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FINAL REPORT

CR 151818

ON

BREADBOARD WASH WATER WASTE RENOVATION SYSTEM
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SUMMARY

Under Contract NAS 9-15369, a breadboard model of a soap wash water renovation system was designed, constructed, and put into operation. The system comprised pretreatment with ferric chloride to remove soap by chemical precipitation, carbon adsorption to remove trace dissolved organics, and ion exchange for removal of dissolved salts.

Using two candidate soaps - Ivory and ML-11 - at several concentrations, operating parameters for the various subsystems and the system as a whole were investigated. Evaluation of the system adequacy was made by monitoring the degree of soap and salt removal throughout the program.

At the conclusion of the program, the entire system was put into continuous operation and carefully monitored to assess overall efficiency and equipment maintenance problems that could be expected in actual use. In addition, the capacity of the carbon adsorbers and the ion-exchange resin was calculated and taken into consideration in the final evaluation of the system adequacy.

The product water produced during this final evaluation was well within the "Tentative Wash Water Standards" with regard to total organic carbon, conductivity, urea content, sodium chloride content, color, odor, and clarity.

I. INTRODUCTION

Over the past decade, reverse osmosis has emerged as a convenient and efficient technique for purification of brackish and waste waters. The RO systems are generally compact, the energy requirements are relatively low since the water is not forced through a phase change, and with proper system design it is possible to obtain potable water (i.e., less than 500 ppm dissolved solids) in a single pass.

With increasingly long space flights taking place, and with the possibility of orbiting space stations, it has become necessary for NASA to develop techniques to conserve and reclaim water. Perhaps the single greatest source of contaminated water from such missions is wash water from bathing and clothes washing. A typical wash water might contain approximately 0.10 percent detergent or soap and 0.005 percent NaCl; lesser amounts of urea, lactic acid, and phosphate builders; and trace amounts of miscellaneous suspended and colloidal materials such as lint, viruses, bacteria, grease, and soil. It is only natural that NASA is considering membrane separation as a basis for such a development.

Unfortunately, most of the membranes currently available have been designed primarily for salt rejection, and their operational life is adversely affected by wash water components such as detergents, bacteria, soaps, and divalent metal compounds, as well as the 175°F pasteurization temperature that is sometimes employed. It was proposed that, if the objectionable constituents could be removed by a pretreatment scheme before the wash water passed through the membrane, the membrane's operation and durability would be enhanced.

Under Contract NAS 9-13536, Springborn Laboratories (formerly DeBell & Richardson, Inc.) studied various techniques for eliminating objectionable wash water waste constituents, and determined that removal of such constituents by a pretreatment scheme is a feasible approach.

Laboratory scale tests demonstrated successful pretreatment schemes for chemical precipitation, filtration, and adsorption, using a simulated wash water containing Olive Leaf Soap.

Under Contract NAS 9-14518, the pretreatment work was expanded to include other soaps such as Ivory and Palmato. In addition, preliminary work was started on selecting antifoam agents for use with the various candidate surfactants.

Under Contract NAS 9-14965 there was continued development of a wash water pretreatment scheme, resulting in an optimum concept for removing objectionable materials from spacecraft wash water waste prior to its introduction into a reverse osmosis membrane system (currently under development by NASA).

During this study, optimum cleansing and antifoam agents were selected.

The purpose of this recently completed program was to develop a total wash water waste renovation system concept for removing objectionable materials from spacecraft wash water in order to make the water reusable. This concept included not only pre-treatment to remove suspended solids such as soap and lint, but also carbon adsorption and ion exchange to remove trace dissolved organics and inorganic salts. Ion exchange was used here in place of reverse osmosis.

To develop the system concept, we designed and constructed a bread-board model of a total water renovation system which was then used to demonstrate the design adequacy of the various system components as well as the limits on system capacities and efficiencies. For demonstration testing, synthetic wash waters based on both Ivory Soap and ML-11 liquid soap were used. Chemical precipitation was with ferric chloride.

II. BREADBOARD DESIGN AND CONSTRUCTION

The wash water renovation system that was designed and constructed is essentially the same as that originally proposed; a schematic diagram of the system appears in Figure 1 (page 8). The major components of the system and their functions are as follows:

Components

1. Reservoir (Nalgene Aspirator Bottle: 2302-0020; 1/2 Gallon Capacity)

The reservoir contained the synthetic wash water feed which, for the purpose of the breadboard, was fed to the system by gravity.

2. Prefilter (AMF-Cuno Auto-Klean Filter: Type DS)

This unit acted as a prefilter for the system to remove hair, lint, and other suspended matter.

3. Centrifugal Liquid/Gas Separator

This unit, designed and built by Hamilton Standard as a urine separator, was used to assess the foaming problem.

5. Centrifugal Pump (Jabsco Centrifugal Pump Model 17860-0000)

This pump picked up the water from the separator and carried it to the mixing chamber.

8. Mixing Chamber

This custom-built, cylindrical, all clear acrylic plastic chamber with free-floating piston was used to mix the ferric chloride coagulant with the soapy wash water to ensure adequate precipitation. Water from the centrifugal pump (5) entered the cylinder from the bottom, forcing the piston upward by positive

displacement. Once the mixing had been completed, air pressure applied through the top of the cylinder by solenoid valve drove the piston downward, forcing the water through the remainder of the renovation system. Check valves in the system prevented backflow during discharge from the cylinder.

9. Filter (Cuno Filter Housing; Model CT-101)

This filter, fitted with a Micro-Wynd Filter Cartridge rated for 1-micron nominal pore diameter, was used to remove the suspended ferric chloride/soap precipitate. Driving pressure came from the air pressure applied to the piston in the mixing chamber.

10. Carbon Adsorber Columns (Cuno Filter Housings; Models CT-101 or Type 1M)

These columns were fitted with AMF-Cuno Activated Carbon Cartridges. In the original system, we proposed the use of one activated charcoal column. We later chose to employ the cartridge type units to facilitate handling and replacement of the charcoal.

