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Water Vapor Diffusion Membrane Development

by

Michael K. Tan

January 1976

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IONICS, INCORPORATED
65 Grove Street
Watertown, Massachusetts

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Ames Research Center
NASA

IONICS INCORPORATED

CR137804

TO: NASA
AMES Research Center
Moffett Field
California 94035
Attention: Mr. C. Malich, Tech. Mgr.

FINAL REPORT

covering period

February 1975 - January 1976

WATER VAPOR DIFFUSION MEMBRANE DEVELOPMENT
NAS 2-7651

Submitted by: IONICS, INCORPORATED
65 Grove Street
Watertown, Massachusetts 02172
Tel: (617) 926-2500

Prepared by: M. K. Tan

Approved by: Edgardo J. Parsi
Director of Research

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INTRODUCTION AND BACKGROUND

The maintenance of adequate supply of potable water for astronauts on space missions of extended duration is an important problem. Limitations of launch weight and on-board storage volume preclude the carrying of large supplies of water, necessitating the recycle and reuse of waste-water produced by the astronauts.

Urine constitutes 60% of the waste water produced by a normal male (3.1 pounds water in urine, 2.0 pounds in respired air and 0.2 pound in feces are the average daily waste). Various systems have been conceived and successfully demonstrated by past investigators to recover water from urine. In terms of energy and weight requirements, the various distillation techniques have been most successful.

Hamilton Standard, a division of United Aircraft Corp. had built a prototype vapor diffusion water reclamation unit under contractual agreement with NASA-Ames Research Center. The system had been successfully demonstrated on a 90 day test to be capable of reclaiming 98% of the water (with the addition of a precipitate-solids filter sub-system) and producing water of potable quality.

However, there are a few components in the system that showed marginal performance. One of these was the membrane used as a phase-separator. These membranes, polyvinyl chloride and cellophane, showed limited life and limited structural strength. The overall objective of this program is to develop membrane technology specifically for this water recovery process.

The VDR system is essentially a combination of distillation and membrane permeation. The process involves three interconnected steps:

(i) the selective dissolution of the charge species into the membrane structure. (This is independent of pressure and dependent on temperature and solubility coefficient. The solvent then proceeds to "plasticize" the membrane, loosening the network of polymeric chains);

(ii) the selective diffusion of the dissolved species through the membrane (where sufficient energy of activation is available, the species "wiggle" through the polymeric chain-barrier, from one position to another. The evaporation of the diffused species establishes a concentration gradient across the membrane, resulting in a net flux); and

(iii) the selective evaporation and condensation of the diffused species (evaporation occurs at the "upstream" end of the membrane structure at a rate which is a function of the temperature and the relative concentration and vapor pressure of the different species. The temperature difference between the up-stream membrane surface and the condenser establishes a partial pressure gradient, resulting in a selective distillation.

The evaporated species proceed to diffuse through the vapor phase zone at a rate several times slower than that of liquid diffusion. This, presumably, becomes the overall rate - determining step.

The initial phase of contract #NAS2-7651 concluded that,

(i) a high operating temperature of 85° C (185° F) be used;

(ii) pretreatment of the urine charge, as recommended by Hamilton Standards be omitted,

(iii) membrane fouling is the major problem in the development of membrane technology specifically for VDR applications.

This continuation contract was conducted along this line.

SUMMARY AND CONCLUSIONS

A total of 18 different membranes were procured , characterized and tested in a modified bench-scale VDR unit. Four membranes were selected for further studies involving membrane fouling. The bulk of the work performed under this contract dealt with the problem of flux decline due to membrane fouling. This is discussed in greater details under "Summary and Discussion on Membrane Fouling Studies" presented in pages 47-51. The system was also investigated for low temperature application on wash-water where the permeated water is not recovered but vented into space vacuum.

The following conclusions were reached:

(i) Pretreatment will not significantly increase flux, product quality and membrane fouling and should be omitted in future design considerations. In the case of microporous hydrophobic 'membranes', such as Goretex's Teflon membrane, and in the presence of wetting agent, such as soap, pretreatment will be required to prevent wetting.

(ii) Permeate removal by means of vacuum appear to be superior over removal by sweep gas for several reasons.

(iii) The VDR is applicable for lower temperature treatment of wash water whereby the product water is vented to space vacuum. Microporous hydrophobic membranes, however, are not suitable for this application.

(iv) The level of water removal from the urine charge should preferably not exceed 80% (i.e. total solids should not exceed 15%) for best result.

(v) For any system some turbulence promotion will be required especially when the charge concentration is relatively high.

In the flat sheet configuration, two-channel flow path with ridges is the preferred design for maximizing mixing and decreasing the boundary layer. However, the optimum pumping rate has not been determined.

(vi) Ionics had developed a full-scale VDR unit for the treatment of various food products. This stack arrangement is briefly discussed on the last page of the report. Scale reduction to fit spacecraft requirement can be readily performed.

I. MEMBRANE COMPARATIVE STUDIES

A total of 18 different membranes were procured and characterized. Although several of these had been tested under the previous program, their performance was sufficiently promising to warrant further investigation. All 18 membranes are listed in Table I together with such relevant data as the manufacturers and their code, basic chemical structure, film thickness, Mullen's burst strength, water sorption, porosity and recommended temperature.

The bench-scale VDR unit was also modified to include a prefiltration subsystem. A new charge recirculation pump was used to increase the maximum charge flow rate from 1 liter/min (0.25 gpm) to 5 liters/min (1.25 gpm). This modified system is shown in Figure 1.

The membrane candidates were tested inthe VDR unit, two at a time, using the following operating conditions:

Charge	untreated human urine, 2.8-3.1% total solids
Charge Flow	3 liters/min (0.8 gpm)
Charge Pressure	310 Torrs (6 psi)
Charge Temperature	80-85° C (176-185° F)
Operating Vacuum	700 Torrs (28 inches Hg)
Operation Mode	Recovered water continuously removed for a period of 48 hours until at least 40% by volume of the water in the charge has been removed.

Flux measurement was made at the beginning and the end of each test. Samples were also collected and assayed for total dissolved solids. These data are summarized in Table II. The following paragraphs describe the performance of the membrane candidates in greater detail.

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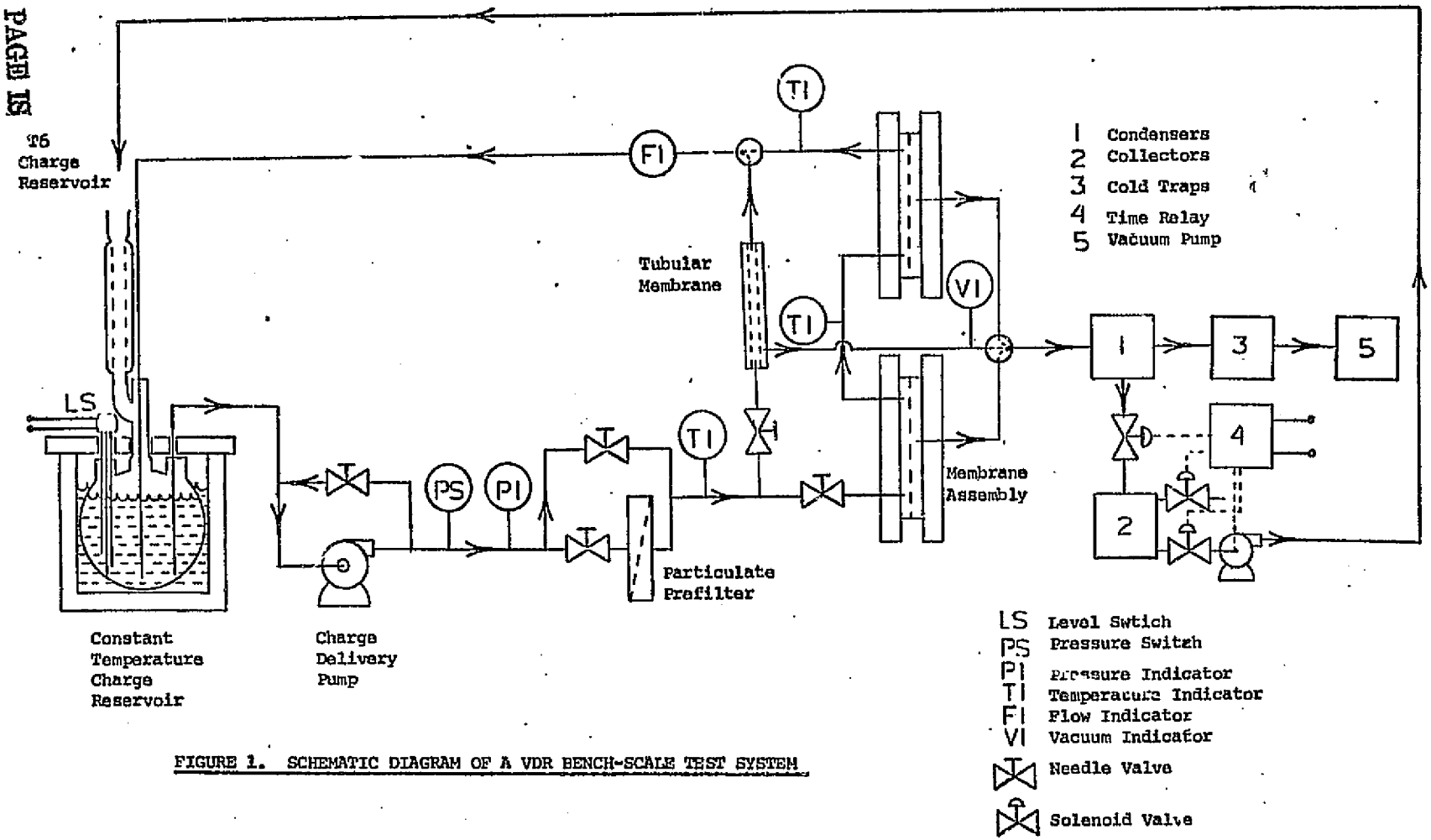

FIGURE 1. SCHEMATIC DIAGRAM OF A VDR BENCH-SCALE TEST SYSTEM

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TABLE I: PROPERTIES OF MEMBRANE CANDIDATES USED IN PRELIMINARY TESTS

Membranes	Manufacturer & Their Code		Basic Structure	Film Thickness* mm (mils)	Water Sorption % Wet Weight	Maximum*** Pore Size μm (mils	Mullen's Torr	Burst* (psi)
M-1a	Ionics	61AZL	Styrene-DVB	.54 (21)	38	-	6800	(130)
M-1b	"	61KQL	" "	.58 (23)	40	-	5700	(110)
M-2a	ENRO	CAB-95	Cellulose Acetate	.087 (3.5)**	64	-	2100	(40)
M-2b	"	CAB-99	" "	.087 (3.5)**	64	-	2100	(40)
M-3	GE	PPOS	Polyphenylene oxide	.026 (1.0)**	68	-	2600	(50)
M-4	NorthStar	NS100	Polysulfone	.062 (2.5)**	73	-	1600	(30)
M-5a	RAI	P1019-37-2	Grafted PTFE	.030 (1.2)	5	-	2600	(50)
M-5b	RAI	P1019-38	Grafted PTFE	.026 (1.0)	7	-	2600	(50)
M-6	RAI	JL200	Grafted HDPE	.038 (1.5)	12	-	2100	(40)
M-7	RAI	P2291	Grafted X-LDPE	.030 (1.2)	10	-	3600	(70)
M-8	Chemplast	Zitex	PTFE	.14 (5.5)	0	1.0(.04)	****	
M-9a	Nuclepore	86A3812	Polycarbonate	.018 (0.7)	20	0.6(.024)	1000	(20)
M-9b	"	86B3A9	"	.018 (0.7)	16	0.03(.0012)	1000	(20)
M-10	Dayco	105	Polyvinyl chloride	.038 (1.5)	10	0.02(.008)	2600	(50)
M-11a	Goretex	S10109	PTFE	.10 (4.0)	0	0.2 (.08)	****	
M-11b	"	S10381	"	.081 (3.2)	0	0.02(.008)	****	
M-11c	"	L10272	"	.23 (9.2)**	0	0.2 (.08)	****	
M-12	Ionics	204RZL	Substituted vinyl polymer	.55 (22)	45	-	7300	(140)

* Measurements made after membrane samples have been equilibrated in H_2O at room temperature
** Thickness includes supporting structure and does not reflect true membrane thickness
*** Values are reported by manufacturer
**** Membrane candidates stretch but did not burst

PTFE = Polytetrafluoro Ethylene
HDPE = High Density Polyethylene
X-LDPE = Crosslinked Low Density Polyethylene

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TABLE II. PERMEATION DATA FROM PRELIMINARY VDR TESTS

Membranes	Mfg. & Their Code		Initial Flux * gm/cm^2-hr	(lb/ft^2-hr)	% Water Removed***	Final Flux gm/cm^2-hr	(lb/ft^2-hr)	Distillate Quality (ppm solids) Initial	Final	REMARKS
M-1a	Ionics	61AZL	0.90	(1.8)	65	0.60	(1.2)	140	160	Marginal performance
M-1b	Ionics	61KWL	2.3	(4.6)	65	1.3	(2.6)	520	490	Inadequate solute retention
M-2a	ENRO	CAB-95	1.7	(3.3)	68	1.3	(2.6)	140	150	Good (requires wet storage)
M-2b	ENRO	CAB-99	1.4	(2.8)	72	1.2	(2.4)	120	110	Good (requires wet storage)
M-3	G.E.	PPOS	1.4	(2.7)	68	1.1	(2.2)	150	180	Good
M-4	North Star	NS100	2.3	(4.6)	72	1.6	(3.2)	130	120	Good
M-5a	RAI	P1019-37-2	>25**	(>50)	Test terminated-low solute retention			>10,000	-	Inadequate solute retention
M-5b	RAI	P1019-38	.035**	(.07)	~0	.035	(.07)	100	110	Inadequate flux
M-6	RAI	JL200	.55	(1.1)	42	.45	(.90)	160	170	Marginal performance
M-7	RAI	P2291	.55	(1.1)	42	.40	(.80)	180	210	Marginal performance
M-8	Chemplast	ZiTex	>25	(>50)	Test terminated -low solute retention			>10,000	-	Inadequate solute retention
M-9a	Nuclepore	86A3812	.35	(0.7)	Test terminated-membrane failure			160	-	Membrane hydrolyzed by ammonia present
M-9b	"	86B3A9	.15	(0.3)	Test terminated-membrane failure			110	-	Membrane hydrolyzed by ammonia present
M-10	Dayco	105	0.00	(0.00)	Test terminated - no flux			-	-	Inadequate flux
M-11a	Goretex	S10109	4.0	(7.9)	Test terminated-low solute retention			13,000	-	Inadequate solute retention
M-11b	"	S10381	1.7	(3.3)	62	1.6	(3.2)	210	190	Good
M-11c	"	L10272	3.5	(7.0)	Test terminated-low solute retention			12,000	-	Inadequate solute retention
M-12	Ionics	204RZL	0.90	(1.8)	45	0.60	(1.2)	200	190	Marginal performance

* Operating at 80°C (180° F) using actual human urine as the charge
** Operated at 49° C (120° F)
*** The system was charged such that after 48 hours of continuous operation 50% or more of the water would have been removed.

M-1 a,b Ionics' NEPTON. These membranes are essentially copolymers of ethyl vinyl benzene, styrene, and divinyl benzene which have been polymerized, crosslinked, and sulfonated prior to their commercial use in electrodialysis stacks. The degree of crosslinking can be controlled by adjusting the amount of divinyl benzene used, and the amount of interstitial polymerizate space can be tailored by using appropriate amount and type of non-polymerizable solvent. The membrane thickness is controlled by using different reinforcing cloth backings.

Two of these membranes were tested - 61AZL and 61 KWL which use diethyl benzene and diglyme (bis-2-methoxyethyl ether) respectively, as the non-polymerizable solvent. The difference in porosity of these two membranes was thought to be sufficiently large as to illustrate the relationship between porosity and membrane fouling.

As seen from Table II the flux decline during the 48 hour operation (65% of the water being removed) is quite significant - 33% for 61AZL and 43% for 61KWL! Moreover, the product quality for the 61KWL is unacceptably high (490-520 ppm solids), and the final flux for the 61AZL (0.60 gm/cm^2-hr or 1.2 lb/ft^2-hr) is very marginal.

Attempt was made to recover the flux decline by repeatedly flushing the membrane with hot water. No significant flux recovery was observed. On disassembling the unit a permanent brown stain was observed, corresponding to the charge flow pattern. Apparently, foulin occurred because of the infusion of organic foulants into the interstices of the membrane and became physically or chemically entrapped there.

For these reasons, M-1a and M-1b were not selected for further investigations.

M-2a,b Envirogenics' Cellulose Acetate Membranes. These are essentially cellulose diacetate-triacetate blend membranes heat-treated at 90° C. Commercially used in spiral-wound modules in reverse osmosis systems, these membranes are of the Loeb-Sourirajan type with a thin dense s elective surface layer supported by a microporous, thicker layer of the same polymer. The membranes used were "CAB-95" and "CAB-99" with the suffix denoting the salt rejection in 1% salt solution at 600 psi in an RO system.

The performance of these membranes in the VDR unit is summarized in Table II. The flux at 70% water removal is quite high (1.3 gm/cm^2-hr) and the flux decline is significantly better than M-1a,b (23% and 14% flux loss, respectively). Moreover, the flux decline is recoverable by repeated water flush. As such, M-2a and M-2b were selected for further investigation.

M-3 General Electric's Sulfonated Polyphenylene Oxide Membrane. This membrane consists of a thin, active sulfonated 2, 6-dimethyl polyphenylene oxide layer (which is 5-12 micrometer thick, i.e. 0.2-0.5 mil) cast on a microporous polypropylene substrate. Its water permeability is claimed to surpass those of cellulose acetate membranes in RO system. Ionic species are retained by the presence of pendant negative sulfonate groups which make the thin layer permselective.

The VDR performance of this membrane is comparable to that of M-2a and M-2b; and while the M-2a,b membranes require wet storage, M-3 does not. As such, this membrane was also selected for further investigation.

M-4 North Star Research's Polysulfone Membrane. This is an asymmetric (Loeb-Sourirajan type) membrane used in reverse osmosis system. It is claimed to exhibit significantly higher flux, higher temperature resistance, and wider pH operation range than conventional cellulose acetate membranes. Developed by North Star Research it is available in flat sheet, as well as tubular configuration.

This membrane exhibits the highest flux of all membranes tested in the program and also exhibits good solute retention. Although, the decline in flux with increasing water removal is particularly severe, the flux still surpasses that of most other membranes tested. It was, therefore, selected for detail studies.

M-5a, M-5b, M-6, M-7 - RAI Research Corporation's PERMION Films. These are essentially polyethylene films (M-5a, b being polytetrafluoroethylene, M-6 being high density polyethylene, and M-7, crosslinked low density polyethylene) with grafted side chains (M-5a,b have different amounts of negative sulfate groups, M-6 has positive pyridine groups, while M-7 has negative methacrylate groups). These are commercially used as battery separators. They are hydrophilic and possess adequate mechanical strength that it was thought they would be suitable for VDR application because of these favorable properties.

