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POTABLE WATER BACTERICIDE AGENT DEVELOPMENT

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CHEMTRIC Final Report 3097

POTABLE WATER

BACTERICIDE AGENT DEVELOPMENT

(Contract NAS 9-12104)

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FOREWORD

The report summarizes the results of the work performed by CHEMTRIC Incorporated under Contract NAS 9-12104 for the development of a bactericide agent/system concept capable of being used in the Space Shuttle Potable Water System. This program was sponsored by and performed for the Crew Systems Division of the NASA MSC. Mr. A. F. Behrend (EC39) was the designated Technical Monitor.

The work reported herein was started in June 1971 and completed in December 1971. Chief program personnel were Thomas L. Hurley (Project Biochemist) and Robert A. Bambenek (Program Manager). Other personnel that made substantial contributions to this program are: Phillip P. Nuccio (Design Supervisor), Mahendrasinh D. Rana (Design Engineer) and Edward T. Allen (Technician). Mr. Charles Verostko of the NASA MSC provided invaluable assistance by coordinating and supervising well over 1000 individual water analyses.

ABSTRACT

This report summarizes the results of the work performed under Contract NAS 9-12104 for the development and evaluation of a bactericide agent/system concept capable of being used in the Space Shuttle potable water system. The concept selected for evaluation "doses" fuel cell water with silver ions before the water is stored and used, by passing this water through columns packed with silver chloride and silver bromide particles, respectively.

Four simulated Space Shuttle potable water system tests, each of seven days duration, were performed to demonstrate that this concept is capable of delivering sterile water - even though $3 \pm 1 \times 10^9$ Type IIIa or Pseudomonas aeruginosa bacteria, two types which have been found in the Apollo potable water system, are purposely injected into the system each day. This result, coupled with the fact that silver ions do not have to be periodically added to the stored water, indicates that this concept is superior to the chlorine and iodine techniques used on Apollo. These tests also demonstrated that if the Space Shuttle potable water system is constructed from stainless steel, the system will have a useful life substantially longer than five 7-day missions.

A fifth simulated mission test, lasting 10-days, was performed to determine if the silver-ion concept can effectively kill Bacillus subtilis spores. It was found that by heating the silver chloride column to about 120°F as the spore-laden water passed through the column resulted in a death rate greater than a 4-log change in concentration per day.

This work also demonstrated that if the Space Shuttle fuel cell water contains appreciable quantities of contaminants such as ethylene glycol and hydrofluoric acid, the Water Treatment System will have to contain sorbents other than activated carbon, and weak-base ion-exchange resins. Some new design requirements for the Space Shuttle potable water system were also determined from the results of these tests.

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INTRODUCTION & SUMMARY

1.1 Background

The Space Shuttle vehicle is to use hydrogen-oxygen fuel cells to generate electric power, and water for consumption and personal hygiene. In general, fuel cell water is relatively pure because it is synthesized from hydrogen and oxygen and has undergone a phase change from an alkaline electrolyte. However, the experience gained from Project Gemini and Project Apollo indicates that fuel cell water can contain (1) trace contaminants which affect it's taste and odor, and (2) microbial contaminants. In addition, even presterilized water stored onboard a spacecraft is easily contaminated via the crew use points unless the water contains a residual bactericide or is stored at an elevated temperature. Consequently, the Space Shuttle vehicle should be provided with a water treatment system which assures the availability of fuel cell water which is sterile, potable and acceptable to the crew.

The Gemini fuel cell water was not consumed by the crew because it had a low pH, poor taste and poor color. Furthermore, limitations prevented the development of a suitable treatment system. Instead, the crew consumed potable water stored in bladder-type tanks which were also used to accumulate the fuel cell water. The Apollo 11 and later crews have all been supplied with water obtained from fuel cells, located in the Service Module, while the crew occupied the Command Module. However, a different type of fuel cell was selected, and the water was dosed with sodium hypochlorite (chlorine) and sodium dihydrogen phosphate (buffer) to avoid the problems encountered with the Gemini fuel cell water.*

Corrosion, poor taste and free gas problems have been encountered with the Apollo fuel cell water.** The chlorine, which is added to assure sterility, accelerates corrosion. In addition, chlorine must be added once every day during the mission because the chlorine reacts with trace contaminants and the construction materials. A "chlorine" taste has been noted whenever the crew failed to follow precise dosing procedures; in addition, a "nickel" taste, which is attributed to accelerated corrosion of stainless steel in the water heater, has been present.

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*Private communication with personnel at the NASA MSC.

**Samonski, F. H. and Tucker, E. M., "Apollo Experience Report - Command and Service Module Environmental Control System", NASA TN D-6718, March 1972

The Apollo Lunar Module crews have all consumed stored water dosed with iodine. Initially, sodium hypochlorite was tried, but abandoned when it was found that chlorine hydrate accumulated on the sublimators which also use the stored water.* It was also discovered that iodine diffuses through the silicone-rubber bladders in the tanks and reacts with the anodized aluminum tank wall. Fortunately, the iodine depletion and corrosion rates are slow, so that in-flight maintenance and failures can be avoided by not exposing the tanks to iodine until just before the vehicle is launched.

All of the water management problems encountered by Project Gemini and Project Apollo indicate that a more durable system should be provided for future spacecraft - and especially for the reusable Space Shuttle. Also, a residual bactericide, which can be passively added to the fuel cell water as the water is produced, should be used instead of chlorine or iodine. In addition, the water quality should be improved to avoid any noxious tastes and/or odors which can be introduced by the fuel cell materials - especially when the fuel cells are operated at any off-design conditions.

Prior to the receipt of Contract NAS 9-12104, CHEMTRIC had evaluated the use of silver ions for sterilizing water distilled from urine, treated flush water and concentrated wash water.**, *** In all cases, condensate passed through a column containing silver chloride particles was found to be sterile, for months - even though the condensate contained some organic contaminants and it was exposed to the laboratory atmosphere. In addition, no corrosion problems were observed in stainless steel hardware which had been exposed to silver-saturated water for up to four years. Thus, silver ions were selected as the residual bactericide to be evaluated under this program for use in the Space Shuttle Water Treatment System.

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*Gillen, R. J., Brady, J. C. and Collier, F., "Apollo Experience Report - Lunar Module Environmental Control Subsystem", NASA TN D-6724, March 1972.

**Nuccio, P. P. et. al., "Refurbishment and Testing of the Integrated Waste Management System", AMGLO Report 3080, Chicago Illinois, October 1969.

***Bambenek, R. A., et. al., "Upgrading and Extended Testing of the MSC Integrated Water and Waste Hardware", CHEMTRIC report 3084, Rosemont, Illinois, May 1972.

1.2 Objectives

The detail objectives of this program are delineated by the following Task Descriptions, as defined in Section 3.2 of the Statement of Work for Contract NAS 9-12104.

Par 3.2.1 Bactericide Agent Selection

Many bactericide agents have been utilized in the past; including halogen base chemicals, silver ions, heat, ultraviolet radiation and ozone. No restrictions are being placed upon the type of bactericide agent selected as long as it meets the following criteria:

a. It will kill or positively entrap bacteria of the types normally encountered in spacecraft water systems.

b. It must not be harmful to the human body in concentrations required to insure 100% kill of bacteria.

c. It can be used in a manner consistent with the flight safety requirements and mission objectives of the Space Shuttle System.

d. It will not affect or degrade the operation of any other spacecraft system (EMI, fumes, etc.).

Par 3.2.2 Materials Compatibility

Since the ultimate objective of this effort is to provide the Shuttle contractor with design data required to develop a total spacecraft water system, it is imperative that the total effect of the bactericide agent/system be known. A study to determine the system's materials compatibility must include at least the following:

a. Select an acceptable flushing fluid and drying gas that are compatible with the bactericide agent/system. Define which metals and non-metals are compatible with the agent/system and which are not, and perform necessary tests to verify these selections.

b. Determine the extent/severity of adverse reactions to any material with which the bactericide agent could conceivably come in contact, particularly concerningits toxicity, flammability and corrosiveness.

c. Determine any unique effects the bactericide agent/system may have on any candidate material such as permeability, galvanic reactions, crevice corrosion, etc. This should include any temperature and flow rate effects.

Par 3.2.3 Breadboard Testing

A breadboard unit shall be assembled to demonstrate that the concept selected performs as expected. The unit will be rigorously tested to determine its performance capability. Testing will include extended exposure to pure water, contaminated water, water system materials, proposed flushing agent(s) and similar key parameters.

Similarly, the Design Considerations are defined as follows.

Par 3.3.1 Configuration

The configuration of the bactericide agent/system should be such that the following Space Shuttle constraints are taken into account:

a. Low weight and volume
b. Minimal power requirements
c. Ease in maintenance
d. High reusability rate (i.e.,long life time)
e. Low cost (Both initial and operational costs)
f. Zero G and $\pm$ 6 G (Any direction) application

The system configuration also should be such that it complies with the present Shuttle potable water system concept.

Par 3.3.2 Application Techniques

During Space Shuttle flights, the bactericide agent must be applied/utilized in a fully automatic manner requiring no flight-crew participation in either normal or off-design operation. The flight unit must fail operational - fail safe. In addition, no viable organisms from the water system must be present when sampled at the crew/passenger use point. These objectives should be kept in mind during the tasks outlined in this Statement of Work.

Par 3.3.3 System Specifications

The Space Shuttle water system has characteristics as follows:

1.	Water Supply	From Fuel Cells
2.	Supply Flow Rate	8.7 lb/hr.(max)@10KW
3.	Fuel Cell Exit Temp.	150 to 175 °F
4.	System Pressure	15 to 60 PSIA
5.	Known Contaminants	6.5 to 7.5 pH
6.	Water Delivery Flowrate	Up to 60 lb/hr
7.	Water Delivery Temp.	
	Hot	150 to 160 °F
	Cold	40 to 60 °F
8.	Water Delivery Pressure	30 PSID @50 lb/hr

Finally, after receipt of the contract, it was decided that the breadboard system should be designed to provide 63 liters (138.6 lbs) of potable water each day for seven days - when treating fuel cell water which has the composition and characteristics defined in Appendix A. This "worst case" water was selected because the exact properties of the Space Shuttle fuel cell water will not be known until the first flight is completed - and because it is less costly to fly an overdesigned system than one which jeopardizes crew safety. In addition, it was agreed that (1) the water treatment system will be located a sufficient distance away from the fuel cells so that the water will be at cabin temperature when treated, and (2) to achieve the desired total flow, the peak input rate of 8.7 lb/hr would exist for 8 hours while the input rate during the other 16 hours of each day would be 4.35 lb/hr. Since "real", worst-case, fuel cell water could not be obtained for this program, it was also agreed that simulated fuel cell water would be used for the breadboard tests.

1.3 Accomplishments

The breadboard system assembled under Contract NAS 9-12104, to evaluate the use of silver ions in the Space Shuttle potable water system, is described in Figures 2 and 3 on pages 2-17 and 2-19, respectively. As shown in these figures, the system includes (1) a biological filter for removing particulates, (2) an activated carbon filter for adsorbing organic contaminants, (3) a canister of silver chloride particles for dosing the water to be stored with silver ions, (4) water storage and material immersion tanks, (5) a deionizer for removing silver ions and inorganic contaminants, and (6) a canister of silver bromide particles for dosing the product water with a smaller quantity of silver ions. Support equipment included (1) holding tanks for simulated fuel cell water and product water, (2) a feed pump, and (3) miscellaneous items such as valves, gages and septums.

The simulated mission tests performed with the breadboard system indicate that the silver ions concept is capable of being used in the Space Shuttle potable water system. The specific accomplishments made are listed as follows - according to potential problem areas, to justify the recommendations listed in Section 1.4. The pages listed in paranthesis indicate the location of detailed information.

1.3.1 Water Sterility

The work performed on water steriliy demonstrated that:

A. Silver ions, which are passively added to simulated fuel cell water, are capable of maintaining a breadboard water system sterile for seven days - even though $3 \pm 1 \times 10^9$ Type IIIa or Pseudomonas aeruginosa bacteria, which have been previously found in the Apollo water system, are injected into the system each day at locations where silver ions are present (pp 4-1 to 4-7).

B. Silver ions are effective in killing Type IIIa and P. aeruginosa bacteria if the gas used to pressurize the water system is either oxygen or nitrogen (pp 4-1 to 4-7).

C. If 0.04 to 0.12 ppm of silver ions are present in the water system, Type IIIa and P. aeruginosa bacteria will experience a death rate greater than a 6-log change ($> 10^4$/ml to $< 10^{-2}$/ml) in periods as short as 15 minutes (pp 4-4 and 4-7).

D. If $3 \pm 1 \times 10^9$ Type IIIa or P. aeruginosa bacteria are injected into stagnant water at a location where silver ions have been removed from the water, samples of this water drawn 23 hours later through a column containing silver bromide particles will contain up to 200 bacteria per ml (pp 4-1 and 4-7).

E. Water drawn from an infrequently used, remote outlet valve is easily contaminated by microbes from an external source (pp 4-1 and 4-7).

F. The simulated fuel cell water has a negligible effect on three types of microbes, even though the water contains lead, copper, chromate and organics (pp 4-4 and 4-9).

G. A biological filter containing silver chloride particles has a useful life of at least ten days when the water being filtered contains $3 \pm 1 \times 10^4$ Bacillus subtilis spores per ml (p 4-10).

H. Silver ion concentrations ranging from 0.04 to 0.12 ppm are not capable of killing B. subtilis spores (p 4-8).

I. A silver ion concentration of 1.21 ppm and a temperature of 118 - 122°F are required to achieve a 4-log reduction of B. subtilis spores in 21 hours (p 4-10).

1.3.2 Water Quality

Experimental evaluations of the activated carbon filter and deionizer, using simulated "worst-case"fuel cell water, demonstrated that:

A. The product water will contain an excessive amount of organic carbon if the fuel cell water contains an excessive amount of ethylene glycol because activated carbon cannot effectively adsorb highly soluble contaminants like ethylene glycol (p 4-11).

B. Activated carbon must be extensively "washed" during preparation to avoid increasing the alkalinity of the filtered water (p-411).

C. If the fuel cell water contains chlorides, the amount of silver added to the water by silver chloride or silver bromide particles is suppressed by the common-ion effect (pp 4-10 and 4-11).

D. If the concentration of silver ions is suppressed by the common-ion effect, the desired level can be easily achieved by raising the temperature of the silver salt column (p 4-9).

E. If the deionizer resins are not washed properly before being installed in the system, amines which evolve during the presterilization process will cause the silver bromide column to add an excessive amount of silver to the product water (p 4-12).

F. If weak-base resins are used in the deionizer, and the fuel cell water contains hydrofluoric acid, the product water will have an excessive fluoride content and low pH (p 4-11).

1.3.3 Materials Compatibility

Candidate metals and elastomers were soaked in filtered simulant and pressurized by nitrogen and oxygen gas, to determine by inspection and weight change if these materials are affected by this exposure. The custom-designed components were pressure tested after each simulated mission test to demonstrate that their construction materials are acceptable. This work demonstrated that:

A. Type 410 stainless steel (SS) experiences less weight gain than Grade A-55 titanium when soaked in filtered simulant (p 4-14).

B. Both Type 410 SS and A-55 titanium experience less weight gain when the filtered simulant is pressurized by nitrogen instead of oxygen (p 4-14).

C Polyisoprene, silicone and EPT (Ethyl Propylene Terpolymer) experience less weight change than Buna-N, Butyl and Viton when exposed to filtered simulant or deionized water (p 4-14).

D. Filtered simulant, pressurized by nitrogen or oxygen, does not have any significantly larger effect on elastomers than does deionized water exposed to the atmosphere (p 4-14).

E. The only material found to be obviously unacceptable is a cast aluminum alloy, which was used in fabricating part of a filter housing (p 4-12).

F. Castings of Type 304 SS may be unacceptable, because parts made from this material did develop "rust" spots (p 4-12).

1.3.4 Component Compatibility

All of the components used in the Breadboard System, except a biological filter, proved to be reliable, have sufficiently low resistance to flow, and have the desired useful life. The small biological filter initially used had a pressure drop near 200 mm Hg (3.9 psi) after seven days of use at the fuel cell water flow anticipated for the Space Shuttle (8.7 lb/hr). A larger filter had a pressure drop of only 60 mm Hg (1.2 psi) after ten days at the same conditions. Since particulates were observed to have "settled-out" in both filter housings, larger pressure drops would have been experienced if the testing had been performed under weightless conditions.

1.4 Recommendations

This report shows that (1) silver ions are extremely effective in killing two types of bacteria previously found in the Apollo water systems, (2) silver ions can be passively added to water, (3) stored water does not have to be periodically dosed with silver ions, and (4) the use of silver ions and the components required do not present any corrosion problems with austenitic stainless steel. Thus, the bactericide concept selected for evaluation during this program avoids the sterility, maintenance and corrosion problems encountered with chlorine and/or iodine in the Apollo Potable Water System. In addition, this report shows that martensitic stainless steel and A-55 titanium, which have more "specific stiffness" than austenitic stainless steel, would also be acceptable materials for constructing the

Space Shuttle Potable Water System. Consequently, it is recommended that the NASA continue the development of this concept for the Space Shuttle.

The next logical step in the development of this concept is to abandon the Breadboard System - and design, fabricate and evaluate a preliminary flight-prototype model of a Water Treatment System. This step is strongly recommended because (1) the Space Shuttle Potable Water System must be reusable, and (2) the Space Shuttle launch environment may have an effect upon the performance of the granular material required in the respective canisters. Thus, if a preliminary flight-prototype model of the Water Treatment System is constructed it can be used to determine the exact procedure required to "recharge" the canisters between each mission. In addition, if this model is subjected to the vibration environment anticipated for the Space Shuttle, subsequent simulated mission tests will indicate any degradation in performance due to granule attrition and/or rupture. Fortunately, the activated carbon filter, silver chloride column, deionizer and silver bromide column canisters developed for the program reported herein are preliminary flight prototype designs - and can be used "as is" in the recommended model. Other details which should be considered in the design of this model are:

A. The outlet of the deionizer should be welded to the inlet of the AgBr Column to positively avoid contaminating the relatively silver-free water between these two components.

B. The capability for periodic heating of the water outlets should be provided to assist low concentrations of silver ($\sim$ 75 ppb) in killing spores, especially if the outlets are remotely located and infrequently used.

C. If the fuel cell water contains more than 0.1 ppm of chloride ion, and the Water Treatment System must be capable of killing B. subtilis spores, the silver chloride column must be heated - or part of the activated carbon replaced by ion-exchange resins.

D. If the Space Shuttle fuel cells are designed so that ethylene glycol or any other highly soluble organics can contaminate the product water, part or all of the activated carbon in the Water Treatment System will have to be replaced by another adsorbent such as activated alumina or synthetic zeolites.

E. If the Space Shuttle fuel cell water contains low atomic weight inorganics such as hydrofluoric acid, the deionizer must contain strong-base resins to assure a product water pH in the range of 6 to 8.

F. A large biological filter cartridge should be provided to avoid excessive resistance to flow, and the cartridge housing should not include any cast stainless steel parts. Also, provisions should be made for by-passing or back-flushing the filter if the fuel cell water contains an appreciable quantity of particulates.

G. To avoid material failures all components containing the fuel cell water should be constructed from A-55 titanium, martensitic stainless steel or austenitic stainless steel. Elastomers such as polyisoprene, silicone and EPT are preferred over Buna-N, Butyl and Viton.

Finally, the recommended Water Treatment System will provide extremely pure water - which, of course, is essentially "tasteless". To avoid any in-flight acceptability problems the system should also include a "mineralizer" - that is, a device which doses the water with the same types of minerals found in domestic water supplies.

SECTION 2

BREADBOARD SYSTEM DEFINITION

The justification and design of the breadboard water treatment system are summarized in the following subsections.

2.1 Concept Selection

As mentioned in Section 1.1, the bactericide proposed and approved for evaluation under this program was silver ions - which are added to the fuel cell water by passing it through a column containing silver chloride particles. Silver dosing has been used to control bacteria in aqueous systems since late in the 19th century. Cliver* recently demonstrated that a 250 ppb dose of silver ions is capable of killing all of the microbial species found so far in spacecraft water systems - but not spores nor all viruses; he also demonstrated that the kill rate is a strong function of silver ion concentration.

The use of silver chloride particles to dose water with silver ions was developed by Melpar Incorporated under Contracts NAS 9-3565 and NAS 9-5119 - and evaluated by CHEMTRIC personnel under Contracts NAS 9-9014 and NAS 9-9191. An electrolytic device for dosing water with silver ions was developed by the AiResearch Manufacturing Company under Contract NAS9-3541 - and used by Cliver and Putnam.** Since a column of silver chloride particles is inherently more reliable than an electrolytic generator, and since Cliver and Putnam both had problems with the AiResearch device, the silver chloride approach was selected for this program.

When water is passed through a column of silver chloride particles, the silver chloride dissolves into the water as silver and chloride ions. However, like common table salt (NaCl), this process continues only until the water is "saturated" with silver and chloride ions. If there are no other interferring ions present, the concentration of these ions at saturation is defined as:

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* Cliver, D. O. et. al., "Biocidal Effects of Silver", Final Report on Contract NAS 9-9300, U. Wisconsin, February 1971 (N71-24436).

** Putnam, D. F. et. al., "Water Management Results for a 90-day Space Station Simulator", ASME Publication 71-Av-6, Presented at the SAE/ASME/AIAA LS and EC Conference in July 1971.

$$C_{Ag^+} \times C_{Cl^-} = K_{sp}$$

where C denotes ion concentration
K_{sp} is the solubility product constant

Since the ion concentrations are equal, this expression can also be written as:

$$C_{Ag^+} = \sqrt{K_{sp}}$$

At 25°C the reported K_{sp} for AgCl in water is 1.56×10^{-10} gram molecular concentrations per liter. (e.g., see page 1117 of the tenth edition of the Handbook of Chemistry by N. A. Lang). Therefore, under these conditions, the concentration of silver ions should be:

$$C_{Ag^+ @ 25^oC} = \sqrt{1.56 \times 10^{-10}} = 1.25 \times 10^{-5} \text{ moles/liter}$$

or

$$1.25 \times 10^{-5} \text{ moles/liter} \times 107.87 \text{ grams/mole} = 1.35 \times 10^{-3} \text{g/l}$$

Under Contracts NAS 9-9014 and NAS 9-9101, CHEMTRIC personnel demonstrated that this concentration is easily achieved in practice - using water distilled from pretreated urine and urinal flush water. Thus, the AgCl column approach is capable of providing larger concentrations of silver ions than used by Cliver to demonstrate that silver ions are capable of killing the types of bacteria found so far in spacecraft water systems.