These adsorber units were contained in conventional housings such as those employed for the cartridge depth filter. In order to ensure adequate contact time of the water with the activated charcoal, we used two units in series. While a contact time of 3-4 minutes may have been adequate, a period as long as 12-16 minutes was thought to be more desirable. Most of the contact came during "down time" of the system between batches of water; we estimate that each cartridge held approximately one batch of water.

11. Ion Exchange Column (AMF-Cuno Demineralizer Housing; Model SM 04010)

This ion exchange column used AMF-Cuno mixed strong anion/strong cation exchange resin. The only major modification to the

system as originally proposed came in the deionizer subsystem; this change was forced by the nature of the deionization process, which does not lend itself to intermittent flow.

For efficient removal of electrolytes, a deionizer is best operated continuously, in a flow rate range of approximately 1-5 gallons per minute per cubic foot of bed. For this reason, we included an optional hold tank and recirculation line in the deionizer subsystem, to provide the capability to collect several batches of water which could then be recirculated through the deionizer at the appropriate flow rate for two or three full cycles and then be discharged from the system. The recirculation was thought to be necessary in order to:

- a. Treat the water which has lain dormant in the deionizer and become contaminated with salt migrating from the resin.
- b. Allow the bed to reach "steady state" where it is producing good-quality effluent.

12.,

14. Reservoirs (Nalgene Aspirator Bottles: 2302-0001, 1-Quart Capacity)

These reservoirs were used to store antifoam agent and the ferric chloride solution, respectively. For the purpose of the breadboard, the solutions were fed by gravity.

13.,

15. Metering Pumps (Cuno; Model 10611-321)

These pumps were used for addition of antifoam agent and ferric chloride solution into the wash water.

Check valves were placed in the system to prevent back flow (6), and pressure gauges were included in order to monitor pressure drop across key components such as the depth filter cartridge. A flow gauge was inserted

in the deionizer subsystem to ensure that the water recirculated within the desired range of flow rates.

The components were wired so that the operational sequence for the renovation system was as follows:

- . The initial position, "ON" - "OFF", of motors and cylinders at start was:

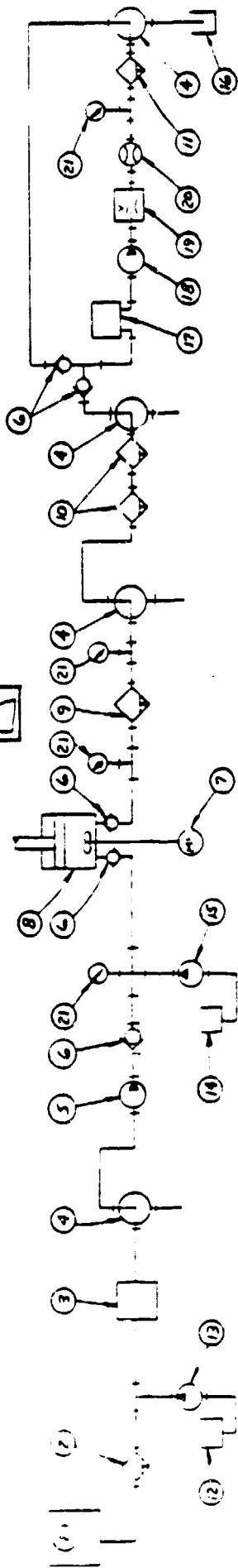
<u>Component Number</u>	<u>Description</u>	<u>Position</u>
13	Metering Pump 1	Off
3	Centrifugal Separator	Off
5	Centrifugal Pump 1	Off
15	Metering Pump 2	Off
-	Solenoid Valve	For air pressure to cylinder - deenergized
8	Mixing Chamber Pistor.	Down
8	Mixer Motor	Off
18	Centrifugal Pump 2	Off

- . When the system was started by a manual switch, metering pumps 1 and 2, the centrifugal separator, and the centrifugal pump turned on. All remained on until the mixing chamber piston tripped a limit switch. At that time the centrifugal separator, both metering pumps, and the centrifugal pump 1 turned off. A manual switch energized the solenoid valve, which introduced air behind the mixing chamber piston and forced it down. When the piston reached the bottom position, and tripped the other limit switch, the entire system stopped.

The breadboard model was constructed in accordance with the design presented above. To facilitate handling and moving the system, all components were mounted on an open box-shaped plywood frame. To aid in observing the mixing action of the antifoam and the ferric chloride, to identify areas where ferric chloride/soap sludge might hang up in the system, and also to facilitate replacing or moving components within the system, we used clear plastic tubing (Tygon, plasticized polyvinyl chloride) for all of the fluid lines.

To join the tubing to the various components, we used plastic reducing nipples and plastic tubing connectors.

DBA-F17-B



ITEM NO. DESCRIPTION

- 1 RESERVOIR
- 2 FILTER
- 3 URINE SEPARATOR
- 4 SAMPLING VALVE
- 5 CENTRIFUGAL PUMP
- 6 CHECK VALVE
- 7 STIRrer MOTOR
- 8 MIXING CHAMBER
- 9 FILTER
- 10 CARBON ABSORPTION COLUMN
- 11 ION-EXCHANGE COLUMN
- 12 ANTI-FOAM RESERVOIR
- 13 METERING PUMP
- 14 FERRIC CHLORIDE RESERVOIR
- 15 METERING PUMP
- 16 STORAGE RESERVOIR
- 17 HOLDING TANK
- 18 RECIRCULATING CENTRIFUGAL PUMP
- 19 FLOW CONTROL VALVE
- 20 FLOW METER
- 21 PRESSURE GAUGE

FIGURE 1

Schematic Diagram of the Wash Water Renovation System

△ VENDOR NO. & DWG. NO. NEXT ASSEMBLY

UNLESS OTHERWISE SPECIFIED SCALE		SCHEMATIC		SPEERBORGH LABORATORIES, INC. 375 FIELD CREST, TOWSON, MD.	
TOLERANCE ON FRACTIONS ± 1/8"		WATER FILTRATION UNIT		ENG. APPROVAL	
TOLERANCE ON DECIMALS ± .005		MATERIAL		DATE	
TOLERANCE ON ANGLES ± 1/2°		CHECKED		DATE	
				DBA-F17-B	

III. ANTIFOAM STUDIES

Once the breadboard had been constructed, we conducted demonstration experiments based on the requirements presented in the statement of work of the contract. These experiments were used to demonstrate the design adequacy of the various system components as well as the limits on system capacities and efficiencies.

