Ionomer-membrane Water Processor (IWP) Engineering Development Unit (EDU) Brine Water Recovery Test Results

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Closing the water loop on long duration spaceflight missions is a key aspect of reducing mission mass and logistics support for orbiting facilities and is a necessity for interplanetary spacecraft. Closing that water loop cannot be achieved without recovery of water from brine. As such, NASA has identified brine processing as an enabling technology for exploring other worlds. Paragon Space Development Corporation is developing the patent-pending Ionomer-membrane Water Processor (IWP) to enable water recovery from urine and brine. Through a NASA SBIR Phase 2 effort, Paragon developed and tested a large-scale IWP Engineering Development Unit (EDU). Testing with urine brine pretreated with the International Space Station (ISS) pretreatment formulation was conducted by both Paragon and NASA Ames Research Center. The IWP EDU successfully recovered over 85% of the water from brine, boosting total water recovery from urine up to 98%. Testing also demonstrated 99% removal of contaminants in brine in a configuration designed for microgravity application.

Nomenclature

ARC = Ames Research Center
ARFTA = Advanced Recycle Filter Tank Assembly
CHX = Condensing Heat Exchanger
COTR = Contracting Officer Technical Representative
dP = delta Pressure
EDU = Engineering Development Unit
ePTFE = expanded Polytetrafluoroethylene
ISS = International Space Station
IWP = Ionomer-membrane Water Processor
NASA = National Aeronautics and Space Administration
RH = Relative Humidity
SBIR = Small Business Innovative Research
SMAC = Spacecraft Maximum Allowable Concentration
TC = Thermocouple
TCCA = Trace Contaminant Control Assembly
TDS = Total Dissolved Solids
TOC = Total Organic Carbon
TSS = Total Suspended Solids
UPA = Urine Processor Assembly
VCD = Vapor Compression Distillation
WFRD = Wiped-Film Rotating-Disk
WPA = Water Processor Assembly

I. Introduction

Closing the water loop on long duration spaceflight missions is a key aspect of reducing mission mass and logistics support for orbiting facilities and interplanetary spacecraft. Currently, no single practical process exists that is capable of extracting purified water from urine in a single step. The vapor compression distillation (VCD) system currently in use onboard the International Spaces Station (ISS) distills water from pretreated urine, recovering ~75% of the water in the urine. However water recovery is restricted to the solubility limit of the various compounds in urine such as calcium sulfate, thus producing concentrated brine that requires further processing for water recovery. Even with switching to new pretreatment, ~15% of the water is left behind in the brine. Paragon Space Development Corporation (Paragon) seeks to recover a higher percentage of water

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from urine by processing the brine with the Ionomer-membrane Water Processor (IWP), and thus take a significant step towards closing the water loop necessary for moving beyond Low Earth Orbit and into deep space.

Paragon has developed a patent pending ionomer-microporous membrane technology known as IWP to simplify and improve the robustness and effectiveness of water recovery processes for human spaceflight applications. Urine or concentrated brine is retained within a multi-layer "brine bag" (i.e. the membrane pair) that safely contains the brine and any harmful compounds and only passes purified water vapor to the external environment. In its simplest "passive" implementation, and as shown in Figure 1, IWP only requires the movement of a purge gas (typically cabin air) across the surface of the membrane pair to extract purified water from urine or brine. As detailed later in this document, nearly all inorganic and organic contaminants are rejected by the membranes and retained within the brine bag. Water transport across the membrane pair is driven solely by the water partial pressure differential between the interior and exterior of the brine bag. Purified water transferred to the air stream is then recovered as humidity condensate and processed nominally by the spacecraft's water recovery system. The water recovery rate can be increased by implementing "active" control features such as increasing the temperature at which the process operates, decreasing the water partial pressure of the incoming air stream, and/or increasing the sweep velocity of the air.