One disadvantage of the silver chloride technique is the fact that the resultant dose level exceeds the maximum allowable concentration for silver in potable water. The U. S. Public Health Service and MSC Spec-C35 both agree that potable water should not contain more than 50 ppb of silver. Thus, the Water Treatment System must also contain a deionizer to remove the excess silver before the water is consumed. However, this may not be a disadvantage if other inorganic constituents must be removed to assure the availability of potable and acceptable water.

With a deionizer in the system, the water located between the deionizer and the outlets (use points) is highly susceptible to biological contamination - because a well designed deionizer will have essentially removed all of the silver ions from this water. Initially it was planned to "protect" this water by periodically heating this section of the water system. However, since the effect of time and temperature on microbial population is well known, it was finally decided to also evaluate the use of silver ions in this part of the system - but at concentrations near the acceptable limit specified by the U. S. Public Health Service and MSC Spec-C35.

The technique selected for dosing the water downstream of the deionizer with silver ions is "passing the water through a column packed with silver bromide (AgBr) particles". Since silver bromide is less soluble than silver chloride in water, this technique should yield lower concentrations of silver ions. The following table of published data* shows that the resultant silver ion dose should be near 50 ppb when the water is at normal room temperature.

Temperature	AgBr Concentration	Ag^+ Dose
15°C	0.000072 g/l	0.042 ppm
20	0.000097	0.056
25	0.000135	0.078
30	0.000180	0.103
50	0.000477	0.274
100	0.003700	2.130

The "worst-case" fuel cell water defined in Appendix A contains 100 ppm of total organics (TO). If these contaminants are all CH_2 groups, the most common group in organic molecules, the Chemical Oxygen Demand (COD) of this water would be 340 ppm. However, experience in analyzing recovered water indicates that a TO of 100 ppm corresponds to a COD of approximately 250 ppm. In either case, the COD of the fuel cell water is substantially higher than the 100 ppm limit established by the National Academy of Sciences Ad Hoc Committee on Water Quality; therefore, the location of this filter should be upstream of the silver chloride column - to minimize any effects of the organics on the solubility of silver chloride, and to avoid storing water which contains a substantial amount of nutrients for bacteria.

Previous work** has shown that activated carbon filters have a useful life of (1) less than 3 days if the input is biologically contaminated, (2) about ten days if the input is passed through a biological filter, and (3) more than 30 days if the biological filter contains silver chloride particles. Since the "worst-case" fuel cell water will be biologically contaminated, and the Space Shuttle mission duration will probably be at least seven days, it was decided that a biological filter containing silver chloride particles should be located upstream of the activated carbon filter. In this location, a biological filter also serves to remove most of the particulates assumed to be present in the "worst-case" fuel cell water.

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* Linke, W. F., SOLUBILITIES, Vol. 1, 4th Edition, American Chemical Society, Washington, D. C. 1958

** Ibid Bambenek, R. A., et. al.

In summary, an analysis of the requirements for the Space Shuttle water system and the possible contaminants in the fuel cell water indicated that a Water Treatment System containing the following components, in the order listed, should be evaluated during this program.

A. Biological Filter (containing AgCl particles)
B. Activated Carbon Filter
C. Silver Chloride Column
D. Water Storage Tank(s)
E. Deionizer
F. Silver Bromide Column

2.2 Component Design

All of the aforementioned components, except the biological filter and the storage tanks, were custom-designed. The following paragraphs describe these designs, and the calculations performed to estimate the required size of each canister or column.

The custom-designed components differ from each other only in size and contents. The same canister design was used for each component. Figure 1 contains a photograph of the three different sizes of canisters; the silver chloride canister (identical to the silver bromide canister) is shown disassembled to reveal the internal parts arrangement.

Each canister contains the following, in sequence from the outlet (back end) to the inlet:

A. A perforated metal disc welded to the end cap which in turn is welded to the cylindrical body.

B. Four discs of 10 mesh, 316 SS screen followed by a layer of pyrex wool 1/8 to 1/4 inches thick and a single disc of the same screen. This combination of screens and pyrex wool retains the particulate contents of the canister and provides a lower pressure drop than pyrex wool alone.

C. The media (charcoal, ion exchange resins, etc.) is packed into the canister on top of the above items.

D. The inlet particle retainer is similar to B above, but the order is reversed - that is, a screen disc is placed on top of the media followed by pyrex wool and 4 screen discs.

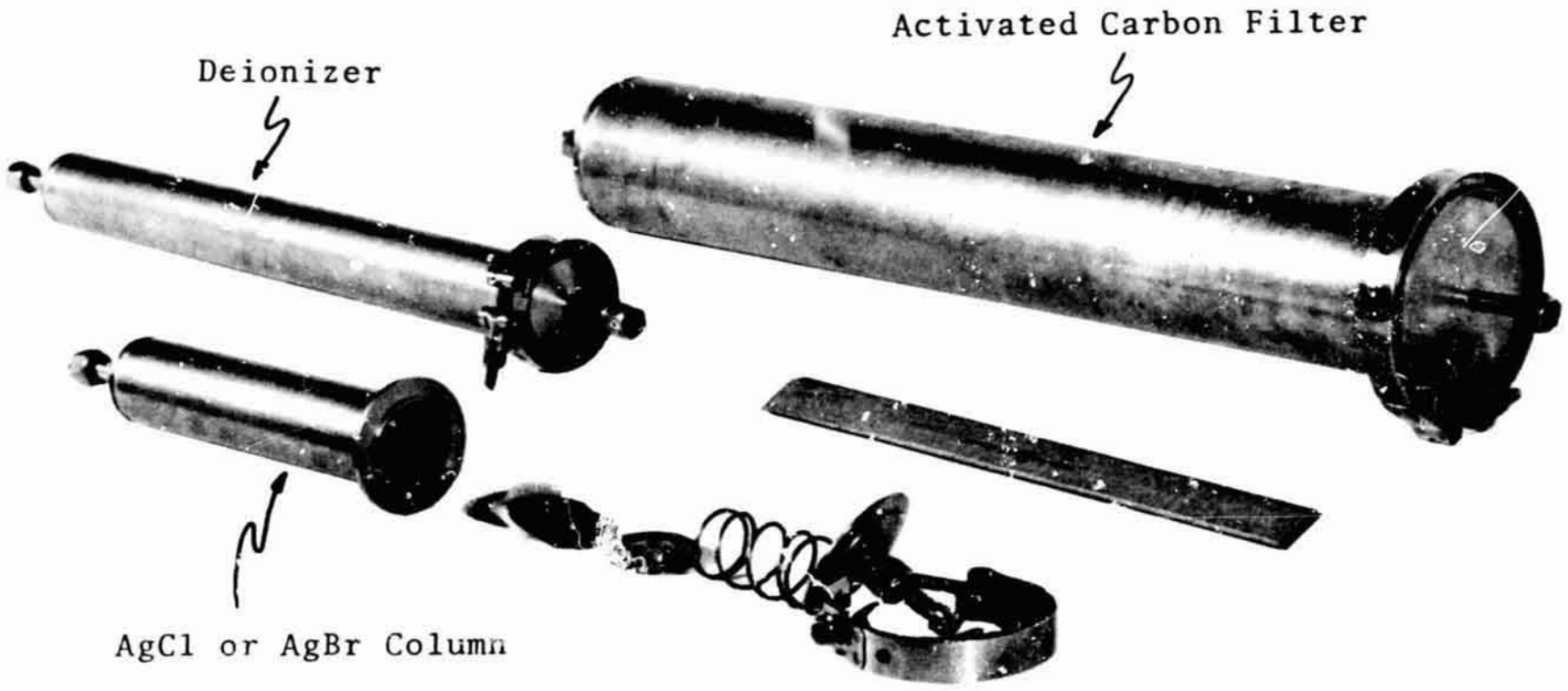

Figure 1 PHOTOGRAPH OF CUSTOM-DESIGNED COMPONENTS

E. A stainless steel, doughnut-shaped disc backs up the above items. The disc provides an "O" ring seal between the disc periphery and the cylinder ID. The center of the ring is covered with a perforated metal disc, tack welded in place. A ridge at the periphery of the center hole serves to locate the media compression spring.

F. A spring fabricated from 18-8 SS is trapped between the above disc and the removable end cap. The spring provides a compressive load on the media to retard media particle movement which could result in particle disintegration and/or channeling.

G. The end cap provides for centering the spring and an "O" ring seal between the end cap and the flange welded to the canister body. The end cap is held in place by a "V" band clamp.

The design effort on the canisters emphasized control of potential corrosion sites. Tubing nipples welded from the inside of the end cap were used to eliminate threaded connectors on the canister body. The fixed end cap was butt welded as opposed to a lap-type union. The canisters were heat treated and annealed to minimize the potential for intergranular corrosion. Finally, all canister parts were passivated. Subsequent cleaning and decontamination procedures were executed, without the use of abrasives, to preserve the integrity of the passivated surfaces.

Although a reliability study was not performed, related CHEMTRIC efforts on the development of the water and waste management system for a modular Space Station* provided direction in the subject design effort. The failure modes of the canisters are (1) external leakage, (2) channeling, and (3) clogging. The probability for external leakage is reduced through the use of welded connections and a single removable seal of the "O" ring variety. Corrosion induced leakage is minimized through material selection, elimination of crevices, heat treatment and passivation. The media compression spring retards bed shifting which can promote channeling; the media restraint also reduces the probability of particle disintegration which if unchecked could lead to off-quality water and clogging of the outlet media retainer. The combination of perforated metal discs, screens

*Subcontract SS-863762-KO with the Hamilton Standard Division of the United Aircraft Company, under Contract NAS 9-10273.

and pyrex wool as media retainers provides a "depth" type filtration capability; this arrangement is less prone to clogging than a "membrane filter" type retainer.

A safety hazards analysis was not conducted but safety goals were considered in the design activity. In regard to the canisters, the only safety element identifiable at this time is the spring loaded end cap. Once the "V" band clamp is removed, the media compression spring can propel the end cap outward. However, sterility requirements dictate no in-flight maintenance, especially at the component repair level. The design of the flight system should provide positive means to prevent canister disassembly.

2.2.1 Biological Filter (BF)

This component consists of a filtering element or cartridge, and a housing. Two filter sizes were evaluated; both were manufactured by the Pall Trinity Micro Corporation (PTM) of Cortland, New York.

The first BF evaluated was the PTM "Junior Size". The filter element (P/N MCS4463UW) in this unit is 5.3 inches long, and has an outside diameter (OD) of 2.2 inches; it is a pleated, membrane-type filter which has a filter area of 1.1 ft^2. This element is rated for absolute retention of all particles 0.35 microns and larger, and 98% of all particles as small as 0.15 microns. The filter media is a proprietary blend of a cellulose base material, inorganic fibers and epoxy resins; type 304 SS is used to fabricate the end caps and the perforated internal support tube. The housing (P/N MDE4463G4) for the filter element has an OD of 2-7/8 inches, and an overall length of 7-5/8 inches; it is fabricated from an aluminum alloy and coated with Teflon.

The second BF evaluated is a larger PTM unit. The cartridge in this unit, which is identified as P/N MCY1001UR, has an OD of 2-3/4 inches, and a length of 9-13/16 inches; the filtration characteristics of this cartridge are equal to those described above for the smaller cartridge - except, it has a filter area of 2.2 ft^2 instead of only 1.1 ft^2. The filter media appears to be the same as above - however, the support tube and end caps are fabricated from polypropylene instead of stainless steel. The housing (P/N MCS1001G16), which is fabricated from type 304 SS, has an OD of 4 inches and a length of 14 inches.

The "dirt" capacity of the two BF cartridges, as reported by the manufacturer, are 20 and 43 grams, respectively.* The anticipated dirt load was 100 mg/liter (suspended solids in the simulant) x 63 liters/day x 7 days (mission duration), or 44.1 grams. Therefore, the larger BF is the one that appears to be required. However, the smaller BF was tried initially because it has substantially less weight and volume than the larger unit - and the maximum water flow rate (66 cc/min or 8.7 lb/hr) is extremely low for either one of these filters.

The following table lists various characteristics of each of two filters evaluated.

Filter Characteristics	Junior Filter	Standard Filter
Mass of Complete, Water-Filled Assembly - kg.	1.17	5.46
Mass of Complete, Drained Assembly - kg.	0.95	4.13
Dry Mass of Cartridge Without AgCl-Glass Bead Mixture - kg.	0.12	0.31
Wet Mass of AgCl-Glass Bead Mixture - kg.	0.42	1.17
Volume - liters.	0.84	2.86

2.2.2 Activated Carbon Filter (ACF)

As mentioned earlier, the canisters differ only in contents and size. The methodology for determining the required activated carbon canister dimensions and the spring characteristics are discussed in the following paragraphs.

Sizing of the ACF was based primarily on prior experience with water recovery systems. Much of this experience has been with a single type of charcoal - that is, Barnaby-Cheney type 365, 20 x 50 mesh.

The sizing methodology consists of (1) estimation of the chemical oxygen demand (COD) of the water to be purified, (2) estimation of the probable COD removal capacity considering the nature of the contaminants and the ratio of influent COD to effluent COD required, (3) estimation of the charcoal quantity required using the estimates generated in 1 and 2 above,

*Determined on the basis of MIL-F-25682 (USAF).

and (4) determining the canister dimensions from the as-packed charcoal density and a target length to diameter ratio of 8 (this ratio is selected to minimize the probability for channeling).

The above technique is at best semi-quantitative. The best method for charcoal canister design would be to rely on data from bench-scale evaluations of charcoal capacity with the water to be purified. In addition, detailed knowledge of the organic contaminants should be available. With such information, an optimized rather than a conservatively designed canister could be realized.

The organic constituent of the simulant was not specified other than to identify the concentration as 100 mg/l. Lacking information relative to the identity of the organic contaminant, the COD was estimated to be 250 mg/l.

The water requirement was established at 63 liters/day - and seven days was established as the probable Shuttle mission duration. Adding one day as a contingency margin, the total quantity of water to be processed during each mission is 63 x 8, or 504 liters. The total COD involved would be 126 grams (0.278 lbs).

A firm potability standard for organic contamination has not been established by agencies normally active in such areas. The Ad Hoc Committee for Water Standards of the National Academy of Science has recommended a value equivalent to 100 ppm COD. Accepting this standard would require the charcoal bed to remove 60% (250 mg/l influent and 100 mg/l effluent) of the incident COD throughout the test period.

The removal capacity of a column is a strong function of the target COD delta. Based on prior experience, the 60% delta requirement is not severe nor is the relative concentration level; based on these considerations a COD removal capacity of 0.09 pounds COD per pound of charcoal should be attainable.

The charcoal quantity estimated on the basis of the above quantities would be 0.278 lbs (total COD) ÷ 0.09 lbs COD/lb charcoal, or 3.09 lbs (1406 grams). Based on the as-packed density of 29.8 lb/ft^3, the volume of charcoal would be 0.104 ft^3 (2.95 liters, or 180 in^3).

The required dimensions of the charcoal portion of the columnar bed were determined from the target length to diameter ratio of 8, and the estimated charcoal volume - namely:

Volume = 180 in^3 = $\pi D^2 L/4$

Substituting L = 8D

Volume = 180 in^3 = $2\pi D^3$

Diameter = $\sqrt[3]{180/2\pi}$ = 3.06 inches

Length = 8 x 3.06 = 24.5 inches

The tubing obtained for the charcoal canister has an inside diameter (ID) of 3.37 inches; it was fabricated from 316 SS. The length of this tubing needed to accommodate the 180 in^3 of charcoal is 20.2 inches; these dimensions provide an L/D value of 6. The only alternative was a similar grade of tubing but with an ID of 2.87 inches; the length would be 27.8 inches yielding an L/D value of 9.7. Either of the two tubing sizes entailed a significant departure from the target L/D value of 8. The 3.37 inch ID tubing was selected since the L/D value provided by this tubing was consistent with the "worst case" concept.

The ACF should be designed to withstand a proof pressure twice the operating maximum, and have a burst pressure at least four times the operating maximum. Based on standard hoop stress equations and the published yield and ultimate strength data for type 316 SS, the maximum wall thickness required is, therefore, 0.004 inches. Obviously, a wall thickness greater than 0.004 inches would be required when dent and puncture resistance along with structural requirements are considered. On the other hand, the .065 inch wall provided by the selected tubing is probably greater than needed to meet reasonable requirements for dent resistance and structural support.

The overall length of the canister excluding the tubing connector is 22-3/8 inches. The OD at the "V" band clamp is 5-3/4 inches.

The data necessary for design of the spring was obtained from the Pittsburgh Activated Carbon Company. In tests conducted by the above organization, a 60 pound compressive load was applied to a 5-inch diameter charcoal bed which was subjected to vibration tests. In these tests, 15 g's at 30 Hz for 2 hours and 3 g's at the same frequency for 24 hours did not affect the integrity of the charcoal granules.

As recommended, the compressive load was scaled down proportionately to the diameter of the activated charcoal column. With the 3.37-inch diameter, a compressive load of 28 lbs would be required.

To permit liberal tolerances for the actual height of the charcoal mass within the canister a low rate spring was selected. The selected spring provided 7.8 lbs per inch of length from 3.589 to 0.892 inches, at which the spring is squeezed down to 1.125 inches. The spring was fabricated from 0.162 inch type 18-8 SS wire. Other characteristics are as follows.

Mean Coil Diameter	= 3.000 in.
Number of Active Coils	= 4
Total Number of Coils	= 5-1/2
Free Length	= 4.725 in. (± 10%)

The following table presents the mass and volume requirements for the activated charcoal canister. Included in the table is the mass of the canister with Pittsburgh Activated Carbon Type CAL 12 x 20 mesh activated charcoal; this type charcoal was used in the last simulated mission test. Barnaby-Cheney Type 365, 20 x 50 mesh charcoal was used in the first four tests.

Canister Characteristics	Barnaby-Cheney Charcoal	Pittsburgh Act.Carbon Charcoal
Mass of Complete, Water-Filled Assembly - kg.	6.79	6.83
Mass of Dry Canister Without Charcoal - kg.	3.18	3.18
Mass of Dry Charcoal - kg.	1.41	1.59
Volume - liters.	3.53	3.53

2.2.3 Deionizer Column

Deionizer sizing is based on the total milliequivalent (MEQ) load to be removed, and the exchange capacity of the ion-exchange resins to be used. The total MEQ load is calculated from the simulant composition and total water quantity to be processed, and the equilibrium resin capacity furnished by the resin manufacturer.

The cationic load provided by the simulant is 0.4 MEQ per liter; 63 liters are processed per day for seven days. Adding an additional day as a contingency margin, the simulant cationic load becomes 0.4 MEQ/liter x 63 liters/day x 8 days or 202 MEQ. Assuming a "worst case" silver dose of 2 ppm (0.019 MEQ/liter), the silver ion load for the 8 days amounts to 9.6 MEQ. The total cationic load becomes 212 MEQ. The anionic load is, of course, identical to the cationic load.

The resins selected are manufactured by Rohm & Haas. The cationic resin is type IR-120, 20 x 50 mesh with a wet exchange capacity of 1.9 MEQ per ml of resin; it is a strong acid-type resin. The anionic resin is type IR-45, 20 x 50 mesh with a wet exchange capacity of 2.0 MEQ per ml of resin; it is a weak base-type resin. The cationic resin is in the hydrogen form while the anionic is in the hydroxide form.

The major feature of the above resins is their high temperature stability. The cationic resin is rated for up to 250°F while the anionic is rated for 212°F. These temperatures permit decontamination of the resins without serious resin degradation.

The cationic resin requirement is 212 MEQ/1.9 MEQ/ml or 111.6 ml; the anionic requirement is 212 MEQ/2.0 MEQ/ml or 106 ml of resin. However, these quantities of resin are based on exchange capacity under equilibrium conditions. Columnar operation does not permit attainment of equilibrium while providing high quality effluent. In addition, the large variety of ionic species in the simulant introduces a large number of potential resin-ionic species equilibria; each equilibria will be different with respect to exchange rate.

The method commonly used to deal with the above problems is to derate equilibrium capacity in rough proportion to the desired ratio of effluent and influent electrolyte concentrations.* For applications requiring maximum purity, the calculated resin requirement would be increased four-fold while for less exacting applications the resin requirement would be increased only slightly beyond that dictated by capacity data.

Potability standards require nearly complete removal of most of the heavy metals but permit liberal concentrations of common electrolyte salts such as sodium and potassium chlorides and sulfates. Deionizer columns using the resin types mentioned above were previously used by CHEMTRIC under Contract NAS 9-9191; these columns were sized on the basis of 3 to 3.5 derating factors. The results of the tests indicate that the selected resins have a marked affinity for heavy metals, most notably silver. In light of this performance and the permissable levels of common electrolytes, a derating factor of two was selected.

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*Kunin, Robert, Ion Exchange Resins, 2nd Edition, John Wiley & Sons, New York, New York, 1958, pp. 114 to 118.

The resin requirement becomes 111.6 ml x 2 or 223.2 ml cationic and 222 ml anionic. For sizing the column, the total resin volume was rounded off to 450 ml.

Current commercial practice as well as previous experience suggests an L/D value of 5 to 7 for the deionizer. Column diameters were calculated in a manner identical to that used for charcoal canister sizing; the pertinent values in the calculation are 450 ml (27.5 in^3) total volume and two values for the length, that is, 5D and 7D; the calculated diameters amount to 1.710 and 1.913 inches.

The type 316 SS tubing selected for the canister has a 1.750-inch OD, and a wall thickness of 0.049 inches. Therefore, the ID is 1.652 inches, and a length of 12.8 inches was selected to accommodate 450 ml (27.5 in^3) of resin. The resultant L/D is 7.8.

The selection process was limited to standard sizes available from local suppliers. The 0.049 inch wall exceeds that required on the basis of hoop stress calculations. As with the charcoal canister, the above wall thickness probably exceeds that required for reasonable resistance to dents and punctures along with support/mounting strength requirements.

The media compression spring was designed along the lines established for the charcoal canister. Strength data could not be obtained for the resins used. However, it appeared reasonable to assume that the smooth resin beads could tolerate a compressive load at least equal to that applied to the jagged, irregularly shaped charcoal particles.