A detailed plan for these demonstration experiments was presented in the Third Quarterly Report under this contract - covering the period January 15, 1978 - April 15, 1978.

For antifoam investigations, we used the breadboard model from the reservoir (1) to the first sampling valve (4), as indicated in Figure 1 (page 8). These investigations were conducted with both Ivory Soap and ML-11 soap based wash waters at a soap concentration of 1,000 ppm or 0.1 percent. Water was fed by gravity to the separator.

The separator was operated at 26 volts to give a nominal speed of 13,000 rpm. At flow rates ranging from 50 to 900 cc/minute there was no evidence of foam exiting from the "gas outlet" of the separator. This was true when using either soap.

At low flow rates, however, above 360 cc/minute but below 600 cc/minute, there were small quantities of foam in the exiting fluid line. At flow rates of 600 cc/minute or greater, the foam disappeared.

As a check on this phenomenon, we repeated the experiment using the ML-11 soap based wash water, and at the same time added 200 ppm SWS-211 antifoam to the system from the first metering pump (13), as identified in Figure 1. This time we continued to get air in the fluid exit line, but there was essentially no foam, although the degree of difference between foam and air bubbles is somewhat judgmental.

We concluded, therefore, that the traces of air/foam in the fluid line were probably a function of the way in which the separator was

being operated, and that we did not appear to have a foaming problem in the form of foam exiting the gas outlet.

With the concurrence of the Technical Project Officer, we conducted the remainder of the breadboard demonstration without the use of antifoam addition.

IV. PRETREATMENT STUDIES

OPTIMUM DOSAGE

Using the results from previous programs on wash water pretreatment as a guide, we investigated the optimum dosage of ferric chloride coagulant. Again, both ML-11 and Ivory soap based wash waters were employed at 1,000 ppm soap concentration.

For this work the breadboard was used from the reservoir (1) up to the second sampling valve (4) after the filter (9), as shown in Figure 1 (page 8). The displacement was adjusted in order to process a nominal "half pound" of water per cycle - namely, 225 cc plus 5 cc to compensate for the volume of 1 percent ferric chloride added per cycle.

For this pretreatment work the metering pump was adjusted to deliver the desired dosage of ferric chloride in one minute, and was operated manually.

The needle valve on the output side of the centrifugal pump (5) was adjusted, in order to provide a mixing chamber fill time of between 1 and 2 minutes.

Slow mix time for this portion of the effort was 5 minutes.

The effect of dosage on soap removal is indicated in Table 1.

With wash waters based on both ML-11 and Ivory soap, there was a high degree of soap removal over a fairly broad range of ferric chloride dosage. In the case of Ivory soap, dosages of from 180 to 210 ppm resulted in soap removals of 96 percent or better; while with the ML-11 soap based wash water, dosages spanning 230 to 260 ppm resulted in soap removals of nearly 95 percent. For the "optimum" dosages, used in subsequent work, we selected the midpoint of these high-removal ranges: 240 ppm ferric chloride for the ML-11 soap based wash water, and 200 ppm ferric chloride for the soap based on Ivory soap.

TABLE 1

Effect of Ferric Chloride
Dosage on Soap Removal Using
1000 ppm Soap Based Wash Water

Sample Number	Ferric Chloride Dosage (ppm)	Residual Soap Concentration (ppm) (1)	Percent Soap Removed
. Ivory Soap Based Wash Water			
A-6089-1	160	> 170/> 170	< 83/< 83
A-6090-2	170	66/64	93.4/93.6
A-6090-3	180	36/39	96.4/96.1
A-8452-1	190	39/36	96.1/96.4
A-8452-2	200	31/31	96.9/96.9
A-8452-3	210	25/28	97.5/97.2
A-8453-1	220	61/59	93.9/94.1
. ML-11 Soap Based Wash Water			
A-6088-1	220	85/87	91.5/91.3
A-8459-1	230	42/49	95.8/95.1
A-8460-2	240	53/52	94.7/94.8
A-8460-3	250	52/47	94.8/95.3
A-6087-D	260	51/54	94.9/94.6
A-6088-2	270	59/57	94.1/94.3

(1) Based on carbon tetrachloride extraction/
IR analysis for carbonyl.

We also determined the effect of shorter slow-mix periods on the degree of soap removal. This work was conducted at the optimum ferric chloride dosage for the soap in question. Mixing time was varied from no mixing (other than that done during filling of the mixing chamber) to the original 5-minute mix time.

The evaluation was conducted with wash waters based on both candidate soaps. All other operating parameters were as before.

The results are presented in Table 2. There are some minor variations in the soap removal results - particularly in the case of sample A-8455-3, where a soap removal of 97.9 was determined. Based on prior experience with removal work and analysis, these variations are not abnormal and are likely due to experimental error inherent in the analysis procedure.

The trend in the data for both wash waters clearly indicates that no slow mixing is required beyond that which occurs during the filling of the mixing chamber, or approximately 90 seconds.

For the remainder of the work we employed no slow mixing beyond that done during the fill cycle.

Once ferric chloride pretreatment dosage parameters for 1000 ppm soap wash water had been determined, further optimum dosage experiments were carried out using both Ivory soap and ML-11 based wash water at concentrations of 500 ppm and 2500 ppm. Again, no slow mix time other than agitation during the chamber fill cycle was employed, and the breadboard operating conditions were the same as with previous pretreatment studies.

The results are presented in Table 3 and Table 4.

For the 500 ppm Ivory soap based wash water, the optimum dosage of ferric chloride comes at 110 ppm, while the maximum soap removal for the ML-11 based wash water is at a ferric chloride dosage of 130 ppm. As was experienced with bench-level experiments during previous work, the residual level of soap in the pretreated water is less for the 500 ppm soap based wash water than for the 1000 ppm soap based solution, while the percent soap remaining is greater.

