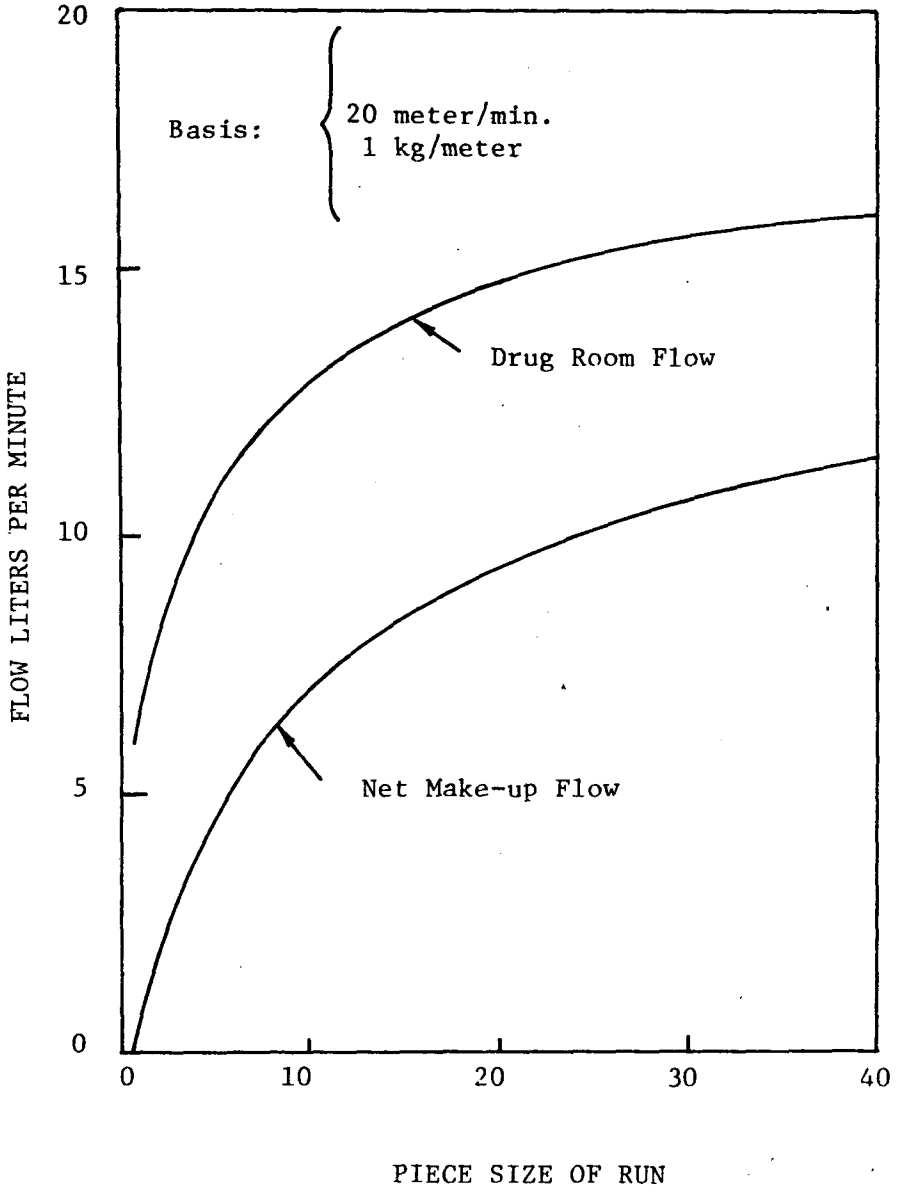

Figure 5. Estimated Drug Room and Net Make-up Flowrates

SECTION 6

MEMBRANE PERFORMANCE TESTS

A series of tests were conducted to evaluate the candidate membrane modules, Table 7, with a continuous fresh supply of wash water for an extended time. The test conditions were altered to systematically increase the fractional recovery of wash water as purified hot process water. At each of three recovery levels (selected to be typical of inlet, middle, and exit of a full-scale membrane system), the effects of velocity, pressure, and temperature were measured. The objective of this test program was to obtain field data covering the range of expected operating conditions so that vendors could design a membrane unit with pretreatment for the recovery system.

Flux decline of a membrane due to wastewater constituents and/or due to particulate fouling cannot be predicted exactly. Experience suggests a test period sufficiently long for a treatment of about 20 m^3 per square meter (500 gallons per square foot) of membrane surface. Because of the membrane flux and the availability of prototype modules, the three candidate membranes processed widely differing volumes of rinse water. The tests are summarized in Table 7.

While the volume of wastewater processed was low the PA-300 membrane (spiral wound module), apparently stable flux levels were achieved within about 5 days at each set of operating conditions. The necessity of at least 1-micron prefiltration was demonstrated by the plugging of one module.

The flux reached nearly stable levels for the Kusters membrane (formed on the external surface by carbon tubes). A small continuing flux decline may have been the result of plugging of the flow passage that culminated in an excessive pressure drop after about 4 weeks. Mechanical cleaning, carried out by the vendor, was reported to have restored the module. Prefiltration in addition to that provided on the dye range may prevent module plugging.

The ZOPA membrane (formed on the internal surface of a single channel stainless steel tube) achieved stable fluxes at each set of operating conditions. There was no indication of plugging during the 720 hours of operation.

GENERAL MEMBRANE PERFORMANCE

Experience indicates that the parameters that determine flux when processing a particular fluid stream are operating time, concentration (e.g., recovery level), pressure, velocity, and temperature.

TABLE 7. SUMMARY COMPARISON OF MODULES TESTED

Manufacturer	Kusters	Mott-Brandon	U.O.P.
Module No.	480	452	380
Flow Geometry	External Tube	Internal Tube	Spiral Wrap
Membrane Material	Zirconium oxide/ polyacrylate/ Carbon tube	Zirconium oxide/ polyacrylate/ Sintered Stainless steel	Poly(ether) amid
Test Operating Time (Hours)	510	720	520
Permeate Processed (Gal/ft^2)	275	900	170
Filtration Required	Finer than 40 mesh screen	40 mesh screen	Finer than 1 μm cartridge
Average Flux[1] (GFD)	13	30	8
Average Color Rejection[1] (%)	∿99	≥99[2]	≥99[2]
Average Conductivity Rejection[1] (%)	60-85	60-80	≥96

[1] Appropriate average over test operating time. Test conditions shown in Figure 13 for Mott-Brandon, Figure 17 for Kusters and Figure 9 for U.O.P.

[2] Exclude data taken with fiber reactive dyes. Some unexplained effect influenced results with fiber reactive dyes. Data before and since test reported here have not indicated any problem with fiber reactive dyes.

Time

Flux decline with time can usually be divided into two parts: rapid initial decline and steady long term decline.

When a new membrane is initially exposed to a waste stream a rapid flux decline usually occurs within a short time (2). This decline may be due to a physical or chemical 'accommodation' between the membrane and the waste stream. The feed constituents may alter the membrane properties either by reaction or through formation of a self-rejecting layer.

After the rapid initial decline, a slower decline associated with fouling, or membrane blinding, by particulates sometimes occurs. The rate of flux decline during particulate fouling decreases with time. Because of this characteristic, particulate fouling can sometimes be identified by a linear plot of the logarithm of flux versus the logarithm of time.

Pressure

Flux is expected to be a linear function of pressure. Depending on flow configuration, velocity and concentration, nonlinearities can occur at high pressure in the presence of large molecular weight feed solutes. At high fluxes (at higher pressures) the large molecular weight solute forms a boundary layer at the membrane surface with a significant resistance to permeate flow causing an apparent decrease in permeability. This boundary layer is dependent on the solute, flow velocity, solute concentration and module configuration.

Velocity

Velocity effects can be characterized as either long term irreversible effects associated with membrane fouling or short term effects which are reversible and are associated with module hydraulics, e.g., fluid boundary layer.

The hydrodynamic effects become particularly noticeable if the flow is changed from turbulent to laminar. Decreasing flow velocity usually results in a decreased flux and in some cases a decreased rejection (as observed by a comparison between mixed feed and permeate samples).

A secondary effect of increasing velocity at a constant inlet pressure is the reduction of module average pressure. Since pressure drop is a strong function of the velocity, small changes in velocity in some modules may result in large variations in module average pressure. The result is an apparent loss in permeability with increasing velocity.

The long term effects of velocity on membrane performance usually are associated with membrane fouling, or membrane blinding by the deposition of particulates. If the membrane fouling is the result of particle or colloid deposition, the rate at which the deposition occurs can be influenced by velocity (8, 9). Higher velocities usually result in lower fouling rate.

If feed constituents become insoluble at higher concentrations, the precipitate can deposit on the membrane surface. Lower velocities increase concentration polarization (10) thereby increasing the probability of precipitates.

Concentration

Like velocity effects, concentration effects may be either short term reversible or long term irreversible.

Short term effects are associated with the apparent lower permeability of the more concentrated boundary layer adjacent to the membrane surface. These effects are usually more pronounced large molecular weight species that are present in the fluid.

Long term effects of concentration may result from the formation of insoluble precipitates or colloids due to increasing concentration. Particles thus formed may deposit on the membrane surface causing fouling.

Of course, concentration is related to system volumetric recovery and thus is expected to have the most effect in downstream portions of a membrane system.

Temperature

Experience (3, 4, 5) has shown that a plot of the logarithm of flux versus the inverse of absolute temperature is a linear plot over a wide range of temperatures. The slope of this plot is usually a constant for a specific waste stream and is expected to have a value between 2000 and 4000 ^{o}L. A slope of 3000 ^{o}K results in a doubling of flux with each $\sim$25 ^{o}C increase in temperature.

TEST EQUIPMENT

High Pressure Test Unit

Shown in Figure 6 is a schematic diagram of the high pressure test unit. The primary mechanical components of this skid-mounted hyperfiltration unit are: (1) feed reservoir, (2) prepressurizing pump, (3) high pressure pump, (4) heat exchanger, (5) hyperfiltration module, and (6) pressure control valve.

The feed reservoir is a 100 liter stainless steel tank. Wastewater flow into the tank is regulated by a liquid level control valve. A centrifugal pump maintains a supply pressure to the suction of the Gaso positive displacement pump.

The Gaso pump is a triplex plunger pump with stainless steel (316) wetted parts. It has a discharge pressure of 1000 psi at a flow rate of 45 ℓ/min with a 15-hp motor and the drive ratios now used.

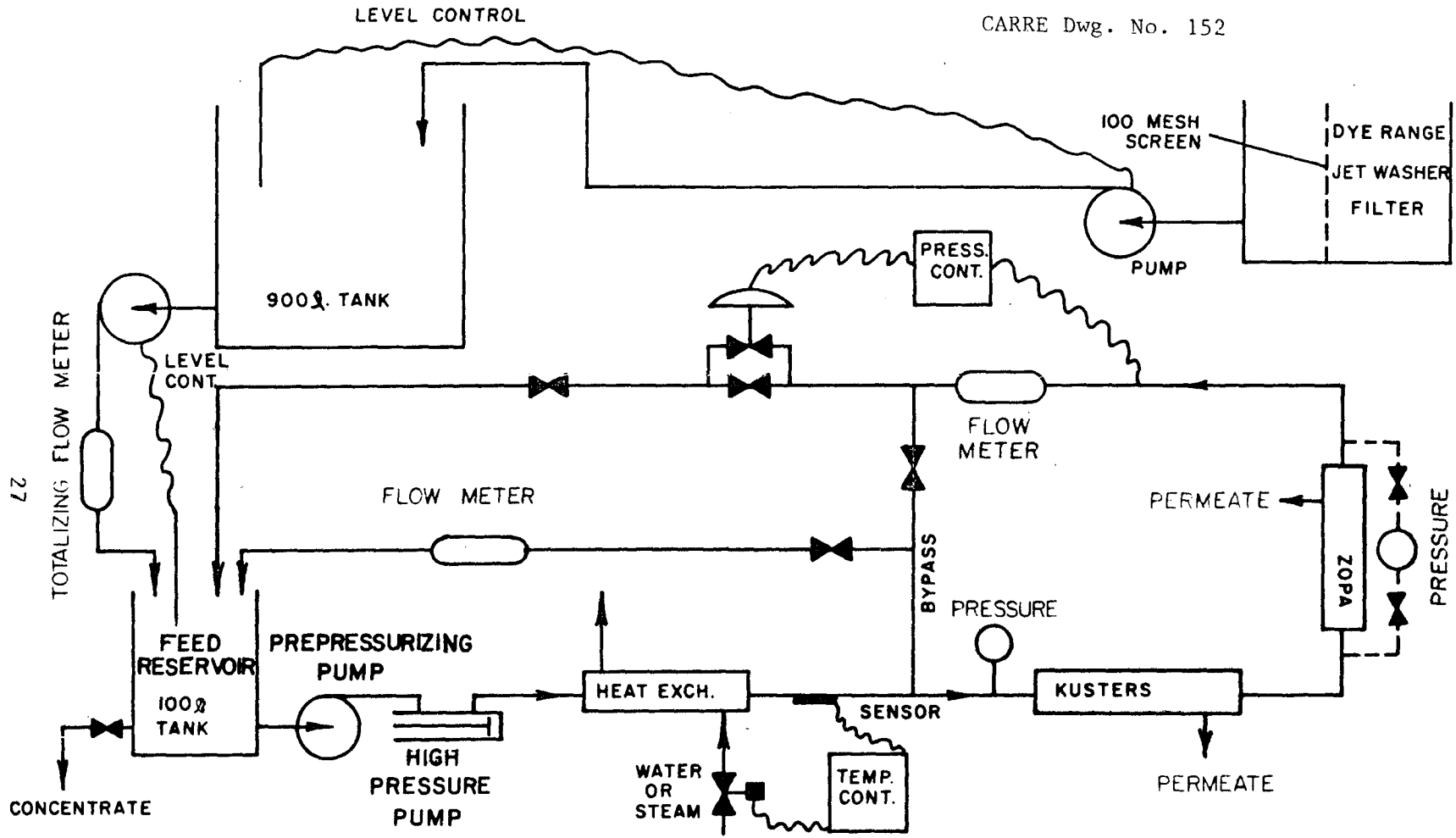

Figure 6. Schematic Diagram of the High Pressure Test Unit.

To protect the system, safety switches are provided to stop the motors in the event of low suction pressure or high discharge pressure. In addition a rupture disc pressure relief device is rated at about 1400 psi.

The heat exchanger is a concentric tube type, the outer tube is 1-inch and the inner is 3/4-inch. The purpose of the heat exchanger is for control of the fluid temperature in this recirculating system.

Modules of various sizes can be tested in this unit. Since the positive displacement pump provides a constant volume of flow a manual bypass valve is used to control the flow to a module. Two valves are in parallel following the module. The air operated pressure control valve maintains a set pressure (up to 1000 psi) in the system. A manual valve is for use in case of malfunction of the automatic valve.

For testing with the pressure above 1000 psi, the high discharge pressure safety switch can be disconnected and the system operated manually.

All pressures are indicated by bourdon-tube devices. The temperature is indicated by thermocouple/resistance bridge devices. Grab samples can be obtained through the indicated sample ports to provide rejection data on specific species. This system was used primarily for testing of ZOPA and Kusters membranes.

Low Pressure Test Unit

Shown in Figure 7 is a schematic diagram of the low pressure test unit. The wastewater, prefiltered to module manufacturer's requirements, is introduced into a 30 gallon reservoir. The reservoir supplies the suction of a Cat positive displacement piston pump that pressurized the flow to between 50 and 400 psi. The pump produces a flow rate of 37 ℓ/min.

The flow to the module is controlled by manual adjustment of a valve to allow a portion of the fluid to bypass to the pump suction. The pressurized flow from the module passes through a pressure control valve ahead of the flow meter. The flow is then routed to either the 100 liter reservoir or directly to the pump suction. Direct routing of the feed to the pump suction creates a small volume system so that concentration to high recovery can be achieved quickly.

The pressure at the module is manually controlled. The temperature of the system is automatically controlled by regulating the flow of steam and cooling water to the heat exchanger.

Pressures are indicated by bourdon-tube type gauges. Temperatures are indicated by a thermocouple/resistance bridge devices. Sample ports for concentrate and permeate are indicated in Figure 7.