However, the permeation data obtained for these membranes (shown in Table II) indicate that M-5a is too microporous, allowing the passage of certain urine solutes and causing significant product water contamination. On the other hand, M-5b yields negligible flux and is also unsuitable for VDR. M-6 and M-7 both exhibit sufficient flux (.55 gm/cm^2-hr or 1.1 lb/ft^2-hr for both membranes) as to be useful for VDR application. However, there are significantly below that of the other membrane candidates so that M-6 and M-7 were not selected for detailed studies.

M-8, Chemplast's ZITEX PTFE Films. These are essentially microporous polytetrafluoroethylene diaphragms. They are hydrophobic in nature and are commercially available in flat-sheet configuration with maximum pore sizes ranging from 1.0 micrometer (.04 mil) to 8.0 micrometers (.32 mil). As the permeation data in Table II show, even the tightest diaphragm (i.e. 1.0 micrometer) is so microporous that it allows passage of urine solutes, causing product water contamination.

M-9a,b Nuclepore Corporation's Polycarbonate Membranes. These are made by a patented irradiation-and-etching process on polycarbonate films. Averaging 10 micrometers in thickness (0.4 mil), these microporous membranes are available in maximum pore sizes ranging from 0.03 μm to 8.0 μm (.012 mil to 0.32 mil).

Initially, it was thought that this wide range in porosities will be particularly interesting in studying membrane fouling and its relationship to membrane porosity. Membranes with maximum pore sizes of 0.03 μm (.0012 mil) and 0.6 μm (.024 mil) were obtained and tested in the VDR unit. Initial

fluxes of .15 gm/cm^2-hr (.30 lb/ft^2-hr) and .35 gm/cm^2-hr (.70 lb/ft^2-hr), respectively, were obtained. However, after 3 hours of operation, membrane failure occurred. On disassembling the unit it was discovered that the hot urine had attacked the membranes. Evidently the urea had decomposed, forming ammonia, and, as reported in literature dealing with plastics, hot ammonia will attack polycarbonates.

M-10, Dayco's Polyvinyl Chloride Films. These are homogeneous films of polyvinyl chloride, available in only one porosity. The thinnest one available, the #105, was selected. (Thickness = 0.38 μm or 1.5 mils). Unfortunately, no flux was observed even after 6 hours of operation.

M-11a,b,c Gore-Tex's PTFE Films. These are homogeneous, microporous films whose structure consists of a matrix of nodules interconnected with fibrils with open pore spaces between the fibrils. The manufacturer claims to be able to control the process to yield a product having almost any desired proportion of pore volume and average pore-size.

Two commercially available films were selected - one with a maximum pore size of 0.2 μm (.08 mil) and another with a maximum pore size of 0.02 μm (.008 mil) and are labelled M-11a and M-11b, respectively A third film, consisting of a composite of M-11a and a porous polypropylene support layer, was also procured for testing (M-11c).

The permeation data are summarized in Table II. M-11a yielded excellent flux. However, the solute retention was quite low. Distillate samples were yellowish, possessing a strong, obnoxious odor and contained 13,000 ppm dissolved solids. The tighter film, M-11b also yielded high-flux. In addition, the distillate quality was acceptable (190-210 ppm) and

the flux decline with the amount of urine processed was significantly less than that observed in all the other membranes. As such, M-11b was selected for detailed studies.

M-11c produced essentially the same data as M-11a.

M-12 Ionics' 204 RZL. This membrane is a product of bulk polymerization of ethylene glycol dimethacrylate (EGDM) and dimethyl aminoethyl dimethacrylate (DMAEM) which has been subjected to a quaternizing process involving an aryl-alkylhalide.

The permeation data shown in Table II indicate that this membrane candidate is not very promising - the flux is marginal and the flux decline with processing time is substantially higher than most of the other membrane candidates.

In summary, a total of 18 membrane candidates were tested in the VDR unit and the 4 most promising candidates were selected for more detailed studies. Emphasis will be placed on membrane fouling. The selected membranes were:

(i) M-2b, ENRO's CAB-99, an asymmetric cellulose acetate membrane;

(ii) M-3, G.E.'s PPOS, a homogeneous sulfonated polyphenylene oxide membrane;

(iii) M-4, North Star Research's NS100, an asymmetric polysulfone membrane; and

(iv) M-11b, Gore-Tex's S10381, a homogeneous polytetrafluoroethylene film.

II. MEMBRANE FOULING STUDIES

Introduction

Central to the efficient operation of any permeation process, assuming acceptable solute retention, is a high water flux and a minimum flux decline with increasing use of the membrane. Flux determines the design considerations of the unit. The higher the magnitude of the flux, the smaller the membrane area would need to be in order to fulfill on-board requirements. Total membrane area determines the size and weight of the hardware and, to a smaller extent, the energy requirement of the unit.

Previous data have demonstrated that flux will decline with increasing urine treatment due to a phenomenon known as membrane fouling. This brings into consideration complicating factors such as the rate of flux decline, the prevention or alleviation of such a decline, the reversibility of this decline and the overall membrane life.

In order to understand the phenomenon which leads to flux decline, it is important to investigate and analyze the cause of this decline, the extent to which it can be alleviated and reversed and to distinguish this from systemic factors that are independent of the membrane such as the energy requirement of selective mass transport. To do this, the work program has been divided into three major areas of investigation:

1. Operating Parameters
2. Fouling Species
3. Recovery of Flux Decline

Operating Parameters

These are the dependent variables such as operating temperature, charge flow rate and the extent of water recovery which can be manipulated within limits.

Operating Temperature: The relationship between the flux and the operating or charge temperature had been shown to be of the Arrhenius type, i.e.

$$J = J_o \, e^{E/RT} .$$

where J is the flux, J_o and E are constants (with E representing the energy of activation for the system), R is the gas constant and T the absolute temperature. The singular importance of a high operating temperature for the attainment of high flux had also been shown in the previous work program.

This investigation concerns the effect of operating temperature on membrane fouling rate. In Experiments 1-4, the four selected membranes were tested at three different temperatures - 60° C, 70° C and 85° C (140° F, 160° F and 185° F) - using untreated human urine. The data are illustrated in Figures 2, 3, 4, 5 for the M-2b, M-3, M-4 and M-11b membranes, respectively. The rate of flux decline is temperature-dependent for the first three membranes and is more severe at higher operating temperature. The hydrophobic M-11b is apparently not appreciably affected by fouling for the particular charge concentration and operation time involved. Despite the higher rate of flux decline, the final water flux is still significantly higher at the higher operating temperatures. As such, unless it can be demonstrated that higher operating

Figure 2. FLUX OF M-2b AS A FUNCTION OF OPERATING TIME AND TEMPERATURE

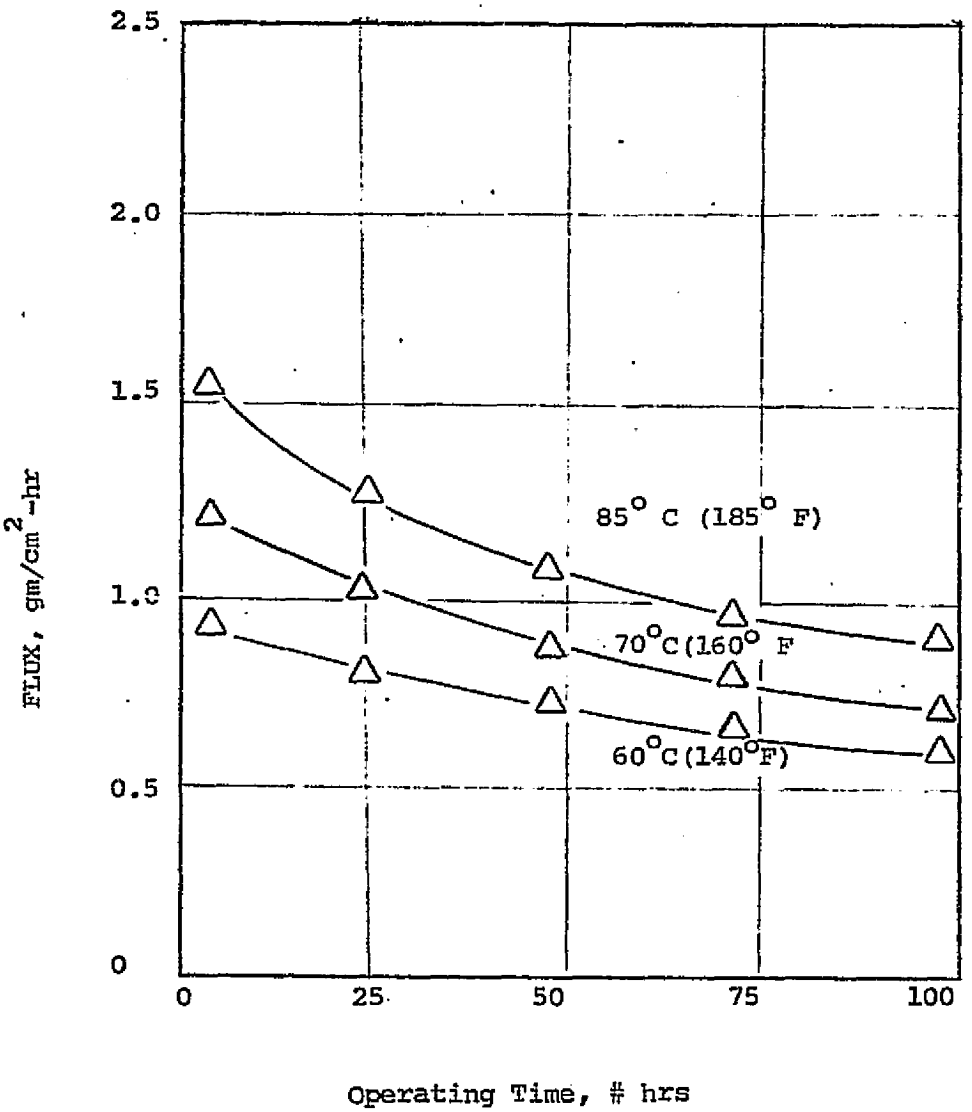


FIGURE 3. FLUX OF M-3 AS A FUNCTION OF OPERATING TIME AND TEMPERATURE

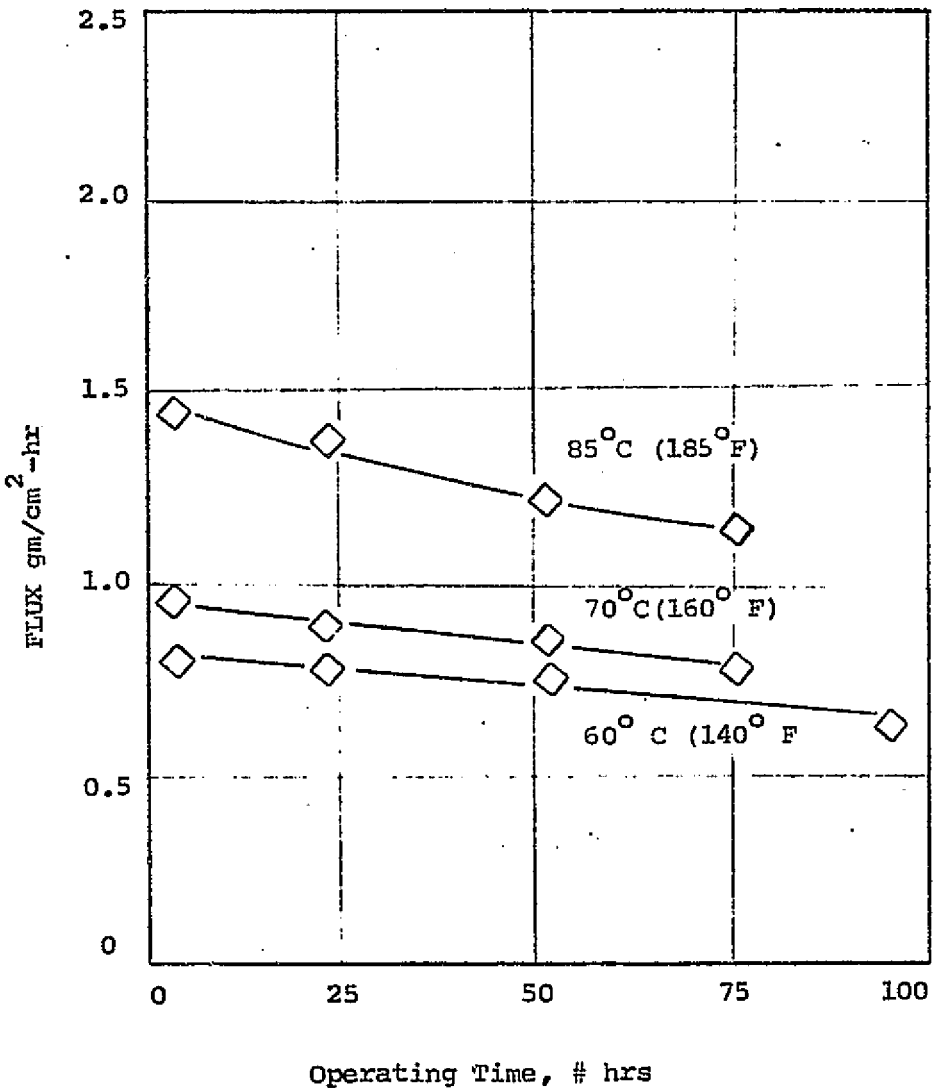


FIGURE 4. FLUX OF M-4 AS A FUNCTION OF OPERATING TIME AND TEMPERATURE

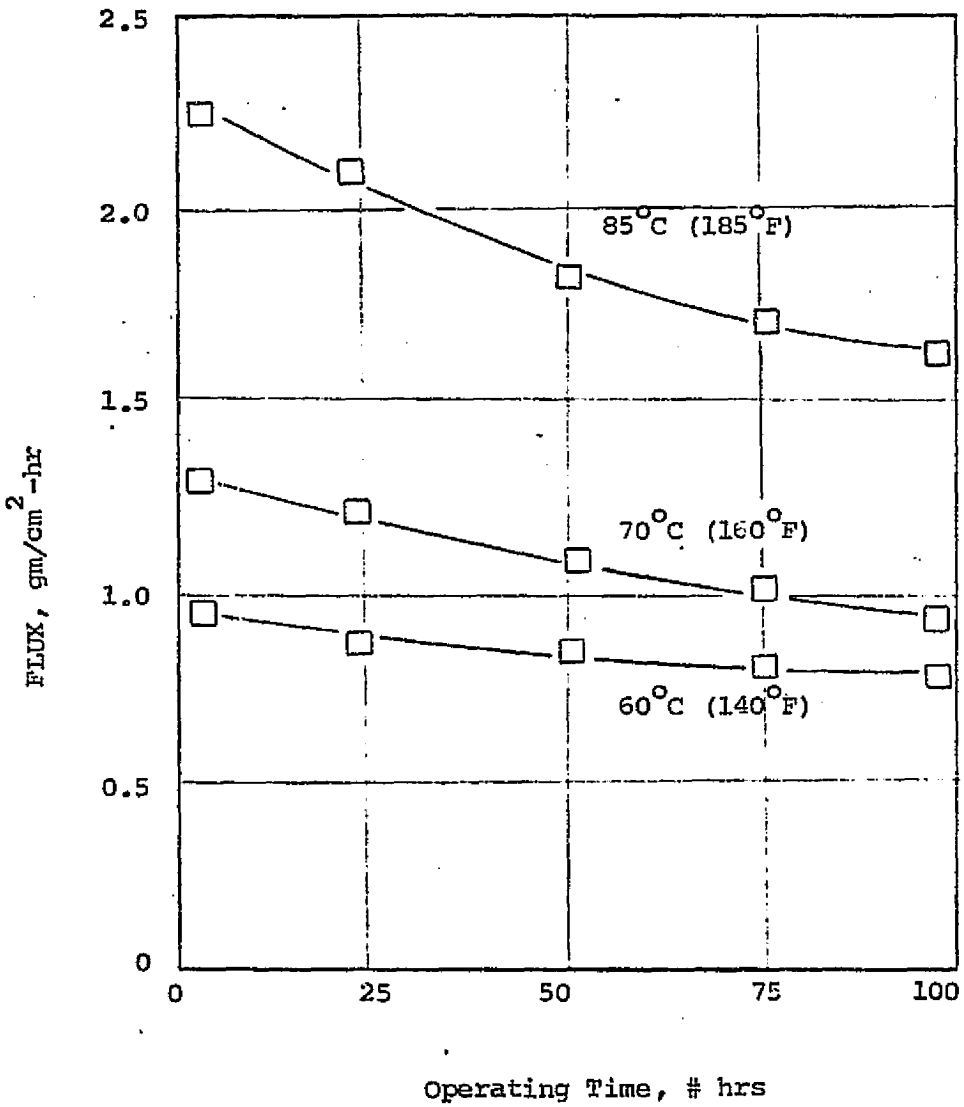
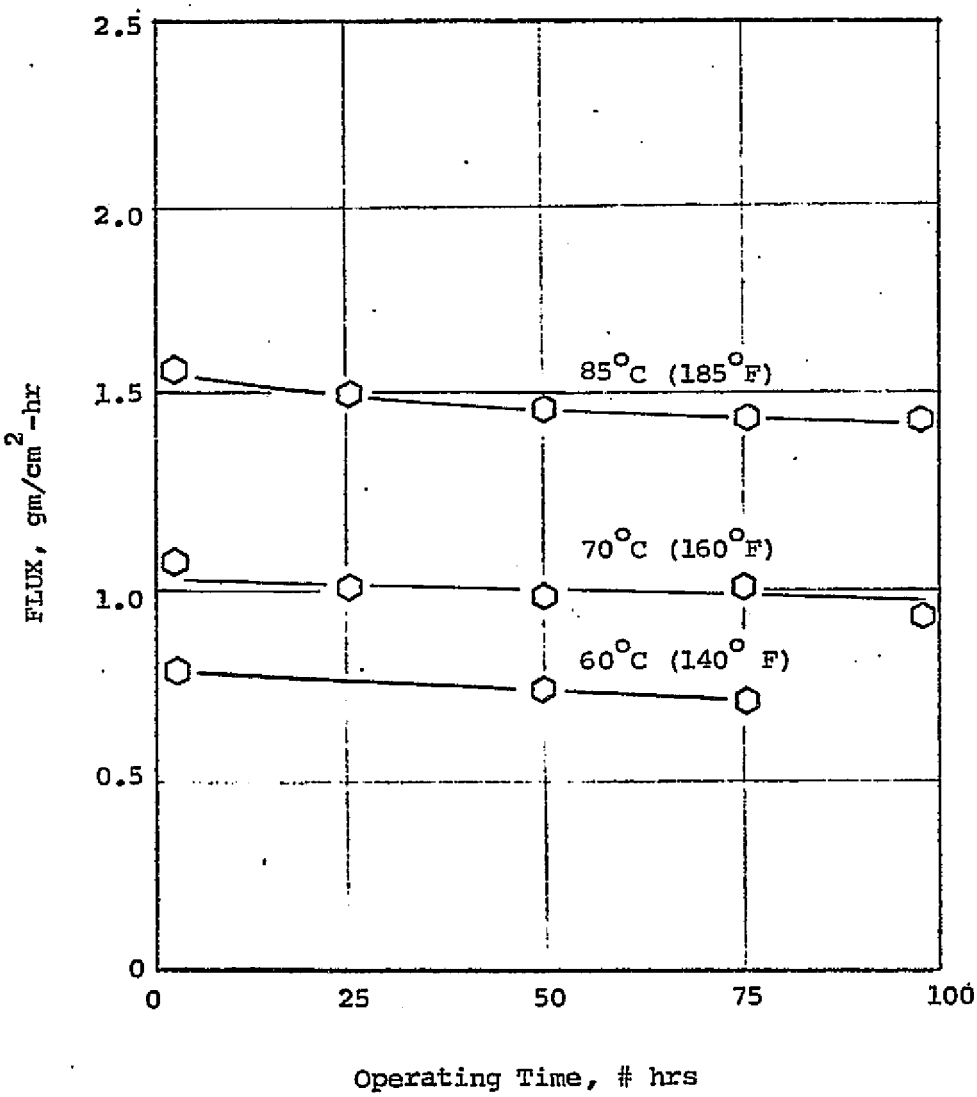


FIGURE 5. FLUX OF M-11b AS A FUNCTION OF OPERATING TIME AND TEMPERATURE



temperatures will have adverse effect on membrane life or lead to other problems, e.g. urine decomposition, the highest permissible temperature should be used.