The compressive load required for the deionizer column diameter is 8 lbs. A low rate spring was also selected for the deionizer column.

The selected spring provides 6.6 lbs of force per inch of length, from 2.34 inches (free length) to 0.44 inches at which the spring is solid; the target compressive load (8 lbs) is applied when the spring is squeezed down to 1.125 inches. The spring was fabricated from 0.080-inch OD, type 18-8 SS wire. Other characteristics are as follows:

Mean Coil Diameter	= 1.24 in.
Number of Active Coils	= 4
Total Number of Coils	= 5-1/2
Free Length	= 2.34 in.

The overall length of the deionizer canister, excluding the tubing connectors is 14-1/4 inches. The OD at the "V" band clamp is 3.83 inches. The effective diamter of the bed is 1.652 inches, and the effective length is 12.8 inches; the funnel shaped end caps, space for the media compression springs, spring retainer, and pyrex wadding bring the overall length up to 14-1/4 inches. The following table presents the mass and volume requirements for the deionizer canister.

Canister Characteristics	Measured Value
Mass of Complete, Water-Filled Assembly - kg.	1.55
Dry Hardware Mass - kg.	0.99
Dry Mass of Resins - kg.	0.25
Volume - liters.	0.56

2.2.4 Silver Halide Canisters

The sizing of these components was based upon previous experience gained by CHEMTRIC in evaluating a silver chloride column developed by Melpar. This unit consists of a cylindrical column containing silver chloride granules in mixture with glass beads. The column provides an effective bed diameter of 1.5 inches and a bed length of 7.5 inches; it contains 162 grams of AgCl particles and 204 grams of glass beads.

The function of the glass beads is not readily discernable from the available literature. However, two functions are suggested - one function would be to retard agglomeration of the particles into a single large mass during quiescent periods, while the other function could be to reduce pressure drop across the bed.

Extensive tests were performed with the column at Melpar. The column was challenged with Escherichia coli, Mycobacterium smegmatis and Clostridium welchii* in concentrations of 10^5 to 10^7 viable cells per ml. Sterile water was produced in a single pass through column.

*This species is a spore former and an obligate anaerobic. The number of spores in the suspension and the type of atmosphere provided for this challenge were not specified. Oxygen is lethal for the vegatative cells of this species.

In the test programs conducted by CHEMTRIC under Contracts NAS 9-9014 and NAS 9-9191, a silver chloride column identical with that column described above was challenged severly on several occasions. On one occasion, while operating the Marquardt distillation unit with badly contaminated urine, the silver column sterilized condensate containing a 10^6 viable bacteria per ml; the bacteria was identified as a species of proteus. In a similar situation, the silver chloride column was continuously challenged with contaminated water containing 150 to 3000 bacteria/ml over a two-day period; this water was sterilized in a single pass through the column.

Because of the excellent performance achieved by the Melpar column, its essential features were duplicated as closely as possible in the canisters fabricated for the breadboard system. The same canister and glass bead mixture was also used for silver bromide because of the close similarity in chemical properties and state of aggregation of the solid.

The same tubing used in deionizer fabrication was used in the fabrication of the AgBr and AgCl column; this tubing has an ID of 1.652 inches, and a wall thickness of 0.049 inches. Since the column diameter of the AgBr and AgCl columns are the same as the deionizer, the media compression springs used are also identical.

The diameter of the above tubing is larger than the tubing used in the Melpar unit. Therefore, a shorter length was required to provide the same bed volume (12.7 in^3). The effective dimensions of the bed are 1.652 inches (diameter) and 6.2 inches (length).

Regarding the structural properties of the tubing used, the wall thickness exceeds that necessary to accommodate the proof and burst pressure requirements mentioned earlier. The liberal wall thickness also would exceed that required for reasonable dent and puncture resistance as well as mounting/structural requirements. However, weight optimization was not a goal in this program.

The overall length (excluding tubing connectors) is 8-1/4 inches; the funnel shaped end caps, space for the media compression spring, and pyrex wool wadding add to the 6.2 inches effective bed length. The diameter at the "V" band clamp is 3.83 inches. The following table presents the mass and volume requirements for the silver halide canisters. The dry weight of the glass bead-silver salt mixture in each column exceeds that used in the Melpar column. The weight difference is attributed to a difference between the glass beads procured for

this program and those used by Melpar. The beads used by Melpar were reported to be 470 microns while those used in this program were 450 to 500 microns.

Canister Characteristics	Measured Value
Mass of Complete Assembly, Including Interstitial Water - kg.	1.24
Mass of Dry Hardware - kg.	0.68
Dry Mass of AgBr & Glass-Beads Mixture - kg.	0.46
Dry Mass of AgCl & Glass-Beads Mixture - kg.	0.42
Volume - liters	0.33

2.3 System Design

Figure 2 illustrates the arrangement of components described in Section 3.2, and the purchased components described in this subsection. A photograph of this system is presented in Figure 3.

The large tanks located on either side of the system are reservoirs for fuel cell water simulant and product water; these items were mounted on dollies to facilitate daily replacement. The twin tanks in the upper portion of the system provided the storage capacity necessary for the anticipated daily water throughput (63 liters/day). The large cylindrical tank in the lower and center portion of the system served as the immersion tank for material testing. These large components were supported by appropriate cross members and were held in place by bulkhead-type tubing connectors. The processor components are mounted on the front face of the structure by means of hose clamps attached to cross members.

The components were spaced on the support structure shown in Figure 3 to facilitate component change-out between tests - and, more importantly, to permit easy access to the system for bacteria injection and water sample acquisition. The need for injection/sampling ports also entailed a larger number of tube fittings than would normally be required.

The supporting components used in this system are described as follows.

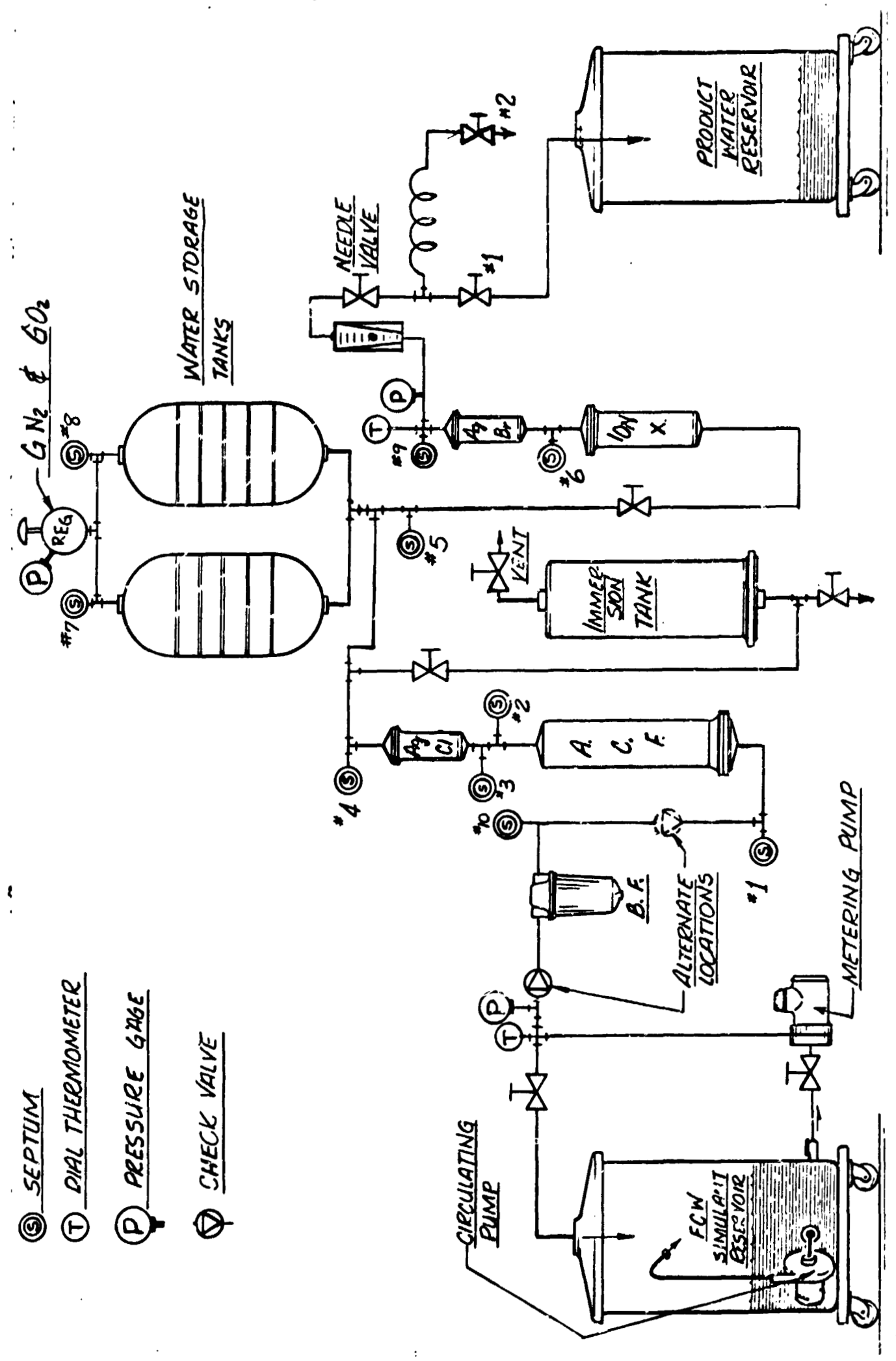

Figure 2 SCHEMATIC ARRANGEMENT OF BREADBOARD SYSTEM

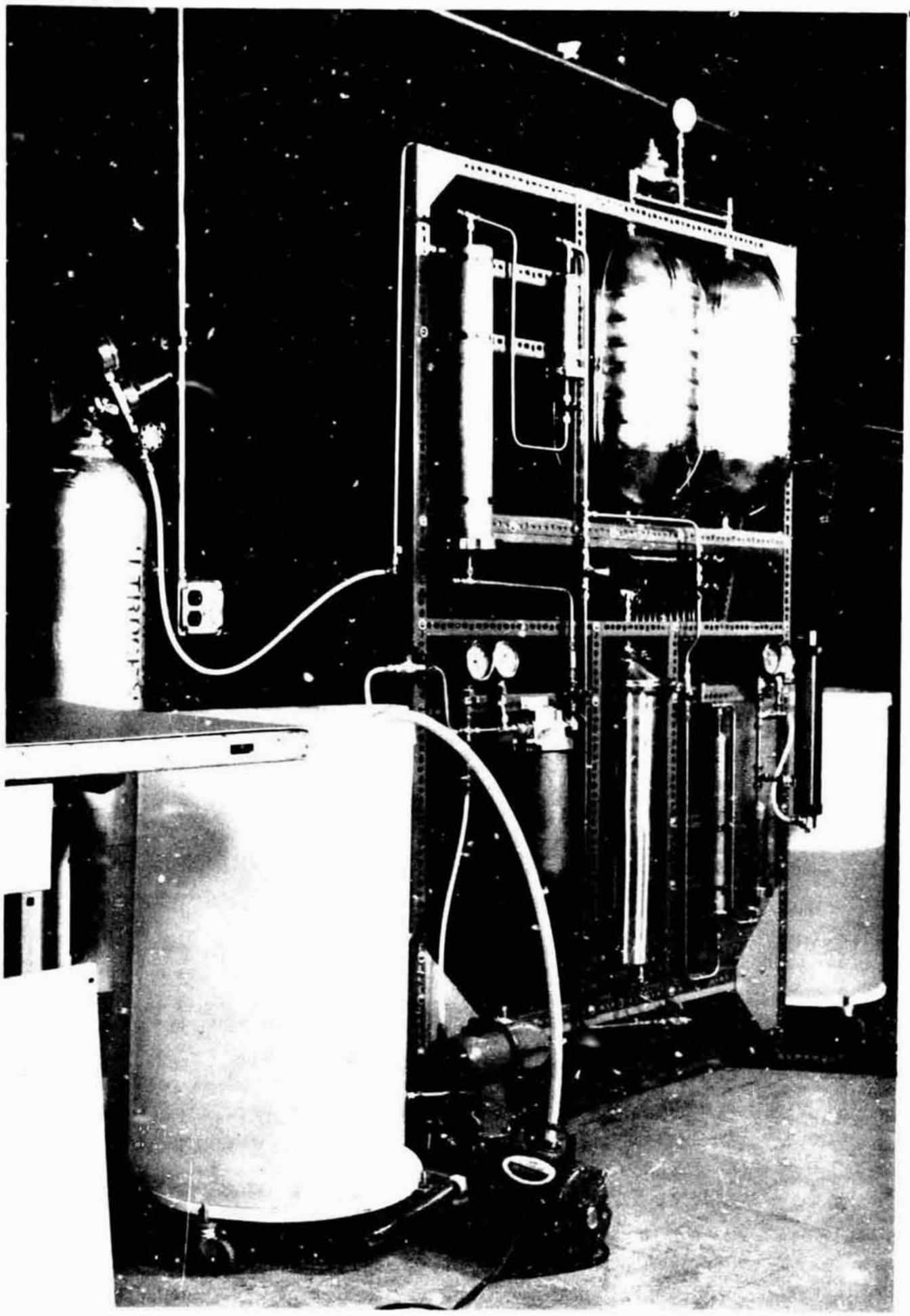

Figure 3 PHOTOGRAPH OF BREADBOARD SYSTEM

A. Metering Pump
Source: Cole-Parmer, Inc.
Catalog Number: 7043
Capacity: 0.9 to 9.5 lb/hr and up to 100 psi
Type: Diaphragm
Function: Pump simulant from reservoir to system
Wetted Materials: Penton Plastic

B. Circulating Pump
Source: Cole-Parmer, Inc.
Catalog Number: 7190-9
Capacity: 1.4 gpm
Type: Centrifugal
Function: Continuous agitation of simulant in reservoir
Wetted Materials: 316 SS and epoxy

C. Shut-Off Valves
Source: Hoke, Inc.
Catalog Number: 7122 G4Y
Type: Ball valve with integral tube fittings
Wetted Materials: 316 SS and Teflon

D. Check Valves
Source: Nupro Co.
Catalog Number: SS-6C-1
Type: Poppet, 1/4 psid cracking pressure
Function: Prevent back flow of water from storage tanks
Wetted Materials: 316 SS and Buna-N rubber

E. Metering Valve
Source: Whitey Research Tool Co.
Catalog Number: IRM-4-316
Function: Regulate flow rate of product water
Wetted Materials: 316 SS and Teflon

F. Flow Meter
Source: Fischer & Porter Co.
Catalog Number: 3535-240, 1010 cc/min
Type: Variable-area glass tube, direct reading
Function: Readout product water drawoff rate
Wetted Materials: Glass, Buna-N and 316 SS

G. Immersion Tank
Source: Millipore Corporation
Catalog Number: YY143200
Type: 6-inch ID x 25-inch long tank with removable end
Function: Immersion testing of construction materials
Wetted Materials: 316 SS and silicone rubber

H. Pressure Regulator
Source: Conoflow
Catalog Number: H-20XT-K
Type: Self-venting, 0.60 psi range
Function: Regulate water storage tank pressure
Wetted Materials: None normally wetted

I. Pressure Gage
Source: Ashcroft Gauge
Type: 2.5-inch dial, 0-6 psi
Function: Readout tank pressure and water pressure
Wetted Materials: 316 SS

J. Thermometer
Source: Weksler Instrument Corp.
Catalog Number: 3504
Type: 3-inch dial, bi-mettallic, 0 to 120 ^{o}F range
Function: Readout system water temperature
Wetted Material: 316 SS

K. Septums
Source: Hamilton Company
Catalog Number: 760-04
Type: Multi-Layer, self-sealing
Function: Provide sterilizable interface sampling
Material: Silicone rubber

L. Septum Holders
Source: Crawford Fitting Co.
Catalog Number: 400-R-6-316 (modified)
Function: Contain Septums
Wetted Material: 316 SS

M. Reservoirs
Source: Cole-Parmer Co.
Catalog Number: 6322-4
Type: Cylindrical graduated tank with cover
Function: Temporary storage of simulant and product water
Material: Polyethylene

N. Storage Tanks
Source: Illinois MFG. & Supply
Catalog Number: G-1
Function: Accumulate silver-dosed water
Material: SS

SECTION 3

TEST PROCEDURES AND METHODS

The breadboard system was subjected to a series of tests designed to simulate a Space Shuttle mission. Each of these tests consisted of seven days of non-stop operation with water input flow rates and product water demands that approximated a typical crew duty cycle. Bacteria were added to the simulated fuel cell water (FCW) or injected into the system at selected locations on each of the seven days.

A total of five simulated mission tests (SMT) were conducted to accommodate the major variables - namely, the type of bacteria injected into the system and the type of gas used to pressurize the water storage tanks.

3.1 Test Routine

3.1.1 System Clean-Up

The initial cleaning of the components and parts consisted of (1) degreasing with chlorinated solvent whereever construction materials permitted, (2) washing in dilute aqueous solutions of laboratory detergent (Alconox) with a soft brush, (3) rinsing in hot tap water, (4) rinsing with an aqueous solution of 50 to 100 ppm hypochlorite, (5) extensive rinsing in deionized water, and (6) air drying at ambient temperature.

The metering pumps, circulating pumps, shut-off valves and reservoirs were exempted from solvent treatment. The normally wetted areas of the thermometers and pressure gauges were dipped in solvent and brushed. Components such as the pressure regulator were disassembled for solvent treatment to remove "O" rings and other non-metallic parts. Metallic piece parts and line segments were soaked in solvent; the water storage tanks were partially filled with solvent and tumbled to permit the solvent to contact all internal areas. The remaining washing and rinsing operations were conducted on the components and piece parts by "pumping" the various fluids through the system.

System clean-up between tests consisted of (1) pumping an aqueous solution of 50 ppm hypochlorite into the system, (2) pumping several large volumes of deionized water through the system, and (3) circulating silver-chloride dosed water through the system. Each of these steps is explained in detail in the following paragraphs.

Upon completion of each SMT, the filters and canisters were removed from the system and replaced by plastic tubing. The septums and septum holders were also removed - but most of them were immediately replaced with new septums and holders which were steam sterilized. Septums number 7 and 8 on top of the storage tanks (see Figure 2) were temporarily replaced with plastic tubing to facilitate flushing the tanks.

After the plumbing changes were made, a clean simulant reservoir was filled with deionized water (25 gallons) and dosed with a sufficient amount of reagent-grade sodium hypochlorite to provide 50 ppm hypochlorite. This solution was then pumped into the system at the normal inlet by a stainless steel centrifugal pump at approximately 0.5 gpm, and accumulated in the storage tanks. Since the total capacity of the storage tanks is 20 gallons, the last 5 gallons flowed out of the tanks through the flexible tubing installed to replace septums 7 and 8.

Next, the tubing was removed from the top of the tanks and some of the solution in the storage tanks was drained through the outlet valves. Septums were then installed at locations 7 and 8 and the pressurant gas turned-on; the pressurant gas used in each cleaning operation was the gas to be used in the next SMT. Finally, the outlet valves (use points) were opened to "blow-down" the system and thereby eliminate as much hypochlorite solution as possible; This procedure was repeated three times with 25 gallon volumes of deionized water.

Next, a "freshly" prepared biological filter and a charcoal canister (see Appendix B) were installed along with the "old" AgCl canister (the same AgCl packing was used in all tests because it was never expended). Using the metering (feed) pump at a rate of 4 liters per hour, 76 liters of deionized water were then pumped into the system. This silver-dosed water was then drained through the normal outlet valves into the simulant reservoir. Finally, pressurant gas was allowed to blow-down the outlet segment of the system.

After the above steps were completed a fresh deionizer and the "old" silver bromide column were installed. At this point the system was ready for the next SMT.

3.1.2 Sequence of Variables

As indicated previously, the primary variables were the type of bacteria used to challenge the system and the type of pressurant gas. The composition of the simulant was fixed and was not altered throughout the test effort. System operat-

ing parameters such as influent flow rate, operating pressure and product water draw-off rate were fixed within a set routine that was applied to each SMT.

The following defines the combinations of variables in each test and the sequence pursued:

SMT No. 1

Bacteria:	Type IIIa*
Pressurant Gas:	Research grade nitrogen
Test Metal:	410 stainless steel heat treated to maximum strength
Test Elastomer:	Polyisoprene rubber

SMT. No. 2

Bacteria:	Type IIIa
Pressurant Gas:	Research grade oxygen
Test Metal:	410 stainless steel heat treated to maximum strength
Test Elastomers:	Silicone, Buna-N and butyl rubber

SMT No. 3

Bacteria:	Pseudomonas aeruginosa, ATCC #14502
Pressurant Gas:	Research grade nitrogen
Test Metal:	Grade A-55 titanium, as-received condition
Test Elastomers:	Polyisoprene, Viton and ethylene-propylene rubber

SMT No. 4

Bacteria:	Pseudomonas aeruginosa, ATCC #14502
Pressurant Gas:	Research grade oxygen
Test Metal:	Grade A-55 titanium, as-received condition
Test Elastomers:	Polyisoprene, Viton and ethylene-propylene rubber

SMT No. 5

Bacteria:	Bacillus subtilis, ATCC #6633
Pressurant Gas:	Research grade nitrogen
Test Metal:	None
Test Elastomers:	Viton and ethylene-propylene rubber

*This bacteria was previously isolated from the Apollo water system, and designated Type IIIa by the Center for Disease Control in Atlanta, Georgia.

The above sequence provided for systematic exposure of the Type IIIa and Pseudomonas species to both aerobic and anaerobic conditions. The aerobic spore forming bacteria, B. subtilis, was subjected to anaerobic conditions to minimize the potential for spore germination - because the spore form of this bacteria is more resistant to sterilization. The sequence also provided systematic exposure of the primary construction material, namely, type 316 SS, to the two types of pressurant gas. Test coupons of the other construction materials were similarly exposed.

The daily test routine was dictated in part by the need to accomplish sampling and simulant preparation within an 8-hour day (single shift). The specific simulant influent flow rates (8.7 lb/hr and 4.35 lb/hr) were used for the time durations specified.

An SMT was initiated at mid-day. The initial simulant input rate was set at 4 liters/hr (approximately equal to 8.7 lb/hr) and was so maintained for four hours. At this time, the rate was reduced to 2 liters/hr (approximately equal to 4.35 lb/hr) and maintained for sixteen hours. During the 16-hour period, the system was unattended except for brief status checks. At the beginning of the 21st hour, the simulant input rate was increased to 4 liters/hr. Product water draw-off was started after 21-1/4 hours. Both product water draw-off and simulant/product water flow regimen was followed on each day of each SMT.