Instruments

Conductivity measurements were made with a Balsbaugh conductivity bridge and dip cell and color comparisons were made using a Bausch and Lomb Spec-

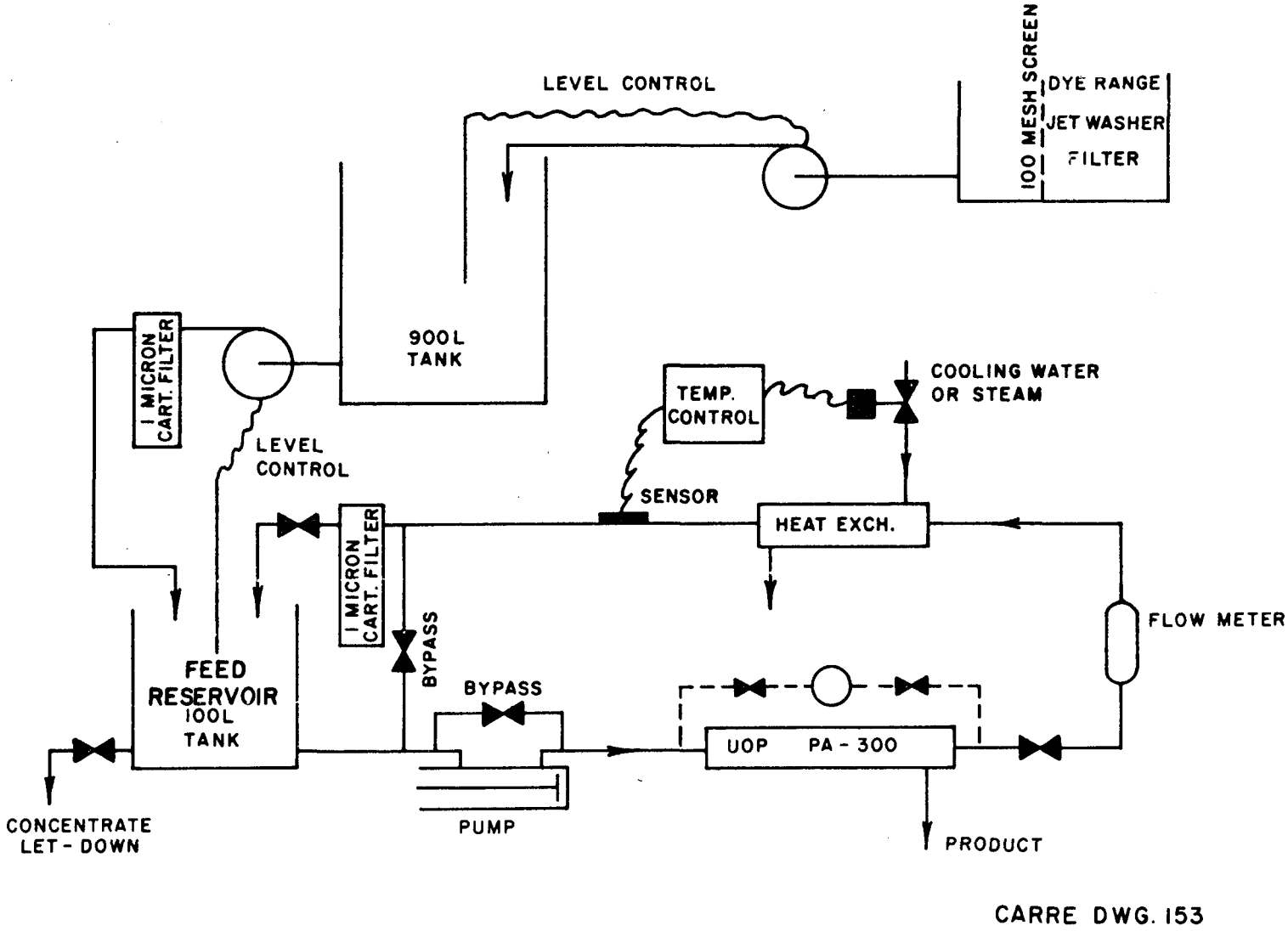

Figure 7. Schematic Diagram of the Low Pressure Test Unit.

tronic 20 spectrophotometer operated at a wave length of 460 m. Permeate flow rate was measured with a graduated cylinder and stop watch.

PROCEDURES

During the tests each module was operated at conditions representing, in so far as practical, conditions in a full scale system. Module pressure, flow velocities, temperature and system recoveries were varied to include conditions expected in full scale system. Membrane flux, color, and conductivity rejections were monitored on site. Chemical analyses were performed on samples of concentrate and permeate to provide additional rejection data. Periodic checks of membrane performance with a standard reference solution of 3 g/ℓ sodium nitrate in tap water were made throughout the period.

Renovation of membranes by washing was attempted periodically. In accordance with manufacturer recommendations, the PA-300 modules were washed with "Solution B" (a solution of 10 g/ℓ of EDTA and 1-mℓ/ℓ of Triton X-100 in tap water with pH adjusted to 9.5 nitric acid). The membranes were exposed to the "Solution B" for about one hour at low pressure and high velocity at a temperature in the range of 30°C to 50°C. The flow was shut off and the membrane was left to soak in the washing solution for approximately one hour. The "Solution B" was then flushed out of the system with tap water.

A clean tap water flush was performed on the zirconium oxide-polyacrylate membranes (ZOPA and Kusters). The washing was done at low pressure, high velocity and at temperatures in the range 30°C to 85°C. The washing was repeated two or three times, changing the washing water as necessary.

Both test units were continuously supplied with the fresh wash water, unless the dye range was not in operation or processes other than dyeing were being performed. During the periods when the range was not used for dyeing, the permeates were returned to the reservoirs. The wash water processed in both test units was filtered through the 100 mesh rotary screen of the jet washer and stored in a 900 liter tank. In addition to the 100 mesh screen, 1-micron cartridge filters (Carborandum 20-inch 1-micron filter Elements No. M39R20A) were installed in the feed line to the PA-300 testing system. The cartridge filters were operated until differential pressure reached recommended limits.

Feed lines to each test unit were equipped with totalizing flow meters to facilitate system recovery calculations. Samples of product, concentrate and feed were collected periodically in one gallon plastic bottles for analysis and reuse evaluation. The samples for analyses were collected and delivered to the laboratory in accordance with quality assurance procedures approved for this project.

MEMBRANES TESTED

Table 8 summarizes the major characteristics of the three membrane modules tested. The characteristics were provided by the vendors.

TABLE 8. CHARACTERISTICS[a] OF HYPERFILTRATION MEMBRANES TESTED

	PA-300[b] U.O.P.	ZOPA[c] Mott-Brandon Corp.	Kusters[d] Corp.
Flow Geometry	Spiral Wound	Tubular porous stainless steel	Carbon Tube bundles
Membrane Material	Poly(ether)amide	Zirconium oxide-polyacrylate	Zirconium oxide-polyacrylate
Method of Replacement	Module (on-site)	Chemically replaced in-situ	Tube replacement on-site
Prefiltration Requirements	1-5 microns	40 mesh screen	40 mesh screen
Maximum Temp., F	150-160	212	212
pH Range	2-12	4-11	4-11
Pressure Limitations, psig	1000 w/S.S. Product Tube	1000	1000
NaCL rejection at Max. Pressure	96.5	80-90	

[a] Characteristics provided by vendors
[b] Universal Oil Products Corporation
[c] Mott-Brandon Corporation
[d] Kusters Corporation

TEST RESULTS AND DISCUSSION

The objectives of these test were: (1) to determine for each membrane the effects of time and recovery level (concentration) on both flux and rejections, (2) to evaluate the vendor prescribed pretreatment and membrane cleaning techniques, and (3) to generate samples of permeate and concentrate for reuse evaluations. Results and discussion of data are presented below. Reuse evaluations are described in Appendix C.

Results

The data obtained are presented. Summary plots showing flux and rejections measured during the operating periods are presented as well as plots showing the effect of pressure, temperature, and flow velocity. More detailed performance plots and complete data are available in project report No. 24-28. Chemical analyses of samples of feed, concentrate and permeate water obtained in the tests are tabulated.

U.O.P. Module--

Performance summaries of the PA-300 modules nos. 90 and 380 are presented in Figures 8 and 9, respectively. Module No. 90 operated 160 hours during a two week period and module No. 380 operated for 520 hours during a one month period. All feed was filtered through 1-micron polypropylene cartridge filters.

Module No. 90 was exposed to recoveries between 0 and 80% at pressures of 300-400 psi, temperatures of 30 to 60^{o}C and a flow rate of 10 GPM. After 100 hours the module differential pressure began to increase while flux increased and rejections decreased. Reversing the flow through the module restored performance for 20 hours before failure occurred. Inspection of the module inlet showed a visible buildup of solids.

Module No. 380 was exposed to recoveries between 0 and 96% at pressures between 200-400 psi, a temperature of 60^{o}C and flow rate of 7 GPM and 10 GPM. A 1-micron cartridge filter was installed in the recirculating concentrate line in addition to the filters on the feed line. These double filters prevented module plugging. Pressures were varied to simulate the system pressure at various recovery levels in a full scale system. Washing with "Solution B" effectively restored fluxes, but subsequent exposure to dye wash water immediately reduced fluxes to the previous levels. Color rejection remained consistently above 99% and conductivity rejection above 96%.

Figures 10, 11, and 12 show the effects of pressure, temperature and flow rate, respectively, on the flux of module No. 380. Figure 10 shows a decreasing permeability with increasing recovery and time. The offset of the lines from the origin is probably the result of plotting flux versus module inlet pressure instead of module average pressure. Flux is a linear function of pressure. Figure 11 shows the effect of temperature on flux at various recovery levels. All lines are parallel with a slope of approximately 2800 K. The effect of circulation flow rate on flux is shown on Figure 12.

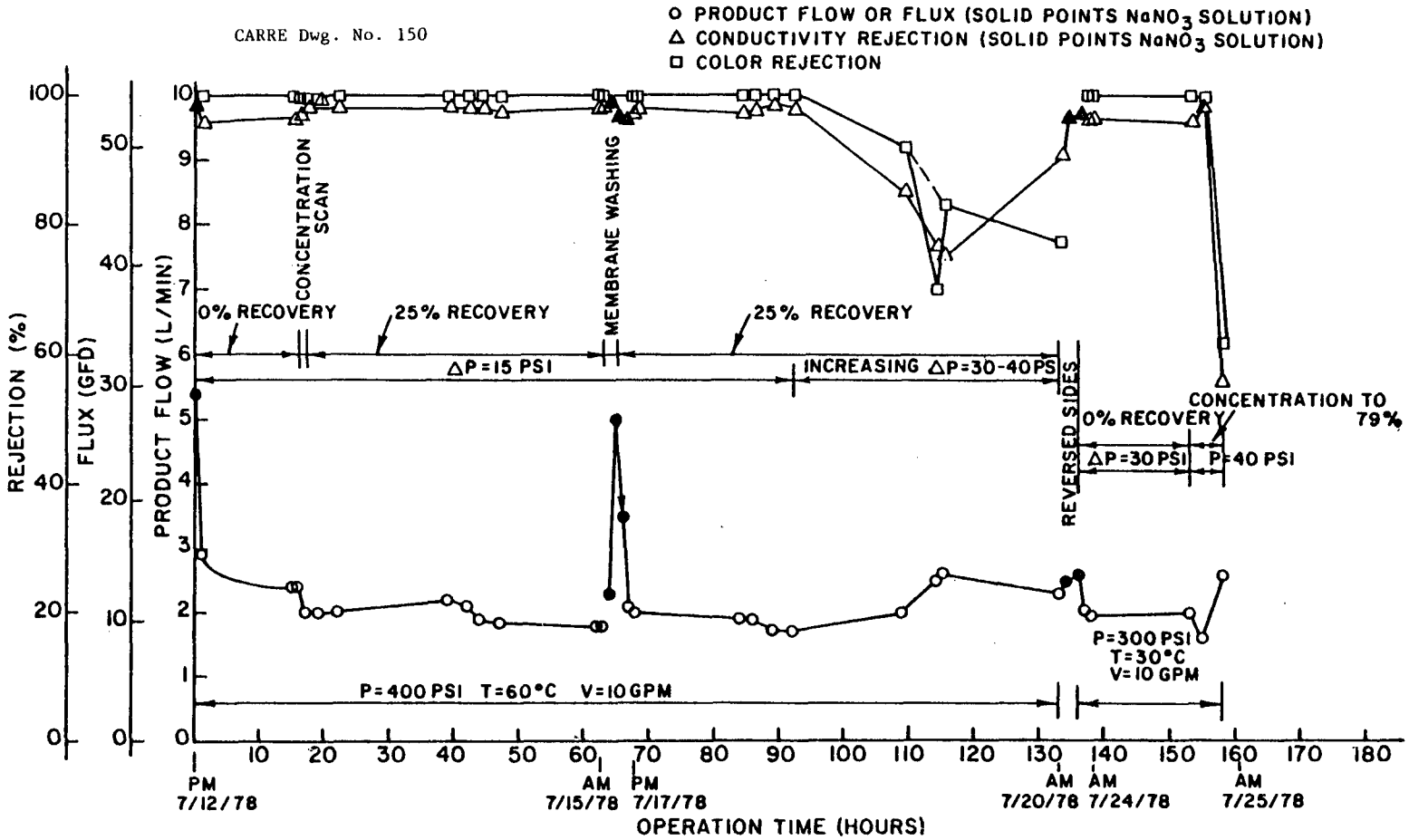

Figure 8. Product Flow and Conductivity and Color Rejection Versus Operation Time for U.O.P. Module No. 90.

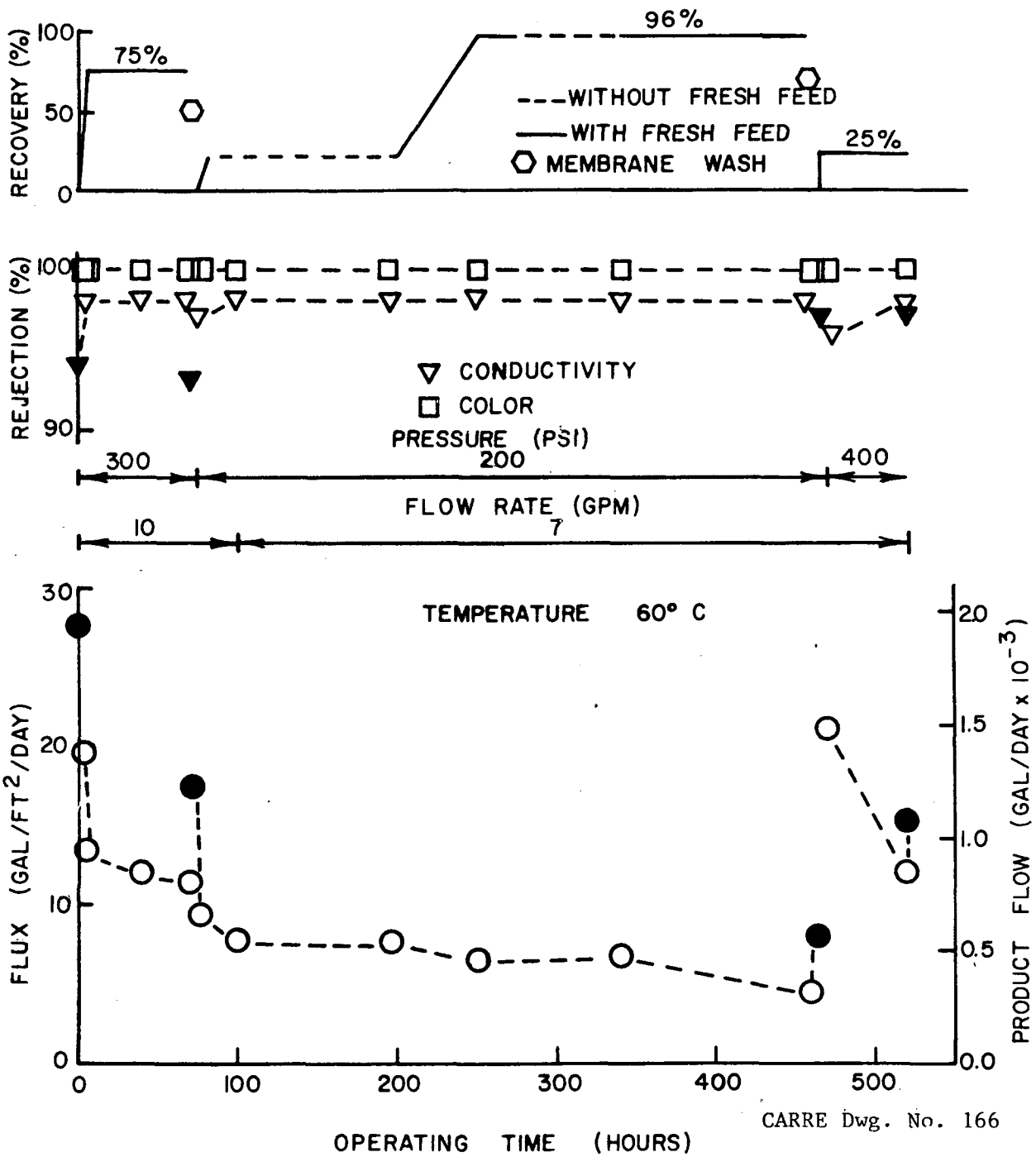

Figure 9. Product Flow and Conductivity and Color Rejection Versus Operation Time for U.O.P. Module No. 380.