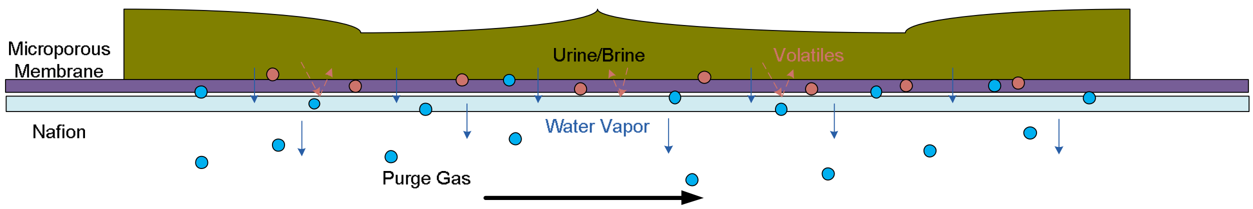

Figure 1: Water transport across IWP membrane pair is driven by the water partial pressure differential.

Through NASA SBIR contracts, commercial spaceflight contracts, and internal product development funding, Paragon has completed: 1) membrane screening and selection, 2) modeling, analysis, and conceptual design of IWP configured as a urine processor as well as an ISS brine processor, 3) extensive water recovery performance testing using real urine and concentrated brine to quantify water transfer rates and contaminant retention effectiveness, and 4) design, manufacture, and test of a full-scale IWP-based urine processor. In addition, the detailed design of an "active" IWP Brine processor engineering development unit (EDU) was recently completed. The EDU was manufactured, tested, and delivered to NASA Ames Research Center (ARC) for further testing to evaluate performance and operation in the active processing mode. This publication will focus on the test results of the IWP brine processor EDU.

A. Background

IWP is a patent-pending dual-membrane distillation process. The first layer, a microporous membrane, allows bulk gas permeation at high rates while retaining the liquid and solids. The second layer is an ionomer membrane with selective permeability to water vapor. The ionomer transports water vapor while trapping harmful volatiles. Water vapor is swept away by a purge gas while residual dehydrated brine and volatiles are fully contained within the membranes. Because the membranes are extremely lightweight, the entire IWP membrane structure is disposable with the brine, maintaining brine containment throughout the life of processing and disposal. Figure 2 demonstrates the dehydrated urine containment in a disposable IWP bag after urine processing. The stages of brine processing from IWP testing are pictured in Figure 3, from original urine to pre-processed brine to recovered water.

The ionomer membrane baselined in the IWP technology is Nafion®, a copolymer of tetrafluoroethylene and perfluro-3,6-dioxa-4-methyl-7-octene-sulfonic acid[1]. Like Teflon, Nafion® is highly resistant to chemical attack, but the presence of its exposed sulfonic acid groups confers unusual properties. "Nafion® very readily absorbs water, from the vapor phase or from the liquid phase. Each sulfonic acid group will absorb up to 13 molecules of water. The sulfonic acid groups form ionic channels through the bulk hydrophobic polymer, and water is very readily transported through these channels. As such, Nafion® functions like a very selective, semi-permeable membrane to water vapor."[1] This ability to selectively allow water to permeate suggests a possible role in water purification processes. The

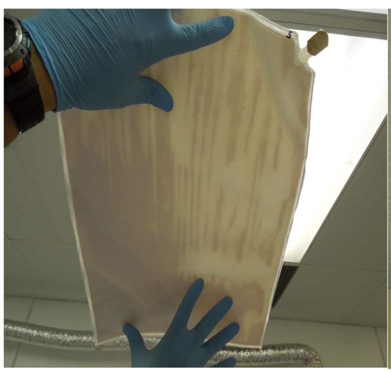

Figure 2: Final stage of urine bag with 98% of water removed; remaining material is "crunchy."

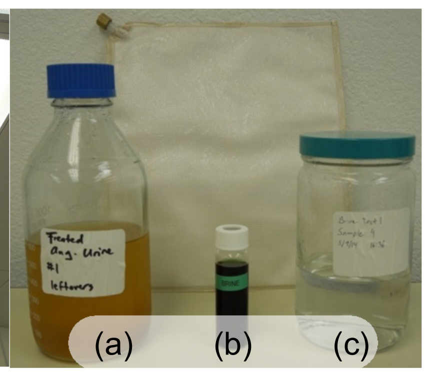

Figure 3: Stages of brine processing, original urine (a), pretreated urine brine (b), and extracted water (c).

sulfonic acid groups pass water, but few other compounds, making it possible to separate water from a contaminated source.