Table 4 presents the results of the pretreatment studies using 2500 ppm soap based wash waters using, again, both Ivory soap and ML-11. In both cases the so-called optimum dosage of ferric chloride spans a fairly wide range - from approximately 500 ppm to 560 ppm for the Ivory soap based wash water, and 560 ppm to 640 ppm ferric chloride for the ML-11 based solution.

It is possible that the high degree of soap removal over a broad dosage range may result from partial adsorption or possibly chemical precipitation of uncoagulated soap, as the pretreated water flows through the ferric chloride/soap filter cake deposited on the filter cartridge.

Again, the soap removal results are similar to what was experienced on the bench with previous pretreatment experiments. While the residual soap contents in the pretreated wash water samples tend to be higher for the 2500 ppm soap based wash water than the 1000 ppm soap based solutions, the percent remaining in the water tends to be less.

Optimum dosage values were generally as expected with the possible exception of the 2500 ppm Ivory soap wash water results. At this high soap concentration, the Ivory soap based wash water had a distinct gel-like nature, causing pumping problems during operation of the breadboard. Results, therefore, may be somewhat suspect. It is very unlikely, however, that an Ivory soap concentration of 2500 ppm would be encountered in a real wash water.

TABLE 2

**Effect of Slow Mix Time on the
Soap Removal at Optimum Ferric Chloride Dosage
With 1000 ppm Soap Based Wash Water**

Sample Number	(1) Mix Time (min)	(2) Residual Soap Conc. (ppm)	Percent Soap Removed
• Ivory Soap Based Wash Water 200 ppm Ferric Chloride			
A-8455-1	5	39/35	96.1/96.5
A-8455-2	3	34/41	96.6/95.9
A-8455-3	1	21/37	97.9/96.3
A-8455-4	0	40/44	96.0/95.6
• ML-11 Soap Based Wash Water 240 ppm Ferric Chloride			
A-8460-2	5	53/52	94.7/94.8
A-8461-1	3	28	97.2
A-8461-2	1	-	-
A-8461-3	0	46	95.4

(1) Does not include the 1.5 minutes of mixing that takes place during filling of the mixing chamber.

(2) Based on carbon tetrachloride extraction/IR analysis for carbonyl

TABLE 3

**Effect of Ferric Chloride Dosage on
Soap Removal Using 500 ppm Soap Based Wash Water**

Sample Number	Ferric Chloride Dosage (ppm)	(1) Residual Soap Conc. (ppm)	Percent Soap Removal
. Ivory Soap Based Wash Water			
A-8482-3	80	38/38	92.4/92.4
A-8482-2	90	20/20	96.0/96.0
A-8482-1	100	19/20	96.2/96.0
A-8481-3	110	11/11	97.8/97.8
A-8481-2	120	14/12	97.2/97.6
A-8481-1	130	28/28	94.4/94.4
. ML-11 Soap Based Wash Water			
A-8468-3	110	114/115	77.2/77.0
A-8468-2	120	51/50	89.8/90.0
A-8468-1	130	27/24	94.6/95.2
A-8467-2	140	30/28	94.0/94.4
A-8467-1	150	78/76	84.4/84.8

(1) Based on carbon tetrachloride extraction/
IR analysis for carbonyl.

TABLE 4

Effect of Ferric Chloride Dosage
on Soap Removal Using 2500 ppm
Soap Based Wash Water

Sample Number	Ferric Chloride Dosage (ppm)	Residual ⁽¹⁾ Soap Conc. (ppm)	Percent Soap Removal
. Ivory Soap Based Wash Water			
A-8485-2	480	>170/>170	< 93.2/<93.2
A-8485-1	500	31/29	98.8/98.8
A-8484-2	520	32/33	98.7/98.7
A-8484-1	540	28/28	98.9/98.9
A-8483-1	560	33/32	98.7/98.7
. ML-11 Soap Based Wash Water			
A-8476-1	540	134/131	94.6/94.8
A-8475-4	560	102/ -	95.9/ -
A-8475-2	580	97/100	96.1/96.0
A-8475-3	600	100/100	96.0/96.0
A-8475-1	620	115/114	95.4/95.4
A-8474-1	640	66/65	97.4/97.4

(1) Based on carbon tetrachloride extraction/
IR analysis for carbonyl.

V. ADSORPTION STUDIES

The adsorption studies were conducted using the breadboard from the feed reservoir (1) to the sampling valve (4) after the second adsorber cartridge (10), as depicted in Figure 1. As discussed in Section II, the system included two adsorber cartridges mounted in series in conventional filter housings. Two units were employed in order to allow adequate contact time of the water with the activated charcoal.

The water entering the adsorbers was pretreated with ferric chloride and filtered, and contained approximately 20 to 50 ppm of residual soap in addition to dissolved salts, urea, and lactic acid.

Water was forced through the adsorbers during pressurization and emptying of the mixing chamber. Flow rate through the adsorbers was a nominal 500 cc/min.

Adsorption studies were conducted with wash waters based on both Ivory soap and ML-11 at soap concentrations of both 1000 and 2500 ppm. The effect of the adsorption on the quality of the water is detailed in Table 5. The data indicates that the level of organic material remaining in the treated water is at a very low level, and well below the specifications of 200 ppm Total Organic Carbon and 50 ppm urea.

There is a slight discrepancy between the values for TOC and residual soap; this is probably due to error in the soap analysis, since these values lie at the lower limit of detectability for the procedure being used.

The values reported here for urea have been determined indirectly from the Total Organic Nitrogen data. While the removal efficiency for the urea is comparatively low, it should be sufficient to keep the level of urea in the water well below the target upper limit of 50 ppm.

TABLE 5

Effect of Carbon Adsorption on
Pretreated Wash Water Quality

Wash Water Type	Residual Soap		(1)	(2)	Residual Urea	
	ppm Soap	Total Percent Removed	ppm TOC	ppm TON	Approx. ppm	Approx. Percent Removed
Ivory soap based wash water - 1000 ppm soap (3)	2/2	99.8/99.8	6/6	3.19/ 3.30	6.8/7.1	32/29
2500 ppm soap (4)	-	-	-	-	-	-
ML-11 soap based wash water - 1000 ppm soap (5)	2/0	99.8/100	8/7	1.62/ 1.64	3.5/3.5	65/65
2500 ppm soap	-	-	20/21	0.84/ 1.23	1.8/2.6	82/74

(1) TOC - Total Organic Carbon

(2) TON - Total Organic Nitrogen

(3) 1000 ppm Ivory soap, treated with 200 ppm ferric chloride, and filtered.