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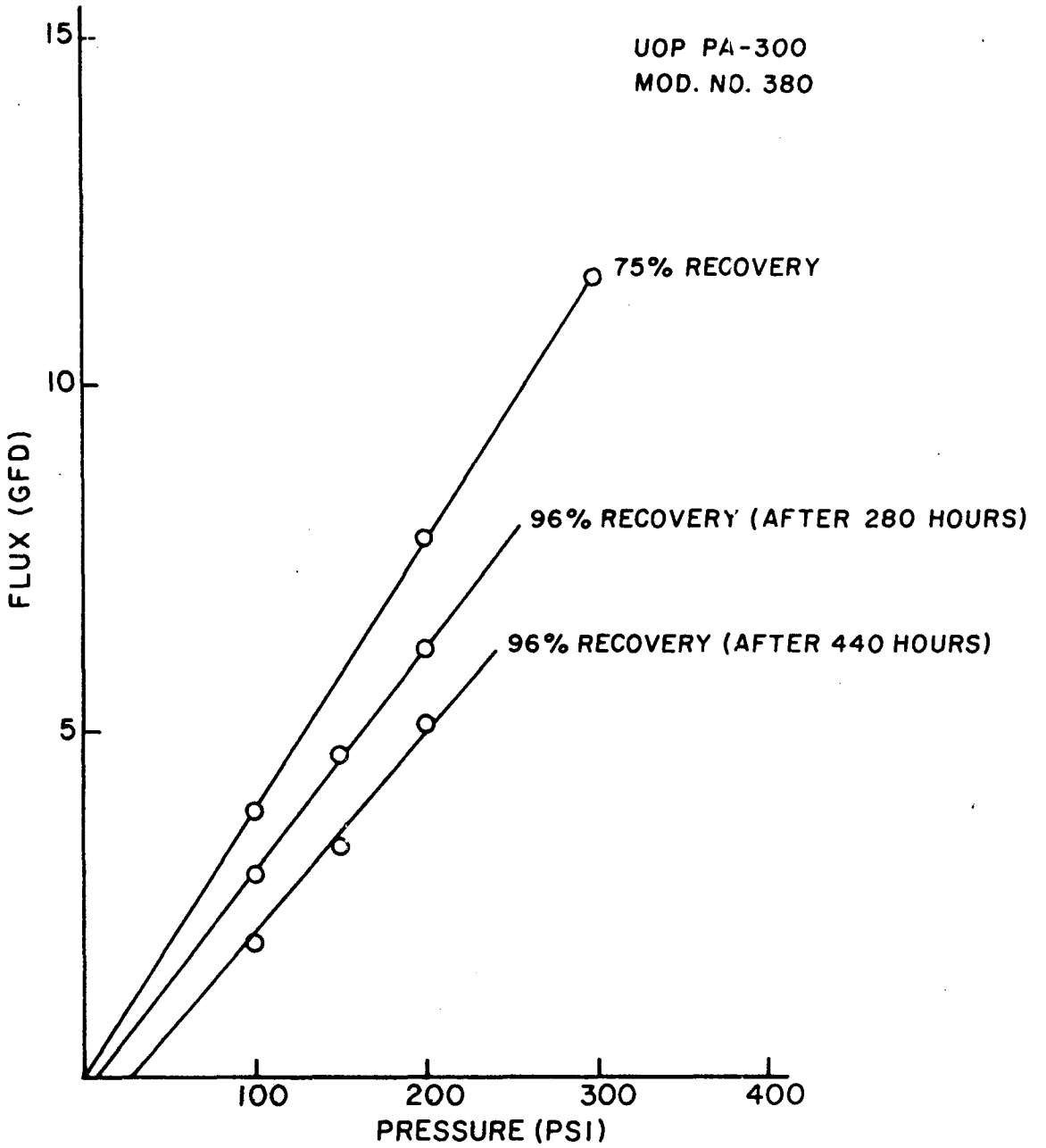

Figure 10. Flux Versus Pressure for U.O.P. Module No. 380.

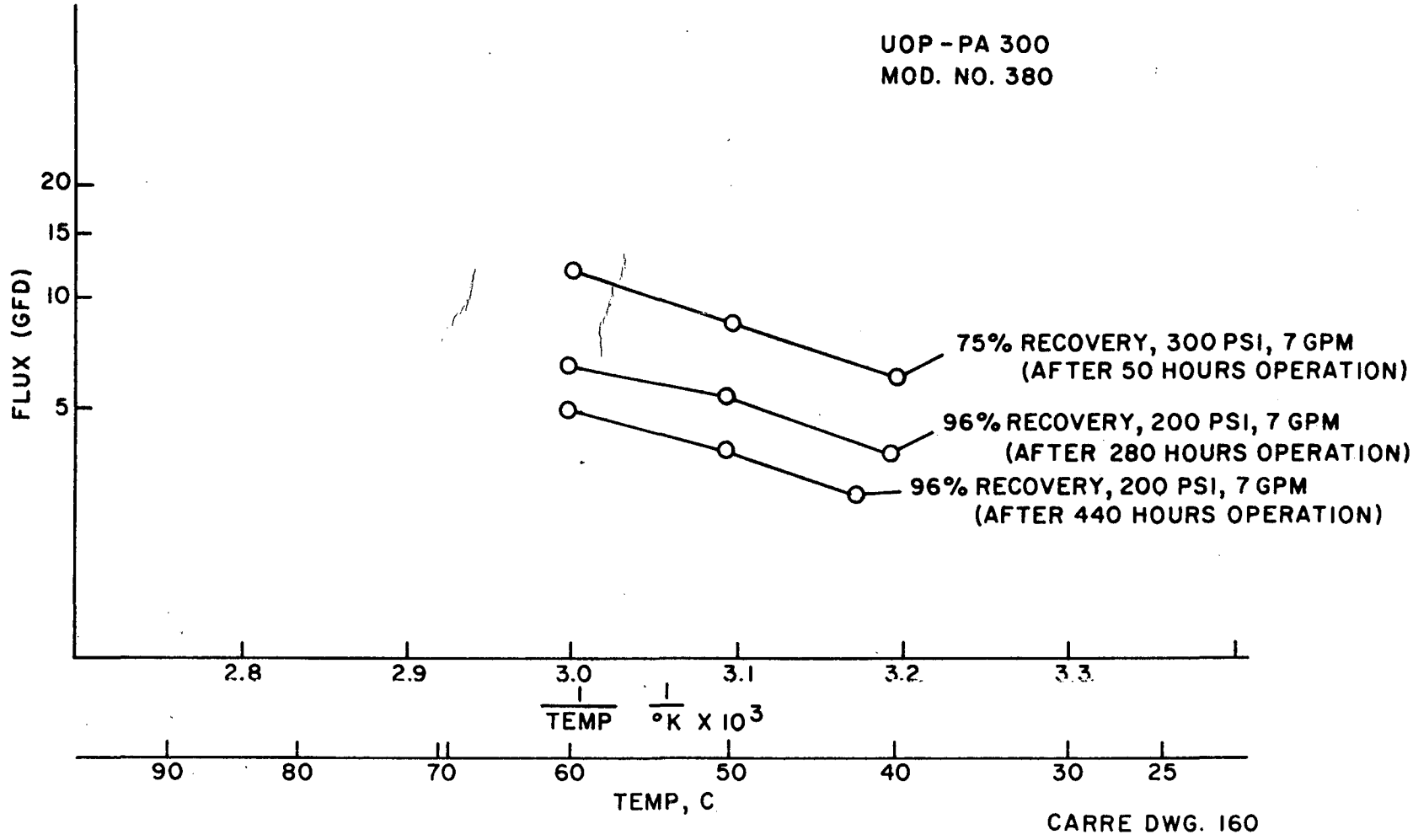

Figure 11. Flux Versus Temperature for U.O.P. Module No. 380.

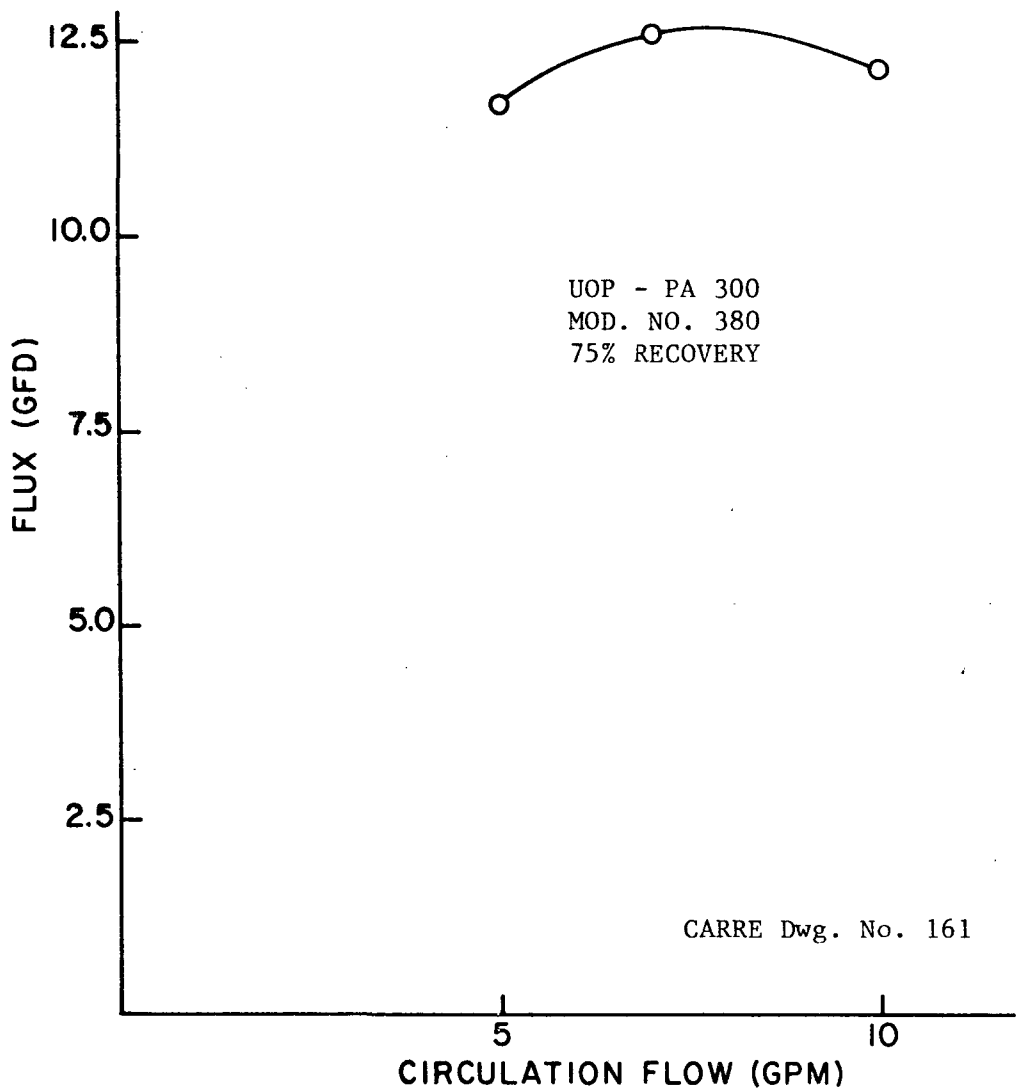

Figure 12. Flux Versus Circulation Flow for U.O.P. Module No. 380.

Table 9 gives the filtration capacity of the 1-micron polypropylene filters. The capacities varied widely depending on the solids loading in the feed. The average capacity is about 315 gallons per filter cartridge.

Chemical analyses of 5 concentrate/permeate sets of samples are presented in Table 10. Rejection of all analyses parameters are greater than 90%.

Mott-Brandon Module--

The performance summary for Mott-Brandon (MBC) ZOPA membrane module No. 452 is presented in Figure 13. The module was operated for 720 hours during a 6 week period from July 18 to September 1. Recoveries were varied between 0 and 96%, velocities between 2 and 6 m/sec. and pressures between 200 and 1300 psi. The temperature was 85°C. The module was washed six times with hot tap water. The only prefiltration was the 100 mesh screen on the dye range. Fluxes were partially restored by the hot water washes.

On July 31, after about 200 hours of operation, the Kusters module No. 480 (below) was installed in the high pressure test unit. Because of the limited flow rate capacity of this test unit pump (∿45 ℓ/min) it was necessary to test the modules in series to assume proper flow velocities. The Mott-Brandon tubular unit had already been tested for 200 hours and shown no evidence of plugging, the Kusters unit was installed upstream to gain similar experience with this unit. Since the test unit employed a high degree of recirculation and the module recoveries are essentially zero for each pass, the chemical composition of the feed was less than 1% difference between the upstream and downstream module positions.

Color rejection rose on initial exposure to the dye wash water to about 99% and remained high throughout the test. Erratic behavior of flux and rejection between 560 and 650 hours is the result of exposure to a fiber reactive dye formulation with a pH of 9 to 10. Steady performance returned with exposure to a direct dye formulation.

Figures 14, 15, and 16 show the effects on membrane flux of pressure, temperature and velocity, respectively. Figure 14 shows a decreasing permeability with increasing recovery. Nonlinear response of flux to pressure is seen at pressures greater than 1100 psi. Figure 15 shows the effects of temperature on flux. All lines are parallel with a slope of about 2800 K. The effect of velocity on flux is seen in Figure 16.

Chemical analyses of six concentrate and permeate samples are given in Table 11. The COD and total solids rejections at all recovery levels are ∿90%.

Chemical analyses of two concentrate and permeate samples are given in Table 12. The COD and total solids rejections at all recovery levels are ∿90%.

Kusters Module--

The Kusters Module No. 480 was installed in the high pressure test unit, upstream of the Mott-Brandon module, on July 31. The two were then operated simultaneously under the same conditions until August 31. The operating

TABLE 9. THE CAPACITY OF THE CORBORUNDUM FILTER ELEMENTS

FILTER NUMBER	CAPACITY[a] (gallons)
1st Filter	662 gallon
2nd Filter	335 gallon
3rd Filter	278 gallon
4th Filter	215 gallon
5th Filter	440 gallon
6th Filter	176 gallon
7th Filter	181 gallon
8th Filter	118 gallon
9th Filter	351 gallon
10th Filter	216 gallon
11th Filter	374 gallon
12th Filter	432 gallon

[a]Gallons of wash water, prefiltered through a 100-mesh screen, at 20 psi differential pressure.