It should also be noted that at higher temperatures the membrane passed significantly larger amounts of water within the particularly time-span noted in the graph. The data were, therefore, replotted to relate flux decline to the amount of water that passed through the membane. Figure 2 is, thus, translated into Figure 6 which shows that flux decline is not significantly more severe at higher temperature.

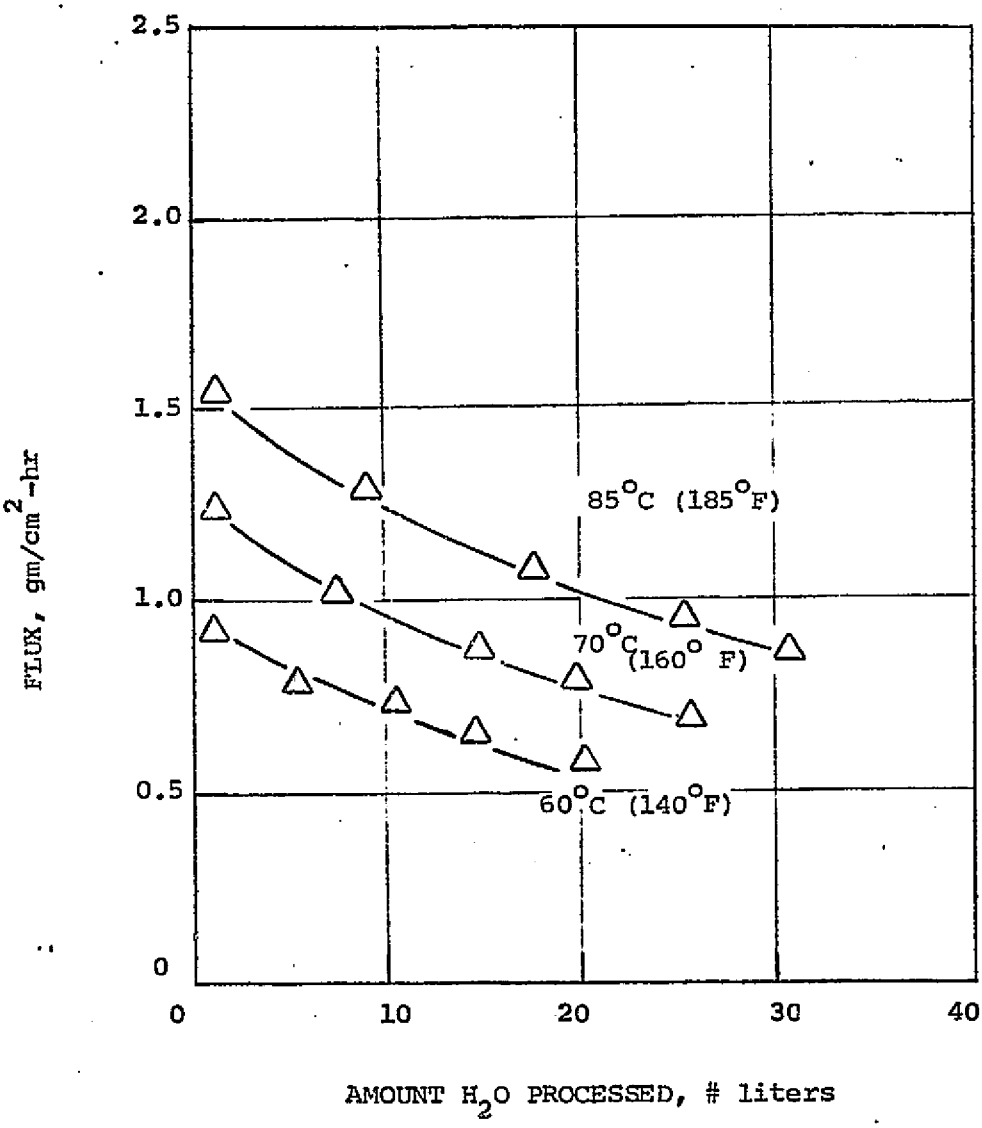
Charge Flow Rate. The effects of charge flow rate or velocity on membrane performance are intimately connected with the concept of concentration polarization. The effects of concentration polarization (the increase in solute concentration at the boundary layer over the concentration in the bulk solution) on selective mass transport have been well documented. Their impact in the VDR system is expected to be threefold:

i) Water flux, being an inverse function of charge solute concentration, will decrease.

ii) Since the selectivity of the membrane is imperfect, solute diffusion through the membrane will also increase. This might lead to plugging up of the membrane pores, as well as an increase in the level of solute contamination in the product water.

iii) The extent of the concentration increment could be such that the solubility limits of some solute species are exceeded causing precipitation at the membrane surface.

FIGURE 6. FLUX OF M-2b AS A FUNCTION OF OPERATING TEMPERATURE AND AMOUNT OF H_2O PROCESSED

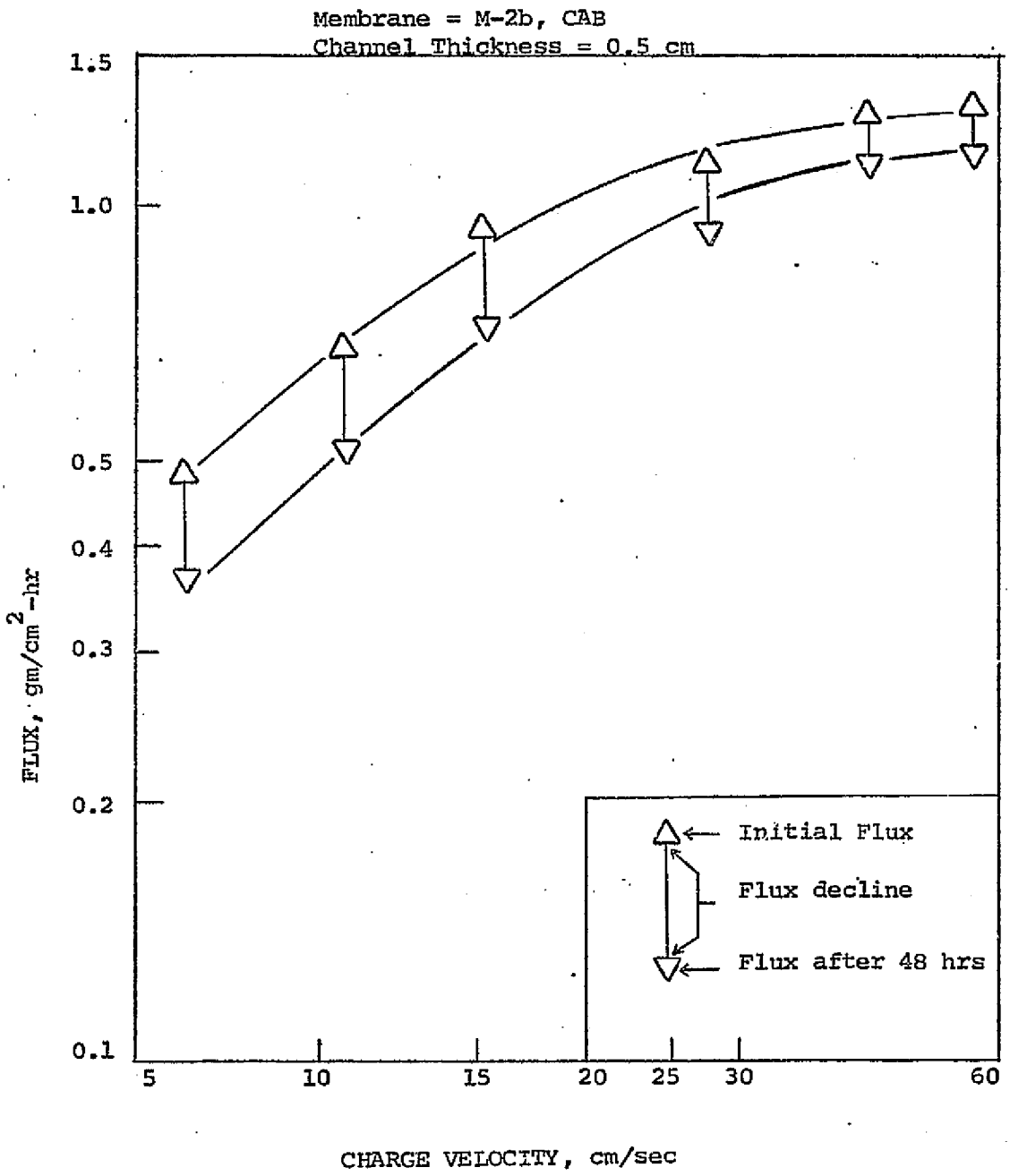


The adverse effects of concentration polarization on mass transport would be significant so long as the diffusion through charge-membrane interface is the limiting condition in the overall permeation process. This may not be the case under certain operating conditions. In fact, several past investigators have suggested that the limiting step is the diffusion of the water vapor through the membrane after phase-change has taken place. However, the membrane may also become plugged up as a result of solute precipitation or crystallization, either on the membrane surface or within the membrane interstices. This effect of concentration polarization may be cumulative and irreversible.

Experiment 5 involved the variation of the charge flow rate while all other operating parameters were kept constant using unconcentrated human urine as the charge. The increase in charge flow would create turbulence in the bulk solution and decrease the effective thickness of the boundary layer, minimizing the effects of concentration polarization. The effects of turbulence promotion might be manifested in (a) the variation in flux; (b) the variation in product quality; and (c) the variation in the rate of flux decline.

Figure 7 summarizes the data obtained in this experiment. Flux was monitored, both initially and after 48 hours of operation, for six different charge velocities. The former series of points (top row of points in Figure 7) shows essentially the "efficiency" of the process, while the difference between it and the latter series of points (vertical lines in Figure 7) reflects the rate of flux decline due to membrane fouling.

FIGURE 7. EFFECTS OF CHARGE VELOCITY ON FLUX



The plot indicates that flux varies as the 0.7 to 0.75 power of the velocity up to 18 cm/sec beyond which increments in velocity lead to progressively smaller increments in flux. Above 30 cm/sec any increase in charge velocity has negligible effect on membrane flux. The same plot also indicates that the flux decline over the 48-hour period is somewhat velocity-dependent, being more severe in the lower velocity ranges. The effects of concentration polarization are expected to be more pronounced as the charge concentration increases with water removal.

Experiment 6 is a series of experiments in which turbulence was promoted by decreasing the thickness of the charge flow channel. This was achieved by stacking different number of pieces of plastic cutouts shown in Figure 8, one on top of another. Maintaining charge flow constant, decrease in channel thickness led to increase in both charge velocity and pressure drop along the flow path. Plotting flux against channel thickness, a curve similar to Figure 7 was obtained (see Figure 9).

The data show that turbulence is achievable through a decrease in the channel thickness leading to an increase in membrane flux. However, a decrease in channel thickness also led to an increase in pressure drop which, in turn, means an increase in the energy requirement of the unit. A trade-off will be needed in the eventual optimization of the unit.

In addition, Experiment 7 was conducted in which permeation was allowed to proceed while maintaining the charge velocity relatively low- at 1.5 cm/sec. Figure 10 shows the flux decline over a five-day period. At the end of the period the cell was carefully disassembled to