The simulant and product water flow rates were verified by means of the graduated reservoirs and elapsed time measurements. Instantaneous read-out of the product water flow rate was provided by a calibrated flow meter.

System pressure was controlled by the pressurant gas regulator. An operating pressure of 33.5 psig was necessary for a water delivery rate of 50 lb/hr with a 30 psi head. Minor trimming of the regulator was required for each SMT to compensate for the slight pressure drop variations encountered with each freshly charged deionizer.

The bactericide system was challenged by injecting known quantities of viable bacteria into the system at selected locations. Bacteria were injected once each day at a single location; the site of injection was different each day. In many cases, the injection was followed by a specific sampling and analysis effort designed to evaluate a specific component. The following describes the pattern of injections followed in each SMT.

Day #1

A bacterial cell/spore suspension was added directly to the simulant reservoir during the first hour of the test day. Sufficient suspension was added to produce a cell/spore concentration in excess of 10,000 per milliliter of simulant. Dosing the reservoir resulted in a continuous 24-hour challenge to the system. The stability of the bacteria dose was monitored by performing plate counts on the dosed simulant during the 4th and 24th hour.

Day #2

Same as above.

Day #3

During the first hour, a bacteria suspension containing $3 \pm 1 \times 10^9$ bacterial cells/spores was injected at the inlet of the activated carbon filter (septum No. 1, Figure 2). The volume used was about 20 cc. Water samples were with-drawn at the outlet (septum No. 2, Figure 2) 30 and 60 minutes after injection.

Day #4

During the first hour, the same bacteria dose was injected at the inlet (septum No. 3) of the AgCl column. Water samples were withdrawn from the AgCl outlet (septum No. 4) 15 and 30 minutes after injection.

Day #5

The same bacteria dose was injected into the water storage tank inlet line (septum No. 4), also during the first hour of operation. The baseline water sampling/analysis routine, described in the next sub-section, provided assessment of system response to this challenge.

Day #6

Again, the same bacteria dose was injected into the tank via the septums located at the pressurant gas inlet (septums 7 and 8) during the first hour of operation. The baseline water sampling/analysis routine, described in the next subsection, provided system assessment.

Day #7

The same bacteria dose was injected at the deionizer outlet (septum No. 6) during the first hour. The baseline product water sampling/analysis routine provided system assessment.

The above routine, along with the water flow management regimen described earlier, defines the SMT. The impact of the main variables, namely bacteria species and pressurant gas were evaluated by the five SMT's in the order described in the first part of this sub-section. The baseline water sampling/analysis routine described in the next sub-section provided performance assessment.

3.1.3 Water Sampling/Analysis

An important objective of the test program was the evaluation of individual component performance as well as overall system performance. To accomplish both types of evaluation, sampling and subsequent analysis of water at intermediate stages in the system were carried out in addition to final product water sampling and analysis. This activity was carried out uniformly in each SMT to insure a common basis of comparison.

Samples were taken twice each day (3 - 4 hours and 20 - 21 hours after start-up) from the septum downstream of the storage tank (septum No. 5). The metering pump was stopped for sampling so that only tank contents were extracted. All samples were taken aseptically. Two separate 500 ml samples were taken at each time, thereby providing individual samples for chemical and bacteriologic analysis. Five ml of 1/10 normal sodium thiosulfate was added to the 500 ml biological sample immediately after sampling to slow down the bactericidal action of silver ions.*

The biological analyses were performed according to the methods described in Section 3.4. The main feature of the selected method is the use of the membrane filter technique which can accommodate large sample volumes. The method also separates the bacterial cells/spores from the silver bearing water and allows washing of the cells to further insure elimination of silver ions.

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*The effectiveness of thiosulfate in arresting the bactericidal action of silver was not evaluated. Prompt analysis of a given sample was relied upon to eliminate the possibility of residual kill. The analytical method of the nutrient media used is capable of eliminating the residual effect.

The following analyses/measurements were performed on each intermediate stage sample each test day:

(1)	Plate Count	(7)	Silver
(2)	Specific Resistance	(8)	Iron
(3)	pH	(9)	Nickel
(4)	Turbidity	(10)	Manganese
(5)	Total Solids	(11)	Chromium
(6)	Total Organic Carbon (TOC)	(12)	Chloride

The methods used for the above are standard techniques, as described in Section 3.3. The metal analyses were performed by atomic absorbtion spectrophotometry. All of the above except TOC were performed in-house by CHEMTRIC personnel; TOC analyses were performed in the Crew Systems Laboratory at the NASA MSC.

Sampling of the final product water was performed at the end of each test day while the product water was being drained from the system. Grab samples were taken for biological analysis. The chemical analyses listed above were performed on the accumulated product water each day.

The first 250 ml of product water (the first grab sample) was taken from the remote outlet valve (valve #2). Two hundred ml of the sample was used for biological analyses while the remainder was used for silver analysis and for detection of corrosion products - namely, chromium, iron, manganese and nickel. The second and third hour grab samples were used exclusively for biological analyses. All grab samples were taken in sterile bottles directly from the respective outlet valve.

Samples of product water were taken directly from the product water reservoir after all of the product water had been drained off. The analyses listed above were performed on the product water each day. In addition, product water was shipped to the NASA MSC for complete analysis on selected days of each SMT.

The analyses performed by the NASA MSC included specific analyses for each constituent listed in the "worst-case" fuel cell water described in Appendix A. This activity involved a great deal of effort and time, and of necessity, had to be reduced in scope. Rather than eliminate specific analyses, the number of samples was reduced. Product water samples from days 1, 4 and 7 were submitted for analyses. When coupled with the daily in-house water analyses, the above limitations did little to interfere with (1) detection of quality trends, (2) evaluation of component useful life, and (3) establishment of compliance with potability requirements.

3.2 Simulant Definition

Fuel cell water could not be supplied in sufficient quantity for use in the tests; consequently, a simulant had to be devised. A tentative "worst-case" composition of fuel cell water was supplied by the NASA MSC. This compilation, presented in Appendix A, contains more than 30 constituents and/or water quality parameters.

The entire list of constituents could not be incorporated into the simulant. The major differences between the simulant composition actually used differed from the NASA supplied composition in the following ways:

A. The simulant was not saturated with hydrogen gas because saturation could not be effected safely within the resource allocation of the program.

B. Silver salts were not added to the simulart for obvious reasons.

C. Iron, nickel, and manganese salts were omitted because their presence could interfere with assessment of possible system corrosion.

D. The alkalinity target level was eliminated because compliance would have seriously reduced the solubility of many of the metallic species. However, the alkalinity of the simulant was measured daily.

E. The particulate load was supplied by powdered silica (50 to 100 micron particles) and bacteria. This combination approximates the particles sizes and quantities required but does not duplicate the requirements.

Except for the deviations noted above, all of the constituents specified in Appendix A were included in the simulant. The constituents selected and their respective target concentrations are described in Table 1.

The concentrations specified in Table 1 are target values. Because of limitations in the scope of the program, simulant preparation relied on reagent purity chemicals and the assay values provided by the chemical manufacturer. Deviations in assay of the species desired as well as variations in water content could produce unavoidable differences between the amount of material calculated to be necessary and the amount of a substance actually obtained. For these reasons, variations in actual composition were anticipated. Such variations could have been virtually eliminated but only through a very substantial increase in technical effort.

Table 1 TARGET COMPOSITION OF SIMULATED FUEL CELL WATER

Cationic Species	Level (ppm)
Cadmium	0.01
Copper	1.0
Lead	0.05
Magnesium	0.17
Mercury	0.005
Platinum	0.05
Potassium	0.50
Sodium	3.30
Titanium	0.20
Zinc	5.0
Anionic Species	
Chloride	8.1
Chromate*	0.1
Fluoride	1.6
Nitrate	0.04
Selenite**	0.08
Sulfate	6.5
Particulates	
Silica (50 - 100μ)	100
Bacteria (1 - 10μ)	100,000/liter
Other	
Total Solids	223
Total Organics	100
pH	6.0-8.0 @ 77 ^{o}F

* Equivalent to 0.15 ppm Cr^{+6}

** Equivalent to 0.05 Se

Because the nature of the organic contaminant was not defined in the NASA list, considerable effort was expended to identify potential contaminants in the fuel cell water. Waste water contamination (cross-over from one loop to another via check valve leakage) was ruled out - leaving non-metallic materials of construction and process fluids as the only potential source of organic substances. Elastomers commonly contain additives to increase useful life; a common additive is butylated hydroxytoluene (an anti-oxidant). The only process fluid capable of contributing organic substances is the coolant fluid circulated through the heat exchanger used to moderate the fuel cell water temperature; a common ingredient in coolant fluid is ethylene glycol. The substances finally selected for simulating the organic contaminant were butylated hydroxytoluene and ethylene glycol. These substances were used in equal quantities to reach the 100 ppm concentration level required.

The simulant was prepared each day by adding the following to 94 liters (25 gallons) of one megaohm quality, deionized water.

A. Aliquots of two concentrated solutions.
B. Weighed portions of the organic substances.
C. Weighed portion of silica
D. Addition of 1 N sodium hydroxide solution to adjust pH.

The concentrated solutions were necessary to eliminate the potential for variations inherent in weighing out small quantities of dry, granular substances each day. Two concentrated solutions were used to permit separation of cationic-anionic pairs that would precipitate out at the concentration level used in the stock or concentrated solution. The two stock solutions were composed as follows.

A. All cationic species, except sodium and potassium as chloride salts.
B. All anionic species, except chloride as sodium and potasium salts.

The dilution factor from the stock solutions to the simulant was 940 - that is, the stock solution was 940 times more concentrated than the simulant. By adding 100 ml of each stock solution to 94 liters of water, the concentration of each constituent was obtained with little error. The same batch of stock solution was used throughout the entire test program.

The potential error is very small and is limited to volumetric type errors. The stock solution aliquot could deviate from 100 ml by ± 2 ml in combination with a deionized water deviation of ± 1000 ml without affecting the stated concentration of a constituent. In actual practice, a class A volumetric pipette was used to measure the stock solution aliquot; such measurements routinely provide an accuracy of ± 1%. The simulant reservoirs were carefully calibrated by adding water in 4 liter increments; the contents of the reservoir could be read within 500 ml.

Aliquots of the two stock solutions were shipped to the NASA MSC for detailed analysis. Using the analysis values and the 940 dilution factor, the anticipated concentrations were computed. Table 2 presents these computed values along with the target values. The differences between actual and target values are due to either deviations from manufacturer's assay data or weighing error.

The simulant was monitored each day by alkalinity, total solids, pH and specific resistance measurements. Plate counts were performed on samples of simulant taken 4 and 24 hours after bacteria injection on the appropriate test days. One liter samples of simulant were also shipped to the NASA MSC for detailed analysis, at least once during each SMT. Table 3 indicates that the composition of the samples was reasonably close to the target values. The low values for total solids is attributed to "settling" of particulates in the reservoir or sample batch - while the nitrate and sulfate deviations are probably due to inter-reactions and/or interference.

3.3 Chemical Analysis Methods

The analytical effort was performed both in-house and at the NASA MSC. Standard methods were used in all analyses.

The analytical methods at the NASA MSC are described in detail in Document No. CSD-A-726 (Procedure Manual for Water Analysis). The procedures contained in the above manual are based on the procedures contained in the Standard Methods* text or on superior instrumental methods.

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*Standard Methods for the Examination of Water and Waste Water, 12th Ed, APHA, Inc., New York, New York

Table 2 ACTUAL COMPOSITION OF DILUTED STOCK SOLUTIONS

Cationic Species	Level (ppm) Target	Actual
Cadmium	0.01	0.01
Copper	1.0	0.96
Lead	0.05	0.053
Magnesium	0.17	0.29
Mercury	0.005	0.001
Platinum	0.05	NA
Sodium	3.3	2.7*
Titanium	0.20	0.04
Zinc	5.0	5.0
Anionic Species		
Chloride	8.1	8.6
Chromate	0.10	0.096
Fluoride	1.6	1.70
Nitrate	0.04	0.09
Selenite	0.08	NA
Sulfate	6.50	1.80

NA = Not analyzed by the NASA MSC
* An Additional 0.2 ± .02 ppm Na added each day via pH adjustment

Table 3 ANALYSIS OF SIMULANT USED FOR EACH TEST

Quality Parameters	Concentration (ppm) SMT 1	SMT 2	SMT 3	SMT 4	SMT 5	TARGET
Aluminum	<0.5	<0.5	<0.5	<0.5	<0.5	NS*
Bromine	<1.0	<1.0	<1.0	<1.0	<1.0	NS
Cadmium	<0.005	≤0.005	0.01	0.01	0.02	0.01
Chloride	8.5	8.3	7.15	8.14	8.03	8.1
Chromium(+6)	0.13	0.13	<0.01	0.06	0.07	0.05
Copper	0.60	0.75	0.63	0.69	0.76	1.0
Flouride	2.3	1.9	1.95	2.0	1.7	1.6
Iron	<0.2	<0.2	<0.2	<0.2	<0.2	None
Lead	<0.5	<0.5	<0.5	<0.5	<0.5	0.05
Magnesium	0.19	0.19	0.32	0.14	0.17	0.17
Manganese	<0.05	<0.05	<0.05	<0.05	<0.05	None
Mercury	<0.005	<0.005	<0.005	<0.005	<0.005	0.005
Nickel	<0.5	<0.5	<0.5	<0.5	<0.5	None
Nitrate	4.0	<0.5	<0.5	0.56	0.5	0.04
Potassium	0.62	0.65	0.75	0.57	0.60	0.50
Silicon	<5.0	<5.0	<5.0	<5.0	<5.0	NS
Silver	<0.05	<0.05	<0.07	<0.05	<0.05	Nc
Sodium	5.0	4.8	3.3	4.3	4.15	3.5
Sulfate	3.9	3.2	2.3	1.6	2.8	6.5
Titanium	<1.0	<1.0	<1.0	<1.0	<1.0	0.20
Zinc	4.5	5.7	6.5	5.0	4.9	5.0
Total Solids (ppm)	48.0	90.7	30.2	158.3	161.4	223.0
Total Carbon	42.5	38.0	38.0	63.6	54.9	NS
pH	6.54	6.26	5.67	5.97	6.09	6.0-8.0
Resistance(Mohm-cm)	0.027	0.021	0.029	0.029	0.034	NS

*NS = Not Specified

The chemical analysis methods performed in-house were as follows:

Acidity

(a) Method - Titrimetric, phenolphthalein at room temperature as per Standard Methods text pages 46-48.
(b) Instrument - None
(c) Accuracy - ±1 mg/l as $CaCO_3$

Alkalinity

(a) Method - Titrimetric, methyl orange end point as per Standard Methods text pages 43-52.
(b) Instrument - None
(c) Accuracy - ±1 mg/l as $CaCO_3$

Specific Resistance:

(a) Method - Standard Methods text pages 280-284.
(b) Instrument - YSI Model #31, Conductivity Bridge
(c) Accuracy - ±1% in range of 2 ohms to 2 megaohms

pH

(a) Method - Standard Methods text pages 225-228.
(b) Instrument - Corning Model 7 pH meter
(c) Accuracy - ±0.05 pH (relative)

Turbidity

(a) Method - Nephelometric as per instrument manufacturer's manual.
(b) Instrument - Hach Chemical Co. Model No. 2100 A laboratory turbidimeter
(c) Accuracy - ±2% of full scale

Total Solids

(a) Method - Gravimetric as per Standard Methods text pages 244-245.
(b) Instrument - None
(c) Accuracy - ±5%

Chloride

(a) Method - Titrimetric, mercuric nitrate method as per Standard Methods text pages 87-89.
(b) Instrument - None
(c) Accuracy - ±4 ppm

Trace Metals (Ag, Cr, Fe, Mn, Ni)

(a) Method - Atomic absorption as per instrument manufacturer's manual of analytical methods.
(b) Instrument - Perkin-Elmer Model 103, Atomic absorption Spectrophotometer
(c) Accuracy - ±0.2%

3.4 Bacteriologic Analysis and Methods

The methodology for (1) culturing the test bacteria, (2) plate counts, and (3) challenge dose preparation are described in the following paragraphs. Standard techniques were employed exclusively.

The test bacteria were obtained as follows:

1. Type IIIa from the National Center for Disease Control, Atlanta, Georgia.

2. Pseudomonas aeruginosa from the American Type Culture Collection (ATCC) - type #14502.

3. Bacillus subtilis from the American Type Culture Collection - type #6633.

Type IIIa was received on an agar slant (Brain, Heart Infusion agar). Five APT agar (BBL #10916) slants were inoculated (streaked) with aliquots of the as-received culture. After 48 hours of incubation at 35°C, the slants were checked for purity by gram stains and streak plates. These cultures were then refrigerated (2 - 6°C) and used as working stock.

The ATCC cultures were lyophilized. Both cultures were rehydrated in sterile, double-strength skim milk - followed by incubation at 35°C for 48 hours. The pseudomonas species were transferred to APT agar slants; these stock cultures were refrigerated after incubation. Stock bacillus cultures were prepared in similar fashion, but with AK agar #2 (BBL #10912); this agar is the recommended* maintenance medium for the bacillus species.

* Arret and Kirshbaum, J. Milk & Food Tech., 22:329, 1959.

The plate counts made on the water samples and the challenge doses utilized the membrane filter (MF) method. The MF method is a standard procedure; the general analytical requirements specified in the Standard Methods text (pages 610-615) were followed. Some latitude is allowed for filter porosity and nutrient media selection. The salient features of the procedure used are as follows.

A. Materials

1. Sterile, phosphate-buffered, physiological saline (PBS).
2. Sterile, 0.001 N sodium thiosulfate.
3. Millipore filter disc holders (Cat. #XX11-047-00).
4. Sterile, 0.22 micron membrane filter discs (Millipore Cat. #GSWP-047-S0).
5. Pre-prepared, sterile APT agar plates less than 1 week old.

B. Method

1. Two hundred ml and one ml quantities of analysate were routinely used. The small quantities of analysate were added to the filter holder containing at least 100 ml of PBS prior to filtration.

2. The filter funnel and disc were washed with 100 ml of 0.001 N sodium thiosulfate solution followed by washing with two, 200 ml aliquots of PBS.

3. The filter disc was rolled onto the APT agar plate and incubated in a humidified cabinet at 35°C.

4. Counts were made after 24 and 48 hours of incubation.

The most attractive feature of the selected plate count method is the ability to eliminate the residual kill capability of dissolved silver salts. Filtration separates bacteria cells/spores from the water sample, thereby separating the cells from residual dissolved silver salts. The thiosulfate wash aids in removing silver salts trapped in the filter; thiosulfate forms a complex ion with silver, thereby greatly increasing the solubility of silver halides. Finally, the nutrient media (APT agar) used in the plates neutralizes the lethal effects of silvers. This neutralization capability was established during a NASA-sponsored program* involved with evaluation of the electrolytic silver-ion generator.

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* Ibid, Cliver, D. O. et. al.

The challenge doses were prepared from 48 to 72 hour APT agar cultures. In general, the preparation consisted of (1) harvesting the culture, (2) dispersion of the culture in sterile PBS, (3) cell suspension washing via repeated centrifugations and decantations, and (4) plate counts on the suspensions. The following paragraphs describe these procedures in greater detail.

The harvesting and washing operations were as follows.

A. Inoculated a Kolle flask containing 300 ml of APT agar followed by incubation in a humidified incubator at 35°C for 72 - 96 hours.

B. Harvested the culture by teasing the agar surface with a wire loop in the presence of 25 ml sterile PBS.

C. The above suspension was pipetted into a sterile capped centrifuge tube containing 1 to 2 mm glass beads. The tube was shaken vigorously to disperse the bacterial cells. An additional 25 ml of PBS was added to aid in dispersion.

D. The above tube was centrifuged, and the supernatant liquid decanted off. The cells were then resuspended in 50 ml PBS. This washing procedure was performed at least three times on all suspensions.

E. The cell mass resulting from the last centrifugation was resuspended in 25 ml of phosphate-buffered deionized water. Aliquots of this suspension were then subjected to plate counts to identify the viable cell concentration; these plate counts used the MF technique described previously.

F. Based on the plate count data, the final suspension was diluted with buffered deionized water so that 20 ml of suspension contained at least 1 x 10^6 bacteria.

The above procedure was followed for all of the bacteria species used. However, since only the spores of B. subtilis were to be used an additional preparation step was required. The washed suspension resulting from step D above was heat treated at 70°C in a thermostatically controlled water bath for 30 minutes to kill off the vegatative cells. Upon completion of the heat treatment, the remaining steps (E&F) were performed.

In actual practice, the time delay inherent in the plate count procedure was intolerable in preparing challenge doses. To eliminate the time delay, a correlation between plate count values and cell suspension turbidity was established to permit preparation of challenge doses accurate to within an order of magnitude. However, plate counts were also performed on each suspension for verification.

The procedures involved in preparing the turbidity correlation were as follows.

A. One ml of the final suspension was transferred to 99 ml of PBS contained within a standard milk dilution bottle.

B. From the above 1:100 dilution, serial, decade dilutions were made up to 10^8 by transferring 10 ml to milk dilution bottles containing 90 ml PBS.

C. MF type plate counts were performed on the 10^7 and 10^8 dilutions.

D. Intermediate dilution (10^3 to 10^5) levels were subjected to turbidity measurements.

To prepare a challenge dose, an aliquot of washed suspension was subjected to serial dilution as specified above. Plate counts were performed along with the turbidity measurements. Using the turbidity values and the established correlation, the viable cell count of the washed suspension was calculated; with B. subtilis it was assumed that the vegatative cell-spore ratio was one. The washed suspension was diluted with sufficient sterile buffered water so that 20 ml of the dilution would contain at least 1 x 10^9 bacteria. From 2 to 4 challenge doses were obtained from each batch of suspension.

Twenty-five ml aliquots of the diluted suspension were dispensed into sterile serum bottles; the bottles were sealed with a rubber serum cap and refrigerated for future use. Sterile disposable syringes were used to extract the 20 ml challenge dose from the serum bottle and to inject the dose into the breadboard system. With Type IIIa and P. aeruginosa, the challenge doses were never stored for more than 3 days before use. A large batch of B. subtilis suspension was prepared because the spores were known to be unaffected by storage; more than 15 challenge doses were prepared from a single batch of suspension derived from four Kolle flask cultures. Regardless of the storage time, each challenge dose was evaluated by plate count; the values obtained showed that both the duration and conditions of storage had no effect on cell count.