TABLE 10. CHEMICAL ANLAYSIS OF CONCENTRATE AND PERMEATE FOR MODULE NO.'S 90 AND 380

RECOVERY LEVEL	25% - #90		25% - #90		75% - #380		75% - #380		96% - #380	
SAMPLE TYPE AND NUMBER	Concen. #1158	Permeate #1157	Concen. #1168	Permeate #1169	Concen. #1180	Permeate #1181	Concen. #1185	Permeate #1186	Concen. #1204	Permeate #1205
DATE TAKEN	7/14/78	7/14/78	7/18/78	7/18/78	7/26/78	7/26/78	7/28/78	7/28/78	8/11/78	8/11/78
COD, mg/ℓ	510	4	260	16	560	23	1270	8	1530	10
Conductivity, μmho/cm	420	15	195	12	840	17	1200	19	1990	21
pH	7.7	5.9	7.1	6.0	7.2	5.6	7.2	5.4	7.6	6.4
Hardness, mg/ℓ	14	0	19	0	79	0	83	0	125	0
Dissolved Solids, mg/ℓ	518	5	316	26	817	24	1571	11	2445	15
Total Solids, mg/ℓ	520	5	320	26	830	32	1580	15	2470	28
Volitile Solids, mg/ℓ	346	5	177	24	388	21	779	11	1140	27
Suspended Solids, mg/ℓ	2	0	4	0	13	8	9	4	25	0
Chromium, mg/ℓ	0.186	0.003	0.021	0.001	0.025	0.003	0.320	0.013	3.300	0.002
Iron, mg/ℓ	0.520	0.019	0.970	0.020	0.180	0.034	0.550	0.045	1.050	0.025
Calcium, mg/ℓ	3.200	0.008	2.160	0.005	17.500	0.002	25.300	0.002	31.000	0.003
Magnesium, mg/ℓ	2.400	0.009	2.880	0.006	9.800	0.011	10.800	0.005	22.400	0.005

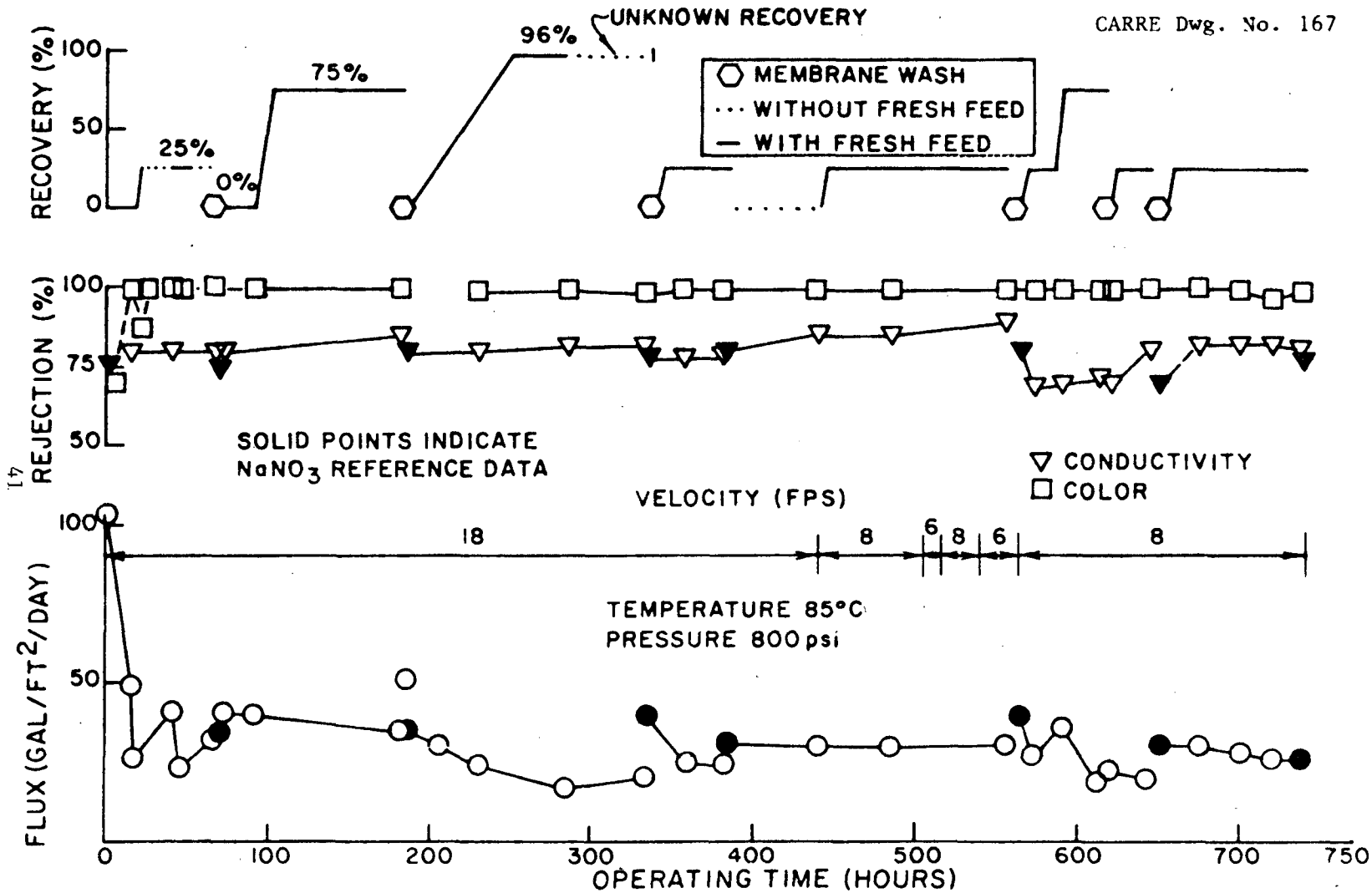

Figure 13. Product Flow and Conductivity and Color Rejection Versus Operation Time for Mott-Brandon Module No. 452.

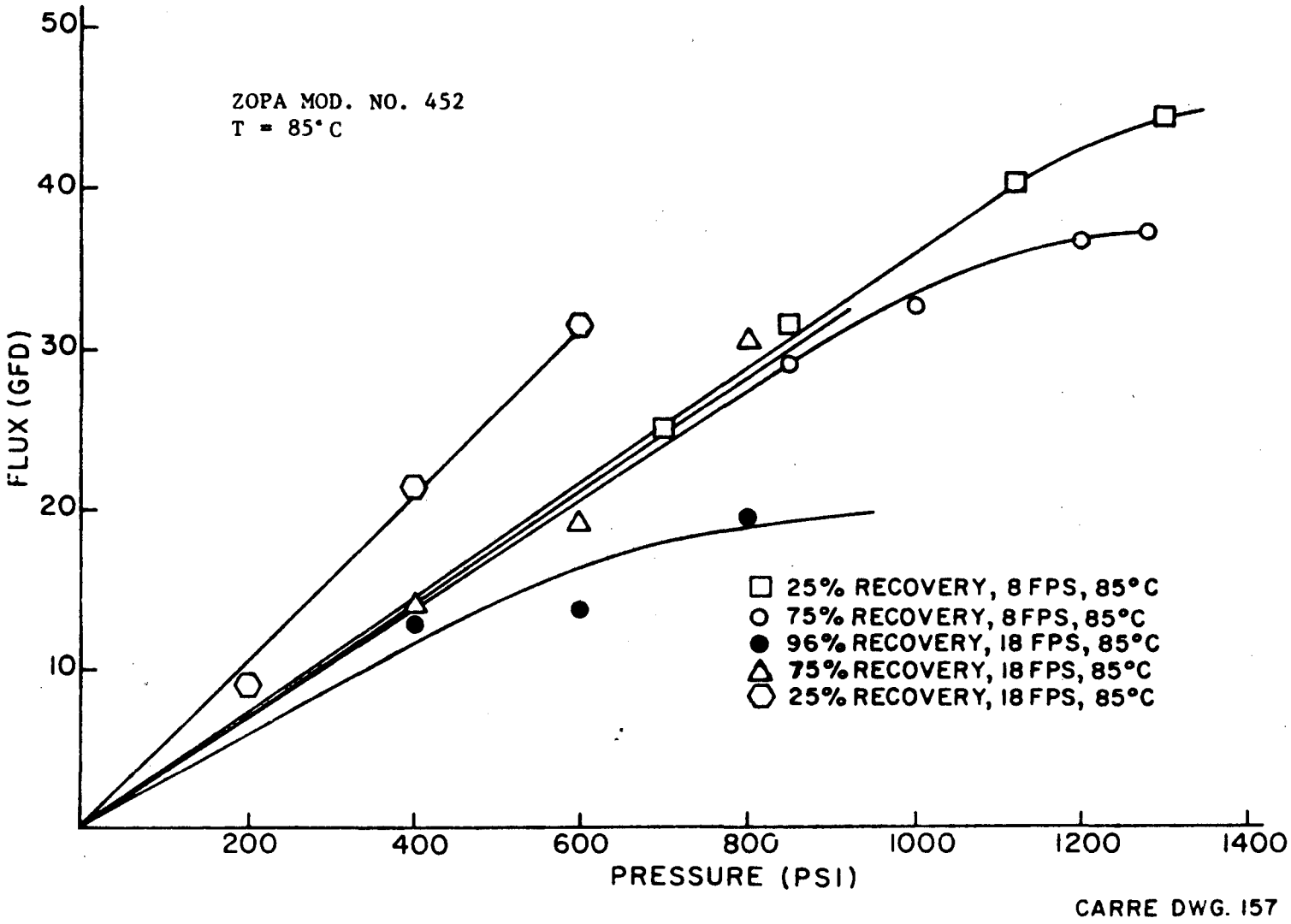

Figure 14. Flux Versus Pressure for Mott-Brandon Module No. 452.

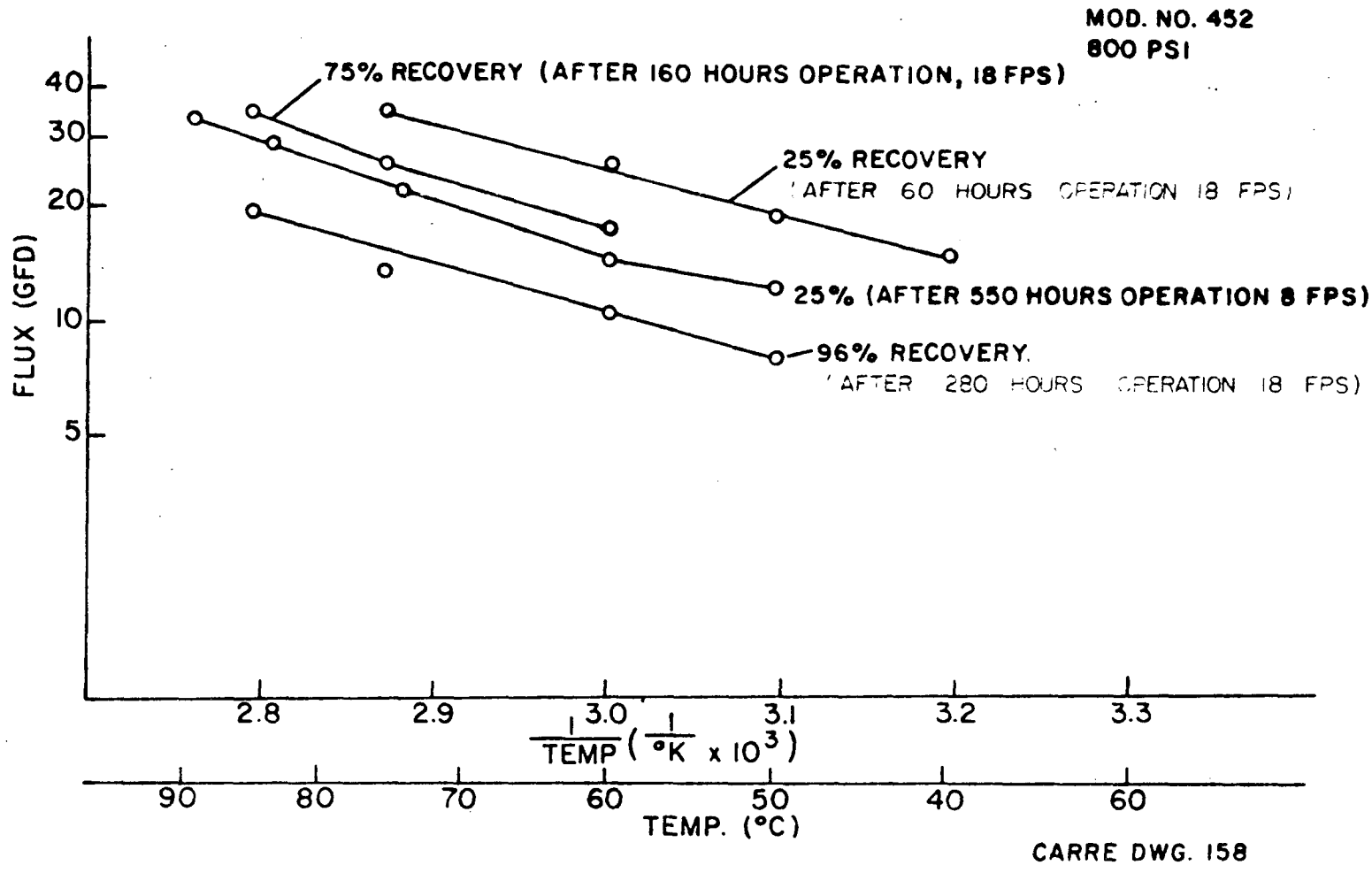

Figure 15. Flux Versus Temperature for Mott-Brandon Module No. 452.

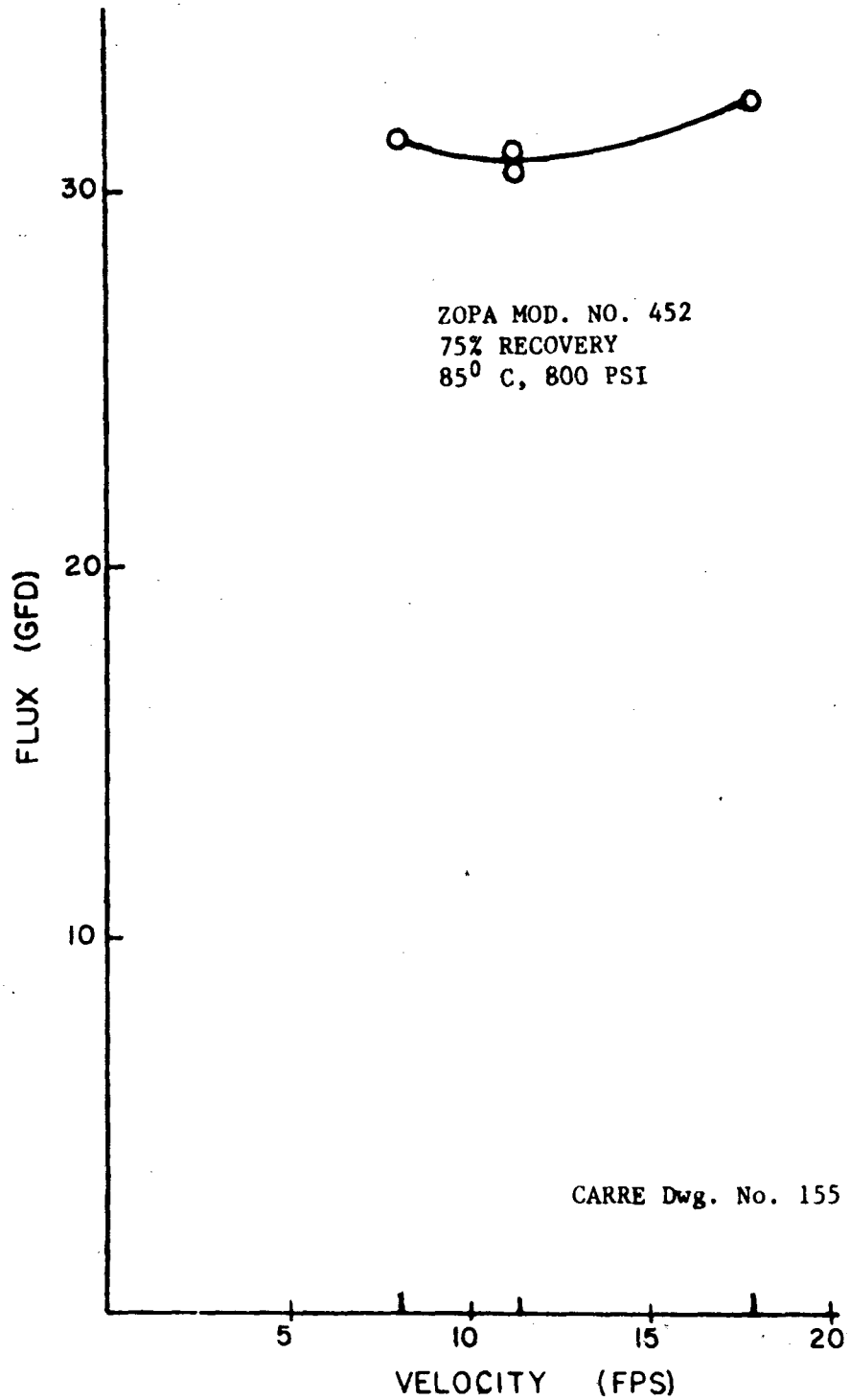

Figure 16. Flux Versus Flow Velocity for Mott-Brandon Module No. 452.