(4) No data; 2500 ppm Ivory solution not homogeneous

(5) 1000 ppm ML-11, treated with 240 ppm ferric chloride, and filtered.

VI. ION EXCHANGE STUDIES

Ion exchange studies with the breadboard were conducted from the supply reservoir (1) to the end of the system (16). Although the design included the option of operating the deionizer in a recirculating loop, this proved to be unnecessary. All ion exchange was done with the exchange bed in series in the system, and with a single pass through the resin.

The water entering the ion exchange bed contained essentially no residual soap, but did contain some urea as well as dissolved salts. The salts included not only those added to the synthetic wash water, but also those resulting from the ion interchange between the soap (potassium or sodium fatty acid salt) and the ferric chloride. These salts were present as additional quantities of either potassium or sodium chloride as reflected by the initial conductivity on the feed water entering the ion exchange unit (see Table 6).

Again, water was forced through the ion exchange bed during pressurization and discharge of water from the mixing chamber. Flow rate was again a nominal 500 cc per minute.

The results of ion exchange on the quality of the product water are presented in Table 6. As both conductivity measurements and chloride ion titrations indicate, ion exchange is extremely effective, lowering the salt concentration to virtually nothing.

TABLE 6

**Effect of Ion Exchange on
Pretreated Wash Water Quality (1)**

Wash Water Type	Conductivity		Cl ⁻ (2) Conc. (ppm)
	μ mho-cm ⁻¹	Approx. Salt Conc. (ppm)	
Ivory soap based wash water -			
Before treatment	600	320	160
After treatment	2.22	< 1	1.1
ML-11 soap based wash water -			
Before treatment	760	410	185
After treatment	1.93	< 1	0.5

- (1) 1000 ppm soap based wash water, pretreated with ferric chloride, filtered, and exposed to activated charcoal.
- (2) By titration using Mohr's method with silver nitrate and potassium chromate.

VII. CAPACITY STUDIES

In this portion of the demonstration, we determined the capacities of the various components of the breadboard, including the filter, adsorbers, and ion exchange bed.

The capacity of the ion exchange resin was determined indirectly from the stated capacity as provided by the resin supplier. The mixed-bed strong anion/strong cation exchange resin employed in the breadboard has a stated capacity of 12,000 grains (as CaCO_3) per cubic foot of bed.

The water employed in our work has a total salt concentration of 310 ppm when expressed as CaCO_3 equivalent. Ironically, most of this salt - approximately 220 ppm, comes from the ion interchange between the soap and ferric chloride.

Based on these parameters, an exchange column of the size used on the breadboard (1/16 cubic foot) would have a capacity of 33.4 gallons (126 liters), when using a typical 1000 ppm soap based wash water. Assuming a nominal 40 shots per day, the unit would last approximately 14 days.

To determine the capacity of the activated charcoal, we conducted a small-scale evaluation on the bench using 25 grams of the activated charcoal from one of the adsorber cartridges. The charcoal was packed into a 1.5 cm inner diameter glass column; a 2500 ppm ML-11 soap based wash water was allowed to flow through the charcoal bed at a rate of 2 cc per minute, providing a dwell time in the bed of approximately 12 minutes.

Samples of the effluent were taken periodically and evaluated for soap content. A plot of residual soap and percent soap removed versus cumulative volume of water processed appears in Figure 2. There was a fairly dramatic drop in the percent soap removed after approximately 1500 cc of water had been processed.

If we assume that the practical capacity of the activated charcoal is equivalent to 1500 cc of water for 25 grams of charcoal when using a

2500 ppm soap based feed, then two adsorber cartridges containing 340 grams of charcoal each would have a combined capacity of 2040 liters (540 gallons) when processing a pretreated and filtered wash water with a nominal residual soap content of 50 ppm.

Assuming a nominal 40 "half-pound" shots per day, the two adsorber cartridges should last 225 days.

During extended operation of the breadboard (see Section VIII of this report), we monitored the pressure drop across the filter as well as the discharge time from the mixing chamber as a measure of the capacity of the filter cartridge. In addition, at the end of the five-day operation, 40 shorts per day, we examined the filter cake on the filter.