The fact that Nafion® acts as an ion exchange resin when exposed to liquids suggests that Nafion® is most effective processing gases rather than liquid solutions. Solutions containing positive ions will reduce the effectiveness of Nafion®'s permeability function by approximately 66% by supplanting the hydrogen ions of the sulfonic acid group with that of the solution cations[1]. As such, the Nafion®-based membrane pair solution is designed to deliver a vapor stream to the Nafion® surface; the ionomer is paired with a microporous membrane to prevent contamination of the ionomer. The IWP membrane pairing and water transport is shown in Figure 1.

II. IWP EDU Testing at Paragon

A. EDU Description

A conceptual system design and detailed EDU design was performed and reported in *Development of Ionomer-membrane Water Processor (IWP) technology for water recovery from brine* during the 44th International Conference on Environmental Systems (ICES).[5] A brief description of the system and EDU design is presented here. Via the SBIR program, Paragon has been developing a patent-pending Active IWP design solution to recover water from brine produced aboard the International Space Station (ISS) by the Urine Processor Assembly (UPA). Based upon current performance, approximately 569 liters of brine wastewater is generated annually. Future planned improvements are expected to reduce this loss to a still significant 329 liters per year. This waste must be removed from ISS and disposed of, and an equal quantity of water must be transported up to ISS to replenish the lost water.[2]

Currently, the ISS UPA processes urine and stores the residual brine in the Advanced Recycle Filter Tank Assembly (ARFTA), which has a capacity of 22L. Once the ARFTA is full, the brine is emptied into a storage container and stored on station or returned to Earth. A brine processor is needed to recover this lost water. A conceptual system design and analysis was performed to facilitate integration of an Active IWP into ISS operations to recover water from brine. Various implementations were evaluated including direct integration into the UPA and stand-alone systems. Integration with the ISS UPA can be achieved by replacing the existing ARFTA with an IWP design solution optimized for continual brine processing. This would have the advantage of continuous water recovery as opposed to batch processing, and potentially minimize crew interaction and operations. While this approach is ideal for integrating IWP into a next-generation urine processing system (e.g. aboard an exploration spacecraft), the logistics and complexity of trying to integrate directly into the existing operational ISS UPA has been initially considered to be cost-prohibitive. Further discussions with NASA are required to determine the validity of this assumption. Stand-alone systems that do not require changes to the UPA operations or hardware were considered more feasible for implementation on ISS, and have the added bonus of lending themselves well to flight experiments to demonstrate the technology in the real environment prior to operational implementation. Stand-alone systems were conceptualized that included continuous feed from the intermediate brine storage containers currently used on ISS for brine storage and transport. This method allows for smaller IWP bags, however requires a pump or compressor, plus a control system to feed the brine into the IWP bags. Additionally, the volume of the intermediate brine storage tank has to be taken into account. A complete batch mode that contains and processes the entire volume from the ARFTA was then analyzed. In the batch design, the brine can be transferred directly from the ARFTA into the IWP bladder for processing, eliminating the necessity and volume of the intermediate storage container. In the end, the batch mode design was selected for further development into an EDU because of the simplicity of its design and implementation.

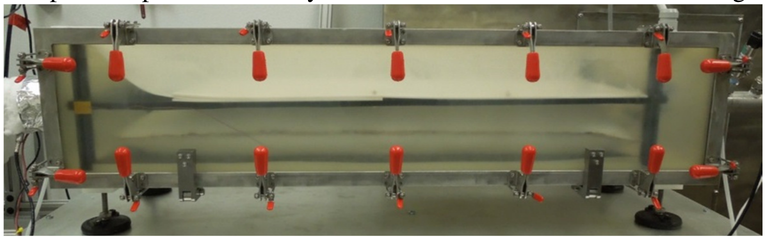

Figure 4: EDU for Active IWP System.

Brine is produced by the UPA in 22L batches in a timeframe of 2-3 weeks, depending on crew size and water recovery in the UPA. For the analysis, an 18 day cycle and 85% initial water recovery from urine was assumed to set the brine processing requirements for the IWP assembly. To implement the Active IWP, the brine is emptied directly from the ARFTA into an IWP bladder, and thus does not impact current urine processor operations or hardware. The bladder is then placed in the housing for processing. A blower pulls an air stream from the Condensing Heat Exchanger (CHX) outlet to obtain the driest air available for the sweep gas. The sweep gas is heated by an inline heater prior to entering the housing to raise the brine temperature to encourage evaporation and counteract the evaporative cooling. The sweep gas and water vapor vent directly into the cabin for the water to be recovered by the CHX. Alternatively, a "Passive" IWP brine processor could be implemented by utilizing existing ventilation and air flows on ISS as the sweep gas and removing the heater, thus requiring no power or support equipment outside of the brine bladder and a fixture to secure the bladder.