TABLE 11. CHEMICAL ANLAYSES OF CONCENTRATE AND PERMEATE SAMPLES FOR MOTT-BRANDON MODULE NO. 452

RECOVERY LEVEL	25%		25%		75%		75%		96%		25%	
SAMPLE TYPE AND NUMBER	Concen. #1170	Permeate #1171	Concen. #1173	Permeate #1174	Concen. #1177	Permeate #1178	Concen. #1185	Permeate #1186	Concen. #1189	Permeate #1190	Concen. #1217	Permeate #1218
DATE TAKEN	7/18/78	7/18/78	7/21/78	7/21/78	7/26/78	7/26/78	7/28/78	7/28/78	8/3/78	8/3/78	8/29/78	8/29/78
COD, mg/ℓ	150	38	470	46	570	38	1320	69	4270	180	336	24
Conductivity, μmho/cm	135	35	315	67	570	120	815	162	1700	255	395	86
pH	6.5	7.0	7.0	7.4	7.1	7.1	6.9	7.2	5.1	5.6	7.7	7.4
Hardness, mg/ℓ	13	0	29	0	75	0	84	0	232	2.5	18	0
Dissolved Solids, mg/ℓ	187	45	459	42	710	67	1330	122	3280	250	487	45
Total Solids, mg/ℓ	200	45	470	51	750	80	1360	124	3330	250	494	45
Volatile Solids, mg/ℓ	104	37	288	37	405	37	791	52	2150	118	281	18
Suspended Solids, mg/ℓ	13	0	11	9	40	13	30	2	50	0	7	0
Chromium, mg/ℓ	0.041	0.002	0.020	0.003	0.051	0.004	0.340	0.004	0.010	0.027	-*	-*
Copper, mg/ℓ	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	0.483	0.009
Iron, mg/ℓ	0.240	0.018	0.560	0.015	0.660	0.026	1.900	0.024	1.300	0.016	-*	-*
Calcium, mg/ℓ	1.710	0.017	2.800	0.037	16.40	0.32	31.20	0.23	40.00	0.43	4.00	0.0175
Magnesium, mg/ℓ	1.750	0.030	4.780	0.240	9.40	0.39	10.60	0.29	31.40	0.76	2.87	0.030

* Not analyzed

TABLE 12. CHEMICAL ANALYSIS OF CONCENTRATE AND PERMEATE SAMPLES FOR KUSTERS MODULE NO. 480

Recovery Level	96%		25%	
Sample Type and Number Date Taken	Concentrate #1189 8/3/78	Permeate #1191 8/3/78	Concentrate #1217 8/29/78	Permeate #1204A 8/28/78
COD, mg/ℓ	4270	140	336	56
Conductivity, μmho/cm	1700	255	395	87
pH	5.1	5.7	7.7	7.5
Hardness, mg/ℓ	232	2.5	18	0
Dissolved Solids, mg/ℓ	3280	120	487	155
Total Solids, mg/ℓ	3330	120	494	157
Volatile Solids, mg/ℓ	2150	118	281	
Suspended Solids, mg/ℓ	50	0	7	2
Zinc, mg/ℓ	-*	-*	0.131	0.007
Chromium, mg/ℓ	0.010	0.003	-*	-*
Copper, mg/ℓ	-*	-*	0.483	0.008
Iron, mg/ℓ	1.300	0.008	-*	-*
Calcium, mg/ℓ	40.00	0.56	4.00	0.14
Magnesium, mg/ℓ	31.40	0.75	2.87	0.16

*Not analyzed.

conditions for the Kusters module required testing in the high pressure test unit. The flow rate specification was such that it was not possible to divide the 45 ℓ/min output of the test pump between the Kusters module and the MBC (above) module. After consultation with the Kusters technical representative, it was decided to place the module in series upstream of the MBC module. This would provide comparable data since the MBC module has already been operated for 200 hours as the only module in the unit. Figure 17 shows flux and rejection performance of the module No. 480 during the 510 hours of operation. Recovery values were varied between 0 and 85%, pressures between 400 and 130 psi and volume flow rates between 5 GPM and 12 GPM. The temperature was 85°C. Feed prefiltration was the 100 mesh screen.

At 510 hours the module was removed from service after its differential pressure rose to 90 psi. Four hot tap water washes were performed. Between the 390 and 470 hour points the membrane was exposed to wash water from a fiber reactive dye formulation at pH of 9 to 10 and exhibited erratic rejection behavior. Stable operation was restored with a tap water wash. Color rejection remained about 99% throughout the tests.

Figures 18 and 19 show the effects of pressure and temperature on flux. A nonlinear response of flux to pressure is seen at pressures greater than 900 psi. The higher permeability for 96% test occurred first. Like other modules tested, temperature effects generated parallel lines with a slope of about 2800 K (Figure 19).

Discussion

The results are generally those expected for membrane processing of the dye wash water. The formulation contained several low molecular weight species, i.e., the dyes and most of the auxiliary chemicals. The use of the guar gum thickener in the dye pad introduced a high molecular weight constituent which influenced flux performance.

Operation Time and Concentration--

The long periods of time are required to concentrate the test solution to high values of recovery. Therefore the effects of concentration are sometimes not differentiated from the effect of operation time. An increase in recovery level (concentration) generally results in a decrease in flux. These decreases may be irreversible; therefore, the flux observed at 25% recovery after operation at 96% recovery may be lower than the flux initially observed at 25%. This membrane 'memory' necessitates a careful examination of the membrane history before conclusions are made concerning membrane flux.

U.O.P. modules-- Figure 8 shows the flux history of module No. 90. The 67% reduction in flux in the first 20 hours of operation was accelerated by the increase in recovery to 25%. The washing at 65 hours operation restored the flux, but the flux declined quickly after subsequent exposure to the effluent stream.

Figure 9 shows the history of module No. 380. After the failure of module No. 90, a 1-micron cartridge filter was installed in the recirculation line in addition to the 1-micron filter at the system supply line. This second filter removed particulate build-up within the system. A flux decline

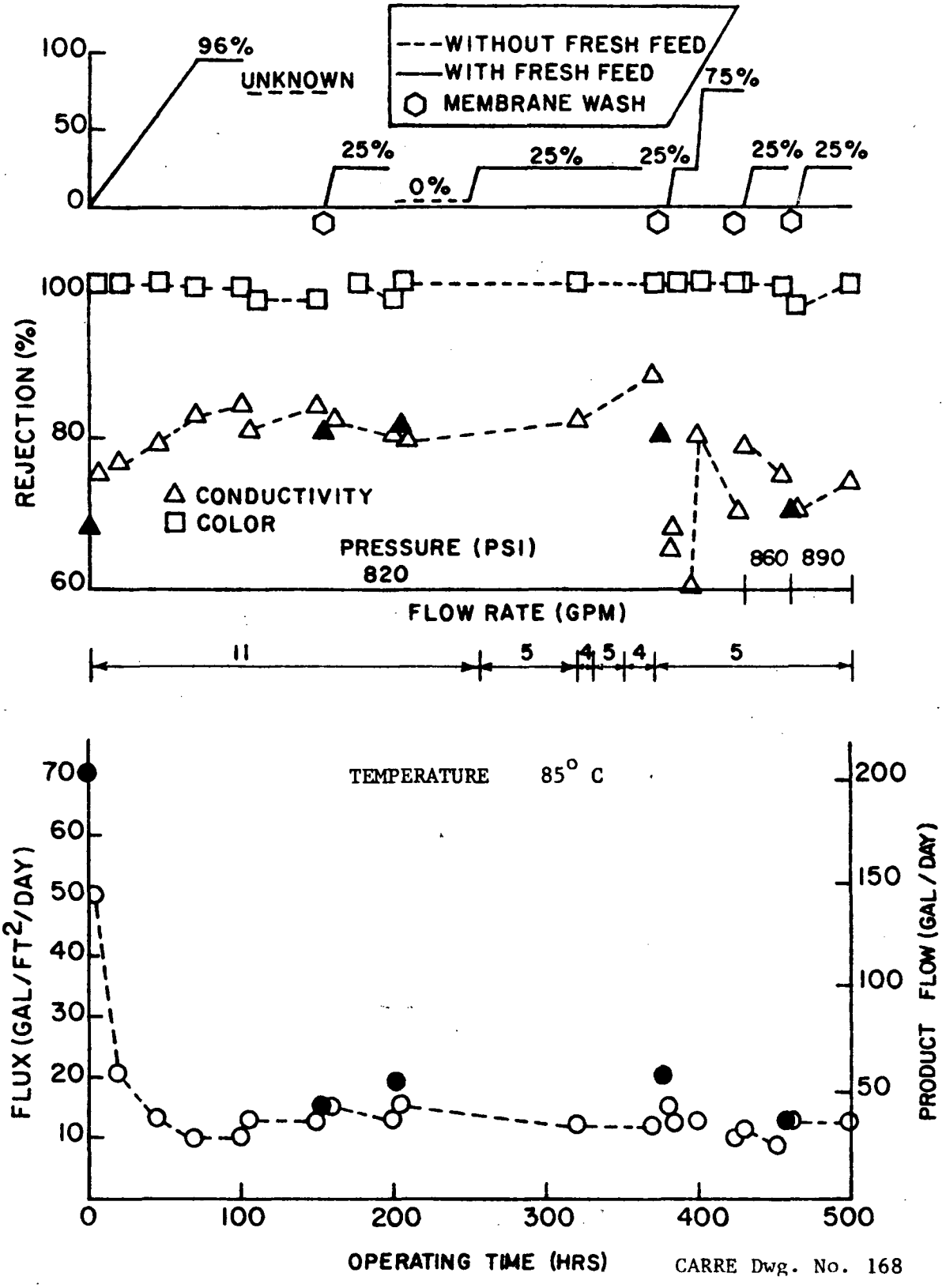

Figure 17. Product Flow and Conductivity and Color Rejection Versus Operation Time for Kusters Module No. 480.

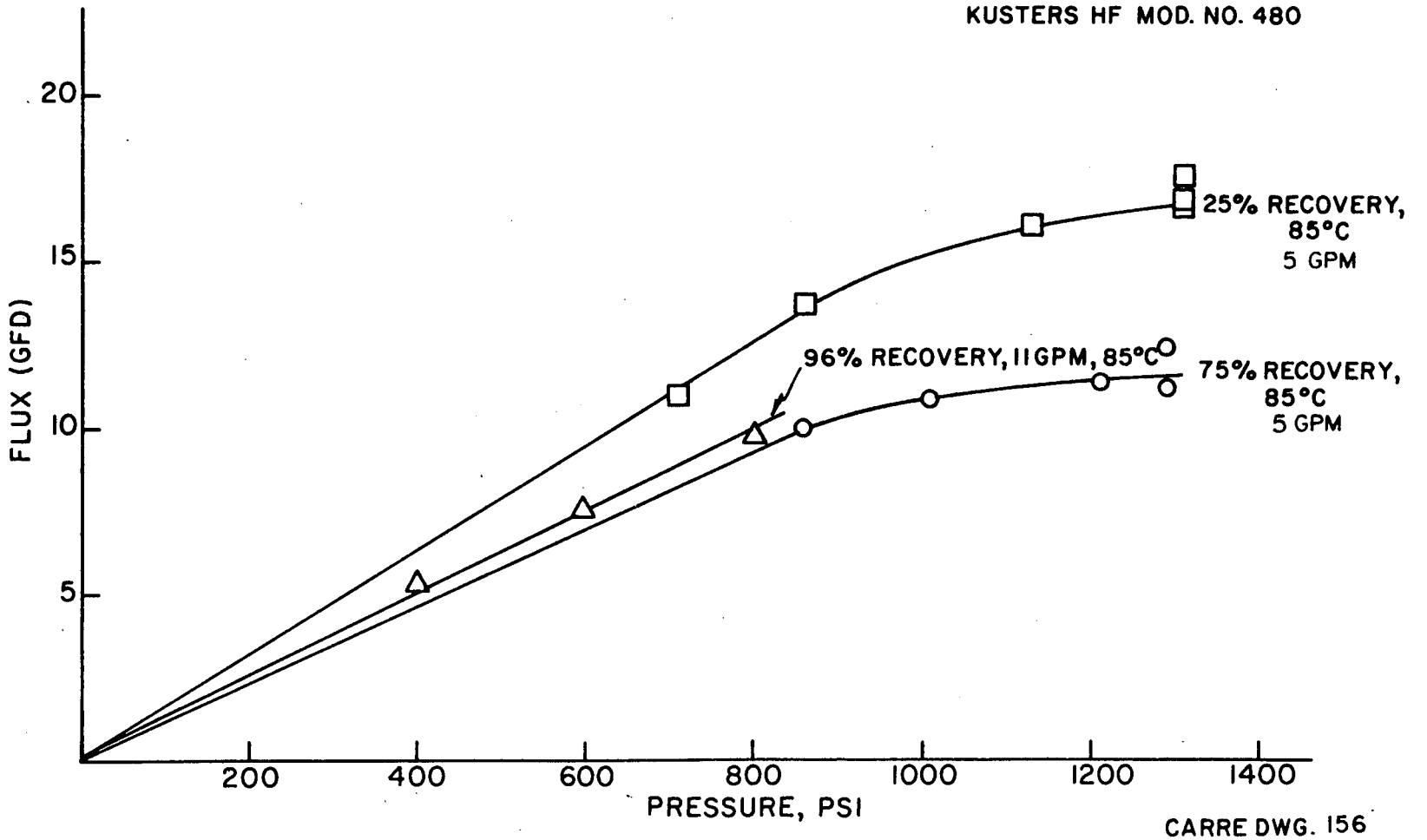

Figure 18. Flux Versus Pressure for Kusters Module No. 480.

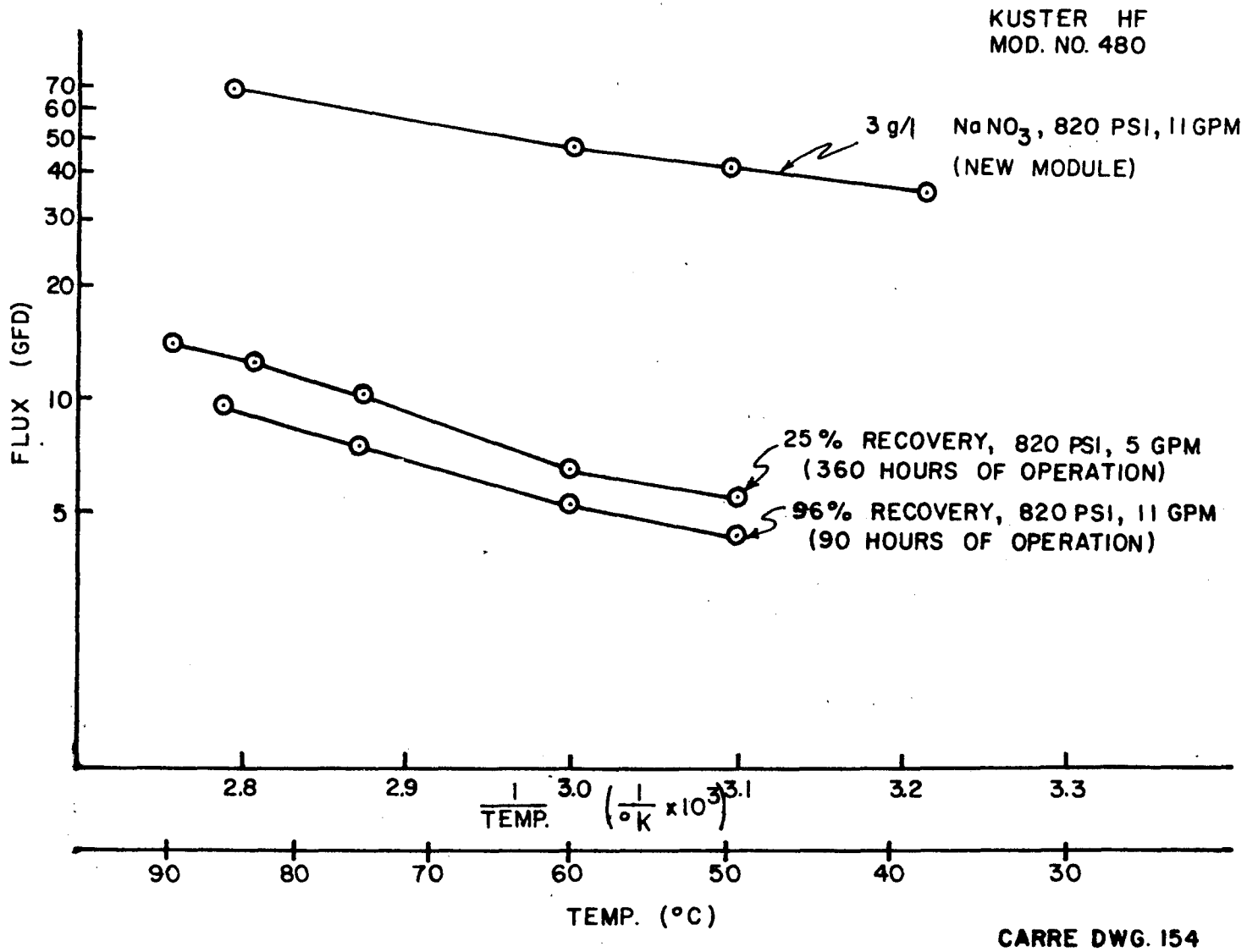

Figure 19. Flux Versus Temperature for Kusters Module No. 480.

similar to the initial decline seen in Figure 8 was again observed. Following the washing of the membrane with tap water, the pressure was lowered from 300 psi to 200 psi. Over the next 300 hours of operation, the flux declined from about 10 GFD to 5 GFD. A wash with UOP "Solution B" resulted in a flux increase. Operation thereafter at a pressure of 400 psi produced a flux decline from 22 GFD to 12 GFD.

A flux increase is sometimes observed for recirculation without the addition of fresh feed. The operating period between 275 hours and 340 hours shows this effect. The flux increase at the 465 hour mark is attributable to a combination of washing the membrane, raising the pressures and reducing the concentration.