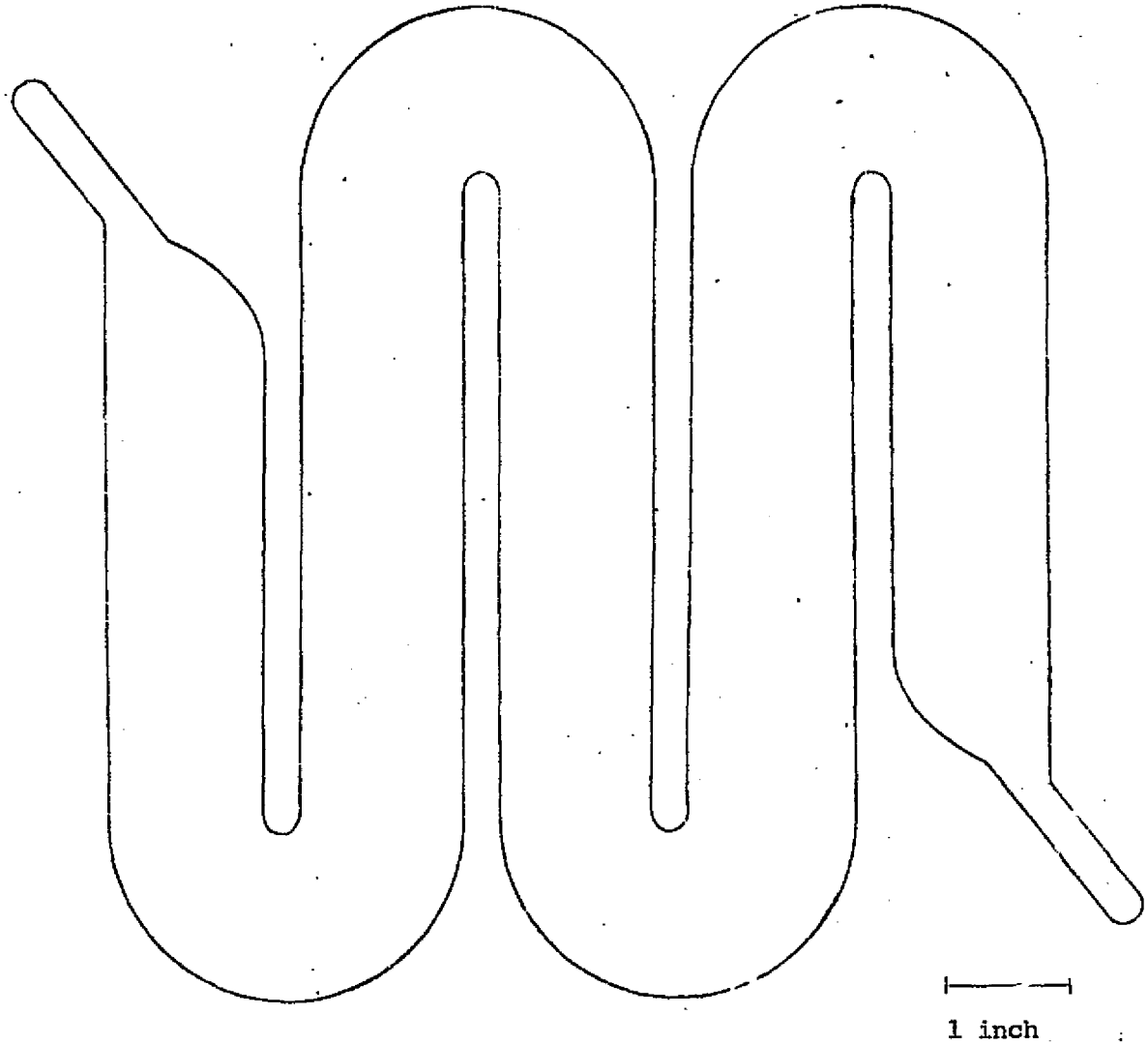


Figure 8: DIAGRAM OF THE CHARGE FLOW PATH

FIGURE 9. EFFECTS OF CHANNEL THICKNESS ON FLUX

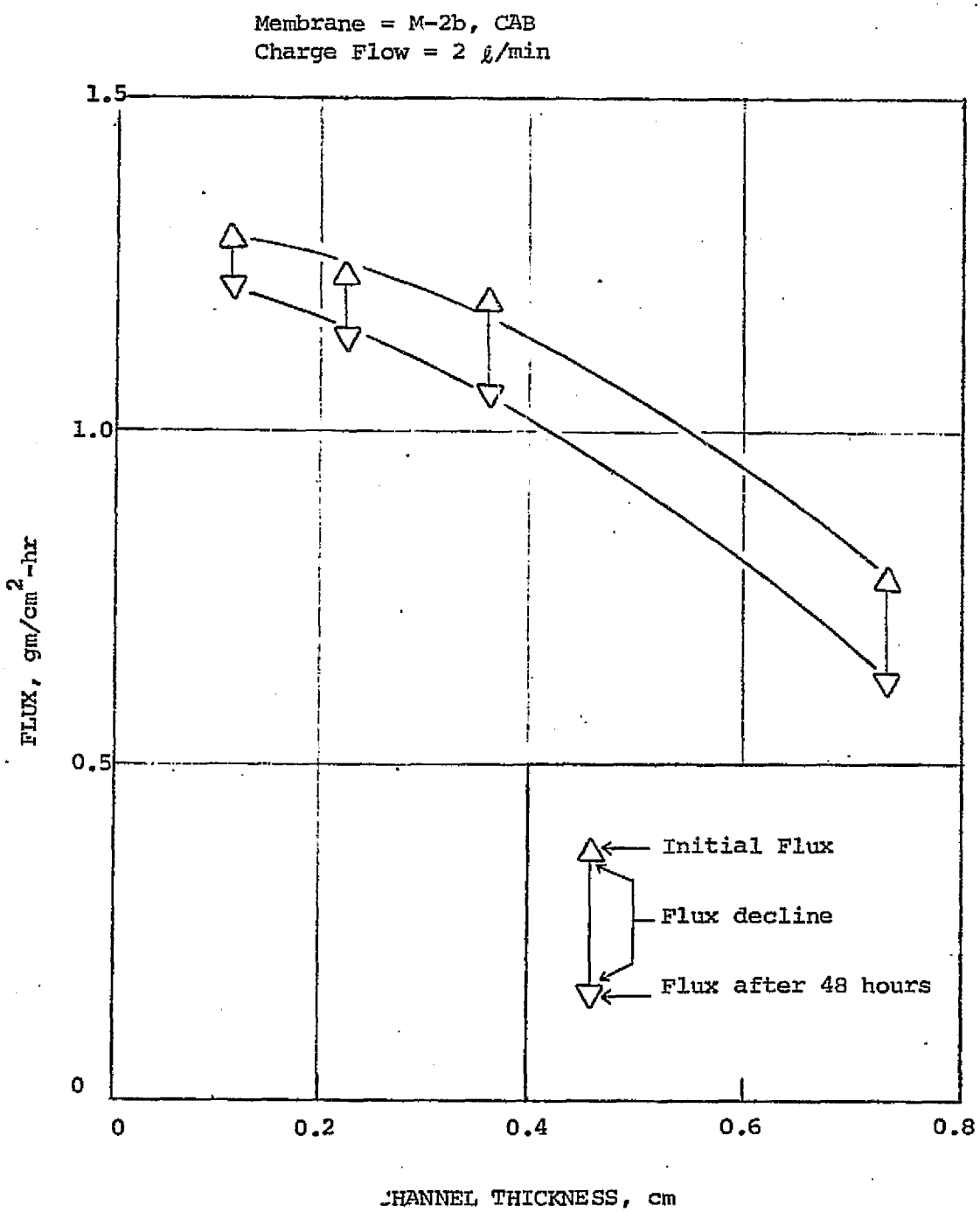
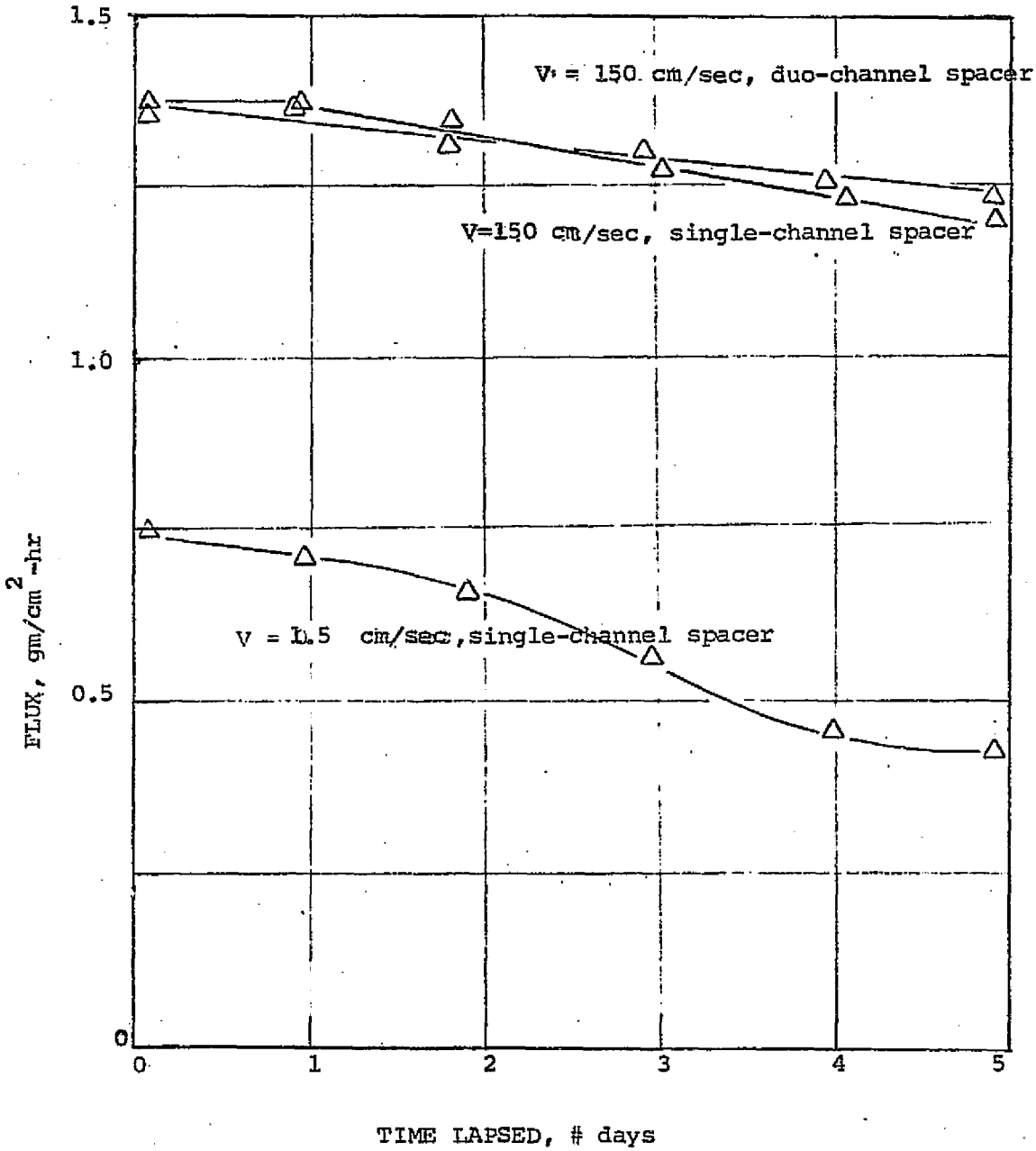


Figure 10. MEMBRANE FOULING AT DIFFERENT CHARGE VELOCITIES



observe the deposition of precipitate on the membrane surface. Figure 11 shows the pattern of the deposition.

The membrane was carefully returned to the cell and the unit reassembled. Flux was restored by repeated flushing with hot water. The experiment was then repeated, maintaining the charge at the maximum velocity permitted by the pump. The data were plotted in Figure 10 for comparison with the data from the low velocity run. The corresponding deposition pattern is shown in Figure 12.

To further enhance mixing Experiment 8 was conducted in which a partition was installed to break up the flow channel into two smaller channels as shown in Figure 13. The flux decline with increasing water removal is shown in Figure 14. The corresponding data for a single-channel spacer is shown in the same figure for comparison. On disassembling the cell, no brownish deposit was noted on the membrane. A three-channel run was also performed and the data also plotted in Figure 14. No brownish deposit was observed also.

These data showed that dividing the channel into two parallel subchannels enhanced mixing, alleviating flux decline significantly. However, division into three subchannels yielded no further improvement.

Fouling Species

Membrane fouling was further investigated by attempting to pin-point the particular foulants.

The first of these experiements (#9) used a 1% salt solution as the charge and M-2b, the cellulose acetate membrane as the separator. Flux was reasonably stable for a period of 100 hours of operation in the

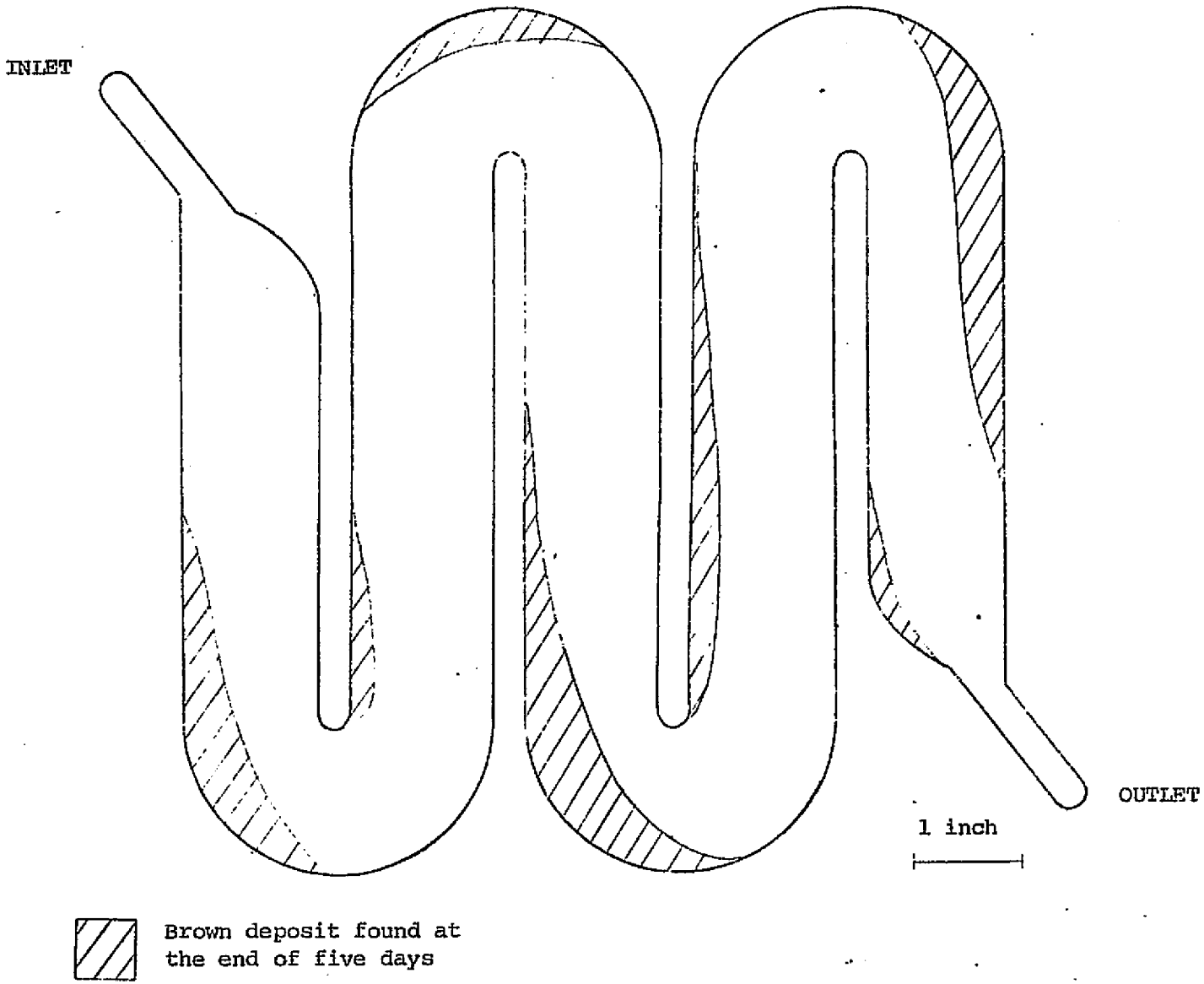


Figure 11 DIAGRAM SHOWING DEPOSITION OF PRECIPITATE AT LOW CHARGE VELOCITY

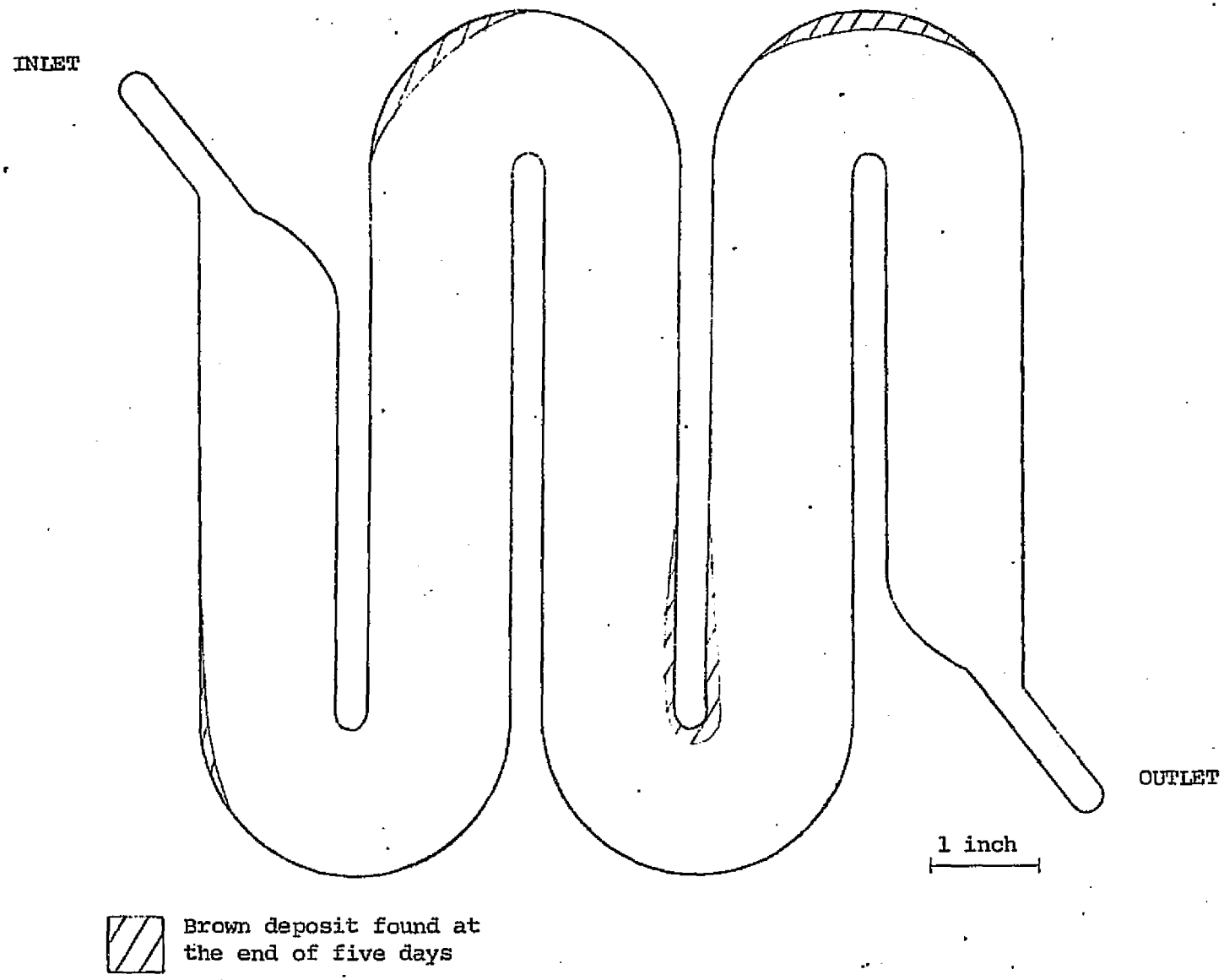


Figure 12. DIAGRAM SHOWING DEPOSITION OF PRECIPITATE AT HIGH CHARGE VELOCITY

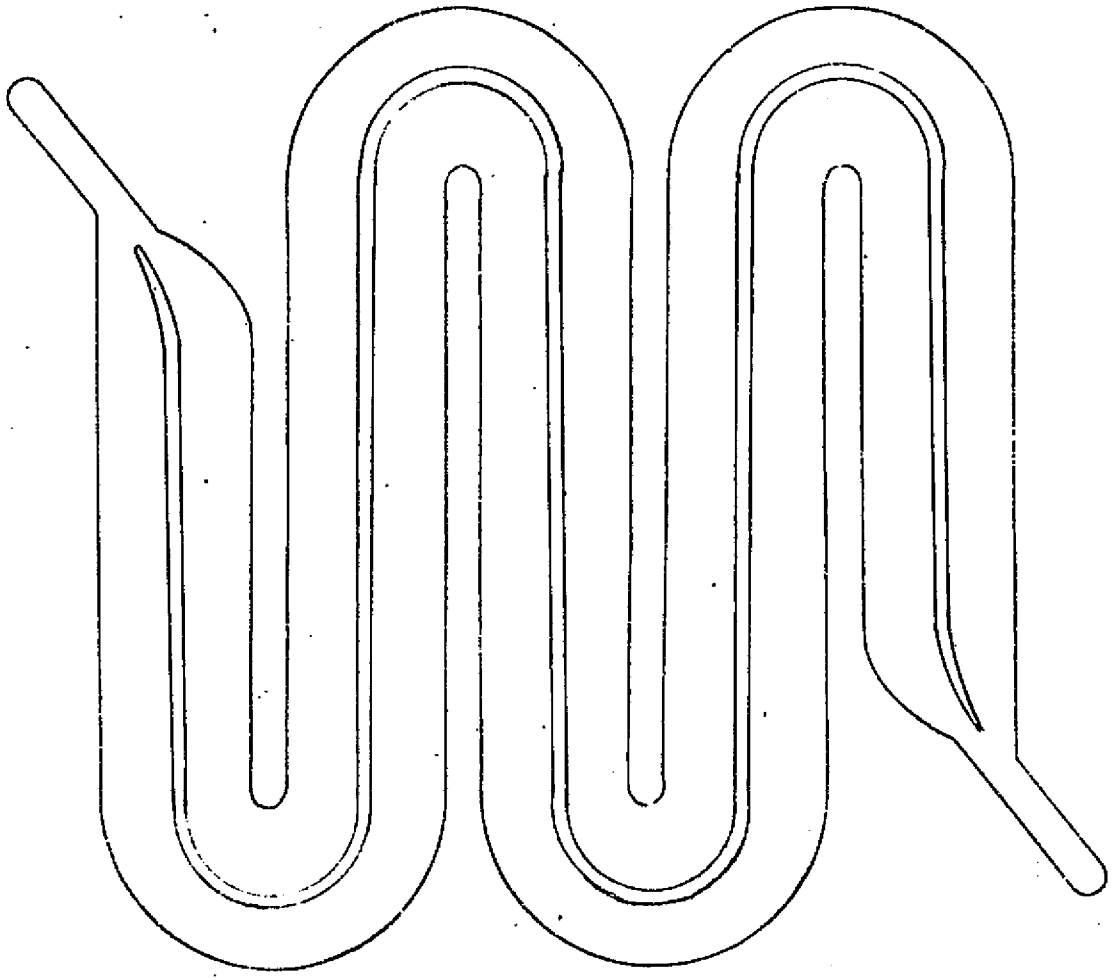
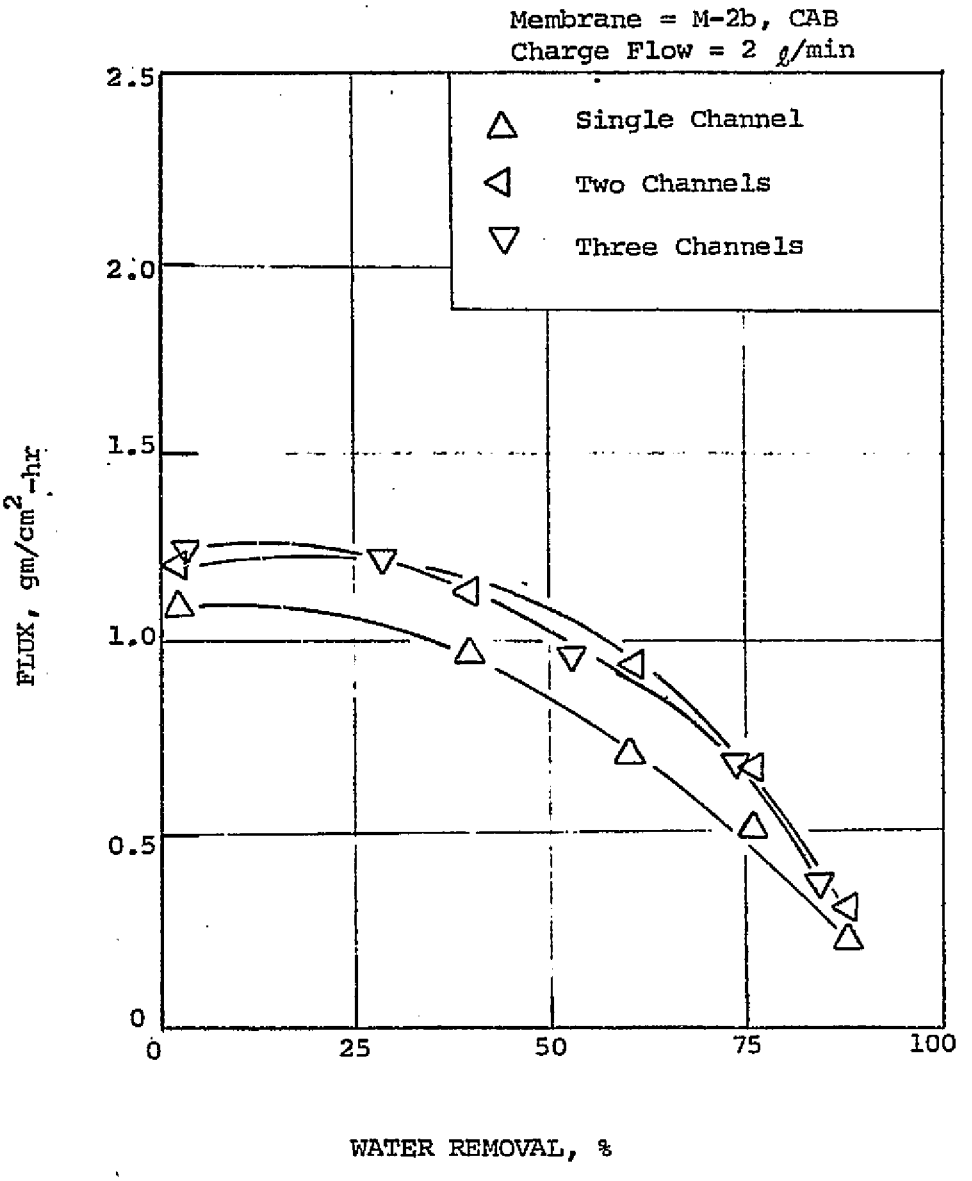