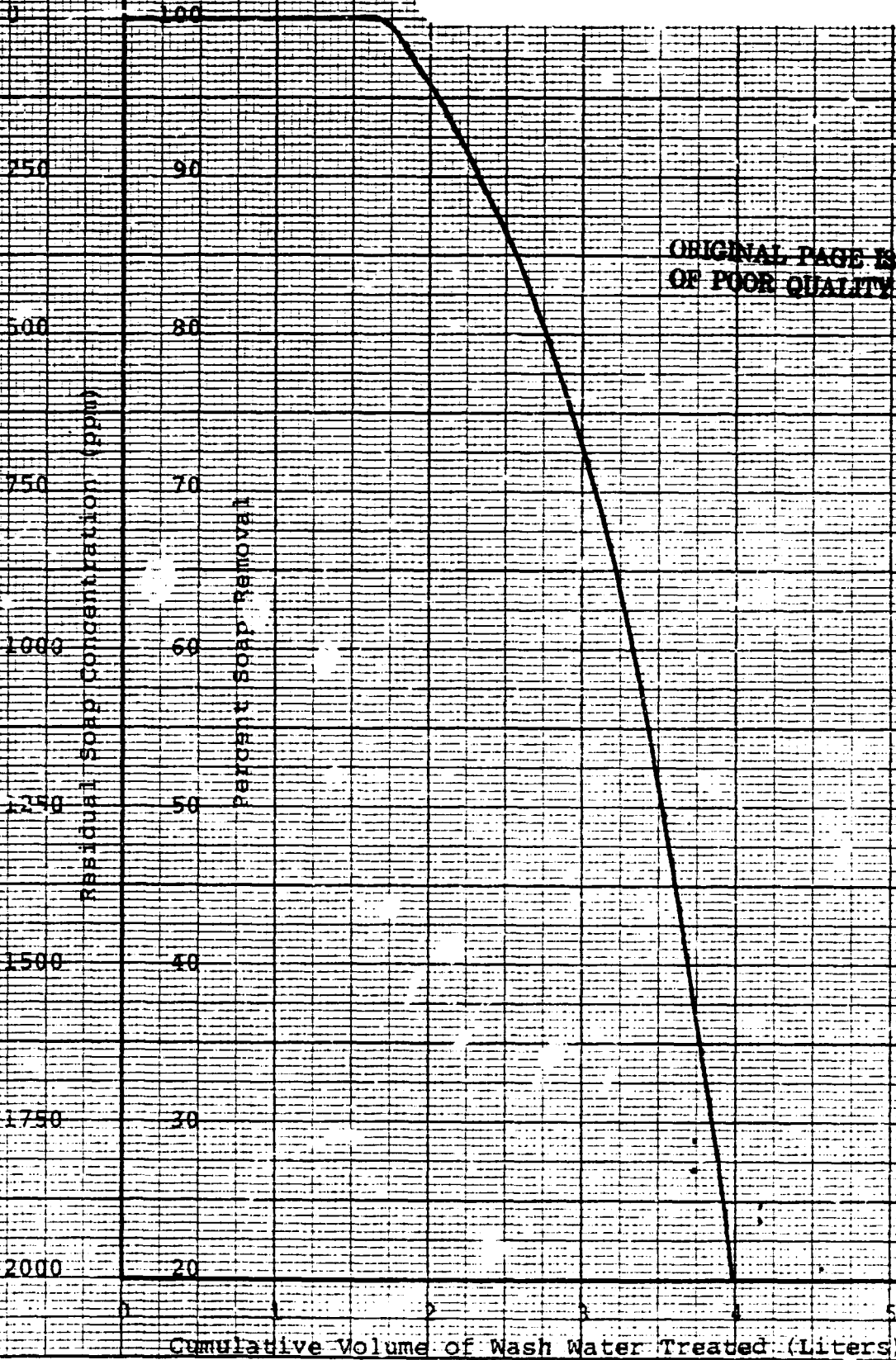
At the completion of the five-day operation, the filter housing was approximately 10 percent filled, indicating that the capacity of the unit would likely be a nominal 50 days, or 450 liters (120 gallons).

We envision that all of the components would be installed in the renovation system, full of water. In the case of expendable components such as adsorbers or ion exchangers, we would expect that extra units with housings would be installed so that they could be valved into the system as needed to replace exhausted units.

With a system such as this there would be essentially 100 percent water utilization or "recovery efficiency". That is, for every "half pound" of water entering the system, an equivalent amount would exit from the end of the system.

FIGURE 2

Residual Soap Concentration and % Soap
Removed Vs. Cumulative Volume of
Wash Water Treated
(2500 ppm ML-11 Soap)



VIII. EXTENDED OPERATION AND OVERALL EFFICIENCY

In order to evaluate the ability of the breadboard to function properly during extended operation as well as the overall ability of the system to treat water to the Tentative Wash Water Standards (presented in Table 1 of the Statement of Work of the Contract, and included here as Table 7), we employed the breadboard from the feed reservoir (1) to the end of the system (16).

For wash water we used a synthetic mixture containing 1000 ppm of ML-11. Coagulation was with 240 ppm ferric chloride.

The breadboard was operated over a five-day period with forty 225 cc shots per day. During this time we routinely monitored soap removal both before and after the adsorber, pressure drop across the filter, discharge time for the mixing chamber - a measure of the pressure drop through the rest of the system, and conductivity and chloride ion titration after the ion exchange bed.

Table 8 presents the residual soap concentration and soap removal both before and after the adsorber over the duration of the test. Although there was some slight variation in the soap removal after the filter on start-up of the breadboard each day, the percent removal was still quite high. Total removal after the adsorber remained excellent throughout the test, registering "no detectable soap" in most cases.

Table 9 details the operating performance of the unit throughout the five-day evaluation. There was little or no increase in pressure drop across the filter or in discharge time for the mixing chamber over the course of the test. As was mentioned previously, at the conclusion of the test the filter housing was filled only to about 10 percent of capacity with soap/ferric chloride sludge.

The overall soap removal for the system, when checked at the end of each day, remained a consistent 100 percent over the five-day period. The residual salt content of the water as indicated by the conductivity measurements and chloride ion titration remained at a very low level during the entire test. The conductivity of the water (i.e., less than $2 \mu \text{ mho-cm}^{-1}$) is equivalent to a salt concentration of less than one ppm.

In summation, the concept has been successfully demonstrated on a systems basis over an extended period of time with no serious problems arising from basic design considerations. There were some minor maintenance problems, however, which will be discussed in the following section (IX).

The final task of the demonstration was to determine the ability of the breadboard to treat water to the "Tentative Wash Water Standards" as presented in Table 7. A full characterization of the product water from the breadboard is detailed in Table 10; data is presented for wash waters based on both ML-11 and Ivory soap. A comparison of this data with Table 7 reveals that the product water from the breadboard is well within the Tentative Wash Water Standards.