Experience suggests that when 500 gallons of fresh effluent per square foot of membrane area is processed, values of flux and rejection are usually stable. Sometimes stable performance is achieved for shorter periods of operation, however.

The module No. 380 processed only 125 gallons of effluent per square foot of membrane area. The results presented in Figure 9 indicate stable values of flux and rejection. The previous experience with module No. 90 indicates that prefiltration requirements for the spiral configuration should be examined carefully.

Mott-Brandon module--Figure 13 shows the history of Mott-Brandon module No. 452 over 700 hours of operation time. The initial decrease in flux from greater than 100 GFD to 50 GFD occurred during the first 20 hours of exposure to the dye wash water. A recovery (concentration) increase to 25% resulted in another rapid flux reduction to ($\sim$30 GFD) at the 20 hour point. Periodic tests with a standard sodium nitrate solution showed this initial loss in flux was not recovered by tap water washing. Color rejection rose significantly during the initial flux decline period while conductivity rejection increased slowly.

Operation without the addition of fresh feed resulted in a flux increase with time. Periodic washing of the membrane with tap water resulted in a partial restoration of flux each time. It appears that the tap water washing may be sufficient to maintain flux at a relatively constant value.

Color rejection remained high throughout the test period. Conductivity rejection remained constant at near 80% except during high pH operation.

Operation at 25% recovery between 340 hours and 390 hours indicate a lower flux than operation at the 75% recovery level between 100 and 180 hours previously. This reduction in flux is probably due to the 96% recovery level operation between 225 and 285 hours.

During the 700 hours of testing the Mott-Brandon module processed 760 gallons of effluent per square foot of membrane area.

Kusters module--The Kusters module was installed, in series, upstream of the Mott-Brandon module on July 31, 1978. Its performance is shown in Figure

17. The two membranes were operated at the same conditions until September 1, when the Kusters module was removed for repairs after the module pressure drop unexpectedly rose. The Kusters module displayed an initial flux decline in the first 30 hours from 70 GFD (with salt water) to 30 GFD. The processing of fiber reactive (high pH) dye wash water resulted in erratic conductivity performance while color rejection remained high.

The Kusters membrane is a zirconium oxide-polyacrylate membrane similar to the ZOPA membrane, therefore, qualitatively similar performance would be expected. Dissimilarities in performance may be attributed to differing hydrodynamic conditions that exist in flux through tubes and flow outside tubes in tube bundles. Hot tap water washes partially restored fluxes.

The Kusters module 488 processes 275 gallons of effluent per square foot of membrane area. The increase in module pressure drop seen at 500 hours indicates prefiltration requirements should be examined carefully.

Pressure--

Flux is expected to be a linear function pressure unless boundary layer resistances to permeate flow become significant compared to membrane resistances. Nonlinearities were seen at high pressures with the Mott-Brandon and Kusters module. The U.O.P. modules were not operated at sufficiently high pressures to observe this phenomenon.

U.O.P. modules--Flux versus module inlet pressure shown in Figure 10. The permeability decreases with increasing time and concentration. The change in permeability from 0.034 to 0.029 GFD/psi during 160 hours of operation is probably a time effect due to fouling or compaction. The offset of the lines from the origin are the result of either inaccuracy in the date or of plotting versus inlet pressure rather than average module pressure.

Mott-Brandon module--Figure 14 shows the nonlinearities of flux versus pressure at approximately 1100 psi where boundary layer flux resistance become significant compared to membrane resistance.

The initial permeability at 25% recovery and 60 hours, is 0.05 GFD/psi. At 530 hours the 25% recovery permeability was 0.035 GFD/psi following an exposure at 96% at 275 hours. The lower permeability is believed to be a result of the intervening exposure to higher concentration.

Kusters module--Figure 18 shows the nonlinearities of flux versus pressure at approximately 900 psi. A higher permeability is shown for 96% recovery at 275 hours. The lower permeability is believed to be a result of the intervening exposure to the higher concentrations.

Temperature--

Figures 11, 15, and 19 show the logarithm of flux plotted versus the inverse of absolute temperature for the three membranes. The slope is nearly constant for all membranes with a value near $2800^{o}K$. Therefore a rise in temperature from 56 to $85^{o}C$ or from 36 to $60^{o}C$ would result in a doubling of flux.

Velocity--

Figures 12 and 16 show the short term effect of velocity on flux. The reason for the shapes of the curve is unclear, but it should be noted that the velocity scans were performed with a constant inlet pressure. In Figure 12 the secondary effect resulting from increasing pressure drop with increasing velocity probably influenced the low flux at the highest velocity.

Long term velocity effect as seen in the flux versus time plots (Figures 8, 9, 13, and 17) are minimal in the range of velocities observed.

SECTION 7

ECONOMICS

The economics of recovery are based on a comparison of the operating cost of the recovery system and the savings due to recycle. Of course, both savings and costs are only estimates at this Phase in the studies. It is the purpose of Phase II and Phase III of this demonstration project to generate empirical data on the economics. In this section are presented estimates of savings for reduced use of energy, water, and chemicals due to recycle. The operating costs for the membrane recovery unit are estimated by the equipment vendors. The payback period is based on the quoted installed costs for the ZOPA membrane system minus the $85,000 estimated to be non-representative for a typical commercial installation.

SAVINGS

The basis for the calculation of the potential savings is the 1979 La France operating budget. Since the membrane system was designed to handle the dye range production based on 120 hours per week including 33% down time, the savings are estimated on this same basis. The savings estimates are tabulated in Table 13.

Reduced Water Reuse

The annual savings of $22,870 attributed to reduced water use is about equally divided between water supply and waste treatment cost, i.e., $11,570 and $11,300 respectively. These savings are realized from reduced energy and chemical requirements. Since the water system facilities are already in place at La France, recycle will reduce neither the depreciation nor the labor cost. For a new plant, both the capital and labor requirement for water supply and waste treatment would be effected by the degree of water recycle in the plant.

It is expected that additional treatment facilities will be required at La France to comply with scheduled future effluent guidelines. Closed cycle operation of the dye range will reduce the cost of these tertiary or additional treatment facilities. However, no estimate of these cost reductions has been made and no credit has been taken for these potential savings.

Reduced Energy Use

The annual savings of $62,000 attributed to reduced energy use is essentially all due to recycle of hot process water; 96% recycle is assumed.

The reduced chemical cost for boiler make-up water treatment of $2,000 is the proportionate part of the annual plant budget for this item.

Reduced Dye Formulation Chemicals Use

The actual budget for dyes and chemicals is proprietary information. The savings are estimated based on the 1979 budget. The reuse scenario is that the production will be scheduled so that the concentrate from light shades can be recycled to darker shades. In a normal shift, eight production runs so scheduled will yield 87% recycle of concentrate even if the concentrate from the last run must be discharged for disposal. (Disposal will be studied during Phase II and Phase III).

The technical feasibility of recycling the concentrate was studied (Appendix C). Additional tests are scheduled in Phase II to establish procedures for scheduling and handling the concentrate to accomplish the reuse. The potential savings of chemicals, and their indirect energy equivalence, is a major factor in the economics of this project. The practicality of chemical recycle is a major question to be answered in this demonstration.

Since the reuse plan calls for recycle only within a single 8-hour production shift, the build-up of extraneous materials is not expected to interfere with the planned reuse. However, any build-up will be monitored.

OPERATING COSTS

The operating costs are estimates as supplied by Mott-Brandon Corporation in response to the invitation to bid. Of course, it is the purpose of this demonstration to obtain actual data during Phase III. The major cost items are labor (including payroll tax), electric power and general maintenance. The annual operating cost is estimated to be $16,510. The breakdown per category is given in Table 14.

PAYBACK PERIOD

The payback period for the $400,000 installed cost of the recovery system, calculated by the standard method employed by Riegel Textile Corporation is 3.8 years. If reuse of dye does not prove to be practical the period is 5.2 years. While the actual payback formula is proprietary, it does contain provisions for taxes and the interest cost of money.

TABLE 13. ESTIMATED ANNUAL SAVINGS[1] DUE TO RECYCLE

1.	Reduced Water Use	$
	Water Treatment	11,570
	Waste Treatment	11,300
2.	Reduced Energy Use	
	Hot Water Recycle	60,000
	Boiler Feed Chemicals	2,000
3.	Reduced Dye Formulation Chemical Use	
	Dyes	61,631
	Auxiliary Chemicals	95,053
4.	Total Savings	$241,554

[1]Based on 1979 operating budget.

TABLE 14. ESTIMATED MEMBRANE SYSTEM[1] OPERATING COSTS[2]

1.	Operator Labor (6 man-months/year)	$ 7,000
2.	Cleaning Chemicals	700
3.	Electric Power (40 horsepower)[3]	5,010
4.	Maintenance Parts	3,000
5.	Payroll Tax	800
6.	Total Costs	$16,510

[1]Based on equipment vendor estimate.
[2]Based on 1979 operating budget.
[3]Based on 2.8 per kilowatt hour; February 1979.

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APPENDIX A. DYE RANGE EFFLUENT CHEMICAL ANALYSES

TABLE A1. VARIATION IN DYE RANGE EFFLUENT WITH TIME FOR AUTOMOTIVE FABRIC

Sampling Point	Time (min.)	Suspended Solids (mg/ℓ)	Conductivity (μmho/cm)	pH	Optical Density
Jet Washer	0	23	200	7.4	0.45
	30	65	290	7.4	0.80
	60	60	290	7.5	0.78
	90	68	290	7.4	0.78
	120	62	300	7.4	0.85
	150	64	320	7.4	0.84
	180	60	315	7.4	0.85
	210	64	315	7.4	0.85
	composite	63	300	7.5	0.82
Dip Box	0	13	160	7.6	0.33
	30	61	320	7.4	1.02
	60	60	350	7.6	1.10
	90	60	350	7.5	1.20
	120	68	360	7.6	1.20
	150	68	340	7.6	1.16
	180	58	370	7.6	1.16
	210	66	380	7.5	1.20
	composite	64	350	7.6	1.16
Wash Box 1	0	19	130	7.2	0.40
	30	55	240	7.2	1.06
	60	52	190	7.3	1.14
	90	58	235	7.3	1.30
	120	58	230	7.3	1.20
	150	54	225	7.3	1.24
	180	56	230	7.3	1.28
	210	56	230	7.3	1.16
	composite	55	225	7.3	1.22
Fix Bath	0	24	8400	4.6	0.26
	30	47	7000	4.7	0.28
	60	59	5400	4.8	0.25
	90	55	4100	5.0	0.41
	120	56	3700	5.2	0.39
	150	42	6500	5.0	0.27
	180	57	4900	5.2	0.32
	210	53	4300	5.4	0.41
	composite	60	5300	4.9	0.34

TABLE A2. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR AUTOMOTIVE FORMULA 9917 (DIRECT AND ACID DYES) FEBRUARY 9, 1978

ASSAY	Pad	Steamer	Jet Washer	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	3850	--	50	100	380
COD, mg/ℓ	35,400	81	460	650	3200
Conductivity, μmho/cm	6500	54	250	250	3200
Alkalinity, mg/ℓ	930	--	50	35	100
Color, ADMI	186600	40	690	1360	420
Hardness, mg/ℓ	<25	--	10	5	700
pH	8.1	9.2	7.4	7.8	4.9
Phenols, mg/ℓ	3.9	--	0.09	0.06	0.31
TOC, mg/ℓ	12060	--	90	190	140
Total Solids, mg/ℓ	20760	39	300	520	6900
Suspended Solids, mg/ℓ	2100	18	31	37	50
Dissolved, mg/ℓ	18660	21	269	483	6850
Chromium, mg/ℓ	25	--	0.052	0.19	0.028
Copper, mg/ℓ	32	--	0.14	0.24	0.03
Iron, mg/ℓ	2.8	--	0.10	0.32	1.2
Manganese, mg/ℓ	0.23	--	0.09	0.06	3.8
Nickel, mg/ℓ	<0.08	--	<0.01	<0.01	0.02
Zinc, mg/ℓ	2.3	--	0.13	0.18	1.4
Magnesium	5.5	--	1.2	1.1	115
Calcium, mg/ℓ	10	--	2.1	1.7	8

TABLE A3. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR AUTOMOTIVE FORMULA 9917 (DIRECT AND ACID DYES) MARCH 16, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	7400	--	--	--	--
COD, mg/ℓ	37300	850	1260	1120	3310
Conductivity, μmho/cm	5200	290	320	230	3200
Alkalinity, mg/ℓ	660	30	28	--	--
Color, ADMI	175500	1020	80	1320	180
Hardness, mg/ℓ	*	9.5	6	--	--
pH	7.8	7.6	7.6	7.3	5.0
Phenols, mg/ℓ	2.1	0.07	0.05	--	--
TOC, mg/ℓ	15700	240	470	--	780
Total Solids, mg/ℓ	20500	530	810	660	6980
Suspended Solids, mg/ℓ	1680	33	58	57	87
Dissolved Solids, mg/ℓ	18820	497	752	603	6893
Chromium, mg/ℓ	28	0.09	0.2	--	0.02
Copper, mg/ℓ	24	0.28	0.29	--	0.025
Iron, mg/ℓ	2.9	0.09	0.15	--	1.15
Manganese, mg/ℓ	0.04	0.04	0.04	--	0.35
Nickel, mg/ℓ	<0.001	0.001	<0.001	--	<0.001
Zinc, mg/ℓ	2.6	0.32	0.49	--	208
Magnesium, mg/ℓ	3.1	1.1	1.0	--	816
Calcium, mg/ℓ	2.3	0.95	0.93	--	9.5

*Dye interferes with method.

TABLE A4. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR AUTOMOTIVE FORMULA 9342 (DIRECT AND ACID DYES) APRIL 25, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	1900	--	--	--	--
COD, mg/ℓ	22160	480	500	464	560
Conductivity, μmho/cm	3000	210	200	160	1700
Alkalinity, mg/ℓ	725	45	45	--	--
Color, ADMI	40190	285	270	365	74
Hardness, mg/ℓ	*	14	14	--	--
pH	7.8	7.3	7.3	7.6	5.6
Phenols, mg/ℓ	1.70	0.31	--	--	--
TOC, mg/ℓ	4880	183	154	--	96
Total Solids, mg/ℓ	11270	400	363	325	2000
Suspended Solids, mg/ℓ	710	20	12	12	2
Dissolved Solids, mg/ℓ	10560	380	351	313	1998
Chromium, mg/ℓ	19	0.10	0.10	--	0.008
Copper, mg/ℓ	18	0.11	0.10	--	0.04
Iron, mg/ℓ	3.5	0.09	0.16	--	1.0
Manganese, mg/ℓ	0.14	0.05	0.07	--	0.46
Nickel, mg/ℓ	<0.04	<0.005	<0.005	--	<0.005
Zinc, mg/ℓ	5.1	0.17	0.2	--	1.4
Magnesium, mg/ℓ	4	1.0	2	--	152
Calcium, mg/ℓ	7	2	2.1	--	16

*Dye interferes with method.

TABLE A5. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR DIRECT DYE FORMULA 9811, MARCH 2, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	2600	44	85	170	110
COD, mg/ℓ	11040	150	400	740	970
Conductivity, μmho/cm	1950	110	300	210	1750
Alkalinity, mg/ℓ	130	22	24	24	19
Color, ADMI	49100	90	62	440	44
Hardness, mg/ℓ	*	10	53	13	560
pH	6.2	7.2	7.1	7.1	4.6
Phenols, mg/ℓ	<0.01	<0.01	<0.01	<0.01	<0.01
TOC, mg/ℓ	2050	42	97	165	215
Total Solids, mg/ℓ	5630	190	470	420	3020
Suspended Solids, mg/ℓ	950	3	53	30	29
Dissolved Solids, mg/ℓ	4680	187	417	390	2991
Chromium, mg/ℓ	<0.05	0.001	0.004	0.003	0.004
Copper, mg/ℓ	31	0.11	0.21	0.42	0.09
Iron, mg/ℓ	1.0	0.04	0.09	0.13	0.38
Manganese, mg/ℓ	0.18	0.027	0.080	0.070	0.20
Nickel, mg/ℓ	<0.07	<0.01	<0.01	<0.01	0.01
Zinc, mg/ℓ	0.06	0.02	0.04	0.04	0.06
Magnesium, mg/ℓ	7.5	3.0	16	3.6	1900
Calcium, mg/ℓ	6.0	1.6	1.6	0.9	15

*Dye interferes with method.