Figure 13 A DUO-CHANNEL SPACER

Figure 14 THE EFFECTS OF DIVIDING THE FLOW PATH INTO SUB-CHANNELS



differential mode, in contrast with the flux decline for the corresponding run using human urine as the charge. Figure 15 compared these data. The flux stability in the case of the salt solution indicated that membrane compaction (resulting from the compaction of the porous support structure in RO membranes due to the exertion of the systemic driving force) was not the cause of flux decline in the VDR.

Experiment #10 compared flux decline in the integral mode (i.e. the recovered water is not returned to the charge reservoir) for three different charge solutions - NaCl, NaCl+ Urea, and actual human urine. The data are shown in Figure 16. It is evident that (i) flux declines in the cases of NaCl and NaCl-Urea solutions and (ii) the flux decline in the case of actual human urine is significantly more severe than the other two cases, especially at high solute concentrations.

The first observation indicates that the flux decline is partially due to the increase in solute concentration in the charge leading to a corresponding increase in osmotic pressure and a higher energy requirement for separation. This should be distinguished from membrane fouling because the flux will increase with a corresponding dilution. The second observation indicates the extent of actual membrane fouling. It implies that flux decline due to membrane fouling is a result of solute species than NaCl and Urea.

To determine the molecular size of the foulants and to study the feasibility of fouling prevention through prefiltration, Experiments 11-16 were carried out using M-2, the CAB membrane. As shown in Figure 1, the charge recirculation pump delivered the urine charge through a filter

FIGURE 15. DATA ILLUSTRATING MEMBRANE COMPACTION IN VDR

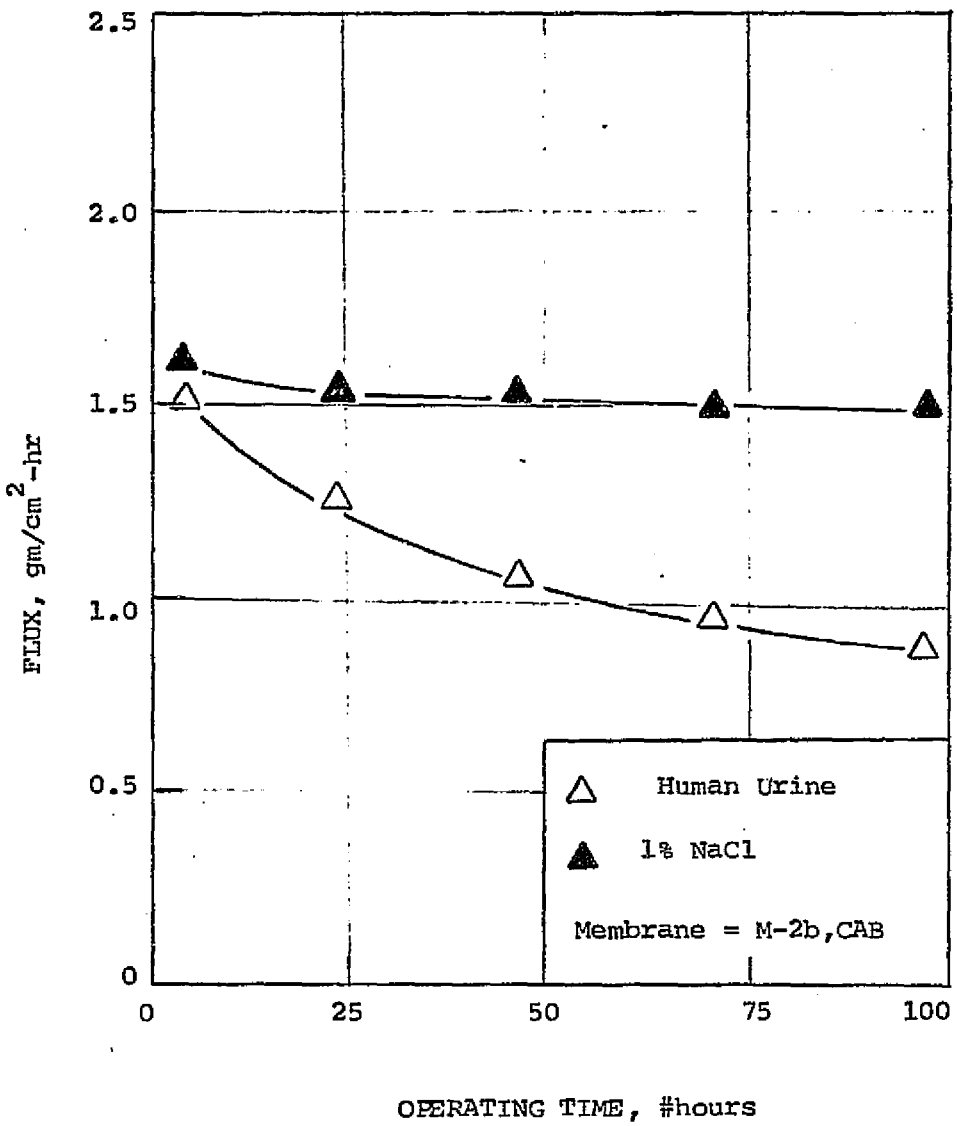
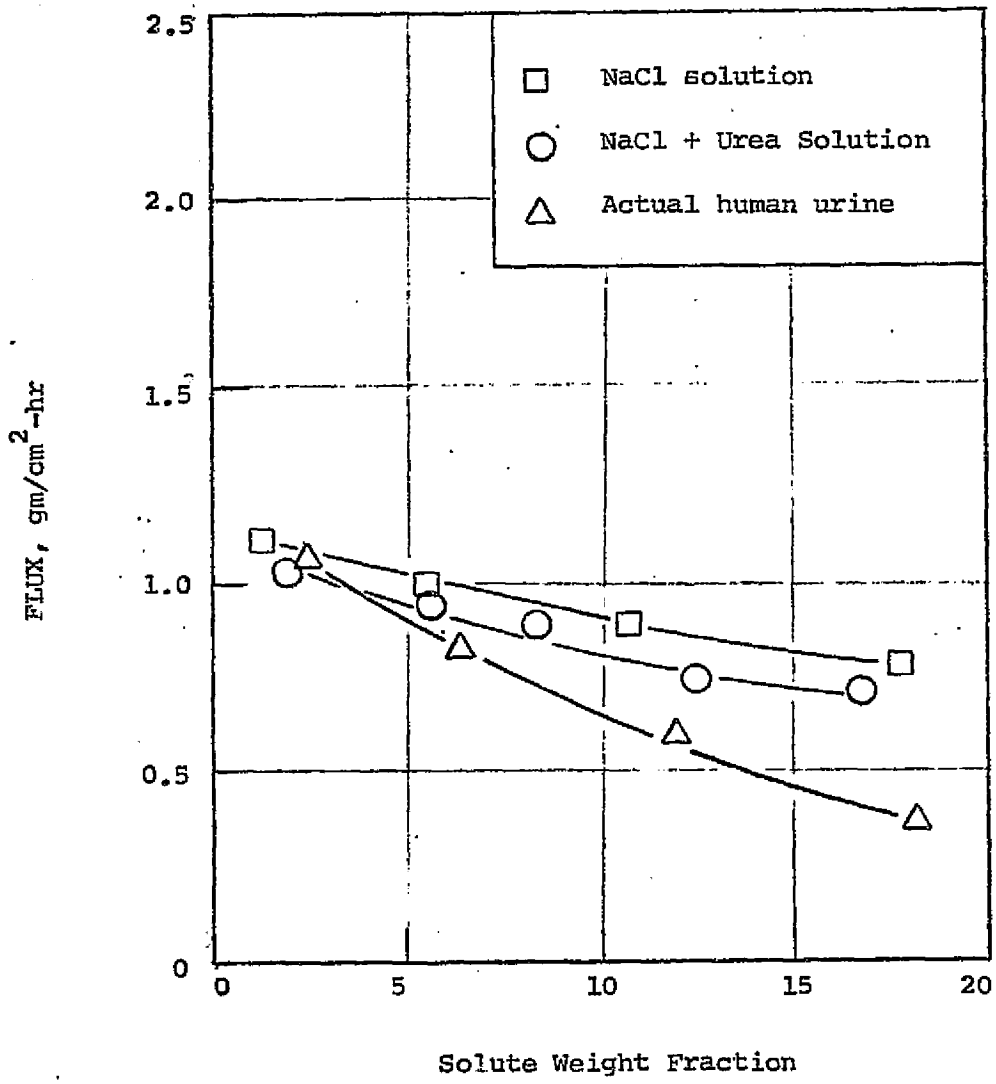


Figure 16 Flux as a Function of Solute Concentration

Membrane = M-2b, CAB



housing to remove urine species of specific molecular sizes. Three different filter cartridges were used - having molecular size cut-offs of 2 μm, 8 μm and 20 μm. Figure 17 illustrates the data obtained when operating the system in a differential mode.

Three additional experiments were conducted using prefiltered urine charge and operating in an integral mode. The data are summarized in Figure 18.

As seen from both Figures, prefiltration with 2 μm, 8 μm and 20 μm filters affects the membrane performance somewhat at low charge concentrations and significantly at higher charge concentrations (i.e. above the 50% water-removal level when the total solids level exceed 7.0%).

Experiments # 17 and #18 study the effects of the charge pH on membrane flux and distillate pH, respectively. As previously noted, pH level of the charge increased with continuous exposure to heat due to the partial decomposition of the urea. Starting at the level of pH6-7, it rose gradually and stabilized at around pH9 due to the buffering action of various urine components. Since flux declined while the pH was increasing it was thought that they might be related to each other.

To investigate the effects of charge pH on membrane performance various charge pH's were obtained by the addition of 2N HCl and 2N NaOH solutions in a random manner)to minimize the effect of operating time).

Figure 19 shows the effect of charge pH on membrane flux. The flux of the M-2 (CAB) membrane and the M-11b (PTFE) membrane is pH independent, while that of the M-3 (PPOS) decreases with increasing pH. The flux at pH 3 is 50% higher than that at pH 9.

Figure 17. Effects of Prefiltartion on Flux
(constant Solute Concentration)

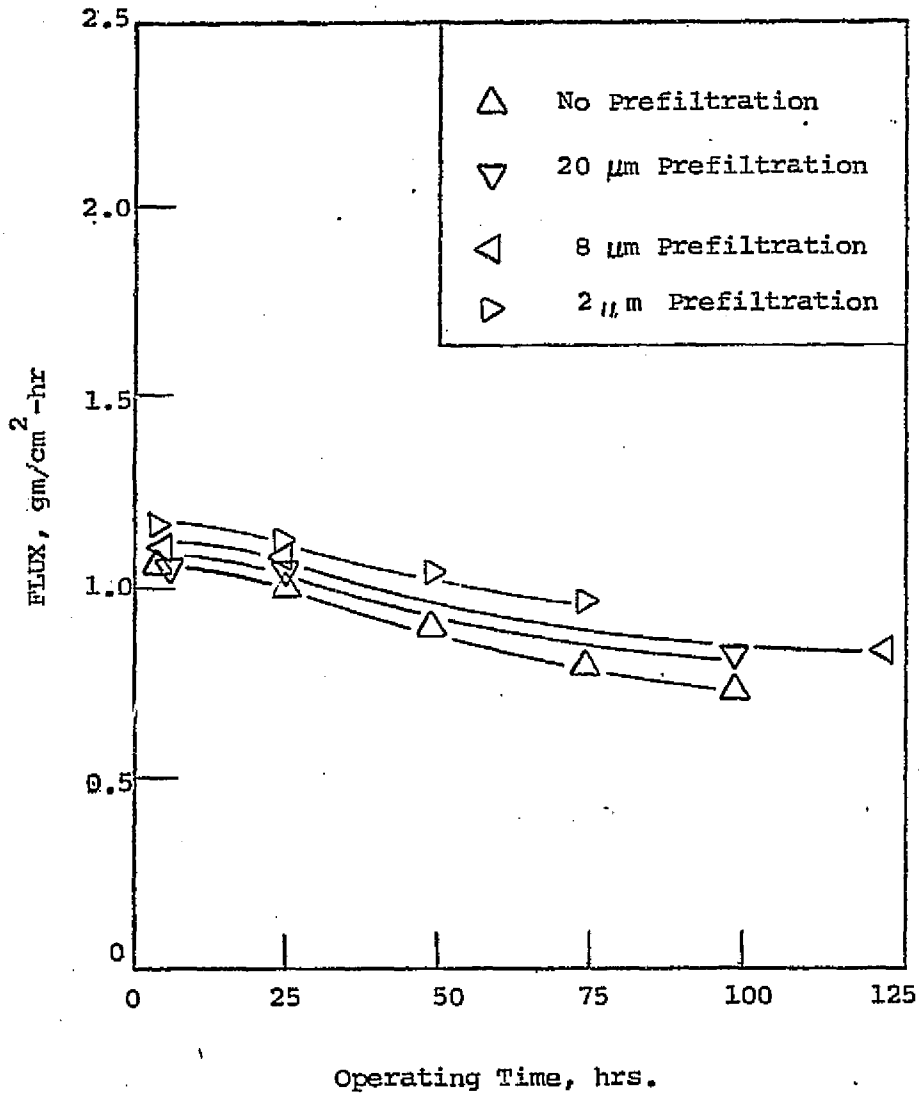
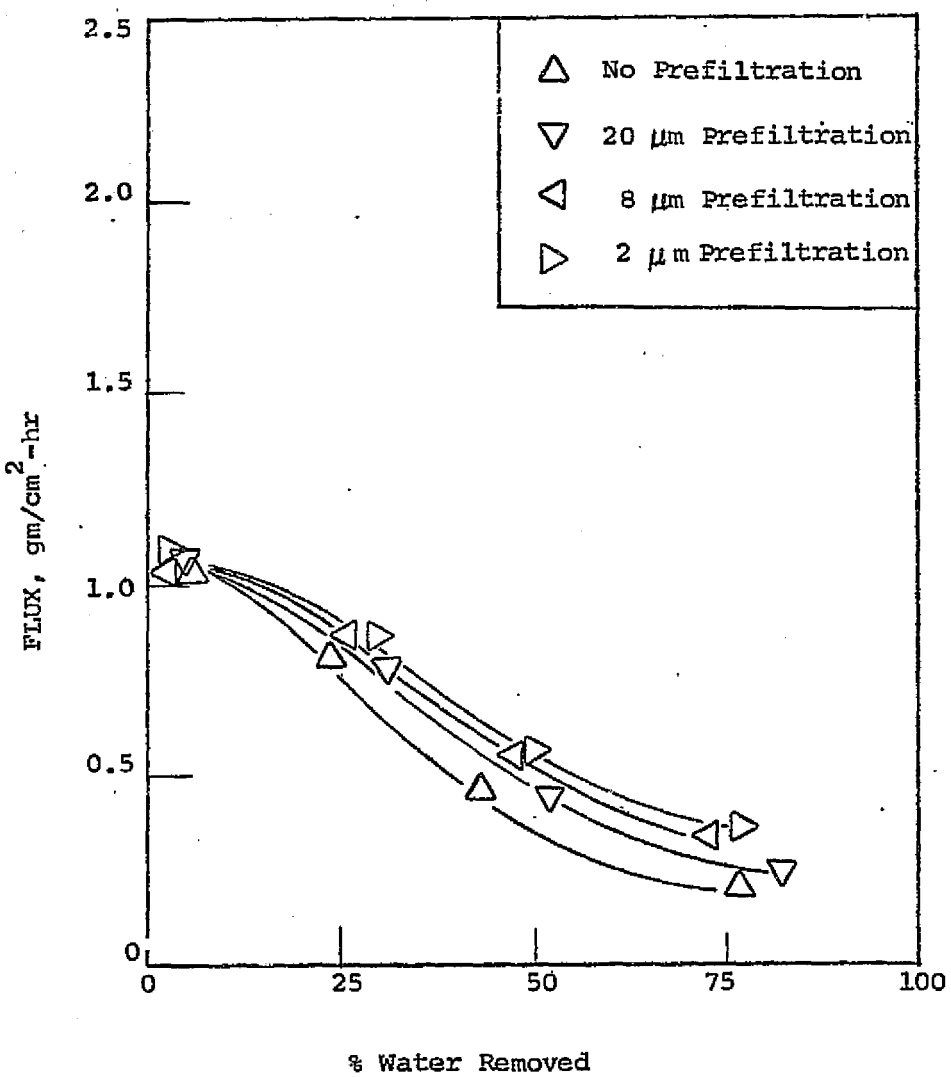