TABLE 7

Tentative Wash Water Standards

Total Organic Carbon (TOC), mg/l	200
Specific Conductivity, $\mu\text{mho-cm}^{-1}$	2000
pH	5 to 7.5
Ammonia, mg/l	5
Turbidity, ppm SiO ₂	10
Color, Pt-Co Units	15
Foaming	Nonpersistent more than 15 seconds
Odor	Nonobjectionable
Total Dissolved Solids (TDS), mg/l	1500
Urea, mg/l	50
Lactic Acid, mg/l	50
NaCl, mg/l	1000
Microorganisms, number per ml	10

TABLE 8

Efficiency of Soap Removal
Over Five Days of Operation⁽¹⁾

Sample Number	Day	Residual ⁽²⁾ Soap Conc. (ppm)	Percent Soap Removed
• Efficiency of Pretreatment and Filtration ⁽³⁾			
A-8494-1	1	64/62	93.6/93.8
A-8498-1	2	29/31	97.1/96.9
A-8500-1	3	53/54	94.7/94.6
A-8603-1	4	63/63	93.7/93.7
A-8605-1	5	30/45	97.0/95.5
• Efficiency of Carbon Adsorption ⁽⁴⁾			
A-8494-5	1	0/0	100.0/100.0
A-8498-6	2	0/0	100.0/100.0
A-8500-6	3	0/0	100.0/100.0
A-8603-6	4	2/1	99.8/99.9
A-8605-6	5	0/2	100.0/99.8

- (1) Five days of operation of the breadboard;
40 "half-pound" shots of 1000 ppm ML-11
based wash water per day.
- (2) Analysis for carbonyl using carbon tetrachloride
extraction/IR method.
- (3) Samples taken at the start of daily operation.
- (4) Samples taken at the end of daily operation.

TABLE 9

Effect of Continuous Operation on
Operating Parameters and Product Water Quality

Sample Number	Shot	Pressure Drop Across Filter (psig)	Mixing Chamber Discharge Time (sec)	(1) Percent Soap Removed	(2) Specific Conductivity ($\mu\text{mho-cm}^{-1}$)	(3) Chloride Ion (ppm)
• Day 1						
A-8494-2	5	3.1	25	-	-	-
	10	3.2	23	-	1.09	-
A-8494-3	15	3.2	22	-	-	-
	20	3.3	21	100.0	1.36	2.6
A-8494-4	25	3.3	21	-	-	-
	30	3.4	21	-	1.77	-
A-8494-5	35	3.4	23	-	-	-
	40	3.4	22	100.0	1.77	2.3
• Day 2						
A-8498-2	45	3.4	22	-	-	-
	50	3.4	24	-	1.95	-
A-8498-3	55	3.5	23	-	-	-
	60	3.5	23	100.0	1.61	2.9
A-8498-4	65	3.5	24	-	-	-
	70	3.5	25	-	1.56	-
A-8498-5	75	3.5	26	-	-	-
	80	3.5	25	100.0	1.54	2.0
• Day 3						
A-8500-2	85	3.5	28	-	-	-
	90	3.5	28	-	7.57	-
A-8500-3	95	3.5	28	-	-	-
	100	3.4	28	100.0	1.46	1.4
A-8500-4	105	3.3	29	-	-	-
	110	3.1	22	-	1.36	-
A-8500-5	115	3.1	19	-	-	-
	120	3.6	24	100.0	1.13	0.8

(1) Based on CCl_4 extraction/IR analysis for carbonyl

(2) Value from conductivity bridge data

(3) Mohr's method of chloride ion determination (titration)

...Continued

TABLE 9 (Continued - 2)

Sample Number	Shot	Pressure Drop Across Filter (psig)	Chamber Discharge Time (sec)	Percent Soap Removed	Specific Conductivity (μ mho-cm ⁻¹)	Chloride Ion (ppm)
. Day 4						
A-8603-2	125	3.3	36	-	-	-
	130	3.7	33	-	1.84	-
A-8603-3	135	3.5	25	-	-	-
	140	3.5	35	100.0	1.41	1.4
A-8603-4	145	3.6	36	-	-	-
	150	3.5	37	-	1.70	-
A-8603-5	155	3.6	39	-	-	-
	160	3.8	40	100.0	1.12	1.7
. Day 5						
A-8605-2	165	3.8	32	-	-	-
	170	3.6	33	-	0.96	-
A-8605-3	175	3.7	33	-	-	-
	180	3.9	32	100.0	0.89	1.4
A-8605-4	185	3.9	32	-	-	-
	190	3.8	32	-	0.90	-
A-8605-5	195	3.9	33	-	-	-
	200	3.9	34	100.0	0.78	2.0

TABLE 10

Characterization of Final Effluent
1000 ppm Soap Based Wash Water

Property	ML-11	Ivory Soap
Total organic carbon (mg/l)	10/11	6/6
Total nitrogen (mg/l)	3.92/3.92	3.92/4.03
Ammonia nitrogen (mg/l)	0.15/0.15	0.73
Organic nitrogen (mg/l)	3.77/3.77	3.19/3.30
Specific conductivity (μ mho-cm ⁻¹)	1.77	2.22
pH	6.3/6.3	-
Turbidity (ppm SiO ₂)	0	0
Color (Pt-Co units)	0	0
Foaming	None	None
Odor	None	None
Total dissolved solids (mg/l)	3.0/2.5	-
Urea (mg/l)	\leq 8.08/8.08	\leq 6.84/7.07
Lactic acid (mg/l)	\leq 25.0/27.5	\leq 15/15
NaCl (mg/l)	< 3.8	< 1.8

IX. MAINTENANCE PROBLEMS

During the program, a series of maintenance problems greatly delayed our pretreatment experiments using the breadboard, requiring that many of the experiments be repeated in order to obtain accurate results. Most of these problems were resolved, however, as discussed below.

Faulty Air Valves

During the filling of the mixing chamber, an air-activated check valve on the "downstream" side of the mixing chamber remains closed to prevent water flow through the remainder of the system (Figure 1, item 6 - page 8 of this report).

During the course of our pretreatment experiments, it was discovered that small quantities of water were bypassing the valve and dripping from the sampling valve (4), just past the filter (9). This was requiring the use of a falsely high dosage of ferric chloride in treating the water in order to obtain efficient soap removal.

The valve was taken apart and the problem traced to the accumulation of soap floc on the valve seat. In the design of a prototype, a more fool-proof type of valve will have to be employed.

During later operation, we routinely watched for any flow from the sampling valve during the fill cycle, which would have indicated further fouling of the valve.

Malfunction of the Centrifugal Pump

During our early pretreatment work we noticed that the delivery pump (5) appeared to be laboring more and more with each series of experiments. Oiling of the pump appeared to help temporarily, but the pump eventually failed. It may be that a heavier duty pump is required for this application, or perhaps a pump that can function better under intermittent operation.