Figure 5: Active IWP EDU Bladder.

Based on the conceptual system design and analysis, an Active IWP EDU was developed to simulate the form, fit, and function of the IWP technology as applied in the system design. It was not intended to be a ground test unit of a flight design and was not optimized for mass, volume, or power. The EDU consisted of two main parts: the brine bladder and housing. The bladder is constructed of the IWP microporous and ionomer membranes. It can hold up to 16.5L of brine, which is 75% of the 22L capacity of the ARFTA, and thus is considered a 75% full-scale brine processor for ISS. During operation, an empty bladder is installed into the housing. A fill port on the bladder is connected to a feedthrough that passes through the housing wall to allow *in situ* filling of brine into the bladder. The housing provides containment of the bladder to direct and control the purge gas flow over the membrane surfaces. The purge gas enters one end of the housing and passes over the length of the brine bladder to promote water vapor permeation. The humid purge gas then exits the other end of the housing to be sampled and condensed. The housing is constructed of various types of plastic compatible with the brine as a precaution in the event of a leak during development testing. A flow-diverting structure is included in the housing to maintain the cross-sectional flow area for the purge gas as the bladder deflates from water removal. The 75% of full-scale, Active IWP EDU is shown in Figure 4 and a bladder is shown in Figure 5. Support equipment for testing the EDU includes a heater and blower, plus a condenser to collect water samples, though a dedicated condenser is not part of the system design. The heater and blower were used for testing purposes and were not optimized to minimize mass, volume, or power.

B. Test Description

The test bed schematic can be found in Figure 6 below. The purpose of the test bed is to evaluate the ability of IWP EDU to extract water from and contain pretreated urine brine.