Figure 18. Effects of Prefiltration on Flux
(increasing solute concentration)



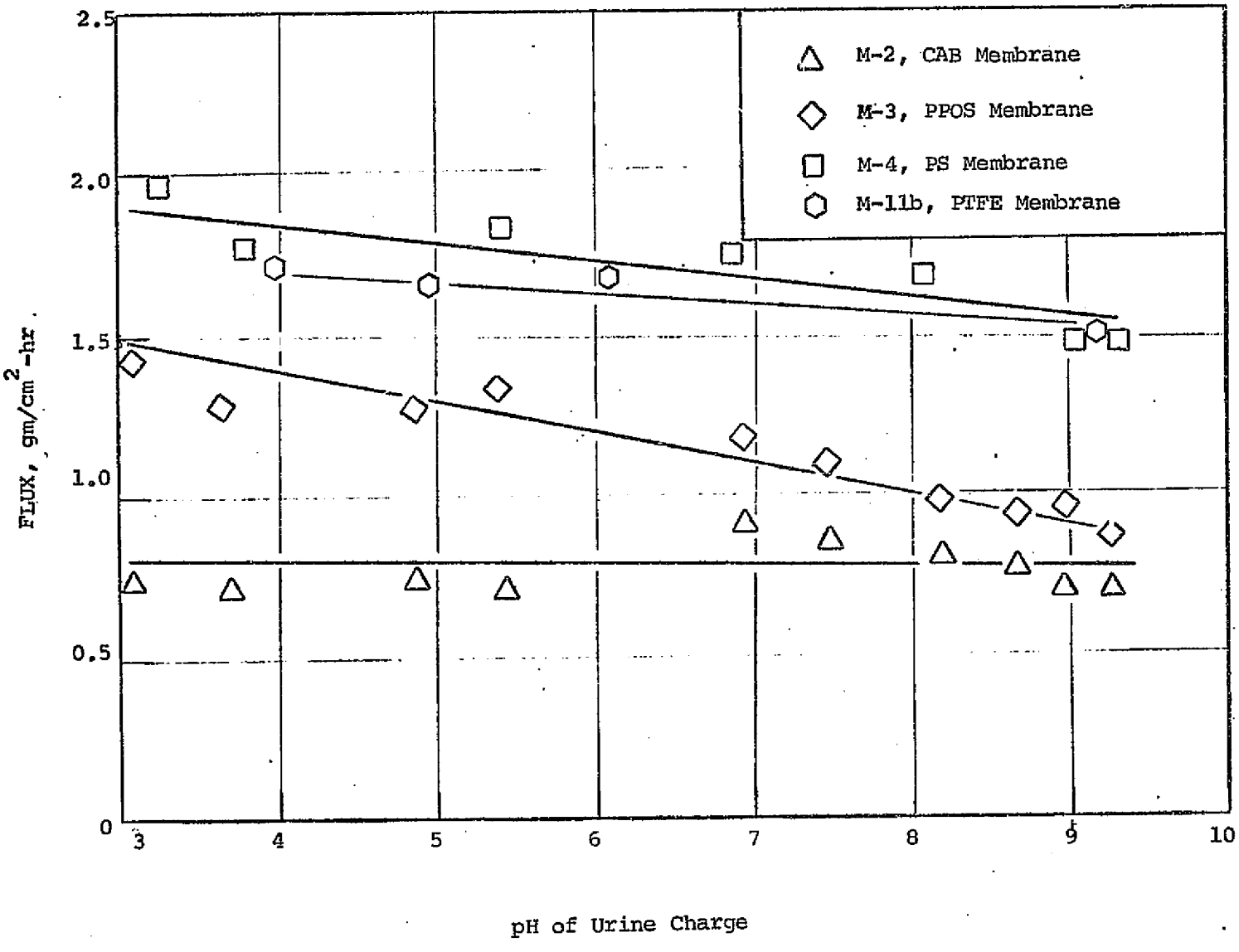


Figure 19 Effect of Charge pH on Membrane Flux

The explanation for the behavior of the M-3 membrane lies in the pendant sulfonate groups. In acidic media these groups are in the hydrogen form, i.e. $-SO_3^-H^+$ and the membrane exhibits higher water sorption, more swelling and higher water permeability. In alkaline media these groups are in the sodium form, i.e. $-SO_3^-Na^+$ and the membrane exhibits significantly lower water sorption, less swelling and lower water permeability.

The flux of the PS membrane appears to decrease somewhat with increasing charge pH. However, the variations are so small that they may not be significant.

Figure 20 shows that increasing charge pH leads to an increase in the pH of the distillate for all membranes. This indicates that ammonia retention of all membranes, including the hydrophobic PTFE membrane is low. Evidently, the ammonia generated by the increase in charge pH, passed through the membrane and was registered in the distillate as an increase in its pH.

Recovery of Flux Decline

The method of flux recovery investigated was the most obvious one - repeated flushing with clean, hot water. This Experiment (#16) is illustrated in Figure 21. A 5-liter batch of human urine was concentrated fivefold (80% of the water being extracted). The concentrate was pumped out and 2 liters of preheated water were added and recirculated along the charge flow path. After 1 hour of flushing, flux measurement was made. The water was then replaced with 2 additional liters of preheated water, and the flux

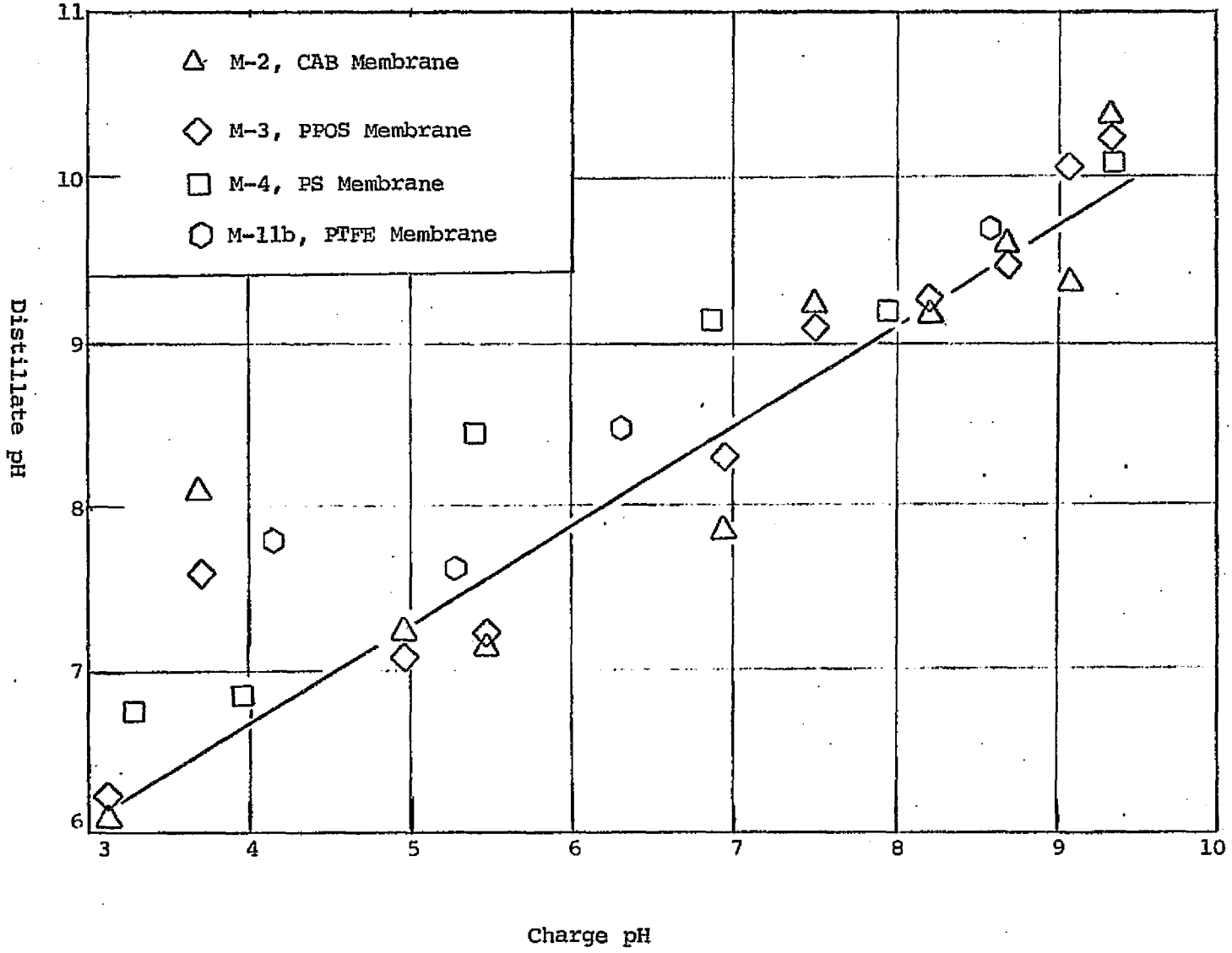


Figure 20 Effect of Charge pH on Distillate pH

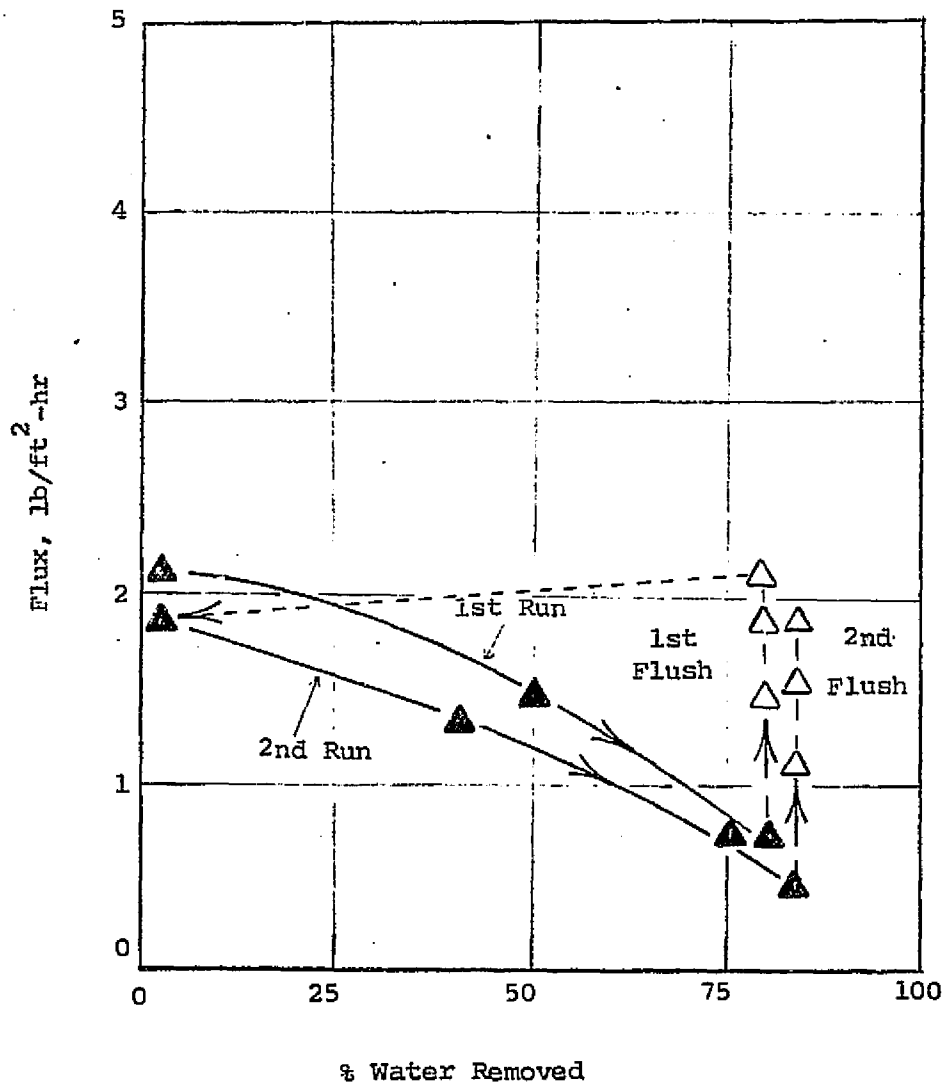
was measured after 2 hours of flushing. The water was then replaced with 5 liters of fresh urine, and the cycle repeated. (See Figure 21)

This procedure required 4 liters of water, approximately the total amount of water extracted and would not be practical in actual space-flight operation. However, it illustrated a point - that the original water flux could be restored through repeated flushing - i.e. that the membrane fouling is a reversible process.

In contrast, previous flux recovery experiments (involving duPont's NAFION and Ionics' 61AZL membranes) performed during the previous contract, showed that repeatedly flushings of the membranes with hot water did not recover the flux completely. (The extent of flux recovery was found to vary with the amount of urine treatment the membrane was used for.)

At the same time, on disassembling the unit, the NAFION and the 61AZL were found to have been permanently stained with a brown color. The CAB membrane, however, looked unchanged. It is believed that the fouling, in the case of the relatively more microporous NAFION and 61AZL, occurred, in part, through the actual partial penetration of the urine components into the interstices of the membranes causing actual plugging. On the other hand, the CAB being a much tighter membrane did not allow the penetration of urine components sufficiently to plug the membrane pores permanently. Probably only a surface deposition occurred in this case. The staining of the NAFION and the NEPTON-AZL can also be attributable to chemical bonding between the foulants if they are positively charged, and the negatively charged functional groups present in these membranes (these negatively charged groups are not present in the CAB).

Figure 21 Data Illustrating Batch Treatment with Periodic Flushing



The fouling of the G.E.'s polyphenylene oxide sulfonate (PPOS) membrane and the N.S.R.'s polysulfone membrane was investigated in a similar batchwise experiment (#19). Water was continuously removed from a batch of human urine. The flux was periodically measured and plotted. After 82% of the water had been removed, the flux dropped considerably.

Flux recovery was performed by repeated water-flush as described in the previous paragraphs. Figure 21a shows the response of the membranes to water flush. After four flushes, the original flux of the PPOS membrane was restored. (The fifth point represents the flux measured after allowing the membrane to relax overnight.) However, the original flux of the PS membrane was not restored. On disassembling the equipment, both membranes were observed to be unstained.

The unit was reassembled and the fouling studies continued with three more experiments. At the end of each experiment flux decline was observed and water flushes were performed to recover flux decline. The water flux at the end of each experiment, after four water flushes were measured and plotted in Figure 22 The corresponding data for the CAB membrane were also shown in the same figure.

While the flux decline in the case of the CAB membrane was completely recoverable, the flux decline in the case of the PS membrane was only partially recovered. The reason for this difference is not known. Since the PS membrane appeared unchanged at the end of the experiments, actual particulate plugging of the pores probably did not occur. However, it should be noted that, despite the appreciable decrease in flux, the PS membrane was still permeating water at a much higher rate than either the CAB or the PPOS membranes.

Figure 21a Flux Recovery Through Water Flush in Batch Treatment of Human Urine

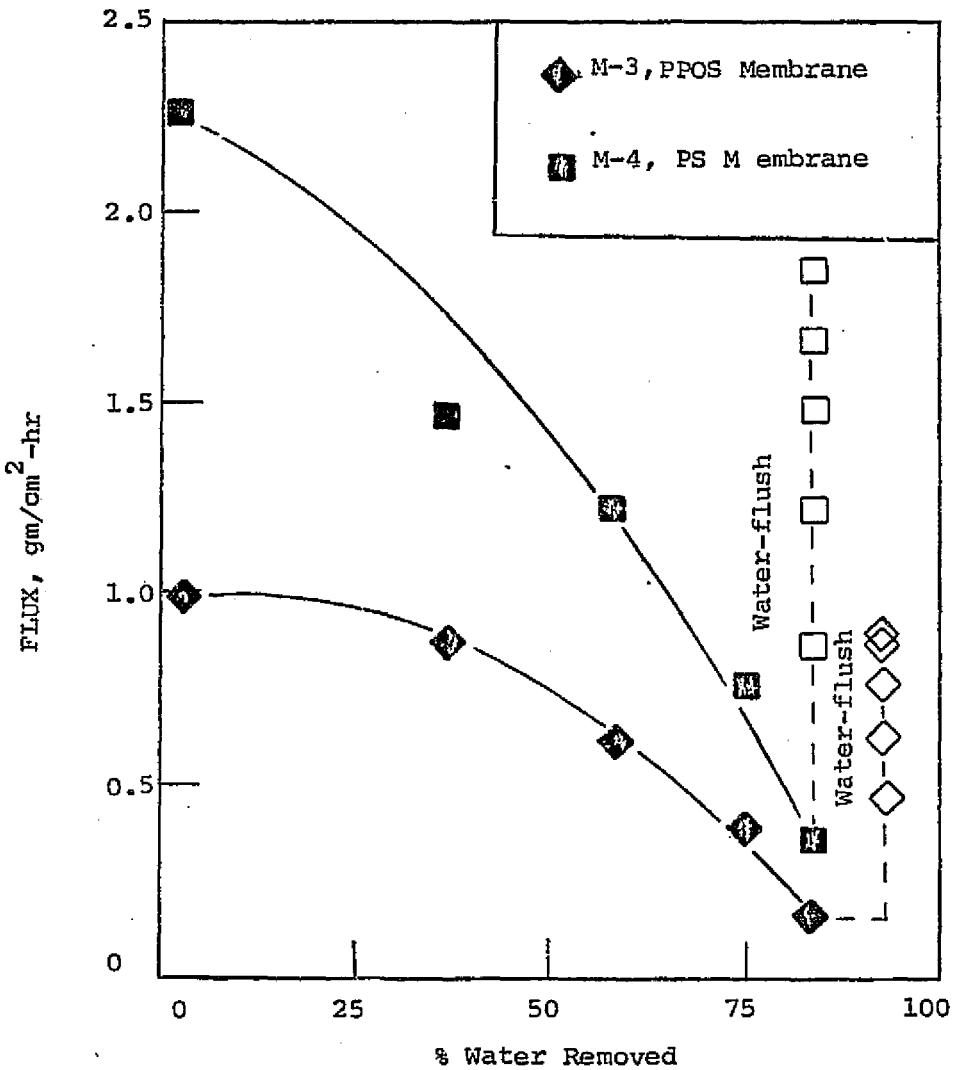
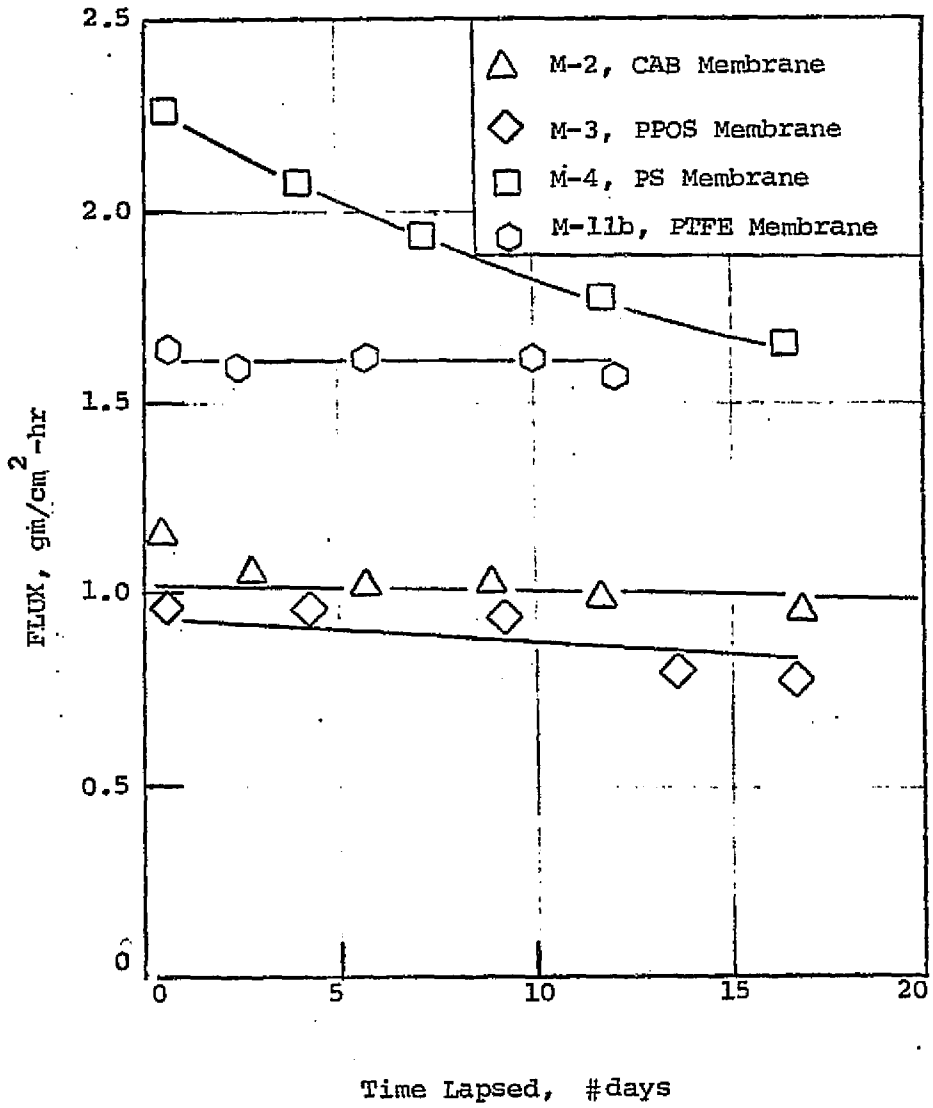