3.5 Construction Materials Evaluation

The breadboard system was fabricated from austenitic stainless steels. Previous experience had indicated that this material would perform well. Additional metallic materials were evaluated for durability by immersing coupons in a special tank included in the system. Five elastomer materials were also evaluated for durability. However, the major effort in materials testing was directed to evaluation of the primary candidate material, namely austenitic stainless steel.

Evaluation of the breadboard system construction material was based on (1) chemical analysis of the water taken from the system, (2) visual inspection of each component unaided and at 10 x magnification wherever possible, and (3) exposing the custom-designed components to 90% of the design yield pressure (117 psi).

The in-house analysis of water taken from the storage tanks was specifically directed to the detection of stainless steel corrosion products. These analyses were performed on samples taken at two different times every test day.

Upon completion of each SMT, the custom-designed components were removed from the system and installed in a high-pressure, bench-type set-up - which consisted of a water reservoir, a high-pressure diaphragm pump, a needle valve and a pressure gage. The inlet of each component was connected to the pump outlet; a tee connector joined the component outlet to a pressure gage and needle valve. Once connected, the pump was turned on; by adjusting the effluent flow rate via the needle valve, the component was subjected to hydraulic pressures equivalent to 90% of design yield pressure or 117 psi. While subjected to this pressure, seals and weldments were carefully inspected for leakage. This test was intended to reveal any significant degradation of structural integrity.

After the above test, the custom-designed components and the immersion tank were disassembled and inspected visually, aided by 10 x magnification wherever possible. Specific searches for corrosion deposits and pits were made.

Additional construction materials, metals and elastomers were evaluated in soak tests. These tests involved immersing coupons of selected materials in the tank provided within the breadboard system. The liquid in the tank was identical to that in the water storage tanks, except for the lack of bacteria.

Figure 4 displays the method used for suspending both the metallic and elastomeric test coupons within the tank. The coupons did not touch the walls or bottom of the tank. With the exception of the coupons providing the crevice, none of the coupons were in contact with each other. Only one metal type was tested at a time - while one or more elastomers were evaluated simultaneously.

The immersion tank, with coupons in place, was filled at the start of each SMT by pumping simulant into the system with the pressurant gas supply turned off. By opening the valve on the immersion tank fill line, silver-dosed water flowed downward into the immersion tank instead of into the water storage tanks. Pumping was continued until water began escaping from the opened immersion tank vent valve. At this time, the simulant feed pump was turned off. The drain valve located below the immersion tank was opened and one liter of water was drained off to provide a gas cap in the tank. The system pressurant gas supply was turned on; this caused pressurant gas to flow into the bubble upward through the water-filled tank. The gas flow rate was controlled by manipulating the immersion tank vent valve. This process was continued for 15 to 20 minutes to displace air in the tank and to saturate the water in the tank with the pressurant gas used. After the above time period, the vent valve was closed causing the gas pressure in the tank to come up to system operating pressure. Finally, the immersion tank fill valve was closed, thereby isolating the tank from the rest of the system.

The two metals evaluated were Grade A-55 titanium and type 410 SS. The metal coupons were all 4-inch squares of 1/32-inch sheet stock. The stainless steel pieces were heat treated to maximum strength while titanium was used in the as-received state. All metal coupons were washed in mild non-abrasive detergent, rinsed in deionized water and dried to constant weight in a 103°C oven.

The elastomeric materials were cut into 4-inch x 7-inch coupons from 1/32-inch sheet stock. The coupons were washed in a weak aequeous solution of tri-sodium phosphate followed by a deionized water rinse. The coupons were allowed to dry in air at room temperature. The coupons were then placed in a desiccator containing calcium chloride, and stored under these conditions until constant weight was achieved.

Once the coupons were cleaned, dried and weighed, they were assembled in the configuration shown in Figure 4 and suspended in the immersion tank. To aid in interpretation, identical elastomeric coupons were also soaked in deionized water contained in 1/2 gallon glass bottles; these control tests paralleled the SMT's.

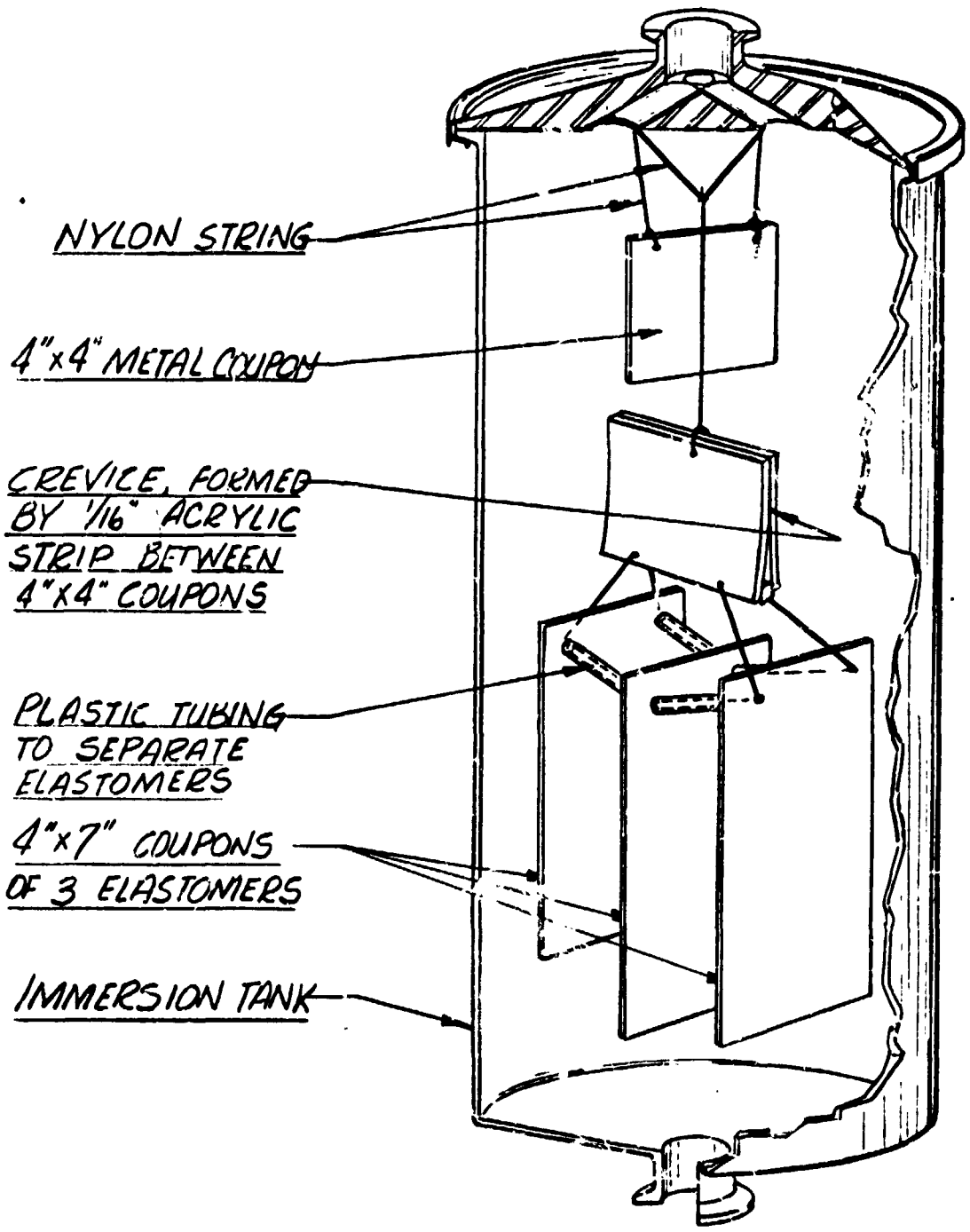

Figure 4 SCHEMATIC OF IMMERSION TEST SET-UP

At the completion of day seven of each SMT, the coupon assembly was removed. Each coupon was inspected visually and then submitted to the respective cleaning and drying operations noted above. Assessment of degradation in both metallic and elastomeric materials was made on the basis of coupon weight change, visual inspection and analysis of the water contained within the immersion tank.

SECTION 4

TEST RESULTS

The Breadboard System was used to perform five simulated Space Shuttle missions for determining (1) the efficacy of silver ions in killing three types of microbes, (2) the quality of the product water, and (3) the effect on materials, when processing simulated "worst-case" fuel cell water. The results of these tests and flow resistance measurements made at the end of the 5th SMT are described in the following subsections.

4.1 Type IIIa Challenge Tests

The first two SMT's were performed to evaluate the effect of silver ions on the Type IIIa bacteria when nitrogen and oxygen, respectively, is the gas used to pressurize the water storage tanks. Since the Type IIIa bacteria is aerobic, SMT #2 provided conditions more conducive to growth than SMT #1. The results of the bacteriological analyses performed during these tests are summarized in Tables 4 and 5.

The lower limit of the technique used to determine the Most Probable Number (MPN) of bacteria in a sample is less than 1 cell per 200 ml ($\leq$ 1/200 ml). Thus, Tables 4 and 5 indicate that all samples taken from septums and the outlet valves were essentially sterile ($\leq$ 1/200 ml) - except outlet valve #2 (OV-2) on the 1st day of SMT #1, and both outlet valves on the 7th day of SMT #2. The explanations for these two anomalies are as follows.

A. The purpose of outlet valve #2 was to determine the efficacy of silver ions in maintaining a remote, infrequently used outlet in a sterile condition. The outlet was located more than 10 feet from the AgBr column, and only 250 ml of water were drawn from this outlet once each day. The contamination detected on Day 1 of SMT #1 could have been from an external source; however, this sample undoubtedly had a low silver content because it was the "first" water drawn from that outlet valve. In either case, the contamination was not Type IIIa bacteria - as evidenced by a markedly different characteristic of a colony.

B. The contamination detected on day 7 of SMT #2 is attributed to a procedural error. The normal procedure on day 7 was to inject bacteria between the deionizer and the AgBr Column, via S-6, where the silver concentration should be less than 50 ppb - and simultaneously withdraw a small amount of water through OV-2, to avoid forcing the bacteria into the deionizer. The technician forgot to "crack" the outlet valve; consequently, the bacteria were not exposed to the normal silver dose until after the 21st hour of the test day.

Table 4 SUMMARY OF BACTERIOLOGIC ANALYSES FOR SMT #1

Day	Sample Points: Sim.Reservoir Hr* = 24	S-2 Hr**	S-2 Count	S-4 Hr**	S-4 Count	S-5 Hr*	S-5 Count	OV-1 Hr*	OV-1 Count	OV-2 Hr* = 22
1	3 ± 1 x 10^4/ml		NA		NA	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	2/100 ml
2	3 ± 1 x 10^4/ml		NA		NA	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
3	NA	0.5 1.0	<1/200 ml <1/200 ml		NA	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
4	NA		NA	0.25 0.5	<1/200 ml <1/200 ml	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
5	NA		NA		NA	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
6	NA		NA		NA	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
7	NA		NA		NA	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml

* Hour of test day
**Hours after injection
NA = Not analyzed

Table 5 SUMMARY OF BACTERIOLOGIC ANALYSES FOR SMT #2

	Sample Points											
	Sim.Reservoir	S-2		S-4		S-5		OV-1		OV-2
Day	Hr* = 24	Hr**	Count	Hr**	Count	Hr*	Count	Hr*	Count	Hr* = 22
1	$3 \pm 1 \times 10^4$/ml	NA		NA		4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
2	$3 \pm 1 \times 10^4$/ml	NA		NA		4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
3	NA	0.5 1.0	<1/200 ml <1/200 ml	NA		4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
4	NA	NA		0.25 0.5	<1/200 ml <1/200 ml	4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
5	NA	NA		NA		4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
6	NA	NA		NA		4 21	<1/200 ml <1/200 ml	23 24	<1/200 ml <1/200 ml	<1/200 ml
7	NA	NA		NA		4 21	<1/200 ml <1/200 ml	23 24	200/ml 98/100 ml	200/ml 200/ml

* Hour of test day
**Hours after injection
NA = Not analyzed

The quantity of bacteria injected into the simulant reservoir on days 1 and 2 of each SMT was $3 \pm 1 \times 10^9$. Since the reservoir contained 94 liters of simulant, the resultant concentration should have been $3 \pm 1 \times 10^4$/ml. Actual counts indicated 1.8×10^4/ml and 2.0×10^4/ml. Therefore, despite the presence of lead, copper and chromate in the simulant, it is concluded that the simulant itself has a negligible effect on Type IIIa bacteria.

The results presented in Tables 4 and 5 indicate that when $3 \pm 1 \times 10^9$ Type IIIa bacteria were injected upstream (S-2) or downstream (S-4) of the Ag Cl Column, samples withdrawn 0.25 to 1.0 hours later from the same septums were essentially sterile ($< 1/200$ ml). Simulant, of course, was diluting and "flushing" these bacteria into the storage tank - but samples taken from S-5 less than 4 hours after bacteria were injected on all test days were also found to be sterile. Thus, silver ions from silver chloride particles in the BF and the AgCl Column were capable of reducing the concentration of Type IIIa bacteria from more than 10^4/ml* to less than 10^{-2}/ml in periods as short as 15 minutes.

4.2 Pseudomonas Challenge Tests

Tests #3 and #4 were performed to evaluate the effect of silver ions on the Pseudomonas aeruginosa bacteria when nitrogen and oxygen, respectively, is the gas used to pressurize the water storage tank. Since this bacteria is aerobic, but "facultatively" anaerobic (i.e., it can also survive under anaerobic conditions), SMT #4 provided conditions more conducive to growth than SMT #3. The results of the bacteriological analyses performed during these tests are summarized in Tables 6 and 7.

The bacteriological results obtained from these tests are essentially the same as those obtained for the Type IIIa Challenge Tests (SMT #1 and #2) - namely:

A. The simulant has a negligible effect on P. aeruginosa, as evidenced by the fact that the concentration of cells in samples withdrawn from the reservoir 24 hours after bacteria injection on the 1st and 2nd day (see Tables 6 and 7) was found to be within the range anticipated after dilution (i.e., $3 \pm 1 \times 10^9$ cells/94 liters or $3 \pm 1 \times 10^4$/ml).

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*Assuming that $3 \pm 1 \times 10^9$ cells are uniformly dispersed in all of the water ($\sim$100 liters) contained in the Breadboard System at any one time.

Table 6 SUMMARY OF BACTERIOLOGIC ANALYSES FOR SMT #3

	Sample Points											
	Sim.Reservoir	S-2		S-4		S-5		OV-1		OV-2
Day	Hr* = 24	Hr**	Count	Hr**	Count	Hr*	Count	Hr*	Count	Hr* = 22
1	$3 \pm 1 \times 10^4$ /ml		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
2	$3 \pm 1 \times 10^4$ /ml		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
3	NA	0.5	<1/200 ml		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
		1.0	<1/200 ml			21	<1/200 ml	24	<1/200 ml	
4	NA		NA	0.25	<1/200 ml	4	<1/200 ml	23	<1/200 ml	<1/200 ml
				0.5	<1/200 ml	21	<1/200 ml	24	<1/200 ml	
5	NA		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
6	NA		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
7	NA		NA		NA	4	<1/200 ml	23	200/ml	200/ml
						21	<1/200 ml	24	15/100 ml	

* Hour of test day
** Hours after injection
NA = Not analyzed

Table 7 SUMMARY OF BACTERIOLOGIC ANALYSES FOR SMT #4

Day	Sample Points: Sim. Reservoir Hr* = 24	S-2 Hr**	S-2 Count	S-4 HR**	S-4 Count	S-5 Hr*	S-5 Count	OV-1 Hr*	OV-1 Count	OV-2 Hr* = 22
1	3 ± 1 x 10^4/ml		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
2	3 ± 1 x 10^4/ml		NA		NA	4	<1/200 ml	23	<1/200 ml	4/100 ml
						21	<1/200 ml	24	<1/200 ml	
3	NA	0.5	<1/200 ml		NA	4	<1/200 ml	23	<1/200 ml	
		1.0	<1/200 ml			21	<1/200 ml	24	<1/200 ml	<1/200 ml
4	NA		NA	0.25	<1/200 ml	4	<1/200 ml	23	<1/200 ml	<1/200 ml
				0.5	<1/200 ml	21	<1/200 ml	24	<1/200 ml	
5	NA		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
6	NA		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	
7	NA		NA		NA	4	<1/200 ml	23	<1/200 ml	<1/200 ml
						21	<1/200 ml	24	<1/200 ml	

* Hour of test day
**Hours after injection
NA = Not analyzed

B. Water drawn from the outlet valves on day 7 of SMT #3 was found to be contaminated, as previously found on day 7 of SMT #2 - because the procedural error described in Section 4.1 was not diagnosed until after SMT #3 had been completed. When this error was rectified, for SMT #4, the water drawn from the outlet valves on day 7 was sterile.

C. The kill-off rate is greater than a six-log change ($> 10^4$/ml to $< 10^{-2}$/ml) in periods as short as 15 minutes.

D. The 250 ml of water drawn from OV-2 on the 2nd day of SMT #4 was found to be contaminated; however, since a colony grown from this sample had a markedly different physical appearance than Type IIIa or P. aeruginosa colonies, it is concluded that the contamination was from an external source. Apparently the amount of silver ions in this infrequently used outlet was insufficient to prevent the external contamination encountered on the 2nd day.

4.3 B. Subtilis Challenge Test

SMT #5 was performed to evaluate the effect of silver ions on the spore form of Bacillus subtilis - and demonstrate that a BF containing AgCl particles has a useful microbial life greater than 7 days. Only one test was necessary, using nitrogen as the tank pressurant, because B. subtilis is aerobic - and it was desired to provide conditions which are conducive to retaining the bacteria in the more "resistant" spore form.* The results of the bacteriological analyses performed during this test are summarized in Table 8.

The duration of this test was extended from 7 to 10 days after it was discovered on the 5th day that the samples withdrawn on the 3rd day were not sterile (plate count results were not available until 2 days after each sample was taken). The test routine was then revised as follows.

*If oxygen were used, the spores may have germinated to the vegatative form which should be more easily killed by silver ions.

Table 8 SUMMARY OF BACTERIOLOGIC ANALYSES FOR SMT #5

Day	Sim.Reserv hr* = 24	S-10 hr*	S-10 Count	S-2 hr**	S-2 Count	S-4 hr**	S-4 Count	S-5 hr*	S-5 Count	OV-1 hr*	OV-1 Count	OV-2 hr* = 22
1	$3\pm1x10^4$/ml		NA		NA		NA	4	<1/200ml	23	<1/200ml	<1/200ml
								21	<1/200ml	24	<1/200ml	
2	$3\pm1x10^4$/ml		NA		NA		NA	4	<1/200ml	23	<1/200ml	<1/200ml
								21	<1/200ml	24	<1/200ml	
3	NA		NA	0.5	$5x10^5$/ml		NA	4	$20x10^4$/ml	23	30/ml	<1/200ml
				1.0	$3x10^6$/ml			21	$17x10^4$/ml	24	18/ml	
4	NA		NA		NA	0.25	$8.3x10^3$/ml	4	$5x10^4$/ml	23	180/ml	1.0/ml
						0.5	$7x10^4$/ml	21	$7x10^4$/ml	24	165/ml	
5	NA		NA		NA		NA	4	$2x10^6$/ml	23	$1x10^4$/ml	$2x10^4$/ml
								21	$1x10^6$/ml	24	$2x10^4$/ml	
6	$3\pm1x10^4$/ml	4	<1/200ml		NA		NA	4	$1.3x10^3$/ml	23	39/ml	$12x10^4$/ml
		21	<1/200ml		NA		NA	21	$3.7x10^3$/ml	24	17/ml	
7	$3\pm1x10^4$/ml	4	<1/200ml		NA		NA	4	$1.3x10^3$/ml	23	18/100ml	$14x10^4$/ml
		21	<1/200ml		NA		NA	21	$3x10^2$/ml	24	56/100ml	
8	$3\pm1x10^4$/ml	4	<1/200ml		NA		NA	4	33/ml	23	18/100ml	$6x10^4$/ml
		21	<1/200ml		NA		NA	21	<1/200ml	24	21/100ml	
9	NA	4	<1/200ml		NA		NA	4	<1/200ml	23	10/100ml	$2x10^3$/ml
		21	<1/200ml		NA		NA	21	32/100ml	24	8/100ml	
10	NA	4	<1/200ml		NA		NA	4	<1/200ml	23	9/100ml	$2x10^3$/ml
		21	<1/200ml		NA		NA	21	32/100ml	24	11/100ml	

* Hour of test day
** Hours after injection
NA = Not analyzed

A. On days 6, 7 and 8 spores were injected in the simulant reservoir only to determine how fast the system would flush-out the previous contamination, and demonstrate that the BF prevents the introduction of additional contamination. At the end of day 5 the BF check valve was moved from the inlet to the outlet side, and another septum (S-10) installed between the BF and the check valve - so that samples of the BF effluent would not contain any water drawn upstream from the contaminated ACF.

B. At the end of day 8 an electrical resistance heater was wrapped around the AgCl Column - to raise the temperature of the column, and thereby increase the concentration of silver ions in the effluent during days 9 and 10. After the SMT was completed it was determined experimentally that the heater raised the effluent temperature to 118 to 122°F; as shown in Section 4.4, this heating increased the silver dose from about 0.07 ppm to 1.2 ppm.

C. On days 9 and 10, $3 \pm 1 \times 10^9$ B. subtilis cells were injected upstream of the AgCl Column during the first hour of operation to simulate a failure of the BF, and to determine if higher concentrations of silver ions could kill these spores.

Table 8 shows that the first two days of this test duplicated the results of the previous tests - namely, (1) the simulant does not significantly enhance or impede the concentration of B. subtilis spores, and (2) if only the input is contaminated, water drawn from the storage tank and both outlet valves is essentially sterile (plate count < 1/200 ml). However, when $3 \pm 1 \times 10^9$ spores were injected upstream of the ACF and the AgCl Column on days 3 and 4, respectively, all samples drawn from the injection septum, outlet valves, and intermediate septums were found to be contaminated. Thus, the concentration of silver ions in the system was not sufficient to effectively kill these spores under the test conditions.