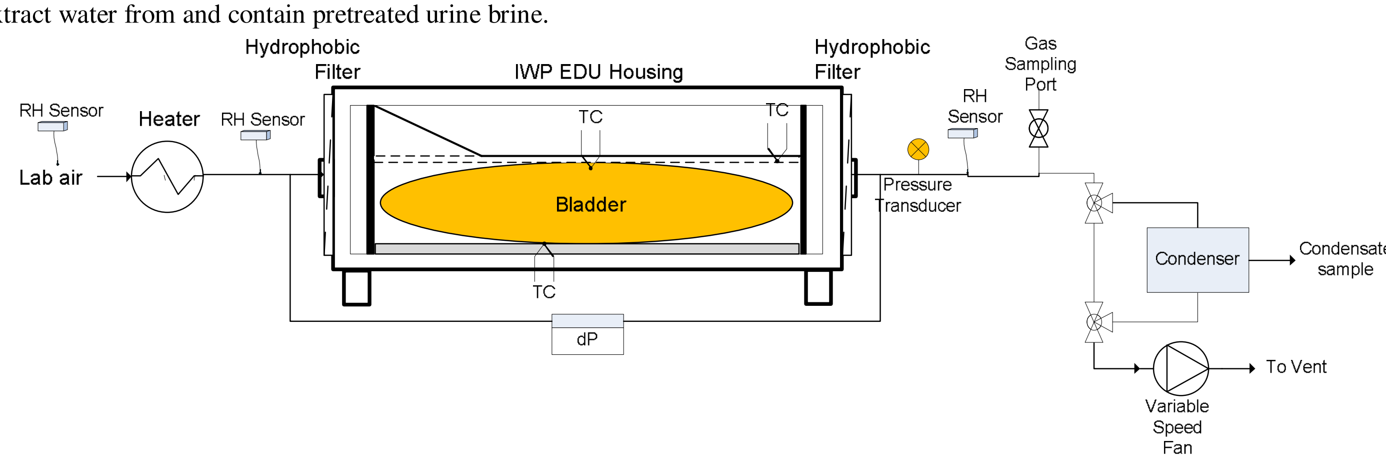

Figure 6: IWP EDU Test Bed Schematic

Lab air was used as the purge gas. The air flow was controlled by a variable frequency drive fan. The frequency set point was determined by flow bench testing. The flow bench was used to match the desired flow with the correct frequency and determine the pressure drop (dP) for the test bed associated with the flow, which was monitored during testing. The purge air flows through the heater before entering the EDU. The heated flow then convectively heats the brine as it flows through the EDU. Thermocouples inside the EDU monitor the temperature of the bladder and housing. A thermocouple located on the bladder surface was attached to the pre-EDU flow heater controller to maintain the brine temperature in the bladder. The purge gas exited the housing after picking up water vapor from the bladder. Pressure, temperature, and humidity of the purge stream were monitored before and after the EDU. A flow diverter feature in the EDU housing maintained the cross-sectional flow area and flow distribution as the brine bladder shrank from water removal during the course of testing. Gas and water condensate samples were collected for analysis.

During operation the bladder was filled with 16L of brine. The empty bladder was placed inside the housing and connected to the fill tubes via self-closing quick disconnect fittings and the EDU door was closed and sealed. The external portion of the quick disconnect was connected to a pump which fed the pretreated urine brine into the bladder. Refer to Figure 7. After filling, the self-closing fitting was disconnected from the pump and the brine processing began. A new bladder was used for each test.

NASA Ames Research Center (ARC) provided brine for EDU testing. The wastewater brine was produced by removing 85% of the water from human urine. The urine was initially pretreated with the Alternative ISS urine pretreatment[3] and salt concentrations adjusted to mimic astronaut urine.[4] The brine was produced by processing urine in the Wiped-film Rotating-disk (WFRD) at ARC. The WFRD is a vapor compression distillation system used to simulate the ISS UPA function. The urine processed by the WFRD was collected from male volunteers at ARC.

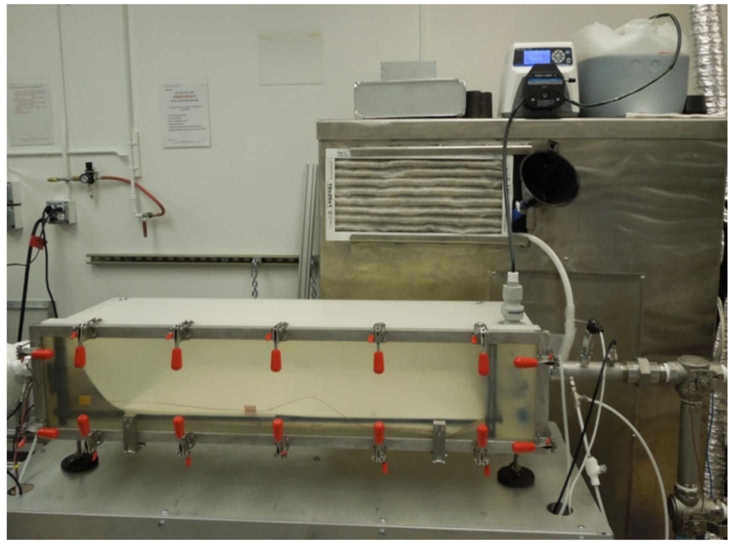
Figure 7: IWP EDU Brine Fill Operation

C. Test Results

The EDU testing was successfully completed at Paragon in May 2014. A summary of tests conducted is presented in Table 1. Water tests were performed before and after the brine tests to establish baseline water and air quality. The last water test was intended to evaluate any radical changes in baseline water quality indicative of cross-contamination or changes to the test bed such as corrosion. Brine Test 1 was stopped when there was no longer any indication of active permeation. Brine Test 2 was continued well after the apparent end of permeation to ensure that the maximum amount of water was extracted from the brine as physically possible without changing the operating parameters.