TABLE A6. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR DIRECT DYE FORMULA 9821, MAY 1, 1978

ASSAY	Pad Box	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	2660	--	--	--	--
COD, mg/ℓ	9040	430	800	830	430
Conductivity, μmho/cm	4350	575	800	750	1250
Alaklinity, mg/ℓ	330	130	190	--	--
Color, ADMI	82530	106	222	248	178
Hardness, mg/ℓ	*	27.5	61.1	--	--
pH	6.4	6.4	6.4	6.4	6.1
Phenols, mg/ℓ	0.25	0.001	--	--	--
TOC, mg/ℓ	1730	188	313	--	154
Total Solids, mg/ℓ	9010	724	1160	1170	1480
Suspended Solids, mg/ℓ	1230	24	36	64	36
Dissolved Solids, mg/ℓ	8780	700	1124	1106	1444
Chromium, mg/ℓ	0.062	<0.003	0.003	--	0.003
Copper, mg/ℓ	18	0.13	0.22	--	0.05
Iron, mg/ℓ	0.72	0.14	0.14	--	0.048
Manganese, mg/ℓ	0.17	0.08	0.12	--	0.26
Nickel, mg/ℓ	<0.036	<0.004	0.007	--	0.007
Zinc, mg/ℓ	0.56	0.09	0.11	--	0.52
Magnesium, mg/ℓ	33	42	17	--	169
Calcium, mg/ℓ	7.4	3.7	6.2	--	10

*Dye interferes with method.

TABLE A7. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR DIRECT DYE FORMULA 9168 MAY 1, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	1650	--	--	--	--
COD, mg/ℓ	9680	252	670	870	385
Conductivity, μmho/cm	5300	410	950	810	1010
Alkalinity, mg/ℓ	310	83	176	--	--
Color, ADMI	117590	140	280	770	62
Hardness, mg/ℓ	*	12.7	53.7	--	--
pH	6.3	6.3	6.3	6.3	6.2
Phenols, mg/ℓ	0.52	0.22	--	--	--
TOC, mg/ℓ	2670	100	290	--	256
Total Solids, mg/ℓ	10040	440	1200	1200	1100
Suspended Solids, mg/ℓ	1725	23	35	79	37
Dissolved Solids, mg/ℓ	8315	417	1165	1121	1063
Chromium, mg/ℓ	0.2	0.003	0.003	--	<0.003
Copper, mg/ℓ	26	0.12	0.4	--	0.07
Iron, mg/ℓ	1.0	4.4	0.14	--	0.1
Manganese, mg/ℓ	0.22	0.13	0.11	--	0.20
Nickel, mg/ℓ	0.036	0.004	0.029	--	0.018
Zinc, mg/ℓ	0.56	0.06	0.13	--	0.31
Magnesium, mg/ℓ	24	4.3	17	--	85
Calcium, mg/ℓ	12	2.5	5.9	--	7.2

*Dye interferes with method.

TABLE A8. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR ACRYLIC FORMULA 8951 (BASIC DYES) MARCH 14, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	16400	200	180	175	110
COD, mg/ℓ	43100	620	520	560	380
Conductivity, μmho/cm	4000	240	190	130	460
Alkalinity, mg/ℓ	*	41	34	21	32
Color, ADMI	191000	970	890	1100	180
Hardness, mg/ℓ	*	0**	1.5	0**	63
pH	4.4	6.7	7.1	7.1	7.0
Phenols, mg/ℓ	0.18	<0.01	<0.01	--	--
TOC, mg/ℓ	4580	170	150	150	120
Total Solids, mg/ℓ	16140	370	340	320	660
Suspended Solids, mg/ℓ	5150	1	4	16	72
Dissolved Solids, mg/ℓ	10990	370	336	304	588
Chromium, mg/ℓ	0.03	<0.001	0.002	0.003	0.003
Copper, mg/ℓ	21	0.055	0.039	0.053	0.058
Iron, mg/ℓ	1.2	0.05	0.05	0.46	0.11
Manganese, mg/ℓ	0.14	0.04	0.04	0.04	0.09
Nickel, mg/ℓ	0.04	0.004	0.001	<0.001	<0.001
Zinc, mg/ℓ	1.8	0.07	0.04	0.05	0.06
Magnesium, mg/ℓ	2.7	3.2	3.3	1.5	18
Calcium, mg/ℓ	6.5	1.3	1.3	1.5	2.4

* Dye interferes with methods.
** Zero by method.

TABLE A9. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR ACRYLIC FORMULA 8960 (BASIC DYES) APRIL 25, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	9400	--	--	--	--
COD, mg/ℓ	47360	1730	1300	1390	144
Conductivity, μmho/cm	6500	660	540	390	130
Alkalinity, mg/ℓ	0*	60	64	--	--
Color, ADMI	146970	6300	850	5230	470
Hardness, mg/ℓ	**	133	78	--	--
pH	3.6	4.8	4.9	5.3	7.2
Phenols, mg/ℓ	0.378	0.016	--	--	--
TOC, mg/ℓ	8540	410	430	--	180
Total Solids, mg/ℓ	58370	1210	910	870	155
Suspended Solids, mg/ℓ	3160	14	15	36	14
Dissolved Solids, mg/ℓ	55210	1196	895	834	141
Chromium, mg/ℓ	<0.042	<0.004	<0.004	--	0.004
Copper, mg/ℓ	34	0.42	0.22	--	0.07
Iron, mg/ℓ	1.1	0.16	0.11	--	0.10
Manganese, mg/ℓ	1.0	0.08	0.09	--	0.06
Nickel, mg/ℓ	<0.04	<0.005	<0.005	--	<0.005
Zinc, mg/ℓ	9	0.96	0.57	--	0.06
Magnesium, mg/ℓ	11	6	5	--	1.0
Calcium, mg/ℓ	12	4.2	4.8	--	2.1

*Zero by method.
**Dye interferes with method.

TABLE A10. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR ACRYLIC FORMULA 8956 MAY 11, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	11400	--	--	--	--
COD, mg/ℓ	25400	800	790	800	95
Conductivity, μmho/cm	1580	210	200	170	90
Alkalinity, mg/ℓ	*	1.0	5.0	--	--
Color, ADMI	56030	790	535	600	122
Hardness, mg/ℓ	**	8.8	12.5	--	--
pH	3.8	5.10	5.30	5.70	6.75
Phenols, mg/ ℓ	0.15	0.05	--	--	--
TOC, mg/ℓ	7130	240	240	--	50
Total Solids, mg/ℓ	11120	490	480	470	133
Suspended Solids, mg/ℓ	1880	9	11	26	15
Dissolved Solids, mg/ℓ	9240	481	469	444	118
Chromium, mg/ℓ	0.025	0.005	0.005	--	0.050
Copper, mg/ℓ	5.56	0.041	0.030	--	0.041
Iron, mg/ℓ	0.76	0.093	0.090	--	0.54
Manganese, mg/ℓ	0.14	0.20	0.17	--	0.10
Nickel, mg/ℓ	0.159	0.016	0.01	--	0.10
Zinc, mg/ℓ	6.57	0.24	0.12	--	0.10
Magnesium, mg/ℓ	5.6	2.32	2.96	--	1.16
Calcium, mg/ℓ	5.93	4.55	4.00	--	1.93

*Zero by method.
**Dye interferes with method.

TABLE A11. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR FORMULA 996 (FIBER REACTIVE), FEBRUARY 1, 1978

ASSAY	Pad	Steamer	Jet Washer	Wash Box #1	Wash Box #2
BOD_5, mg/ℓ	1200	6	58	39	5
COD, mg/ℓ	14150	12	630	340	31
Conductivity, μmho/cm	28000	130	1550	280	95
Alkalinity, mg/ℓ	20400	--	700	83	16
Color, ADMI	4400	8	1400	1490	125
Hardness, mg/ℓ	48	--	6	2	1
pH	10.8	9.6	10.6	10.0	8.0
Phenols, mg/ℓ	0.05	--	0.01	<0.01	<0.01
TOC, mg/ℓ	7500	--	230	80	11
Total Solids, mg/ℓ	40240	50	1240	270	74
Suspended Solids, mg/ℓ	160	<1	6	10	3
Dissolved Solids, mg/ℓ	40080	50	1234	260	71
Chromium, mg/ℓ	0.043	--	0.002	0.001	0.001
Copper, mg/ℓ	0.114	--	0.012	0.005	0.002
Iron, mg/ℓ	0.067	--	0.052	0.074	0.050
Manganese, mg/ℓ	0.104	--	0.08	0.04	0.02
Nickel, mg/ℓ	0.12	--	0.01	0.005	<0.005
Zinc, mg/ℓ	0.122	--	0.044	0.019	0.005
Magnesium, mg/ℓ	12	--	1.4	0.8	0.7
Calcium, mg/ℓ	6.4	--	0.8	0.5	0.2

TABLE A12. CHARACTERIZATION OF DYE RANGE EFFLUENTS FOR FORMULA 995 (FIBER REACTIVE), MARCH 13, 1978

ASSAY	Pad	Jet Washer	Dip Box	Wash Box #1	Fix Bath
BOD_5, mg/ℓ	900	--	--	--	--
COD, mg/ℓ	8150	400	220	330	660
Conductivity, μmho/cm	2250	1500	820	420	700
Alkalinity, mg/ℓ	18000	650	--	--	--
Color, ADMI	36750	450	370	2450	110
Hardness, mg/ℓ	*	3	7.5	**	130
pH	10.9	10.8	10.7	10.4	6.2
Phenols, mg/ℓ	0.042	0.013	--	--	--
TOC, mg/ℓ	1850	120	80	160	240
Total Solids, mg/ℓ	26600	1000	600	370	1070
Suspended Solids, mg/ℓ	291	22	11	19	11
Dissolved Solids, mg/ℓ	26309	978	589	352	1062
Chromium, mg/ℓ	0.01	0.001	0.001	<0.001	0.001
Copper, mg/ℓ	0.19	0.007	0.011	0.009	0.007
Iron, mg/ℓ	0.85	0.11	0.05	0.7	0.08
Manganese, mg/ℓ	0.09	0.04	0.03	0.02	0.11
Nickel, mg/ℓ	0.2	0.004	<0.001	<0.001	<0.001
Zinc, mg/ℓ	0.34	0.02	0.02	0.03	0.05
Magnesium, mg/ℓ	5.8	0.9	0.6	0.5	6.8
Calcium, mg/ℓ	5.6	1.4	1.1	1.0	4.2

*Dye interferes with method.
**Zero by method.

APPENDIX B. MODIFIED WASHING PROCEDURE TESTS

Test of high temperature washing have shown that by lowering the rinse water flow rate by about 50% there is no decrease in cloth quality. Increasing the rinse water temperature from 40°C to 60°C, the cloth quality is improved over standard washing conditions with no effect on fabric shade. Further increase in temperature to 82°C (180°F) still improves the cloth quality, but results in shade reduction to be below standard with some standard dye formulations. The amount of additional dye required ranges between 0 and 20%.

CURRENT WASHING EFFECTIVENESS

The effectiveness of the current washing procedures was determined. On 24 production runs samples of cloth were taken: one at the exit of the steamer (prior to washing) and the second just prior to the fix bath (after washing was complete). The 8 inch x 8 inch square samples were washed in a series of one-liter baths. The wash water was collected and analyzed for conductivity, color and dissolved solids. Table B1 shows results for the twenty-four sets of samples. The removal percentage is calculated by the expression:

$$\text{Removal (\%)} = 100 \left(1 - \frac{\text{Amount Removal from Cloth Sample Washed on Range}}{\text{Amount Removal from Cloth Sample Prior to Washing on Range.}}\right)$$

The results indicate that 32% of the removable color is removed in the wash boxes and 72% of the dissolved solids are removed. The average solids removed from the cloth is 7.35 grams per yard. Dissolved solids were measured rather than total solids due to the fraying of the patches during washing giving erratic total solids data. The results show considerable variation from one sample to another. The variations would not be correlated by fabric pattern or dye color code.

HIGH TEMPERATURE, LOW FLOW RATE WASHING TESTS

The washing effectiveness of the continuous dye range at various rinse water flow rates, flow arrangements and temperatures was evaluated. Five pieces (approx. 55 yards each) of cotton/rayon cloth were dyed using a dark brown shade selected by the dye house supervisors as a critical test of temperature effects.

The first piece of cloth was dyed using normal water flow rate, arrangement, temperature, and range speed. The second piece was dyed using a lower water flow rate with normal temperature and complete counterflow to the jet washer; the third piece was dyed using the lower water flow rates with 140°F water temperature and complete counterflow; the fourth piece was

dyed using the lower water flow rate and 160°F water temperature and complete counterflow; and the fifth piece of cloth was dyed using the lower water flow with 180°F water temperature and complete counterflow.

Patch samples were taken at various points along the washer sections. The patches were washed in the laboratory using the procedures described above. Table B2 shows the washing effectiveness results obtained from analysis of the laboratory wash water. The results indicate little effect of flow rate or temperature on removal of dissolved solids from the fabric. Surface color removal is significantly increased at high temperature. Surface color removal is desirable in that the crocking property of the fabric is improved. However, excessive color removal may result in requiring additional dye for some shades.

TABLE B1. WASHING EFFECTIVENESS AS DETERMINED BY DAILY PATCH SAMPLES FROM NORMAL PRODUCTION RUNS DURING THE PERIOD 3/21/78 to 4/20/78.

PATCH NO.	PATTERN CODE	COLOR CODE	SURFACE COLOR REMOVED %	DISSOLVED (1) SOLIDS REMOVED %	DISSOLVED (1) SOLIDS REMOVED gm/yd
1	1174	689	28	99	10
2	1203	249	23	88	21
3	1198	736	18	83	8.7
4	1174	338	44	67	7.4
5	1174	111	45	57	3.6
6	1175	901	67	73	2.3
7	1222	338	29	93	11.3
8	416	703	25	76	2.7
9	416	225	61	69	3.1
10	1175	737	3	96	14
11	1174	336	20	79	11
12	1198	089	38	81	4.6
13	1152	224	41	95	15
14	1211	2249	89	89	3.8
15	1211	8149	16	90	3.2
16	1211	133	26	62	4.4
17	1211	010	20	70	3.9
18	1174	133	10	80	8.3
19	1174	717	42	72	7.0
21	1152	234	26	33	2.8
22	1174	3325	14	30	1.5
23	1112	402	22	16	1.0
24	1112	133	23	57	6.8
25	1174	249	16	67	19
AVERAGES			32	72	735

[1]Removal percentages determined by comparison between results of laboratory washings of patches taken at the exit of the steamer and just prior to the fix bath.

TABLE B2. WASHING EFFECTIVENESS WITH VARYING WASH WATER FLOWRATE AND TEMPERATURE

Temperature °C	Total Range Flowrate ℓ/min	Surface Color Removed %	Dissolved Solids Removed %	Dissolved Solids Removed gm/yd
43	370	-	47	8.9
43	190	47	51	10.3
60	190	42	48	8.7
71	190	53	44	6.9
82	190	64	51	10.0

APPENDIX C. REUSE EVALUATION

The purpose of the reuse evaluation tasks conducted by Texidyne, Inc. were to evaluate the recovery and reuse potential of concentrate and permeate generated by hyperfiltration and ultrafiltration of dye range effluents.

The following evaluations were conducted to evaluate the recovery potential of membrane concentrate and permeate:

1) Fabric samples were dyed in the La France dye lab using membrane water samples to estimate dye recovery potential.
2) Feed, concentrate and product samples were analyzed in the Texidyne laboratory to determine chemical recovery and reuse.
3) Certain tests (e.g., viscosity and foaming measurements) were conducted in the Texidyne laboratory to estimate membrane recovery of dye bath auxiliaries.
4) Analyses of process chemicals were conducted at Texidyne to determine the suitability of recovery/recycle.

All reuse tests conducted were on a laboratory scale and must be verified during plant scale trials. The ultimate reuse and recovery potential of a given waste stream can only be determined during a long-term operation of the membrane treatment system. However, certain preliminary evaluations were made under the limitations of short-term tests.

Concentrates and permeates generated by membrane treatment of dye range wash water were evaluated for reuse potential.

Three dye pad additives — pad thickeners, dyes, and foaming agents — are found in most dye pad formulations used at La France. One objective of the reuse tests was to determine if these additives could be recovered in hyperfiltration concentrates.

Mass balances studied have shown that 35-45% of the dyes and chemicals applied to the fabric in the pad bath are rinsed out in the wash boxes. This means that 35-45% of the process chemicals used on the dye range at La France are theoretically available for recovery. High temperature counterflow washing should remove essentially all of the dye bath auxiliaries from the fabric. Factors limiting recovery potential include degradation of process chemicals, build-up impurities, and the development of satisfactory reuse procedures and production schedules.

Some estimates of chemical recovery can be made based on membrane performance, reuse tests, and characteristics of the process chemicals. Estimates of process chemical recovery from the continuous dye range at La France are listed in Table C1.