The spore concentrations presented in Table 8 for days 3, 4 and 5 indicate that the spores were diluted and "swept" through the system from the injection point to the outlet valves. However, the low counts at the outlet valves on days 3 and 4 indicate that they "hang-up" on surfaces in the deionizer and/or AgBr Column - so that the fully diluted concentration ($3 \pm 1 \times 10^4$ ml) was not detected in the product water (OV-1 and OV-2) until nearly three days after initial contamination. This probable effect of surfaces on spore retention also may explain why the S-5 samples taken on day 5 had spore concentrations greater than 10^4 ml - i.e., spores from the previous injections caused the concentrations to be greater than the fully-diluted concentration.

During days 6, 7 and 8 samples drawn from S-10 indicated that the BF was preventing further contamination of the system, while the sterile simulant (after the BF) was flushing the previously injected spores from the system. The S-5 counts indicate that during these days the spore concentration was reduced to the point that on day 8 the 21 hour sample was essentially sterile (plate count < 1/200 ml). The concentration of spores in the OV-1 samples also decreased with time, but because of the large surface area afforded by the deionizer and the AgBr Column, all of the spores were not flushed out by the end of day 8. The relatively consistent counts in the OV-2 sample indicates that the spores may have even germinated at this outlet.

On days 9 and 10, when the AgCl Column was heated to about 120°F to obtain a silver dose of 1.2 ppm, and $3 \pm 1 \times 10^9$ spores were injected upstream of the AgCl Column, the 4 hour S-5 samples were essentially sterile - but the 21 hour S-5 samples contained 32 cells/100 ml. Thus, the combined effect of a very small amount of heat and higher silver doses yielded a kill rate greater than 10^4 to 10^0, or a 4 log reduction in less than 21 hours. The outlet samples, of course, were not sterile because of residual contamination and the low concentration of silver between the deionizer and the outlet valves.

4.4 Water Quality

Samples of the simulant, stored water and product water were analyzed once each day for the values of the "key" quality parameters, and periodic samples of the product water were forwarded to the NASA MSC for detailed analyses. The results of these analyses are summarized in Appendixes C & D.

Two quality problems persisted during each of the five SMT's, because the simulant contained contaminants which unexpectedly affected the performance of the ACF and the deionizer. The first problem recognized was the excessively low pH and specific resistance of the product water during SMT #1 (see Table C1). From the detailed analyses of the SMT #1 product water (see Table D1) it was concluded that the deionizer was unable to remove (1) the hydroflouric acid added to the simulant to obtain a flouride level of 1.6 ppm, and (2) the chloride added to obtain a level of 8 ppm. On the other hand, the silver concentration was found to be too low - indicating that the chloride ions were suppressing the equilibrium concentration of Ag.

For SMT #2 the positions of the cationic and anionic resins were reversed. Tables C2 and D1 indicate that this change improved the pH, specific resistance, chloride content and flouride content of the product water - but the pH was still

too low, and the silver content was too high. For SMT's #3, 4 and 5 the contents of the deionizer were "layered" and the resin ratio varied to determine if further improvements were possible with the preselected resins. As shown in Tables C3, C4, C5, D2 and D3 - low pH, high fluoride content, and high silver content continued to be a problem. Since the pH tended to decrease while the fluoride content increased during each of these tests, it is concluded that the preselected resins had insufficient capacity for hydroflouric acid. Since the silver contents were higher than predicted by equilibrium calculations, especially in the early part of each test, it is concluded that residual amines which evolved during presterilization of the resins were swept into the AgBr Column where they increased the solubility for silver by a chelation effect. Other resins were not investigated because an extensive amount of bench experiments would have been required to determine if any other commercially-available resins could withstand the mandatory presterilization procedure and also have more capacity for flourides than the preselected resins.

The second persistent problem was the Total Organic Carbon (TOC) of the stored water and the product water. As indicated by the data in Appendixes C & D, the ACF's had a negligible effect on this parameter until SMT #3 and 5 - and even then the TOC removal efficiency was on the order of 30% instead of the design goal of 60%. Based upon the noticeably sweet taste of the stored water and the product water it was concluded that the ACF's were unable to adsorb the ethylene glycol added to the simulant to achieve part of the desired organic contamination. If none of the ethylene glycol was adsorbed, and 60% of the butylated hydroxytoluene (the other organic contaminant) was adsorbed, an overall removal efficiency of 30% would be expected. This problem, was recognized during SMT #1, but no changes were made to the ACF's until SMT #5 because (1) no other organic contaminants were suggested, and (2) a survey of activated carbon manufacturers indicated that none of them "claim" to have a material which effectively adsorbs ethylene glycol from water. For SMT #5 a different brand was tried - but the results were essentially the same as before.

Another problem which occurred, but was rectified, was high alkalinity of the stored water. During SMT #1 the level was higher than for the simulant. Consequently, it was concluded that the ACF was not washed sufficiently during preparation. For the subsequent tests the washing operation was improved until the alkalinity of the stored water was less than the level in the simulant (see Table C5).

Finally, the concentration of silver in the stored water was always found to be less than the desired minimum level of 1 ppm - until the AgCl Column was heated to about 120°F during days 8, 9 and 10 of SMT #5. This result is attributed to the presence of chloride ions in the simulant, which passed through the ACF and suppressed the equilibrium concentration of silver ions. The following calculation shows that under these conditions the silver ion concentration should be only 0.075 ppm.

$$C_{Ag^+ @ 25^oC} = \frac{K_{sp @ 25^oC}}{C_{Cl^- @ 25^oC}} = \frac{1.56 \times 10^{-10}}{2.25 \times 10^{-4}} = 6.96 \times 10^{-7} \frac{\text{moles}}{\text{liter}}$$

$$C_{Ag^+ @ 25^oC} = 6.96 \times 10^{-7} \times 107.9 = 7.5 \times 10^{-5} \frac{\text{grams}}{\text{liter}} = 0.075 \text{ ppm}$$

A review of Appendix C indicates that the measured concentration in samples of stored water ranged from 0.04 to 0.12 ppm - until the AgCl Column was heated to raise the concentration to 1.21 ppm.

4.5 Materials Evaluation

The construction materials used in the Breadboard System were either austenitic stainless steel or non-metallics. Specifically, the custom-designed components were fabricated from type 316 SS as were the valves, lines and flow meter connectors. The storage tanks, large BF housing and wetted parts of the pressure gauges and thermometers were type 304 SS. The small BF housing was Teflon-coated aluminum.

Evidence of corrosion was associated only with the biological filters. The small BF housing had a mounting boss on the inside of the end cap for attachment of the filter cartridge. During assembly, it was nearly impossible not to abuse the Teflon coating on the end cap boss when inserting the filter cartridge. This boss corroded badly as evidenced by the readily apparent loss of aluminum making up the boss.

The large BF housing, consisting of a cast 304 SS head and a drawn 304 SS bowl, had rust stained areas on the head. However, the bowl portion revealed no evidence of corrosive attack - indicating that drawn 304 SS is acceptable while cast 304 SS is unacceptable.

Visual inspection and hydraulic pressure tests failed to reveal any evidence of corrosion or degradation in the custom-designed components. The extensive water analysis effort directed to detection of stainless steel corrosion products (iron, nickel, manganese and chromium) indicated the absence of

stainless steel corrosion in any of the five tests. It should be pointed out that even though chromium was a simulant ingredient, the chromium concentration in any of the water samples never exceeded the concentrations directly attributable to the chromium added to the simulant.

The immersion tests conducted on 410 SS and A-55 titanium did not evoke dramatic changes in the material. Coupons of these materials were immersed in simulant which had been pumped through the biological filter, charcoal column and silver chloride column under both oxygen and nitrogen atmospheres. Table 9 presents the weight change data obtained from the immersion tests. All metal coupons gained weight, but the gain was limited to one milligram or less. The only defects or changes detectable upon visual examination were limited to several, small, localized "rust" spots on the 410 SS coupons joined in the crevice configuration and tested under nitrogen. Analysis of immersion tank water did not reveal any stainless steel corrosion products in excess of that already in the simulant. However, the 410 SS coupons gained less weight than the titanium coupons - and all coupons gained less weight when pressurized with nitrogen.

The elastomeric materials - namely, polyisoprene silicone, Buna-N, butyl, ethylene-propylene, and Viton - were subjected to the same immersion tests described above. However, control coupons identical to those used in the immersion tests were also soaked in deionized water under air in 1/2 gallon glass bottles. With the exception of butyl rubber, the percentage weight change detected in the test coupons did not differ any more than 0.05 units from the percentage weight change detected in the controls. Butyl rubber gained 0.2% in weight while the controls lost an average of .04%. Viton and Buna-N gained about 0.25% in weight as did their respective controls. Ethylene-propylene and polyisoprene both gained 0.1 % or less while silicone lost weight by a similar margin. The essential point is that deionized water produced essentially the same effect on the elastomers as the "stored" water with the possible exception of butyl rubber. Visual examination, which included manual flexing and stretching, did not reveal any physical degradation. However, the data in Table 9 does indicate that polyisoprene, silicone rubber and EPT experienced less weight change than the other elastomers - regardless of the test solution or ullage gas.

The silver concentration of the immersion tank water was always slightly less than the typical 0.07 ppm silver detected in the stored water; the magnitude of silver loss was 0.01 to 0.02 ppm. Pressurant gas appeared to have no effect on silver dose stability. The cause of the silver loss is obscure and no single material can be clearly identified as a silver

Table 9 SUMMARY OF MATERIAL SOAK TESTS

Pressurant Solvent	% Change by Weight Atmosphere Deionized Water	Nitrogen Simulant	Oxygen Simulant
Metals			
410 Stainless Steel	---	+.0004	+.0007
	---	+.0007	+.0009
	---	+.0011	+.0031
A-55 Titanium	---	+.001	+.017
	---	+.006	+.032
	---	+.005	+.032
Elastomers			
Polyisoprene	-.040	-.158	---
	+.052	---	+.015
Silicone	-.014	---	+.121
	-.028	-.025	---
Buna-N	+.260	---	+.542
	+.114	+.117	---
Butyl	-.256	---	+.223
	-.032	-.064	---
EPT	+.062	---	+.098
	+.132	+.093	---
Viton	+.231	---	+.301
	+.269	+.285	---

ion "sorber". However, the immersion tank, which was fabricated from 316 SS, provided a wetted area at least two times greater than the wetted area provided by all coupons at one time. This tank was cleaned in laboratory detergent before each SMT. Thus, the tank itself may have adsorbed the small amount of silver lost over a seven day period.

4.6 Component Performance

The components used in the breadboard system proved to be relatively easy to fill, prepare and install, and no "leaks" were observed during the five SMT's. However, before the 1st SMT there was some concern about the flow and pressure pulsations produced by the diaphragm-type feed pump. Also during the first four SMT's, when the small BF was used, the pressure drop across that component appeared to be excessively high near the end of each test. The diaphragm-type feed pump was retained to facilitate unattended operation because its output was found to be less sensitive to small changes in back-pressure than a gear-type pump which was also evaluated; in addition, a pulsating flow was considered to be a desirable "extreme" test condition. A larger BF was used during the 5th SMT.

After the 5th SMT was completed all of the filters and canisters used in that test, and the small BF and the ACF used in SMT #4, were operated at different flow rates to determine their actual pressure drop characteristics. A mercury manometer, deionized water, a rotameter and a bench-type set-up were used to obtain this data. Figures 5, 6 and 7 present the results of these tests.

Figure 5 indicates that the large BF has substantially less resistance to flow than the smaller BF, even though the larger filter was onstream for 10 instead of 7 days. This result, of course, was anticipated because the larger BF has more "dirt" capacity. In both cases, this data must be considered as being the mirimum pressure drop for the Space Shuttle because sediment was found in the bottom of the BF housings after each test - and under weightless conditions these particulates would be drawn instead to the face of the filter cartridge and thereby increase the rate of surface fouling.

Figure 6 indicates that the flow resistances of the AgCl column, SMT #4 ACF and SMT #5 ACF are negligible - even if they are "on-stream" for 38, 7 and 10 days, respectively. The finer charcoal (20 x 50 mesh), of course, offers more resistance to flow than the coarser charcoal (12 x 20 mesh).

Figure 7 shows that the pressure drop across the deionizer and the AgBr Column is significant - but not unduly so considering the high flow rates anticipated when water is drawn for use.

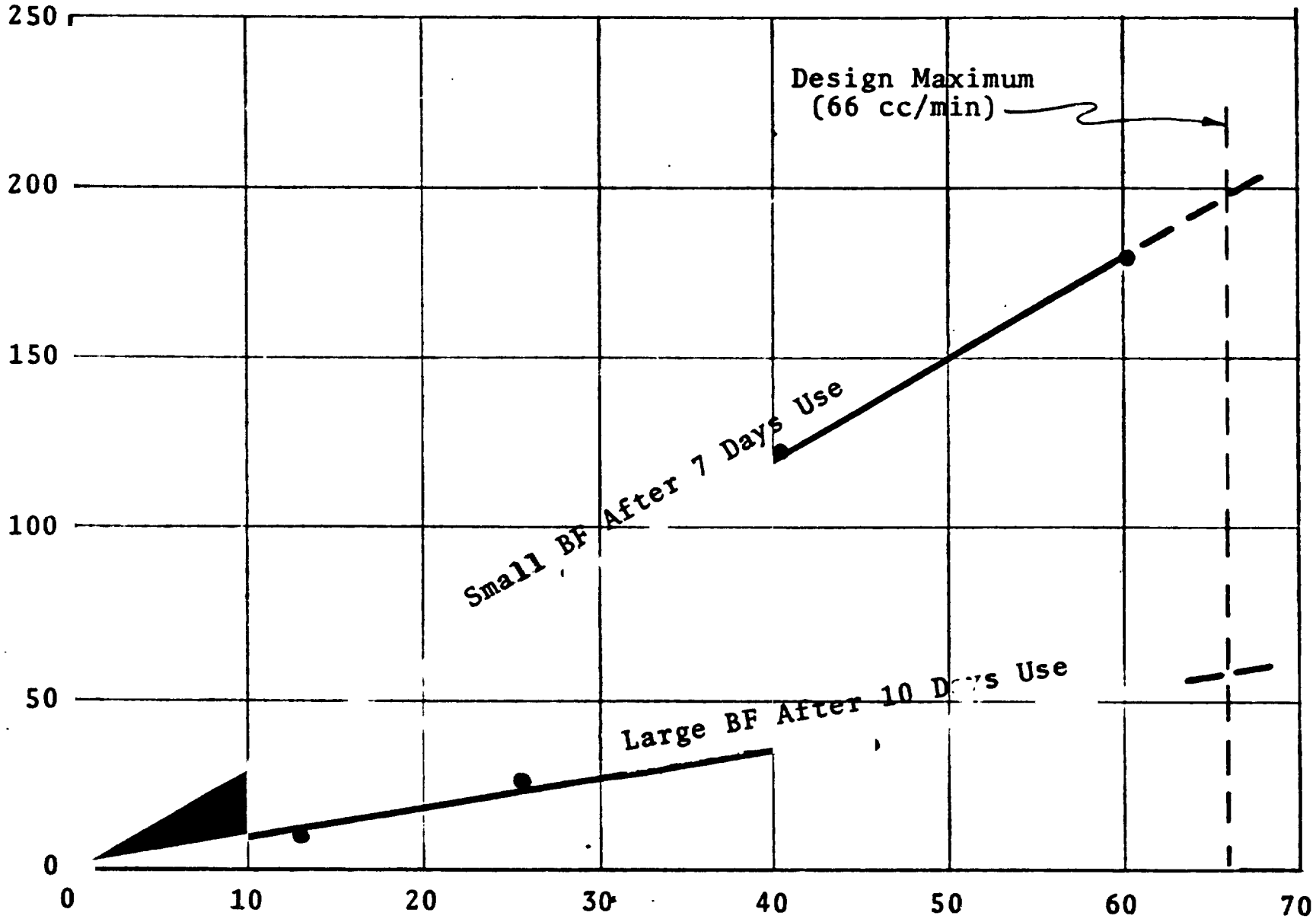

Figure 5 FLOW RESISTANCE OF SMALL AND LARGE BIOLOGICAL FILTERS

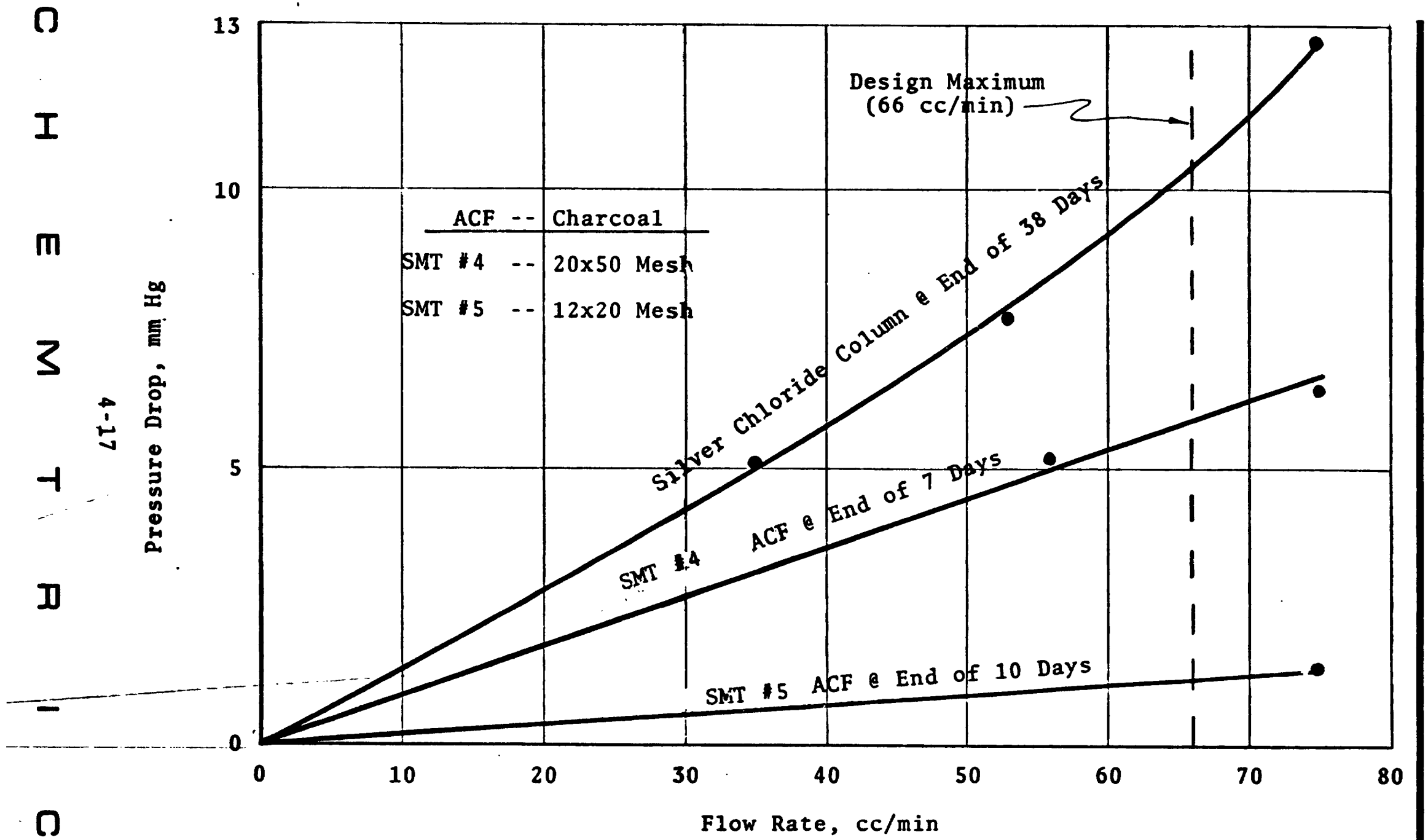

Figure 6 FLOW RESISTANCE OF SILVER CHLORIDE COLUMN AND ACTIVATED CARBON FILTERS

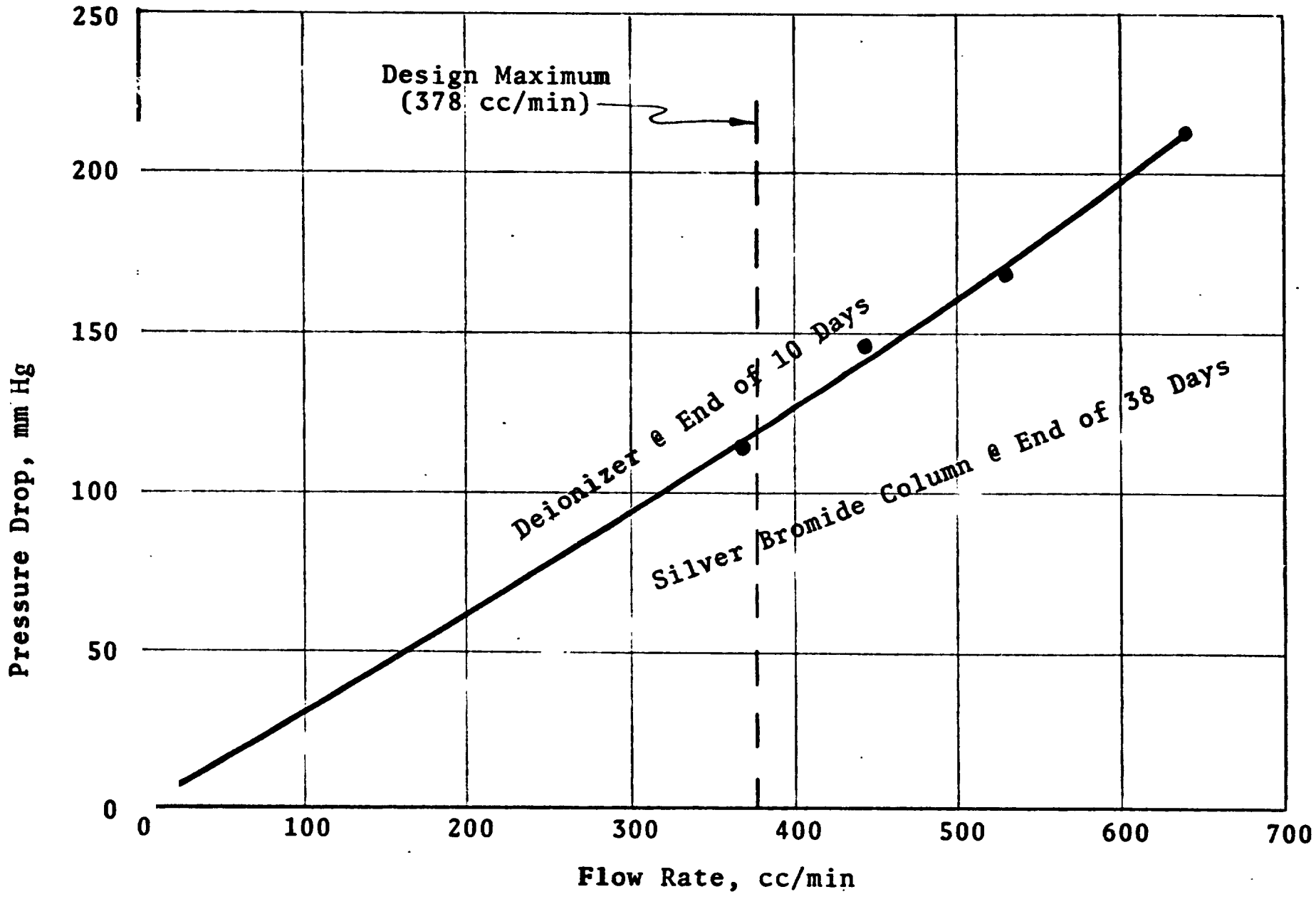

Figure 7 FLOW RESISTANCE OF DEIONIZER AND SILVER BROMIDE COLUMN

SECTION 5

GENERAL DISCUSSION

5.1 Bacteria Challenges

In the first four SMT's, silver ions were repeatedly challenged with bacteria doses containing $3 \pm 1 \times 10^9$ bacteria of the type previously found in spacecraft water systems. This number of bacteria upon maximum dilution would provide a concentration of 3×10^4 bacteria per ml. After exposure to silver ions, plate count data indicated less than one viable cell per 200 ml. This decrease amounts to more than a 6 log reduction in viable bacteria. Based on the challenges conducted on day 4 of each of these four SMT's, the bacteria were killed by a single pass through the AgCl Column. Although the breadboard system was not ideally suited for establishing exposure time requirements, it is evident that Type IIIa and the Pseudomonas species were killed in less than 4 hours.