Table 1: IWP EDU Test Matrix from Paragon Testing

Test #	Date	Duration	Notes/Comment
Water 1	5/3/14	3 days	Baseline water and air quality, system operation, Flow Bench testing
Brine 1	5/6/14	4 days	Permeation rate, total water recovery, water and air quality
Brine 2	5/10/14	9 days	Permeation rate, total water recovery, water and air quality
Water 2	5/19/14	1.5 days	Baseline water quality

1. System Operational Conditions

The average operating conditions for each brine test are presented in Table 2, along with the target values. It was observed during the first test that the top of the bag heated up to the target temperature much faster than the bottom, indicating poor air flow underneath the bladder. A second flow diverter was temporarily installed to attempt to force more air underneath the bladder. This seemed to be a small improvement as indicated by the time for the bottom of the bladder to heat up. During Brine 1, the bladder temperature was maintained such that the bottom temperature was at least 35°C. During Brine 2, the temperature was controlled so that the top was 35°C. The target room/inlet humidity was a dew point of 7.2°C (45°F, 1019 Pa partial pressure), mimicking the humidity of the return air from the ISS condensing heat exchanger. As shown, the average room humidity was slightly low during both brine tests. It was found during Brine 2 that the humidifier wick needed replacement. Once it was replaced, the humidity was maintained above 7.2°C such that the average was 3% lower than the target.

Table 2: EDU Testing Average Operating Conditions

Test #	Bladder Top Temperature (°C)	Bladder Bottom Temperature (°C)	Average Bladder Temperature (°C)	EDU dP (in H_2O)	Outlet Pressure (psia)	Room/Inlet Dew Point (°C)
Target	35	35	35	N/A	N/A	7.2
Brine 1	40.7	36.0	38.4	7.02	13.06	6.1
Brine 2	35.4	32.5	34.0	6.83	13.10	6.97

The relative humidity (RH) at the inlet and outlet of the EDU were measured, but used for reference only as the meters were found to be out of tolerance after testing during a post-test calibration. A third RH meter was used to record the ambient temperature and humidity of the air immediately before entering the system. There are indications of permeation performance other than RH that were recorded during the course of testing. The inlet air flow temperature and heater power are directly coupled to permeation rate by evaporative cooling. Refer to Figure 8. A smooth curve can be made of the decrease in inlet flow temperature from the beginning of testing. The point at which the flow temperature levels toward the end of the test indicates the end of any significant permeation. This point occurs around 7600 minutes. The bladder and housing temperatures also converge at this point, indicating an end to evaporation. The breaks in the data are times during which data was not being recorded, but the system was still operating normally during those time intervals.

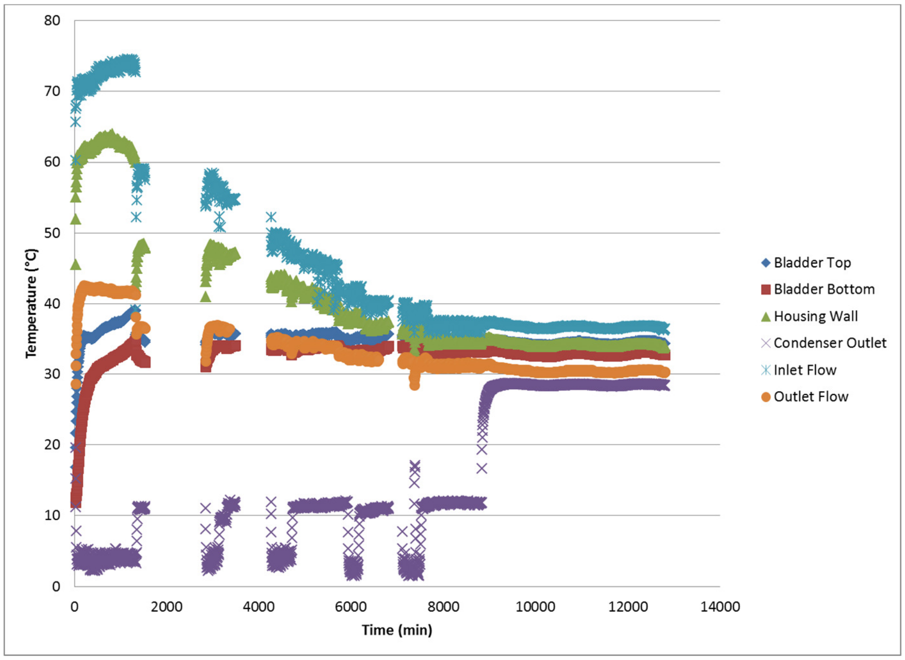

Figure 8: Brine Test 2 Temperatures

The average power consumption for each test is presented in Table 3. The average blower power is the measured blower power during the time frame for each configuration. However, the blower power consumption was calculated assuming the entire test ran in either the condenser-bypass or condenser-flow through mode, since the condenser is considered support test equipment and not part of the IWP system. This was done to demonstrate the difference in total power that would be required for a test run if it was operated in either mode without interruption. Because the pressure drop through the condenser is relatively low, the difference in blower power requirements is small. Additionally, it appears that the average heater power during the condenser bypass interval was higher than during flow through condenser. However, the air flow rate was also higher during condenser bypass testing than it was during the rest of testing. The increased heater power during this time is likely caused by the higher flow rate. The heater power from Brine 2 is a better indication of required heater power for the process because it was more closely regulated during the second test.