In the meantime, we substituted another centrifugal pump which appeared to work satisfactorily, until near the end of the program. However, during the last week of operation this pump began to labor, and all indications were that it, too, would eventually fail.

Inconsistent Dosage from the Metering Pump

The metering pump being employed for the ferric chloride dosage was a piston-type pump with variable stroke length and speed. During operation the strokes come at uniform intervals, approximately every 5-6 seconds.

In order to calibrate the pump, we disconnected the feed line and collected ferric chloride solution for 5-minute intervals. We then adjusted the metering pump until we obtained the desired dosage rate per minute.

During pretreatment, the metering pump was operated for 1 minute per cycle during filling of the cylinder. After observing problems with inconsistent soap removal results, it was discovered that there was a fairly long delay when the pump was activated before the first stroke was delivered.

Further investigation showed that the flow rate was different for five 1-minute intervals separated by intervals of 1 minute "off", than for a collection period of 5 continuous minutes. In other words, the actual dosage being delivered to the system was substantially less than our 5-minute calibration had indicated.

To avoid any further problems with inconsistent dosage, a peristaltic pump was substituted for the piston metering pump. In addition, the peristaltic pump was connected to a buret, which served as a reservoir for the ferric chloride solution. This allowed us to meter in the solution "by eye" to the required dosage.

Instability of the Ferric Chloride

During the early pretreatment work an inconsistency developed in the soap removal results which was traced to an apparent degradation of the

1 percent ferric chloride solution. This "degradation" showed up as a cloudiness in the otherwise clear solution.

Seal for the Mixer Shaft

During the pretreatment work we had problems with excessive leakage through the mixer shaft seal at the bottom of the cylinder. The seal consisted of a stainless steel bushing coated on the inside with hard rubber. Repeated operation, perhaps aggravated by poor alignment of the mixer and shaft, caused the rubber to wear and the seal to leak.

With excessive leakage through the seal - particularly during the fill cycle - we were in effect getting a larger displacement of water than the target 230 cc; therefore, the ferric chloride dosages tended to be in error.

After replacing the seal twice, we constructed an O-ring type of seal which helped to accommodate slight misalignment of the stirrer shaft. No further problem was encountered, even during one week's continuous operation, with 40 shots per day.

Leakage of Water and Air Past the Piston Seal

To prevent water and air from bypassing the piston in the mixing chamber, we employed a rubber seal which was mounted in and traveled with the piston, and sealed against the cylinder wall.

A two-fold problem developed with the use of this design. Small quantities of water began to bypass the piston and accumulate on the air side of the seal during the fill cycle. This problem appeared to have been caused by both rust and floc particles which had lodged between the seal and the cylinder wall and allowed water to bypass. The problem was solved by replacing the steel shaft above the piston, the source of the rust. The new shaft was of stainless steel. In addition, the piston and cylinder were cleaned thoroughly.

Leakage caused by floc along the seal continued to be a problem. During the five days of continuous operation (40 shots per day) water was allowed to accumulate above the piston so that the amount of leakage could be

measured. After a total of 200 shots, plus approximately 50 additional shots made during operational difficulties, about 90 ml of water had been forced by the piston seal. This represents an average "blow-by" of approximately 0.4 ml per shot.

One possible solution to this problem would be to simplify the mixing chamber. Our demonstration work showed that much of the mixing between the ferric chloride and the soap wash water takes place in the lines before the mixing chamber. Perhaps the cylinder/piston design could be substituted with an enlarged longitudinal cylinder in series, with turbulence promoters mounted in the walls to effect the mixing. A positive displacement piston pump could be substituted for the centrifugal pump now used (Item 5 in Figure 1), to provide the driving pressure necessary to force the water through the cylinder, filter, adsorber columns, and ion exchange bed. Such a design would simplify the entire system and obviate the mixer, piston, and problems of sealing both to the mixing chamber.

The second part of the leakage problem involved air bypassing the seal into the water side of the piston during the pressuring part of the cycle. This problem was probably aggravated by the rust particles, but it may have been caused in part by excessive air pressure. The unit was inadvertently being operated at 25 rather than 5 psig. This problem was eliminated by operating at lower air pressure.

Malfunction of the Mixer Motor

During the pretreatment work the mixer motor (7) for the cylinder malfunctioned. The motor operated, but the stirrer would not turn. The problem was traced to a stripped nylon gear in the gear box of the mixer.

The mixer was replaced by another laboratory stirrer motor.

Check Valve Malfunction

This problem occurred periodically throughout the final five-day evaluation of the breadboard. It involved the fouling of the Nalgene plastic check valves with soap/ferric chloride floc.

Most of the problem came with the check valve between the centrifugal pump and the mixing chamber, so that during the pressurization and discharge of the chamber some of the water was forced back through the lines and the pump. As a result, subsequent shots of water were effectively "overdosed" with ferric chloride.

In order to be able to complete the demonstration, we finally substituted a plastic "stop-cock" type of manual valve in the line. Eventually, however, for any future work, this type of check valve would have to be substituted with a more foolproof design.

Buildup of Soap Floc Throughout the System

Floc accumulation caused some minor problems in the operation of the system, due mainly to buildup in narrow constrictions with a resultant pressure increase. The major problem areas were: the ferric chloride injection valve and gauge assembly (21), the lines leading to and away from the mixing chamber, and the air-operated valve on the "downstream" side of the chamber. Excessive floc buildup occurred primarily with 2500 ppm soap based wash waters because of the greater amount of floc formed during the chemical precipitation.

The problem at the ferric chloride injection port was alleviated by boring out the polyethylene ferrule fitting that joins the plastic tubing to the pipe "T". This provided less restricted flow of the soap solution/floc mixture and less accumulation of floc.

Floc buildup in the lines was generally not serious, however. Only once during the processing of 2500 ppm soap based wash water did the lines have to be removed and cleaned - once the ferrule fitting had been bored out.

It appears that the floc accumulation problem can be solved simply by decreasing flow restrictions in the system. Larger valve ports and wider diameter tubing may eliminate the problem completely.