The above performance is most remarkable in light of the depressed silver ion dose. The depressed silver concentration is readily accounted for by the common ion effect afforded by the 8 ppm chloride in the simulant. Since chloride is a common water contaminant, any bactericide must be capable of performing in the presence of chloride ions.

The challenges carried out on day 7 of SMT #1 through 4 evaluated the AgBr Column. When the bacteria ($3 \pm 1 \times 10^9$) were injected so that they were exposed to the AgBr overnight (20 to 21 hours), sterile product water was obtained. In SMT #2 and #3, the injected bacteria were pushed back into the deionizer where the silver ion concentration would be extremely small if not zero. When product water was drawn off 20 to 21 hours after injection, the bacteria moved rapidly through the AgBr Column. In these instances, the AgBr Column was simply overwhelmed. Using the plate count data and the total volume of product water (63 liters), approximately 10^7 of the 3×10^9 bacteria injected survived AgBr treatment. However, this apparent reduction in viable bacteria cannot be attributed exclusively to the silver bromide dose since many bacteria could have been trapped within the system.

During SMT #1 through 4, ample opportunity existed for contamination to develop in the portion of the system downstream of the AgBr Column. The laboratory in which the tests were conducted was immediately adjacent to the laboratory wherein an Integrated Water and Waste Management System was being evaluated. Prior experience has shown that unprotected water bacteria level one would expect in a waste management evaluation laboratory, and the demonstrated ease with which bacteria can invade an outlet valve, AgBr would appear to have done a creditable job in protecting the system. Only two isolated episodes

of outside bacteria contamination were encountered; in both cases a single sample was involved indicating that the bacteria may well have been introduced into the sample at the time of acquisition.

The last SMT, using B. subtilis spores, presented a severe challenge of the system. It is clear from the test results that the biological filter excluded bacillus spores from the system quite effectively. It is also clear that the silver dose realized with 8 ppm chloride was ineffective in fending off the spores injected downstream of the filter. In light of this, it is not surprising that the AgBr dose was incapable of clearing the outlet portion of the system once the spores penetrated beyond the deionizer and AgBr Column.

On days 9 and 10 of the last SMT, the silver dose was elevated to 1.2 ppm (17 times the previous silver dose) by heating the AgCl Column to 120 ± 2°F. As indicated in the test results, a 4 log reduction was realized in four to 21 hours of exposure. The exposure time cannot be fixed beyond the admittedly broad time span; additional samples and analyses would be required to establish an exact kill rate. The rate of the mildly elevated temperature may or may not have been significant in the kill rate observed. The temperature involved is well below the 80 to 100°C temperatures normally used in heat shocking (a method used to cause spore germination). The heated water was accumulated in the storage tanks; the temperature of the water drawn off 21 hours later ranged from 80 to 90°F. Thus, the spores were exposed to a temperature more conducive to spore germination than room temperature. This incubator effect may have been quite significant. In any event, the potential complimentary effect of heat in the kill-off seen during days 9 and 10 cannot be ruled out.

The test results suggest the need for certain design features to be incorporated into the system. The line connecting the AgBr and ion-exchange columns should be welded to the canisters so that positive means are provided to eliminate the probability of outside bacteria entering this sensitive segment of the system. If spore forming bacteria are regarded as a real threat, some means in addition to AgBr must be devised to protect the outlet segment of the system from backward invasion from the outlet valves. Switching outlet lines each day from cold water to hot water (160°F) should be effective.

5.2 Water Quality

The problem areas encountered with product water quality were organic contamination, pH, fluoride and silver; these problems were related to performance of specific components. Excluding the above, contaminant levels were below the most stringent standards for the species analyzed.

The organic contamination was due to ethylene glycol. This substance was detected by its sweet taste in both the ACF effluent and product water. In the last SMT, a different brand of charcoal was used with only a slight improvement. Consultation with several major activated carbon manufacturers revealed that highly water soluble low molecular weight organics are not adsorbed to any real extent by water-type carbons. An activated carbon, such as those used in gas adsorbtion systems, with radically different pore sizes may be useful, but specific tests must be performed to evaluate the suitability of such carbons. Positive means must be provided in the fuel cell design to eliminate any chance for ethylene glycol entering the water supplied to the bactericide system.

The remaining water quality problems are attributable to the deionizer and the resins used. The problems encountered, namely removal of low atomic weight species and resin derived organic contamination, are not unusual for resin beds.

In regard to pH, the product water was so pure that little buffering capability was available. Consequently, small quantities of acid, specifically hydrofluoric, produced dramatic shifts in the pH of the product water. This conclusion was verified by total acidity determinations, which indicated that the product water contained only a small amount of "free" acidity. To eliminate this problem it is necessary to utilize an ion-exchange resin which has a higher affinity for hydrofluoric acid than the weak base-type resin used in these tests.

The silver ion dose obtained from the AgBr Column was always high the first day of operation and in some cases very high (0.32 ppm as opposed to the .07 ppm expected); a definite downward trend was established with time on stream. This suggests a resin derived contaminant, most probably amines, which interferred with the normal AgBr - Ag^+ equilibrium. Amines are capable of interferring with the equilibrium by chelating silver - and are known to be released by anionic exchanges. The resins were washed extensively before packing in the deionizer. However, the sterilization procedure may have caused some resin degradation, or the resins were mechanically abused by the rapid cool down of the packed deionizer after sterilization.

Several attempts were made to correct the above mentioned problems during the tests. Ion-exchange columns were prepared with increasing anion to cation resin volume ratios in an attempt to correct acid component leakage. The two resin types also were alternated with respect to the outlet to eliminate amine leakage. A resin bed was prepared with small alternating layers of cationic and anionic resins. The best performance obtained was in SMT 4; the resin bed in this test was packed

with the cationic resin at the outlet with a 1.4 to 1 ratio of anionic to cationic resin. In this test, the initial silver concentration was the lowest of any tested, and the flouride leakage was acceptable (below 1 ppm) - but the pH was above 6 for only the first two days.

On the basis of the experience to date, it is apparent that substantial improvement in DI performance could be achieved if a strong base resin were used. However, the resin must have the high temperature (160 to 180°F) resistance necessary to survive long term exposure to pasteurization temperatures. In consulations with resin manufacturers, some alternate resins were recommended for evaluation.

The problems encountered with the DI unit can be solved with the resins used by increasing the anionic to cationic ratio. However, this ratio must be established by test and will probably require a larger deionizer canister. The untried resins recommended by manufacturers, also offer a potential solution. In either case, further laboratory evaluation of resins is warranted.

5.3 Construction Materials

Austenitic stainless steel, specifically 316, is the recommended material. Type 304 appeared to perform equally well except for the cast material. Titanium can not be rejected on the basis of the tests conducted. This material does offer the substantial weight savings over 316 SS. Of course, the suitability of titanium for use in pure oxygen environments must be resolved.

The elastomeric materials tested were all qualified by seal and "O" ring manufacturers as suitable for use in water systems. On the basis of the tests conducted the following order of preference is recommended: (1) polyisoprene, (2) ethylene-propylene, (3) silicone, (4) Viton, (5) butyl and (6) Buna N.

5.4 Component Design

The design of the custom components appeared to be more than acceptable. No hardware design deficiencies were encountered. The pressure drop data obtained on the canisters indicates a very acceptable level of flow resistance considering the nature of the packed columns.

The performance of the standard housing used for the biological filters was less than desired; however, the large cartridge or filter element performed quite well. A customized housing for the large filter element should be undertaken.

The useful life of the biological filter is of course dependent on the life of the cartridge. As indicated earlier, one-g evaluation of the filter results in solids settling out rather than being dynamically removed by the filter cartridge. The large filter was on stream for 10 days and still incurred only a 1.2 psi pressure drop, but some solid materials had settled to the bottom of the housing. Consequently, the potential for filter plugging in zero-g must be considered. The system should provide for by-passing the filter or back flushing to eliminate plugging.

5.5 Flushing Fluid and Drying Gas

Between tests the system was successively flushed with (1) a 50 ppm hypochlorite solution, (2) deionized water, and (3) deionized water saturated with silver chloride ions. Since this procedure did not cause any deleterious effects it is concluded that this procedure should be acceptable for refurbishing the Space Shuttle potable water system.

Both nitrogen and oxygen appear to be acceptable gases for pressurizing and drying the Space Shuttle potable water system - because they are compatible with the recommended construction materials, and they do not affect the bactericidal efficacy of silver ions.

APPENDIX "A"

TENTATIVE WORST-CASE FUEL CELL WATER COMPOSITION

TENTATIVE WORST-CASE FUEL CELL WATER COMPOSITION[1]

Constituent	Level (ppm)
Total Solids	500
Total Organics	$\angle$100
pH	6.0 - 8.0 @ 77°F
Particulates	
0-10μ	Unlimited[2]
10-25μ	875
50-100μ	100
$>$ 100μ	2
Ionic Species	
Cadmium	0.01
Chromium (Hexavalent)	0.05
Copper	1.0
Iron	0.3
Lead	0.05
Manganese	0.05
Mercury	0.005
Nickel	0.05
Silver	0.05
Zinc	5.0
Selenium	0.05
Magnesium	0.17
Potassium	0.54
Sodium	3.3

Constituent	Level (ppm)
Sulfate	< 50
Fluoride	1.6
Nitrate	0.04
Titanium	0.2
Platinum	0.05
Chloride	< 25
Silica	20
Dissolved hydrogen	Saturated
Alkalinity	>30
Microbiological	
Coliform organisms	10,000/100 ml
Fecal coliforms	2,000/100 ml

Note 1: The water composition is based on available fuel cell data,and "Water Quality Criteria" Report of the National Technical Advisory Committee to the Secretary of the Interior, April 1, 1968.

Note 2: Unlimited means that particles in this size range are not counted; however, any obscuring of the filter grid lines shall be cause for rejection.

APPENDIX "B"

COMPONENT PREPARATION PROCEDURES

Biological Filter
Activated Carbon Filter
Deionizer
Silver Halide Columns

BIOLOGICAL FILTER

The cartridges in these filters were cleaned before use to remove water-soluble manufacturing residues. The cleaning procedure consisted of multiple extractions in boiling deionized water. In these extractions a cartridge was placed in a metal pan with 2 to 3 liters of water and heated to boiling followed by draining and air cooling of the cartridge. These extractions were repeated at least three times or until no visible color was detectable in the hot water. The cartridge was then installed in the housing which in turn was connected to a deionized (DI) water source and flushed at 0.25 to 0.5 gpm for 30 minutes. The water-laden filter assembly was then allowed to stand idle for 30 minutes after which the water in the housing was drained-off. The flushing process was repeated until the pH and resistance of the drainings were in close agreement with the values obtained for the DI water used in flushing. The final step preparatory to packing with the AgCl glass bead mixture was steam sterilization at 115°C for 15 minutes. This sterilization step was primarily precautionary in the event the in-house DI water system contained viable bacteria.

The final steps include packing the hollow cartridge core with AgCl-glass bead mixture identical with that used in the AgCl Column. The mixture was added wet in small quantities while tapping the cartridge bottom agains a hard surface; water was periodically sprayed down the core to aid in packing. Two discs of 10 mesh screen were then placed on top of the mixture followed by a 1/4-inch thick layer of pyrex wool, and finally another disc of 10 mesh screen. Next, the packed cartridge was installed in the housing. After assembly, the filter was again connected to the DI water source and flushed at 0.25 to 0.5 gpm until the pH of the filter effluent was identical to that of the influent.

ACTIVATED CARBON FILTER

Charcoal requires extensive washing before use to eliminate water soluble substances which can amount to as much as 3% of the dry weight of the charcoal. These soluble substances are mostly minerals (primarily chloride and carbonate salts). Charcoal can also contain a large variety of undesirable metals.

Charcoal preparation consisted of (1) treatment with dilute hydrochloric acid (HCl), (2) extensive washing in hot tap water, (3) washing in deionized water, and (4) steam sterilization. The following paragraphs describe these steps in detail.

The purpose of acid addition is to neutralize the alkaline substances in charcoal. Previous experience with the Barnaby-Cheney (B/C) charcoal had shown that 25 ml of concentrated HCl per pound of charcoal is adequate for neutralization. However, for this program, 45 ml of concentrated HCl per pound of charcoal was found to be necessary.

The charcoal was first mixed with a volume of deionized water equal to twice the charcoal volume. The acid was added slowly while stirring vigorously. Addition of the acid resulted in gas evolution, which gradually diminished with time. A major component of this gas is hydrogen sulfide which was readily identified by its characteristic "rotten-egg" odor. The mixture was stirred for 15 - 20 minutes or until gas evolution ceased. The supernatant liquid was then decanted off and the charcoal added to a flotation tower.

The flotation tower used is a standard polyethylene pipette washing jar which has a diameter of 6-inches, and a height of 31-inches. Wash water was introduced through the side by means of a 3/8-inch bulk-head tube fitting located approximately one inch from the bottom. A media retainer fabricated from 10 and 100 mesh screen was mounted approximately three inches from the bottom. Wash water flow rates of 1 - 2 gpm expanded the charcoal to approximately 6 times the settled or compacted volume, thereby providing vigorous mixing with the incoming fresh wash water.

The acid-treated charcoal was then washed with 130 to 160°F tap water for 20 to 30 minutes with the above flow rate. The water was then turned off and the water within the tower allowed to drain back through the charcoal and out through the wash water inlet. The last 500 ml of the drainings were measured for pH and specific resistance. Washing with tap water was continued until the drainings matched the tap water with respect to pH and specific resistance. The above process was then repeated with deionized water of at least one megaohm purity.

Once the charcoal had been washed, it was steam sterilized at 115°C (250°F). The time at sterilization temperature was adjusted to match the quantity and configuration of the charcoal. Small batches of charcoal were sterilized in 45 minutes at the above temperature. When sterilized in large jars or bottles, approximately 45 minutes per pound of charcoal was required.

Final assembly of the canister entails (1) loading or packing of the charcoal, (2) assembly of media compression and end cap, and (3) sterilization/decontamination.

A clean canister body was mounted in a ring stand with the fixed end cap down. A piece of Tygon tubing was then connected to the end cap tubing connector. Next, the screen discs and pyrex wadding were inserted inside the canister against the fixed end cap, and several hundred ml of deionized water poured into the canister; the Tygon tubing was elevated sufficiently to keep the water from draining out of the canister. The charcoal was then added in 300 to 400 ml quantities while wet. With each addition, the Tygon tubing was lowered to allow water to drain out while tapping the canister/ring stand assembly against a hard surface. Packing was continued until 3000 ± 50 ml of charcoal was added. The charcoal was covered by water at all times to prevent the inclusion of air. The screens, pyrex wadding and spring retainer were then placed on top of the charcoal. At this point, the spring retainer was approximately 1/2 inch below the edge of the canister body, and the spring was placed in position. Next, the end cap with the "O" ring held in place by a thin coating of silicone grease was placed on the spring - and the end cap forced down and held while the "V" band was installed.

Sterilization/decontamination can be effected by either steam sterilization as noted previously or long term (18 - 24 hours) exposure to pasteurization temperatures. Because of the size of the charcoal canister, the latter decontamination procedure was used.

The equipment used for decontamination is as follows: (1) a constant temperature water bath with agitator, (2) a coil of 304 SS, 1/4-inch OD tubing with an equivalent linear length of 30 ft., (3) a washed and sterilized biological filter, and (4) a length of steam sterilized silicone tubing. The biological filter, silicone tubing and coil were connected together and steam sterilized as a unit.

Decontamination was initiated by connecting the biological filter inlet to the deionized water source, and using the silicone tubing to connect the filter outlet with the coil. After the connections were made, deionized water was run through the filter, tubing and coil at 0.25 to 0.5 gpm to displace any trapped air. The coil outlet was connected to the charcoal canister inlet; a length of flexible tubing was then connected to the canister outlet. The coil and canister were immersed in the water bath, and the tubing connected to the canister outlet was routed to a convenient drain. Deionized water was turned on and regulated down to 30 to 50 ml/min, and the water bath controls were adjusted to maintain a temperature in the range of 180 to 200°F. After 18 to 24 hours exposure, the canisters were found to be sterile.

DEIONIZER COLUMN

The resins used require very extensive preparation to eliminate manufacturing residues. Previous experience has indicated that the as-received resins are biologically contaminated - thereby requiring sterilization and decontamination. The following paragraphs describe these washing and sterilization procedures.

Each resin type was washed separately. The initial step consisted of multiple extractions in boiling, very high quality (2 to 3 megaohm) deionized water. In the above process, a resin quantity was mixed with at least five volumes of water in a pyrex glass beaker and heated to boiling. The mixture was boiled for five to ten minutes, after which the container was removed from the heat source and the resins allowed to settle. The supernatant liquid was decanted-off and fresh deionized water added immediately. The resins were again allowed to settle and the water decanted-off and fresh deionized water added again. The above process was repeated at least ten times or until all traces of color and taste were eliminated.

The manipulative procedures for packing the deionizer column were essentially the same as those previously described for charcoal packing. The main requirements were (1) careful measurement of the resin volumes added, (2) adequate vibration of the canister during resin addition, and (3) keeping the resins not only wet but covered by a layer of water.

Deionizer packing differed from charcoal packing in that two types of material were added, and that additional 10 mesh screen discs are used to separate the different resins. Since there are two different materials, there was a preferred orientation of the resin types with respect to the inlet; the strong acid type normally was packed at the inlet end.

Sterilization/decontamination of the deionizer canister was carried out at 180°F over a period of 18 to 24 hours. The apparatus, procedures and operating conditions used were the same as for the activated carbon filter.

SILVER HALIDE COLUMNS

Preparation of the silver halide media entailed grinding the "as-received" silver salts, sizing the ground material with standard sieves, washing the glass beads, and finally mixing the glass beads with the ground silver salts in the specified 1 - 1.25 ratio.

Both AgCl and AgBr (reagent grade) were obtained from Fisher Scientific. The "as-received" material contains granules ranging from dust sized particles to golf ball sized lumps. Although Melpar's tests indicated that careful particle sizing was not required, some degree of uniformity is desirable. Material that passed through a 45 mesh (Tyler) screen was rejected while material retained by a 6 mesh screen was reground.

The silver salts can be photoreduced and consequently care was taken not to expose them to direct light. The grinding and sizing procedure was done at CHEMTRIC under subdued lighting.

The AgCl solid is somewhat ductile and hammering with a pestile was found to be more productive than grinding. The AgBr solid was, on the other hand, readily ground.

The glass beads (450 - 500 microns) were obtained from Sargent-Welch Scientific under catalog number S-61760-30D. The as-received beads were washed in a dilute aqueous solution of laboratory detergent (Alconox) followed by rinsing in hot tap and deionized water. The beads were then added to concentrated chromic acid; the chromic acid was heated to boiling and allowed to "simmer" for one hour. After cooling, the glass beads were washed in deionized water and dried in a 103°C oven overnight.

The glass beads and ground silver salts were blended by weight in the ratio of 1.25 parts glass beads to one part silver salt. Once the proper proportions were weighed-out, the ingredients were added to a common container (preferably glass) along with a volume of water just equal to the volume of the ingredients. The ingredients were stirred manually to achieve uniform distribution of glass beads and silver halide particles.

The packing procedure for loading the canister with the above mixture was somewhat different from that used in packing charcoal or ion-exchange resins. The main difference is that the mixture was loaded into the canister without excess water.

If the canister contained water, the silver halide particles and glass beads would settle to the bottom at different rates, thereby producing stratification. The mixture was added in 50 to 100 ml increments while tapping the canister outlet against a hard surface.

When the packing and assembly were complete, the canister was connected to a deionized water source and flushed at 0.5 to 1.0 gpm for 5 to 10 minutes. The effluent was analyzed for silver content and turbidity, to insure that the column saturated the water with silver ions, and no "fines" were escaping.