Figure 22 Data Showing the Recoverability of Flux Decline



Complete recoverability of the PPOS membrane flux is unclear. As seen from Figure 22 original flux was restored after the initial two batches; but only about 85% of the original flux was recovered after the next two batches.

Summary and Discussion on Membrane Fouling Studies

(i) Although flux decline appears to be more severe at higher operating temperatures, the resulting flux is still higher at those higher operating temperatures. At the same time, flux decline as a function of total amount of recovered water is essentially the same at different operating temperatures. As such, a maximum operating temperature - 85° C (185° F) - is recommended for the VDR unit, subject to other restraints, such as, problems related to urine decomposition in an enclosed reservoir, problems related to the higher energy requirements associated with a higher operating temperature. (Both of these are not within the scope of this program.)

(ii) Acidification reduced the level of suspended solids but did not affect flux except in the case of the M-3, PPOS membrane. As such, it would appear to have no advantage, and is not recommended except for the reduction of ammonia passage through the membrane. (A trade-off between the complication associated with acidification and the ill-effects associated with ammonia passage has not been studied).

(iii) Flux decline is at least partially recoverable by repeated flushing with hot water, an impractical proposition during space mission. However, this implies that no chemical bonding or physical entrapment of the solutes within the interstices of the membrane has occurred. The foulants are probably precipitation of certain solute species which become redissolved in the flush-water.

(iv) Suspended solids appear to be a major contributor to membrane fouling when the charge velocity is significantly below 18 cm/sec (0.6 ft/sec) as indicated by the deposition of a brown precipitate on parts of the membrane surface where mixing is inadequate. At higher charge velocities prefiltration to remove suspended solids will retard membrane fouling, especially when the charge solids concentrations exceed 7%.

A trade-off between the problems associated with the addition of a prefiltration subsystem and the alleviation of flux decline will be needed. The selection of a specific molecular cut-off point is also not clear. Preliminary data in Figure 18 indicates that an 8-20 μm prefilter is warranted.

(v) Concentration polarization is a major factor influencing the flux and its decline (see Figure 7). Increasing the pumping rate of the urine charge, dividing the serpentine flow-path into two equal channels and reducing the channel thickness all lead to improved mixing of the urine charge and an alleviation of the flux decline.

However, higher pumping rate will entail a higher energy requirement; so will a reduction in channel thickness. Division of the flow path into sub-channels will lead to a smaller effective membrane area. A trade-off study is required for optimization. Preliminary studies indicate that using a duo-channel spacer with effective thickness of 0.3 cm (1/8 inch) and a pumping rate of 2 liters/min (0.5 gpm) will yield satisfactory result, (i.e. no solid deposition after 5 days of operation. Flux of 0.6-0.9 gm/cm^2-hr at a charge concentration of 14% solids or 70% water removal.)

(vi) Flux decline is disproportionately severe at high solid concentrations (total solids > 14%). This is probably because at these concentrations, the solubility limits of some of the urine components are exceeded causing precipitation. E.g., at five-fold concentration there is a ten-fold increase in the amount of suspended solids. Table V present some relevant properties of concentrated urine.

This raises the question as to the practical limits of the system as related to the extent of water reclamation. Although the data by the present and past investigators have shown that as high as 95% of the water can be recovered, the flux at such a high concentration is extremely low. In view of the fact that pure water is being generated on-board the space-craft, depending on the duration of the mission and the feasibility of on-board storage of urine concentrates, it may be that an 85-90% water recovery is adequate. The present data indicate that a conservative objective of 85-90% water recovery should be set as the practical limits of the VDR.

(vii) The rate of fouling varies with the particular membrane used. Figure 23 shows the variation of flux as a function of the water removed. At 85% water removal the flux of M-4 and M-11b (the PS and the PTFE membranes) is twice that of M-2b and M-3. (The CAB and the PPOS membranes) However, while the flux decline in the cases of the M-2b and M-11b can be completely recovered by water - flushing, that of the M-4 is clearly not completely

TABLE III: PROPERTIES OF HUMAN URINE CONCENTRATES

	Fraction of Extracted Water				
	0	.8	.9	.95	.98
Boiling Point Elevation, ^{o}F	0	3	6	23	> 40
Osmotic Pressure, psi	500	800	2000	5000	9000
H_2O Partial Pressure	1	1.05	1.10	1.35	1.70
Suspended Solids g/ℓ	0	.5	2	5	12
Viscosity, cps	1	1.3	2	23	> 30

recoverable. (See Figure 22). The M-3 membrane exhibits the lowest flux of all the four membranes selected and also exhibits less than full flux recovery (see Figures 23 and 22, respectively). As such, the data indicated that M-2b and M-11b should be the membranes selected for VDR applications. M-11b has the advantages of dry storage, high flux and lower flux decline. M-2b has the advantage of being more commercially widely used in RO systems. It has been specifically developed as a membrane for water treatment applications (whereas the PTFE "membrane" is essentially a microporous diaphragm developed for non-sticking sheeting applications). M-2b is also commercially available in the tubular and spiral-wound configurations. Most of all, the performance of M-2b is not disturbed by the presence of wetting agents such as pretreatment chemicals, and the soaps used by the crew for hygiene purposes; while the M-11b is not useable at all because of a "wetting" phenomenon. (This will be discussed in a later portion of the report under "Extension to Include Washwater Treatment").

III. MEMBRANE LIFE DATA

A long term continuous test involving a selected membrane would tie up the VDR bench unit for a prolonged period of time and the data obtained may not be of sufficient significance (since, in actual usage, the unit would be subjected to batch-wise treatment instead of a continuous run). At the same time, the stability of a membrane in the presence of hot human urine for a prolonged period of time is an important piece of data.

To obtain some sort of membrane life data without disturbing the scheduled experiments; such as, prefiltration, fouling investigation, flux recovery, etc., a series of ten experiments was designed. A specific piece of M-2b, the cellulose acetate blend membrane, was used throughout the ten experiments. Flux decline occurred during each experiment. The original flux was restored by repeated flushing with hot water. Table VI and Figure 23 summarize the data obtained during this series of experiments.

The result of each experiment had been discussed in various parts of this report. The most relevant data here relates to the stability of the M-2b membrane. The complete recoverability of flux indicates that the M-2b has good stability when exposed to human urine of pH range 6-8.5 and concentration range of 3-18% solids, at 85° C (185° F) under an applied pressure of 1 atmosphere, and the stress and strain inherent in the pumping of the charge and the periodic interruption of the run for experimental purposes, over a period of 40 days.

TABLE IV LIST OF EXPERIMENTS

Code	Description of Experiments: Charge	Prefiltration	Mode	Duration (# days)	Flux Decline	Amt. of Flushing	H_2O Flux
1	H U*	None	H_2O removed	3	severe	5/2**	2.1
2	H U	20 μm	"	2	"	5/2	2.0
3	H U	None	H_2O returned	4	appreciable	4/2	2.0
4	H U	8 μm	H_2O removed	3	severe	6/3	2.0
5	H U	20 μm	H_2O returned	5	appreciable	4/2	1.9
6	H U	8 μm	H_2O removed Batch treatment	2	severe	5/2	1.8
7	H U	"	H_2O returned	4	appreciable	5/2	2.1
8	H U	none	H_2O removed Batch treatment	4	severe	6/3	1.7
9	NaCl	none	H_2O returned	4	some	0	not measured
10	NaCl + $(NH_2)_2CO$	none	H_2O removed	3	some	2/1	2.2

* Actual human urine used

** The first number denotes the # of hours; the second, the # of times fresh H_2O was used.

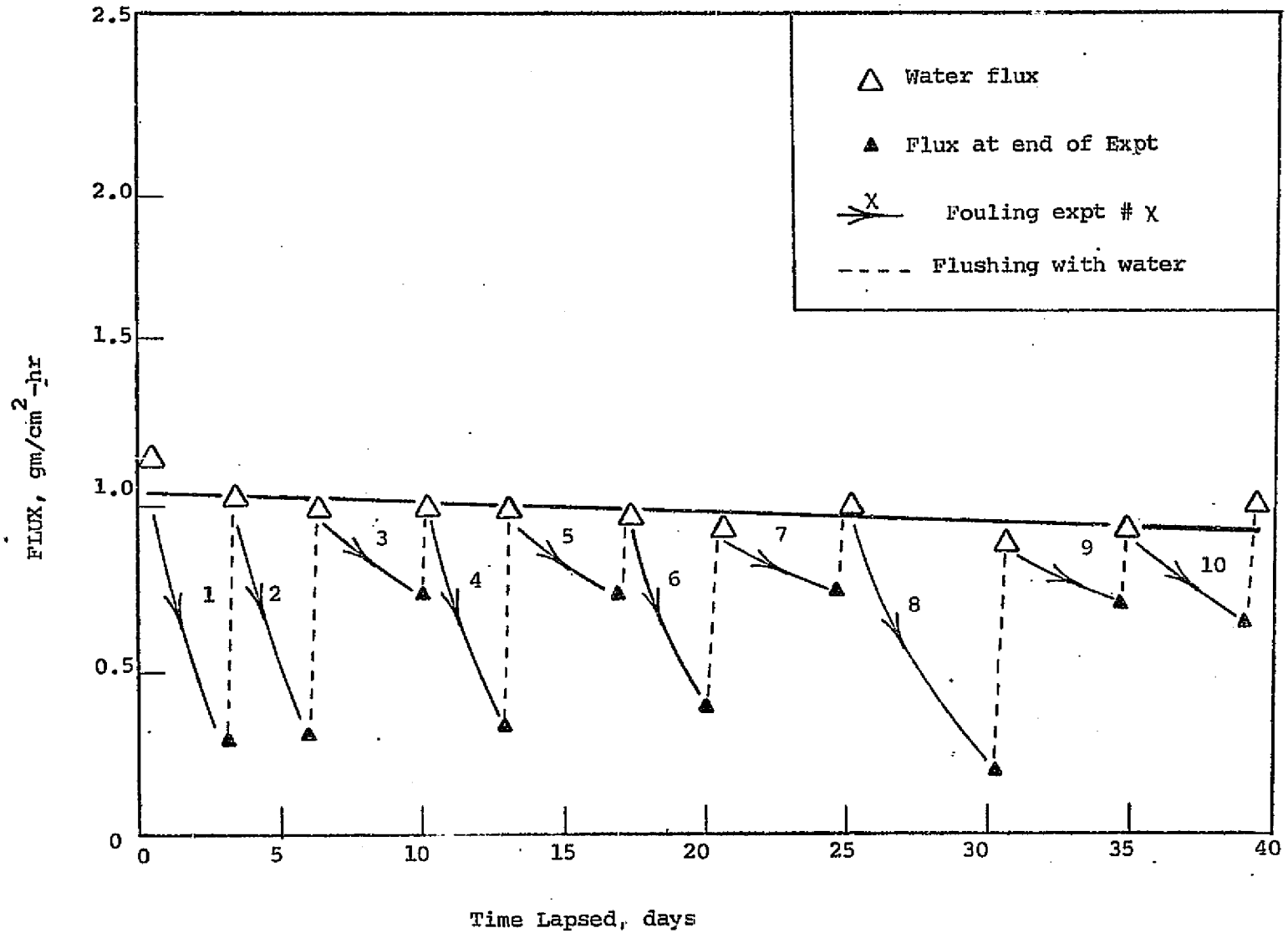


Figure 23. Diagram Showing the Flux Recovery with Water Flush

On disassembling the unit at the end of the 40-day run, the membrane was found to be essentially unchanged. In addition, pieces of M-2b, M-3, M-4 and M-11b were soaked in concentrated urine at 85° C for a 60-day period. After rinsing with water these membranes were tested in the VDR unit. The flux under the usual operating conditions is essentially the same as that of the corresponding untreated membranes.

IV. EXTENSION TO INCLUDE WASHWATER TREATMENT

Introduction. The application of the VDR for urine treatment has been discussed in the previous sections. A second major liquid waste in a space cabin is the washwater stream. This is a more diluted charge containing soaps or detergents produced by the "bathing" of the crew. It was felt that a unit suitable for urine treatment should also be investigated for suitability in treating washwater.

A synthetic washwater charge with the following composition was prepared and tested in the VDR unit (Experiment # 21):

Sodium dodecyl benzene sulfonate	5000 ppm
Lactic Acid	1400 ppm
Urea	800 ppm
Sodium Chloride	2300 ppm
Glucose	100 ppm

(This approximates prefiltered actual washwater and was used in an RO module evaluation contract with NASA-Ames by the present investigator).

Figure 24 shows the variation of flux with water recovery for the four selected membranes. The operating conditions were identical to those used for human urine under this contract. All but the M-11b membrane shows similar performance to the case of human urine. However, after two hours of operation the M-11b became "wetted" and the charge passed through it with no solute retention whatsoever.

The M-2b, M-3 and M-4 are hydrophilic membranes with pores in the range of 0.0001 to 0.001 μm. They function as semi-permeable membranes. They swell with water sorption, allow selective transport of the charge in the liquid phase and a phase-change step near the vacuum side of the membrane after which the permeate is removed in the vapor phase. The presence of

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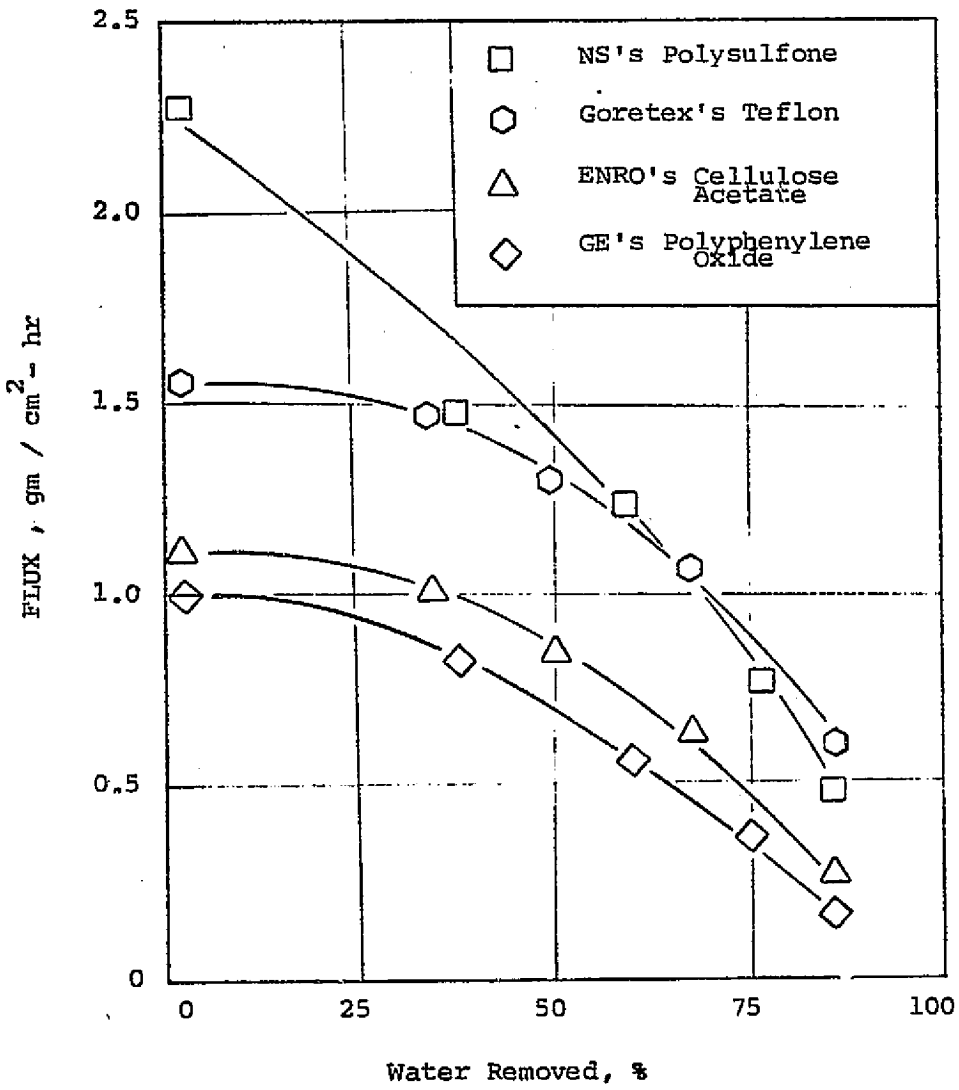
Figure 24 DATA SHOWING FLUX DECLINE WITH INCREASING WATER REMOVAL



wetting agents such as soaps or pretreatment chemicals have no effect on such hydrophilic semi-permeable membranes.