TABLE C1. ESTIMATE OF CHEMICAL RECOVERY VALUE IN LA FRANCE DYE RANGE RINSE WATER HYPERFILTRATION CONCENTRATES

Chemical	% Savings from Recycle[a]
Pad Thickener (guar gum)	Inconclusive[b]
Dyes	0-15%[c]
Foaming Agents	40-60%[d]
Salt	60-80%[e]

[a]Expressed as a percent of chemicals applied to fabric.
[b]Recovery of pad thickener cannot be determined during short-term tests.
[c]Dye savings will depend on compatibilities of pad formulas.
[d]Based on foam tests.
[e]Salt is not added to all pad formulations. Recovery data is based on membrane salt rejection performance.

One specific dye pad additive evaluated for recovery was the dye pad thickener. The thickener used at this textile plant was an industrial grade guar gum. A Brookfield Viscometer was used in laboratory tests to measure guar gum concentrations. Attempts to estimate thickener concentrations in membrane concentrations using viscosity measurements were unsuccessful. This is not surprising since the dye range rinse water was circulated through the membrane for periods of up to seven days at elevated temperatures (160-180°F). A full-scale membrane system with shorter residence time may improve guar gum recovery.

Product water samples were evaluated in the La France laboratory for reuse as process water. The laboratory dyeings indicate that hyperfiltration is more suitable than ultrafiltration for recycle of hot dye range wash water. Ultrafiltration products are not suitable for use as general process water because of poor color (dye) rejection. The residual dye in the product could effect the shades of subsequent dyeings.

Hyperfiltration product water was found to be suitable for reuse as general process water. This was found to be suitable for use as process water to prepare dye formulations and for use as hot wash water. Average permeate characteristics, compared to the plant tap water characteristics, are shown in Table C2.

Foaming agents in concentrate waters were estimated using a semiquantitative test. Water samples were agitated in a graduated cylinder and the resulting foam height was measured as an estimate of foaming agent concentrations. The results, Table C3, show that foaming agents were rejected by the membrane. The foaming agents appear to be relatively stable at the temperature conditions of the membrane system.

Concentrate samples generated by membrane treatment of dye range wash water were evaluated in the La France dye lab for dye recovery. The laboratory dyeings showed that considerable dye could be recovered in the concentrate water. However, the savings due to the dye reuse can only be determined during a long series of production runs. The scheduling of production shades will optimize the value of recovering dyes for reuse. Test results are presented in Table C4.

TABLE C2. COMPARISON OF AVERAGE DYE RANGE WASH WATER HYPERFILTRATION PERMEATE WATERS AND LA FRANCE PROCESS WATER

	Permeate Waters[a] 7/18/78-8/31/78	Process Waters[b] 7/78-8/78
COD, mg/ℓ	50	9
Conductivity, μmho/cm	101	90
Hardness, mg/ℓ	2	9
pH	6.5	7.05
Total Solids, mg/ℓ	78	60
Suspended Solids, mg/ℓ	5	3
Dissolved Solids, mg/ℓ	73	57
Volatile Solids, mg/ℓ	39	18
Chromium, mg/ℓ	0.006	0.002
Iron, mg/ℓ	0.047	0.022
Calcium, mg/ℓ	0.57	2.36
Magnesium, mg/ℓ	0.46	1.00

[a]Represents an average of 14 samples.
[b]Represents an average of 8 samples.

TABLE C3. FOAMING AGENT REUSE EVALUATION

Sample Description	Foam Height[1]
Pad Formulation	20
Pad, diluted 1/2 w/deionized water	34
Pad, diluted 1/4 w/deionized water	34
Feed	17
Concentrate	27
Product	<1

[1]Measured in a graduated cylinder. Heights reported are relative.

TABLE C4. SUMMARY OF REUSE EVALUATIONS: DYE PAD FORMULATION RESIDUAL

Sample No.	Sample Description	Reuse Evaluation/Recovery Potential
1093	Dye Pad Drop Mixture	A sample of wastewater taken from a mixture of dye pad residues was evaluated for color in the La France laboratory. The composite residue gave an olive color when applied to a velour fabric with the Laboratory Kusters pad. The addition of yellow (4 g/ℓ) and grey (4.2 g/ℓ) dye to the pad mixture gave a commercial shade (Pat. 1112).
1100	Dye Pad Drop Mixture UF Concentrate	The concentrate from an ultrafilter separation of the above dye pad mixture (1093) was padded with the laboratory dye machine to evaluate color yield. The color obtained was approximately four times as dark as sample 1093 and had the same shade. This result indicates that pad mixture can be concentrated by ultrafiltration. However, the dyes were not separated from the pad thickener.
1098	Dye Pad Drop Mixture UF Produce	Sample 1098 contained no color. Apparently the dye auxiliaries formed a rejecting layer on the membrane since UF membranes do not normally reject dyes. Sample 1099 contained a small amount of color. These waters could be used to prepare a pad formulation, but have no dye value.
1099		

TABLE C5. ANALYTICAL DATA: DYE PAD FORMULATION MEMBRANE SAMPLES

ASSAY	SAMPLE NUMBER #1093	#1098	#1099	#1100
BOD_5, mg/ℓ	--	630	960	4000
COD, mg/ℓ	--	1138	1684	14,592
Conductivity, μmho/cm	--	1550	2350	3125
pH	--	7.1	7.1	7.1
Phenols, mg/ℓ	--	.224	.206	.326
Dissolved Solids, mg/ℓ	--	996	1892	9962
Total Solids, mg/ℓ	6931	1021	1906	10,367
Suspended Solids, mg/ℓ	920	25	14	405
Zinc, mg/ℓ	--	4.6	0.70	1.9
Chromium, mg/ℓ	--	0.014	0.017	0.11
Copper, mg/ℓ	--	1.40	3.83	31.3
Iron, mg/ℓ	--	0.059	0.210	2.41

TABLE C6. SUMMARY OF REUSE EVALUATIONS: DYE RANGE WASH WATER SAMPLES FROM MEMBRANE SCREENING TEST

Sample No.	Sample Description	Reuse Evaluation/Recovery Potential
1061	Dye Rinse UF Concentrate	This concentrate water contained significant amounts of acid and direct dyes. This concentrate was used to develop a production shade. No problems were observed, although more dye (0.637% OWF) was required for the concentrate bath than for the normal production dyeing (0.599% OWF). For this particular shade a negative dye savings was realized because the dye shades were not compatible.
1062	Dye Rinse UF Product	The product water contained approximately 20% as much dye as the concentrate (1061). This amount of dye would prohibit the use of this product sample as general process water. Water analyses also indicate that this water would not be suitable for general process use (total solids = 270 mg/ℓ, COD = 350 mg/ℓ).
1140	Dye Rinse HF Concentrate	A large quantity of direct red dye was recovered in this concentrate. A deep red dye formula was developed from this water with a resulting dye savings of approximately 10%.
1141	Dye Rinse HF Product	This product water contained a small amount of direct dye. Overall water quality was good, and this water should be suitable for most process requirements.

TABLE C6. CONTINUED

Sample No.	Sample Description	Reuse Evaluation/Recovery Potential
1087	Dye Rinse HF Concentrate	This rinsewater concentrate water contained a mixture of dyes that stained a test cloth of a tan shade. Dye savings would result if this concentrate was used to develop a darker tan or brown shade.
1088	Dye Rinse HF Product	This product water was equal to, or superior to, the quality of textile plant tap water. Can be used in all process applications requiring tap water.

TABLE C7. ANALYTICAL DATA: DYE WASH WATER SAMPLES FROM MEMBRANE SCREENING TEST

ASSAY	SAMPLE NUMBER #1061	#1062	#1087	#1088
BOD_5, mg/ℓ	960	120	289	<1
COD, mg/ℓ	2627	346	853	50
Conductivity, μmho/cm	240	170	369	85
pH	8.2	8.2	7.3	7.5
Phenols, mg/ℓ	.060	.040	0.06	0.002
Dissolved Solids, mg/ℓ	1659	266	790	68
Total Solids, mg/ℓ	1711	269	798	68
Suspended Solids, mg/ℓ	52	3	5	<1
Zinc, mg/ℓ	2.99	0.03	0.56	0.029
Chromium, mg/ℓ	0.17	0.006	0.042	0.003
Copper, mg/ℓ	0.19	0.03	0.237	0.014
Iron, mg/ℓ	4.75	0.04	0.53	0.024

TABLE C7. CONTINUED

SAMPLE	SAMPLE NUMBER #1140	#1141
COD, mg/ℓ	1344	43
Conductivity, μmho/cm	1100	200
pH	6.6	7.0
Dissolved Solids, mg/ℓ	1561	127
Suspended Solids, mg/ℓ	15	6
Chromium, mg/ℓ	0.035	0.0035
Iron, mg/ℓ	0.67	0.068
Calcium, mg/ℓ	24.6	0.535
Magnesium, mg/ℓ	17.0	0.70

TABLE C8. SUMMARY OF REUSE EVALUATIONS: WASH WATER SAMPLES FROM LONG TERM MEMBRANE TESTS

Sample No.	Sample Description	Reuse Evaluation/Recovery Potential
1174 1181 1186 1188 1190 1191 1203 1207	Dye Rinse HF Products	Product sample 1190 and 1191 had a pink color or tint. No other product samples contained any measurable dyes. The product waters were equal or superior to the quality of plant tap water. These waters are suitable for general process use.
1180	Dye Rinse HF Concentrate	The concentrate water contained only a small amount of red dye and gave a pink pastel shade when used for a laboratory Kusters dyeing. No trouble was encountered when the sample was used to prepare a compatible plant formulation. This concentrate has minimal dye recovery value; recovery of auxiliaries (e.g., foaming agents) may be significant.
1185	Dye Rinse HF	This concentrate was essentially colorless. The rinse water probably came from a pastel shade, because very little dye was removed in the rinse water. This concentrate should be compatible with most production shades.
1187	Dye Rinse HF Concentrate	This sample contained a small amount of dyes that tinted the test fabric green. This concentrate should be compatible with most medium and dark production shades.

TABLE C8. CONTINUED

Sample No.	Sample Description	Reuse Evaluation/Recovery Potential
1189	Dye Rinse HF Concentrate	This concentrate contained a significant amount of acid red and direct red dyes. The residual dye concentration was approximately 10-15% of the dye required to match production shade.
1202	Dye Rinse HF Concentrate	This concentrate water contained a mixture of dyes that gave a grey shade on the test fabric. Dye savings will be realized if the concentrate is used to dye a grey to black shade.
1211	Dye Range HF Concentrate	The concentrate contained no measurable dye concentration. This type of concentrate is compatible with most production formulas. Recycle value is the recovery of foaming agents and other dye pad auxiliaries.

TABLE C9. ANALYTICAL DATA: DYE WASH WATER SAMPLES FROM LONG-TERM MEMBRANE TESTS

ASSAY	SAMPLE NUMBER #1174	#1181	#1186	#1188	#1190
COD, mg/ℓ	46	23	69	8	180
Conductivity, μmho/cm	67	17	162	19	255
Hardness, mg/ℓ	0[a]	0[a]	0[a]	0[a]	2.5
pH	7.4	5.6	7.2	5.4	5.6
Dissolved Solids, mg/ℓ	42	24	122	11	250
Total Solids, mg/ℓ	51	32	124	15	250
Suspended Solids, mg/ℓ	9	8	2	4	<1
Chromium, mg/ℓ	0.003	0.003	0.004	0.013	0.027
Iron, mg/ℓ	0.015	0.034	0.024	0.045	0.016
Calcium, mg/ℓ	0.037	0.002	0.23	0.002	0.43
Magnesium, mg/ℓ	0.24	0.011	0.29	0.005	0.76
Volatile Solids, mg/ℓ	37	21	52	11	118

[a]0 by method.

TABLE C9. CONTINUED

ASSAY	SAMPLE NUMBER #1191	#1203	#1207
COD, mg/ℓ	140	29	40
Conductivity, μmho/cm	255	19	670
Hardness, mg/ℓ	2.5	0[a]	0
pH	5.7	5.6	11.2
Dissolved Solids, mg/ℓ	120	25	250
Total Solids, mg/ℓ	120	38	250
Suspended Solids, mg/ℓ	<1	13	<1
Chromium, mg/ℓ	0.003	0.003	0.222
Iron, mg/ℓ	0.008	0.016	0.069
Calcium, mg/ℓ	0.56	0.008	0.005
Magnesium, mg/ℓ	0.75	0.006	0.054
Volatile Solids, mg/ℓ	76	37	44

[a]0 by method.

TABLE C9. CONTINUED

ASSAY	SAMPLE NUMBER #1202	#1211
BOD_5, mg/ℓ	--	74
COD, mg/ℓ	1770	296
Conductivity, μmho/cm	1980	380
Hardness, mg/ℓ	130	17
pH	7.4	7.35
Dissolved Solids, mg/ℓ	2465	417
Total Solids, mg/ℓ	2480	423
Suspended Solids, mg/ℓ	15	6
Zinc, mg/ℓ	--	0.202
Chromium, mg/ℓ	3.8	--
Copper, mg/ℓ	--	0.483
Iron, mg/ℓ	0.59	--
Calcium, mg/ℓ	27.0	4.67
Magnesium, mg/ℓ	22.0	3.00
Volatile Solids, mg/ℓ	1170	203

TABLE C10. IDENTIFICATION OF MEMBRANES USED TO GENERATE WATER SAMPLES EVALUATED FOR REUSE

SAMPLE NUMBER	MEMBRANE		TYPE OF SAMPLE
1093	-		Raw Range Rinse Water
1098	Mott-Brandon	UF	Product
1099	GCDM Co.[1]	UF	Product
1100	[2]		Concentrate
1061	Mott-Brandon	UF	Concentrate
1062	Mott-Brandon	UF	Product
1087	Mott-Brandon	HF	Concentrate
1088	Mott-Brandon	HF	Product
1140	Mott-Brandon	HF	Concentrate
1141	Mott-Brandon	HF	Product
1174	Mott-Brandon	HF	Product
1181	UOP	HF	Product
1186	Mott-Brandon	HF	Product
1188	UOP	HF	Product
1190	Mott-Brandon	HF	Product
1191	Kusters	HF	Product
1203	UOP	HF	Product
1207	Mott-Brandon	HF	Product
1180	UOP	HF	Concentrate
1185	Mott-Brandon	HF	Concentrate
1187	UOP	HF	Concentrate
1189	M-B/Kusters[3]	HF	Concentrate
1202	UOP	HF	
1211	M-B/Kusters[3]	HF	Concentrate

[1]GCDM Co., Gaston County Dyeing Machine Company
[2]Concentrate from feed ($1093) with both Mott-Brandon UF and GCDM Co. UF used simultaneously.
[3]Concentrate with both Mott-Brandon HF and Kusters HF used simultaneously.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/2-80-055		

4. TITLE AND SUBTITLE	5. REPORT DATE
Closed-cycle Textile Dyeing: Full-scale Hyperfiltration Demonstration (Design)	March 1980
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
Craig A. Brandon (Carre, Inc.)	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
LaFrance Industries LaFrance, South Carolina 29656	1BB610
	11. CONTRACT/GRANT NO.
	Grant No. S805182
12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
EPA, Office of Research and Development Industrial Environmental Research Laboratory Research Triangle Park, NC 27711	Phase; 9/77-4/79
	14. SPONSORING AGENCY CODE
	EPA/600/13

15. SUPPLEMENTARY NOTES IERL-RTP project officer is Max Samfield, Mail Drop 62, 919/541-2547. EPA-600/2-76-060 is a related report.

16. ABSTRACT The report describes the first (design) phase of a full-scale demonstration of hyperfiltration for closed-cycle operations of a LaFrance Industries dye house. (The remaining three phases are installation, operation, and maintenance.) The decision to demonstrate the process was based on earlier projects that showed hyperfiltration to be potentially economical for recycle/reuse of energy, water, and chemicals in textile preparation, dyeing, and wet finishing. On-site pilot tests of three hyperfiltration modules led to the selection of the Mott-Brandon ZOPA module. Representative wash waters from LaFrance dyeing operations were characterized as a basis for demonstration equipment design. The dye range is to be converted to counterflow with a water flow rate of 50 gpm at 82 C, with 96% of the wash water recovered as permeate for direct recycle. Reuse and/or disposal of the concentrate and dye pad residuals will require further study. Payback period, without credit for chemicals recovery, is estimated to be 5.2 years.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Textile Finishing	Stationary Sources	13H
Dyeing	Closed Cycle Systems	
Filtration	Hyperfiltration	07D
Regeneration		

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
Release to Public	Unclassified	100
	20. SECURITY CLASS *(This page)*	22. PRICE
	Unclassified	

EPA Form 2220-1 (9-73)