APPENDIX C

SUMMARY OF DAILY WATER ANALYSES

C H E M T R I C

Table C1 SUMMARY OF DAILY WATER QUALITY ANALYSES FOR SMT #1

Test Day No.	Sample Water	pH	Sp. Res. (kΩ-cm)	Turbidity (JTU's)	Alkalinity (ppm)	Cl (ppm)	Cr (+6) (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Ag (ppm)	TOC* (ppm)	TDS (ppm)
1	Simulant	7.1	24.5	8.3	5.0	8.1	0.08	<0.1	<0.05	<0.2	<0.03	--	9
	Stored	6.7	17.5	1.1	31.0	8.1	0.06	<0.1	<0.05	<0.2	0.08	31.5	97
	Product	3.9	11.0	<0.1	<1.0	6.5	<0.05	<0.1	<0.05	<0.2	0.06	32.0	1
2	Simulant	7.1	24.5	8.2	5.0	8.1	0.06	<0.1	<0.05	<0.2	<0.03	--	30
	Stored	6.7	17.1	0.5	31.0	8.1	0.05	<0.1	<0.05	<0.2	0.06	20.0	87
	Product	3.5	7.0	<0.1	<1.0	8.2	<0.01	<0.1	<0.05	<0.2	0.06	--	0
3	Simulant	7.1	25.5	7.9	3.0	8.1	0.07	<0.1	<0.05	<0.2	<0.05	--	18
	Stored	6.9	20.0	0.4	29.0	7.5	0.05	<0.1	<0.05	<0.2	0.07	36.3	14
	Product	3.6	8.4	<0.1	<1.0	8.0	<0.05	<0.1	<0.05	<0.2	0.06	37.0	0
4	Simulant	7.1	25.4	8.1	4.0	8.1	0.07	<0.1	<0.05	<0.2	<0.05	--	21
	Stored	6.8	18.3	0.7	28.5	8.1	0.08	<0.1	<0.05	<0.2	0.08	42.5	10
	Product	3.6	7.7	<0.1	<1.0	8.1	<0.05	<0.1	<0.05	<0.2	0.05	--	0
5	Simulant	7.4	26.0	8.0	4.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	--	45
	Stored	7.1	17.3	0.5	26.0	8.1	0.07	<0.1	<0.05	<0.2	0.08	50.0	15
	Product	3.6	8.4	<0.1	<1.0	8.1	<0.05	<0.1	<0.05	<0.2	0.05	--	0
6	Simulant	7.1	25.5	8.2	5.0	8.1	0.07	<0.1	<0.05	<0.2	<0.05	--	29
	Stored	7.1	17.5	0.6	25.5	8.1	0.05	<0.1	<0.05	<0.2	0.06	42.0	13
	Product	3.6	8.2	<0.1	<1.0	8.2	<0.05	<0.1	<0.05	<0.2	0.15	--	0
7	Simulant	7.1	27.0	8.1	5.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	41.5	27
	Stored	7.1	18.5	0.7	25.0	8.0	0.05	<0.1	<0.05	<0.2	0.06	41.3	12
	Product	3.6	8.2	<0.1	<1.0	8.2	<0.05	<0.1	<0.05	<0.2	0.16	41.0	0

* TOC analyses performed at NASA/MSC. Dashes indicate no analysis conducted.

Table C2 SUMMARY OF DAILY WATER QUALITY ANALYSES FOR SMT #2

Test Day No.	Sample Water	pH	Sp. Res. (kΩ-cm)	Turbidity (JTU's	Alkalinity (ppm)	Cl (ppm)	Cr (+6) (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Ag (ppm)	TOC* (ppm)	TDS (ppm)
1	Simulant	7.2	28.0	8.4	5.0	8.1	--	--	--	--	<0.03	--	7
	Stored	7.1	16.1	<0.1	17.0	8.1	0.05	<0.1	<0.05	<0.2	0.05	40.8	0
	Product	6.0	42.5	<0.1	<1.0	<0.5	<0.05	<0.1	<0.05	<0.2	0.18	39.0	0
2	Simulant	7.1	27.5	8.3	4.0	8.0	--	<0.1	<0.05	<0.2	--	--	18
	Stored	7.1	15.9	<0.1	18.0	8.0	0.06	<0.1	<0.05	<0.2	0.06	40.5	6
	Product	5.6	330.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.12	--	0
3	Simulant	7.1	27.0	6.8	6.0	8.1	--	--	--	--	--	--	12
	Stored	7.1	16.4	<0.1	16.5	8.1	0.05	<0.1	<0.05	<0.2	0.08	38.8	6
	Product	5.7	225.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.12	--	0
4	Simulant	7.1	28.5	8.3	6.0	8.1	0.07	--	<0.05	<0.2	--	--	84
	Stored	7.1	16.9	0.15	14.0	8.1	0.07	<0.1	<0.05	<0.2	0.07	38.3	128
	Product	5.2	185.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.08	40.0	10
5	Simulant	7.1	28.5	8.3	5.0	8.1	0.13	<0.2	<0.05	<0.5	<0.03	36.5	81
	Stored	7.1	17.0	<0.1	13.0	8.1	0.06	<0.1	<0.05	<0.2	0.04	38.3	35
	Product	5.4	130.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.12	--	19
6	Simulant	7.2	29.0	7.9	5.0	8.1	--	--	--	--	--	--	82
	Stored	6.6	18.3	0.2	13.5	8.1	0.06	<0.1	<0.05	<0.2	0.07	41.3	43
	Product	4.7	94.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	--	0.11	--	11
7	Simulant	7.1	29.5	8.3	5.0	8.1	0.06	<0.1	<0.05	<0.2	<0.03	--	88
	Stored	6.2	18.5	<0.1	13.0	8.1	<0.05	<0.1	<0.05	<0.2	0.08	42.8	28
	Product	4.8	82.0	<0.1	<1.0	<1.0	0.07	<0.1	<0.05	<0.2	0.08	40.0	20

*TOC analyses performed at NASA/MSC.
Dashes indicate no analysis conducted.

Table C3 SUMMARY OF DAILY WATER QUALITY ANALYSES FOR SMT #3

Test Day No.	Sample Water	pH	Sp. Res. (Ω-cm)	Turbidity (JTU's)	Alkalinity (ppm)	Cl (ppm)	Cr (+6) (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Ag (ppm)	TOC* (ppm)	TDS (ppm)
1	Simulant	7.6	31.8	5.8	4.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	36.5	63
	Stored	6.6	23.8	<0.1	6.0	7.9	0.06	<0.1	<0.05	<0.2	0.07	36.5	13
	Product	7.1	285.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.12	36.5	4
2	Simulant	7.3	28.0	8.3	5.0	8.1	0.07	--	--	--	<0.03	--	91
	Stored	6.5	22.0	0.6	5.0	8.0	0.08	<0.1	<0.05	<0.2	0.08	36.5	58
	Product	7.1	365.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.09	--	19
3	Simulant	7.2	30.5	8.3	4.0	8.1	<0.01	<0.2	<0.05	<0.5	<0.05	38.0	114
	Stored	7.8	22.8	0.2	8.5	8.0	0.07	<0.1	<0.05	<0.2	0.05	37.3	35
	Product	5.9	725.0	<0.1	2.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.08	--	12
4	Simulant	6.9	30.0	8.2	5.0	8.1	--	--	--	--	--	--	107
	Stored	7.9	23.0	<0.1	7.0	8.1	0.06	<0.1	<0.05	<0.2	0.06	37.0	26
	Product	5.9	425.0	<0.1	1.0	1.0	<0.05	<0.1	<0.05	<0.2	0.06	36.5	1
5	Simulant	7.2	30.1	8.1	5.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	--	101
	Stored	7.9	23.0	<0.1	7.5	8.1	0.07	<0.1	<0.05	<0.2	0.06	37.3	16
	Product	6.2	230.0	<0.1	2.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.06	--	0
6	Simulant	7.1	30.0	8.2	5.0	8.1	0.06	<0.1	<0.05	<0.2	<0.03	--	70
	Stored	7.8	22.8	0.37	5.0	8.1	0.06	<0.1	<0.05	<0.2	0.07	36.0	19
	Product	6.2	220.0	<0.1	<1.0	1.0	<0.05	<0.1	<0.05	<0.2	0.07	--	1
7	Simulant	7.2	28.0	6.7	5.0	8.1	0.06	<0.1	<0.05	<0.2	<0.03	--	105
	Stored	7.7	22.8	<0.1	5.5	8.0	0.06	<0.1	<0.05	<0.2	0.07	36.8	16
	Product	5.7	140.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.06	35.0	16

*TOC analyses performed at NASA/MSC.
Dashes indicate no analysis conducted.

Table C4 SUMMARY OF DAILY WATER QUALITY ANALYSES FOR SMT #4

Test Day No.	Sample Water	pH	Sp. Res. (kΩ-cm)	Turbidity (JTU's)	Alkalinity (ppm)	Cl (ppm)	Cr (+6) (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Ag (ppm)	TOC* (ppm)	TDS (ppm)
1	Simulant	7.8	24.0	8.5	3.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	61.0	168
	Stored	7.7	19.5	<0.1	9.0	8.0	0.08	<0.1	<0.05	<0.2	0.09	41.5	71
	Product	6.8	510.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.07	43.0	8
2	Simulant	7.6	28.5	8.4	5.0	8.1	0.06	<0.2	<0.05	<0.5	<0.05	62.5	180
	Stored	6.9	23.2	<0.1	4.5	8.1	0.07	<0.1	<0.05	<0.2	0.08	43.0	87
	Product	6.5	780.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.06	--	24
3	Simulant	7.0	26.0	8.3	4.0	8.1	0.06	<0.2	<0.05	<0.5	<0.05	63.0	54
	Stored	7.1	22.8	<0.1	5.5	8.1	0.07	<0.1	<0.05	<0.2	0.08	41.8	99
	Product	6.0	630.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.07	--	5
4	Simulant	6.9	26.0	8.4	4.0	8.1	0.06	<0.2	<0.05	<0.5	<0.05	64.0	96
	Stored	7.2	22.0	<0.1	8.0	8.1	0.06	<0.1	<0.05	<0.2	0.08	43.0	86
	Product	5.7	460.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.06	41.0	1
5	Simulant	7.2	27.5	8.4	5.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	64.0	46
	Stored	7.3	23.2	0.21	7.0	8.1	0.07	<0.1	<0.05	<0.2	0.08	41.8	38
	Product	5.5	410.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.04	--	2
6	Simulant	7.5	27.5	8.5	5.0	8.1	0.07	<0.1	<0.05	<0.2	<0.05	61.5	49
	Stored	7.3	22.8	0.18	6.0	8.1	0.08	<0.1	<0.05	<0.2	0.08	42.0	33
	Product	5.3	280.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.07	--	13
7	Simulant	7.5	28.0	8.4	5.0	8.1	0.07	<0.2	<0.05	<0.5	<0.05	61.0	96
	Stored	7.4	23.0	<0.1	7.0	7.8	0.07	<0.1	<0.05	<0.2	0.08	41.3	11
	Product	5.1	180.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.07	41.0	7

*TOC analyses performed at NASA/MSC.
Dashes indicate no analysis conducted.

Table C5 SUMMARY OF DAILY WATER QUALITY ANALYSES FOR SMT #5

Test Day No.	Sample Water	pH	Sp. Res. (kΩ-cm)	Turbidity (JTU's)	Alkalinity (ppm)	Cl (ppm)	Cr (+6) (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Ag (ppm)	TOC* (ppm)	TDS (ppm)
1	Simulant	7.6	26.5	8.3	5.0	8.1	0.06	<0.1	<0.05	<0.2	<0.03	57.0	93
	Stored	6.8	44.0	<0.1	2.0	8.1	0.06	<0.1	<0.05	<0.2	0.08	36.5	46
	Product	6.0	870.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.28	36.0	21
2	Simulant	7.4	31.0	8.1	5.0	8.0	0.06	<0.2	<0.05	<0.5	<0.05	56.0	154
	Stored	6.6	40.3	<0.1	2.0	7.5	0.06	<0.1	<0.05	<0.2	0.08	33.0	49
	Product	5.8	540.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.16	--	5
3	Simulant	7.3	29.0	8.1	4.0	8.1	0.07	<0.2	<0.05	<0.5	<0.05	56.0	202
	Stored	6.6	33.0	0.25	2.5	6.1	0.07	<0.1	<0.05	<0.2	0.08	31.0	20
	Product	5.7	480.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.10	--	2
4	Simulant	7.3	30.0	8.1	4.0	8.1	0.07	<0.2	<0.05	<0.5	<0.03	50.0	301
	Stored	6.8	31.0	0.4	3.0	7.4	0.06	<0.1	<0.05	<0.2	0.08	34.5	3
	Product	5.5	470.0	<0.1	1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.09	39.5	8
5	Simulant	7.2	28.5	8.3	4.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	54.0	238
	Stored	6.8	28.5	0.63	3.5	7.9	0.07	<0.1	<0.05	<0.2	0.07	32.5	6
	Product	5.6	350.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.09	--	1
6	Simulant	7.2	29.5	8.2	4.0	8.1	0.07	<0.2	<0.05	<0.5	<0.05	47.0	173
	Stored	6.8	29.0	0.39	2.5	6.9	0.06	<0.1	<0.05	<0.2	0.08	31.3	95
	Product	5.3	180.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.08	--	13
7	Simulant	7.1	27.0	8.2	5.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	55.0	246
	Stored	6.9	28.8	0.24	5.0	7.8	0.06	<0.1	<0.05	<0.2	0.07	30.5	--
	Product	4.95	120.0	<0.1	<1.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.08	39.0	2

*TOC analyses performed at NASA/MSC.
Dashes indicate no analysis conducted.

Table C5 (Cont.) SUMMARY OF DAILY WATER QUALITY ANALYSES FOR SMT #5

Test Day No.	Sample Water	pH	Sp. Res. (kΩ-cm)	Turbidity (JTU's)	Alkalinity (ppm)	Cl (ppm)	Cr (+6) (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Ag (ppm)	TOC* (ppm)	TDS (ppm)
8	Simulant	7.4	29.5	8.2	3.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	53.0	160
	Stored	6.7	30.5	--	5.0	--	0.07	<0.1	<0.05	<0.2	1.21	28.8	--
	Product	4.5	66.0	--	--	--	<0.05	<0.1	<0.05	<0.2	0.08	37.0	--
9	Simulant	7.5	28.5	8.1	3.0	8.1	0.07	<0.1	<0.05	<0.2	<0.03	51.0	128
	Stored	6.7	27.8	--	2.0	6.5	0.06	<0.1	<0.05	<0.2	1.27	32.5	--
	Product	4.3	35.0	<0.1	0.0	<1.0	<0.05	<0.1	<0.05	<0.2	0.08	45.5	--
10	Simulant	7.5	31.5	8.1	4.0	8.1	0.07	<0.2	<0.05	<0.5	<0.05	50.0	189
	Stored	6.9	25.8	--	2.0	6.3	0.07	<0.1	<0.05	--	1.21	28.0	--
	Product	4.1	25.0	<0.1	0.0	<1.0	<0.05	<0.1	<0.05	<0.5	0.07	45.0	1.7

*TOC analyses performed at NASA/MSC.
Dashes indicate no analysis performed.

APPENDIX D

SUMMARY OF DETAILED

PRODUCT WATER ANALYSES ON

SELECTED TEST DAYS

Table D1 DETAIL ANALYSES OF PRODUCT WATER FOR SMT #1 & #2

Quality Parameters	Concentration (ppm) SMT 1 Day 1	SMT 1 Day 3	SMT 1 Day 7	SMT 2 Day 1	SMT 2 Day 4	SMT 2 Day 7	Specified Limits USPHS (ppm)	ADHOC (ppm)	MSC35 (ppm)
ABS	--	--	--	--	--	--	0.05	NS	NS
Arsenic	--	--	--	--	--	--	0.01	0.50	NS
Barium	--	--	--	--	--	--	1.0	2.0	NS
Cadmium	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.01	0.05	0.01
C(CHCl3ext.)	--	--	--	--	--	--	0.20	NS	NS
Chloride	5.0	5.9	5.9	<0.1	<0.1	<0.1	(1)	450.0	NS
Chromium(+6)	<0.01	<0.01	0.05	<0.01	<0.01	<0.01	0.05	0.05	0.05
Copper	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	1.0	3.0	1.0
Cyanide	--	--	--	--	--	--	0.01	NS	NS
Flouride	1.8	1.9	2.4	0.25	0.66	1.25	1.0	NS	NS
Iron	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.30	NS	0.30
Lead	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	0.05	0.20	0.05
Magnesium	0.09	0.03	0.02	0.02	0.02	<0.02	NS	NS	NS
Manganese	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.05	NS	0.05
Mercury	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	NS	NS	0.005
Nickel	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	NS	NS	0.05
Nitrate	2.35	4.3	<0.5	0.63	<0.5	<0.5	45.0	NS	NS
Phenols	--	--	--	--	--	--	0.001	NS	NS
Potassium	0.07	0.09	0.10	0.08	0.01	0.10	NS	NS	NS
Selenium	--	--	--	--	--	--	0.01	NS	NS
Silver	<0.05	<0.05	<0.05	0.17	0.10	0.09	0.05	0.50	0.05
Sodium	<0.05	0.09	0.12	≤0.05	<0.05	0.08	NS	NS	NS
Sulfate	≤1.0	≤1.0	≤1.0	≤1.0	≤1.0	≤1.0	(1)	250.0	NS
Zinc	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	5.0	NS	5.0
COD	--	--	--	--	--	--	NS	100.0	NS
Total Solids	8.2	9.8	5.3	3.1	2.9	3.3	500.0	1000.0	500.0
Total Carbon	33.0	38.0	42.0	40.0	41.0	41.0	NS	NS	NS
pH	3.60	3.49	3.52	5.31	4.66	4.46	NS	NS	6.0-8.0
Resistance (Mohm-cm)	0.017	0.015	0.015	0.400	0.250	0.126	NS	NS	NS

(1) Sum of chloride and sulfate should not exceed 250 ppm

Table D2 DETAIL ANALYSES OF PRODUCT WATER FOR SMT #3 & #4

Quality Parameters	Concentration (ppm) SMT 3 Day 1	SMT 3 Day 4	SMT 3 Day 7	SMT 4 Day 1	SMT 4 Day 4	SMT 4 Day 7	Specified Limits USPHS (ppm)	ADHOC (ppm)	MSC35 (ppm)
ABS	--	--	--	--	--	--	0.05	NS	NS
Arsenic	--	--	--	--	--	--	0.01	0.50	NS
Barium	--	--	--	--	--	--	1.0	2.0	NS
Cadmium	<0.005	0.01	<0.005	<0.005	<0.005	<0.005	0.01	0.05	0.01
C(CHCl3ext.)	--	--	--	--	--	--	0.20	NS	NS
Chloride	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	(1)	450.0	NS
Chromium(+6)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.05	0.05	0.05
Copper	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	1.0	3.0	1.0
Cyanide	--	--	--	--	--	--	0.01	NS	NS
Flouride	0.18	0.25	0.68	0.24	0.27	0.56	1.0	NS	NS
Iron	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.30	NS	0.30
Lead	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	0.05	0.20	0.05
Magnesium	≤0.01	<0.01	<0.01	<0.01	<0.01	<0.01	NS	NS	NS
Manganese	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.05	NS	0.05
Mercury	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	NS	NS	0.005
Nickel	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	NS	NS	0.05
Nitrate	<0.5	1.55	<0.5	<0.5	<0.5	0.70	45.0	NS	NS
Phenols	--	--	--	--	--	--	0.001	NS	NS
Potassium	0.15	0.07	0.10	0.01	0.04	0.10	NS	NS	NS
Selenium	--	--	--	--	--	--	0.01	NS	NS
Silver	0.23	<0.05	<0.05	0.08	<0.05	<0.05	0.05	0.50	0.05
Sodium	0.13	0.10	0.10	<0.01	<0.01	0.10	NS	NS	NS
Sulfate	≤1.0	≤1.0	1.5	≤1.0	≤1.0	≤1.0	(1)	250.0	NS
Zinc	0.06	0.02	0.03	<0.01	<0.01	<0.01	5.0	NS	5.0
COD	--	--	--	--	--	--	NS	100.0	NS
Total Solids	1.8	2.2	2.6	2.3	1.3	1.2	500.0	1000.0	500.0
Total Carbon	37.0	37.0	36.0	45.0	42.0	42.0	NS	NS	NS
pH	5.27	4.70	4.52	6.18	5.87	5.35	NS	NS	6.0-8.0
Resistance (Mohm-cm)	0.330	0.190	0.150	0.550	0.590	0.350	NS	NS	NS

(1) Sum of chloride and sulfate should not exceed 250 ppm

Table D3 DETAIL ANALYSES OF PRODUCT WATER FOR SMT #5

Quality Parameters	Concentration (ppm) SMT 5 Day 1	Day 4	Day 7	Day 8	Day 9	Day 10	Specified Limits USPHS (ppm)	ADHOC (ppm)	MSC35 (ppm)
ABS	--	--	--	--	--	--	0.05	NS	NS
Arsenic	--	--	--	--	--	--	0.01	0.50	NS
Barium	--	--	--	--	--	--	1.0	2.0	NS
Cadmium	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.01	0.05	0.01
C(CHCl3ext.)	--	--	--	--	--	--	0.20	NS	NS
Chloride	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	(1)	450.0	NS
Chromium(+6)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.05	0.05	0.05
Copper	<0.10	<0.10	<0.10	≤0.10	<0.10	<0.10	1.0	3.0	1.0
Cyanide	--	--	--	--	--	--	0.01	NS	NS
Flouride	0.02	0.04	0.46	0.76	1.40	2.0	1.0	NS	NS
Iron	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.30	NS	0.30
Lead	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	0.05	0.20	0.05
Magnesium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	NS	NS	NS
Manganese	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.05	NS	0.05
Mercury	<0.005	<0.005	<0.005	<0.005	≤0.005	<0.005	NS	NS	0.005
Nickel	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	NS	NS	0.05
Nitrate	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	45.0	NS	NS
Phenols	--	--	--	--	--	--	0.001	NS	NS
Potassium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	NS	NS	NS
Selenium	--	--	--	--	--	--	0.01	NS	NS
Silver	0.32	0.10	0.09	0.08	0.08	0.07	0.05	0.50	0.05
Sodium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	NS	NS	NS
Sulfate	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	(1)	250.0	NS
Zinc	≤0.01	<0.01	<0.01	0.02	0.01	0.02	5.0	NS	5.0
COD	--	--	--	--	--	--	NS	100.0	NS
Total Solids	0.8	1.1	--	--	--	1.7	500.0	1000.0	500.0
Total Carbon	37.0	40.0	40.0	38.0	46.0	46.0	NS	NS	NS
pH	5.28	5.33	4.53	4.36	4.14	4.01	NS	NS	6.0-8.0
Resistance (Mohm-cm)	0.650	0.500	0.180	0.100	0.050	0.040	NS	NS	NS

(1) Sum of chloride and sulfate should not exceed 250 ppm