The PTFE "membranes" are microporous diaphragms with pores in the range of 0.02 to 0.2 μm. The charge is contained by surface tension forces due to the pore size and the hydrophobicity of PTFE. Application of hydrodynamic force sufficient to overcome these forces will render the retentive properties of the diaphragm inoperative, causing liquid "leakage". The presence of wetting agents such as soaps and pretreatment chemicals may reduce the surface tension effects sufficiently to cause such a leakage when vacuum is applied.

In the case of M-11a with maximum pore-size of 0.2 μm the pressure differential alone was sufficient to overcome the surface tension effect and drive the urine charge through the larger pores of the diaphragm, thus, exceeding the bubble point of the membrane. In the case of the tighter M-11b, the pressure differential was insufficient to exceed the bubble point. But in the presence of sodium dodecyl benzene sulfonate the surface tension was reduced sufficiently that the pressure differential became sufficient to exceed the bubble point. It is possible that a tighter PTFE diaphragm may be useable in washwater treatment.

Shuttle Application. In the case of space flights of shorter duration such as the shuttle, a closed-loop water system is not required, especially where on-board fresh water is generated regularly. However, overboard dumping of wastes is prevented because of the interference of the contained solids on the space-craft systems and experiments - optics, star sightings, etc. Since the baseline systems already dump water vapor and volatile organics

overboard, e.g. water flash evaporator and feces inactivation, the VDR unit may be allowed to do the same.

A VDR unit can be designed for the specific application of continuously dumping the permeate, consisting largely of water vapor and volatile organics, into space vacuum. This would greatly alleviate on-board storage problem of the human urine and washwater. It would also have the advantage of not requiring a condensation subsystem and a post-treatment sub-system (the permeate contains a strong obnoxious odor). The energy required to maintain a systemic driving force, such as the vacuum, will also not be required, since this unit will essentially be tapping space vacuum for permeate removal. In addition, the evaporation of waste fluids to space vacuum will provide supplemental spacecraft cooling and, thus, conserve fuel cell water previously required for cooling.

To minimize the energy requirements of this system an experiment was conducted to determine the minimum operating temperature feasible for effective continuous operation. Figure 25 shows the temperature-dependency of the system using the M-2b membrane and a synthetic washwater charge (Experiment #22). Figure 26 is a similar plot and includes the data obtained for M-3 and M-4 also (Experiment #23).

From Figure 25 it appears that the flux-temperature relationship is of the Arrhemius type only down to 50° C (120° F), below which the flux decreases rapidly with decreasing temperature. Operating the unit below 38° C(100° F) is not recommended.

Experiment #24 involves a run at 38° C (100° F) using a urine-wash water combined waste as the charge (2 liters urine and 1 liter washwater).

Figure 25 EFFECTS OF OPERATING TEMPERATURE ON MEMPRANE FLUX

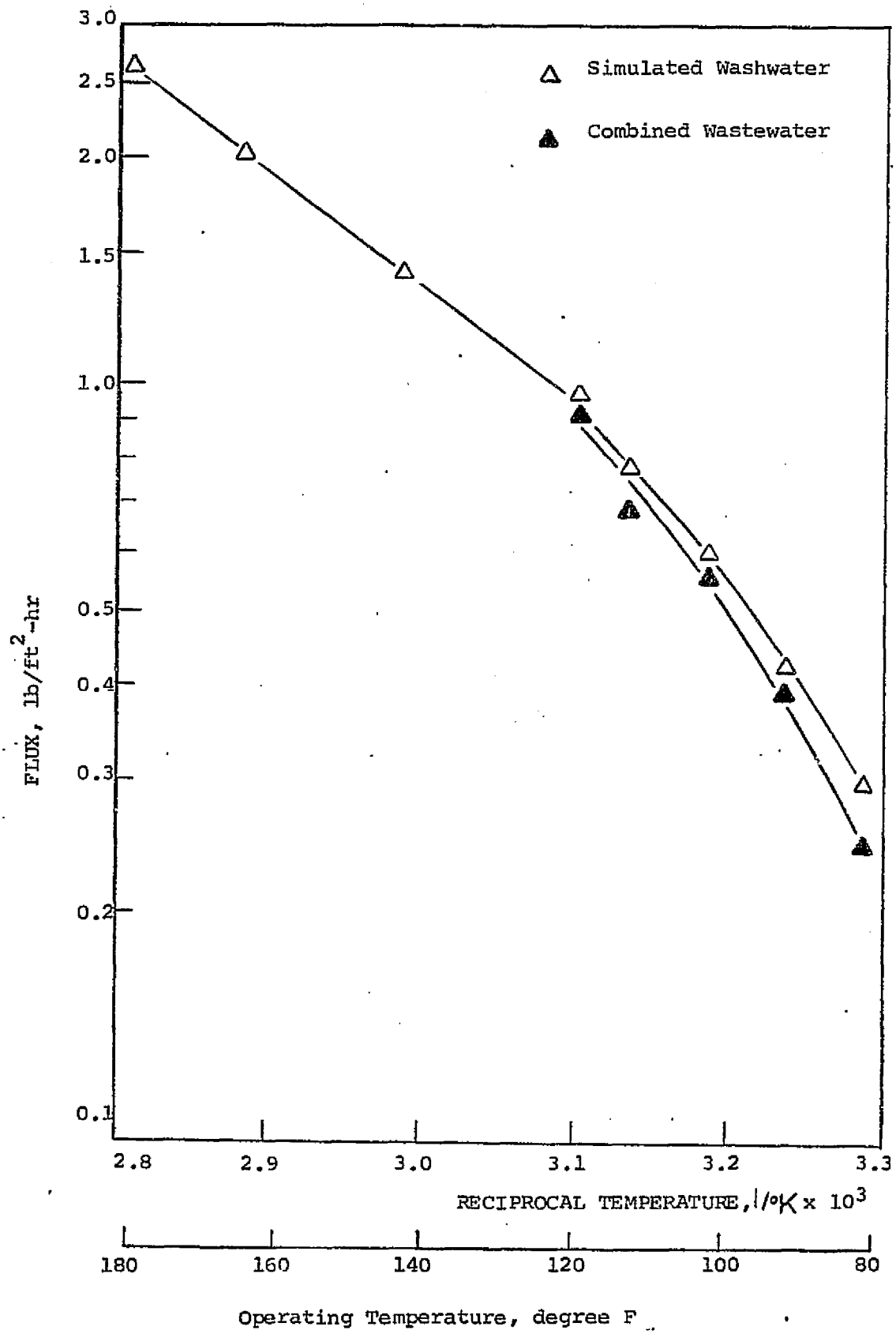
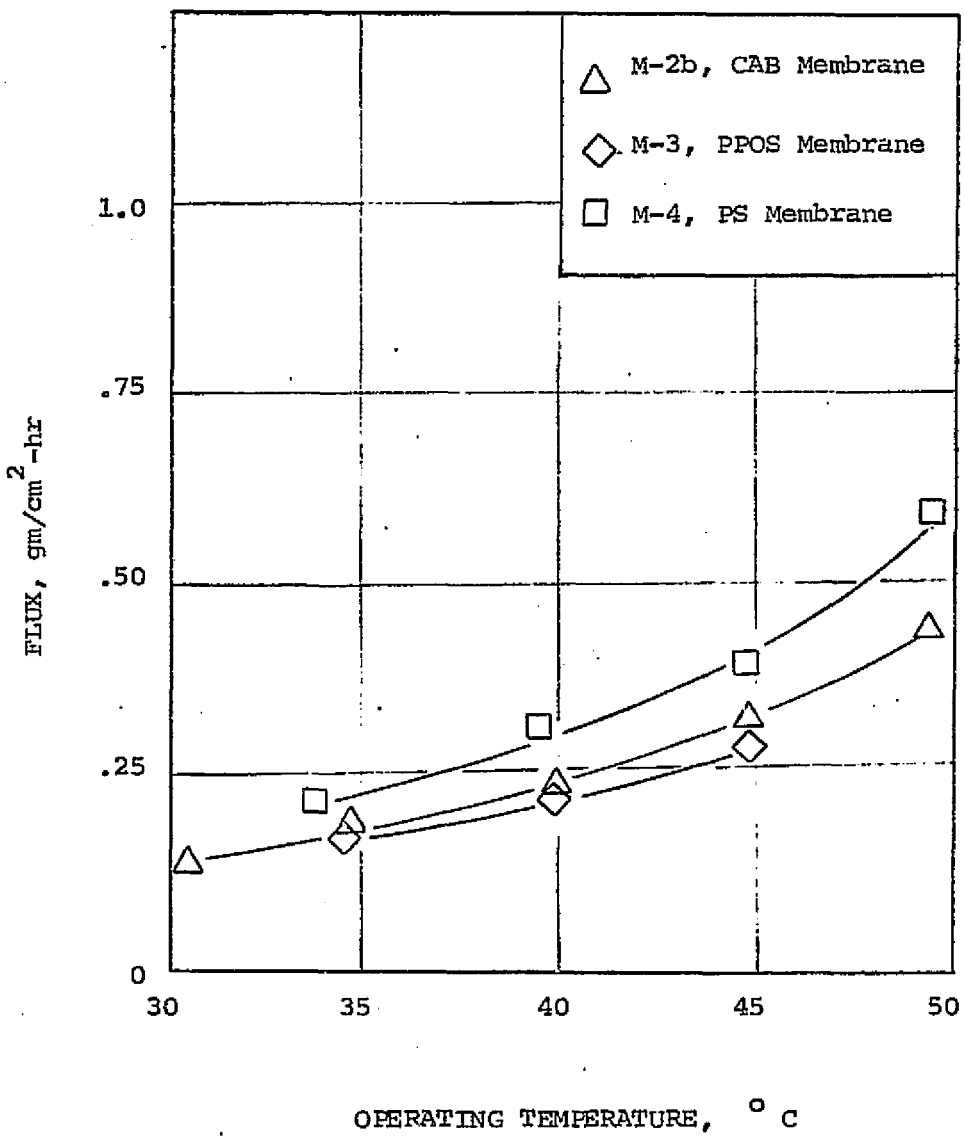


FIGURE 26. LOW TEMPERATURE PERMEATION OF WASHWATER



The M-2b, CAB membrane was selected because of its superior performance in VDR uring tests, as well as, its proven stability on prolonged exposure to both urine (performed during the present contract) and washwater (performed by the present investigator during an RO module-evaluation contrast with NASA-Ames).

It appears that to process 3ℓ of the combined wastewater (the estimated product of 1 man-day) using two VDR cells (each with effective membrane area of 220 cm^2 or 35 $in.^2$) at 38° C(100° F) a total of 40 hours of operation time are required to remove 80% of the water in the charge. Assuming membrane fouling, a total of 1320 cm^2 or 210 $inch^2$ (six pieces of membrane each equivalent to the one used in the bench unit) membrane area will be required to process 1 man-day of combined waste. If the VDR unit is designed in the stack configuration, assuming no further optimization work is performed, the size of the stack for an average flight crew of 4-men will be in the vicinity of a one-foot-cube. Figure 27 is a schematic diagram for such a system. Figure 28 is an exploded view of the stack assembly.

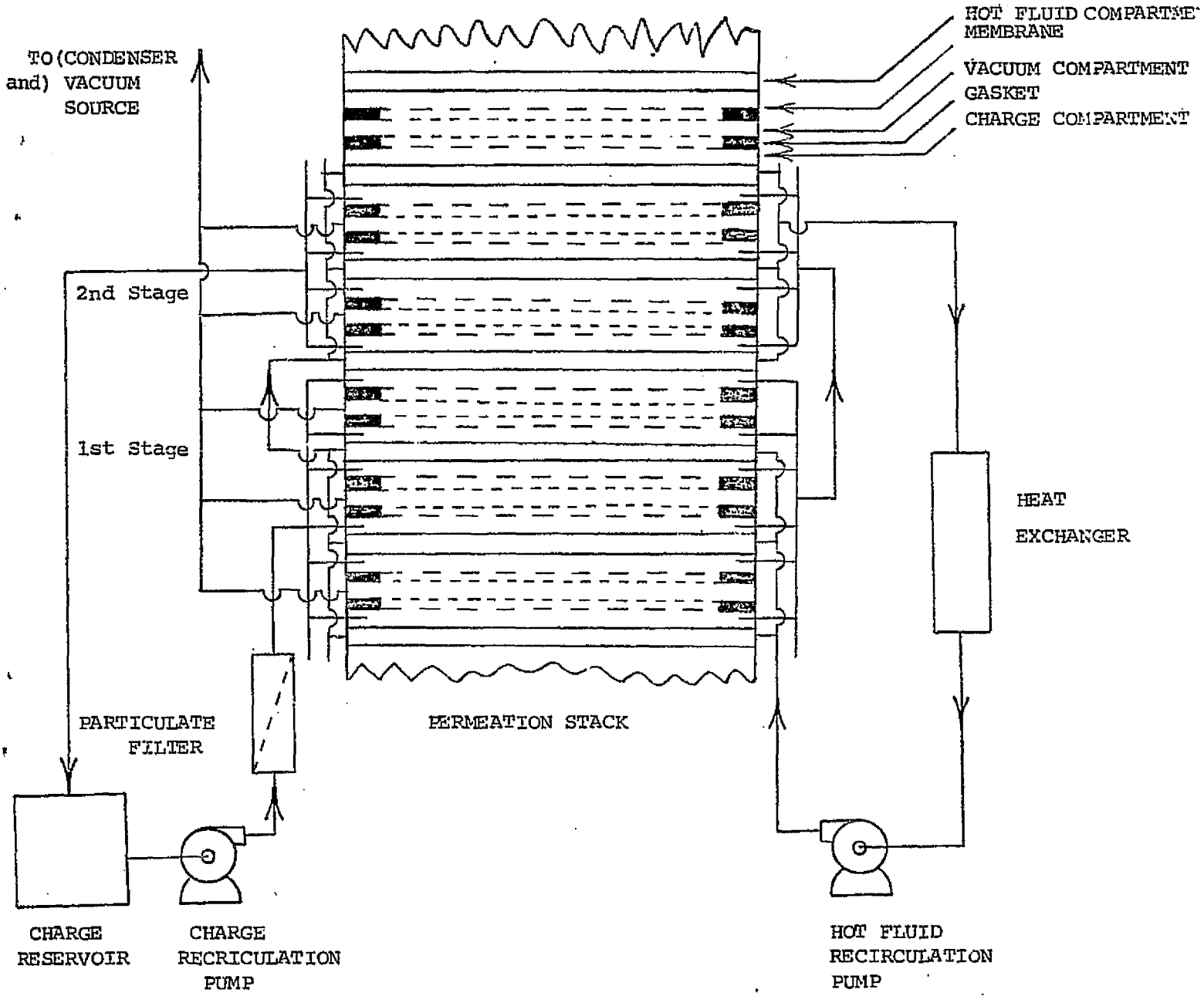


FIGURE 2.7. SCHEMATIC DIAGRAM OF A VDR UNIT IN STACK CONFIGURATION

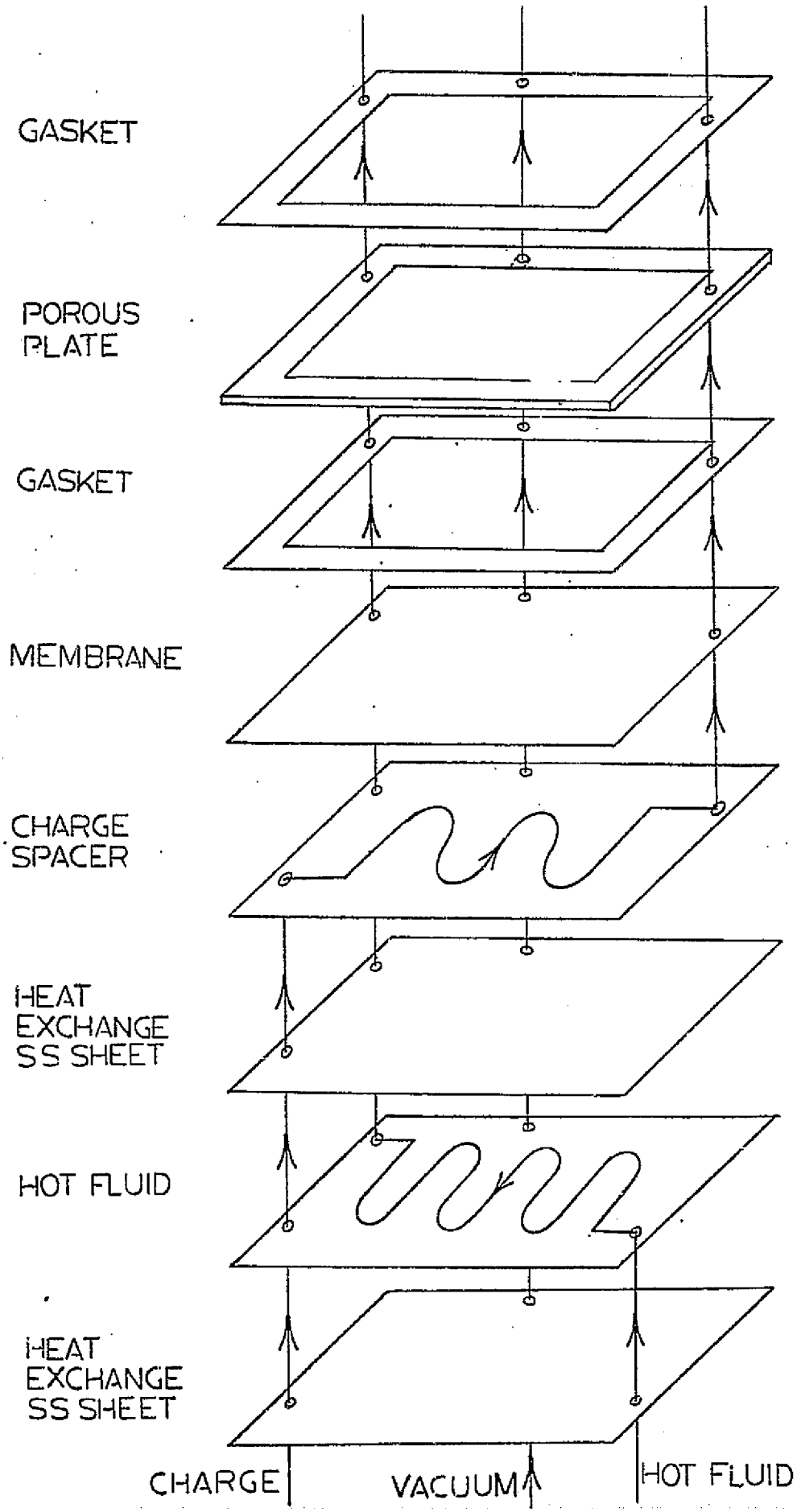



FIGURE 28. EXPLODED VIEW OF A VDR STACK