Table 3: Average Power Consumption

Test #	Average Heater Power (W)	Average Blower Power (W)	Blower Calculation Power Factor	Heater Energy Consumption (kWh)	Blower Energy Consumption (kWh)	Total Power Consumption for System (kWh)
Brine 1 through Condenser	413	429	0.85	37.82	39.28	77.10
Brine 1 through Test Stand (bypass)	458	416	0.85	41.99	38.07	80.05
Brine 2 (through condenser)	300	427	0.85	38.31	54.52	92.83

2. *Water Recovery*

Total water recovery was evaluated by the mass loss over the course of testing. The initial brine mass was taken from the mass of the 16 L brine reservoir. The empty mass of the new bladder was also measured. 16 L of brine was then pumped into the bladder pre-installed in the EDU Housing. Because the bladder is filled in-place with the housing sealed, the full mass of the bladder could not be measured. After testing was complete, the dried-out bladder was removed from the housing and weighed to obtain the final brine mass. From the initial and final brine masses, the extracted water mass was calculated. The starting water mass was determined by assuming the brine was initially 70% water by mass,[5] which is based on an assumed 3.5% mass fraction of the pre-processed and un-augmented human urine[6]. The brine was produced by recovering 85% of the water in urine, leaving behind 15% of the initial water in urine. The percent water recovery from brine is the ratio of extracted water to initial water mass in brine. The overall water recovery from urine (P_U) was calculated by multiplying the percent water recovery from brine (P_B) by the percent of water in brine leftover from initial urine processing (15%). That product was then added to the initial percent water recovery from urine (85%):

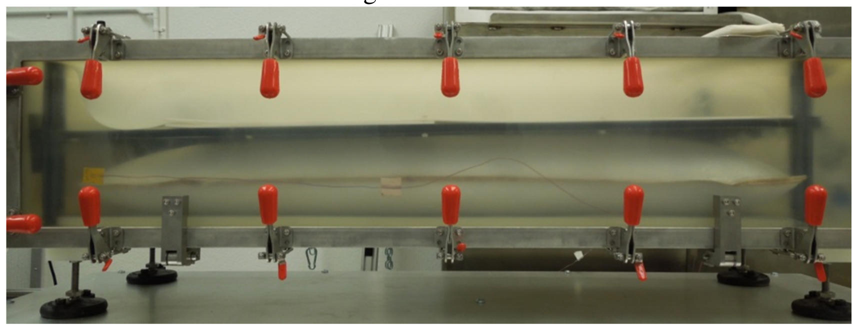
Figure 9: Full Bladder at Beginning of Brine Test 1


Figure 10: Bladder at End of Brine Test 1

$$P_U = 85\% + P_B * 15\%$$
Equation 1

The water recovery results from EDU testing are presented in Table 4. The water recovery from brine during Brine 1 was 82%. Brine 2, which ran for longer than Brine 1, recovered 86% of the initial water in brine. When looking at water recovery from urine, Brine 1 boosts overall water recovery to 97%. Brine 2 increased water recovery from urine up to 98%. This is in line with the small-scale testing which saw 98% total water recovery from urine. Figure 9 and Figure 10 demonstrate the difference in the bladder size from the beginning of testing to the end of Brine Test 1.

Table 4: EDU Brine Testing Water Recovery

Test #	Empty Bladder Mass (kg)	Final Bladder Mass (kg)	Initial Brine Mass (kg)	Final Brine Mass (kg)	Initial Water Mass (kg)	Extracted Water Mass (kg)	Percent Water Recovery from Brine	Overall Water Recovery from Urine
Brine 1	0.314	8.332	18.785	8.018	13.150	10.767	82%	97%
Brine 2	0.314	7.748	18.8	7.43	13.2	11.351	86%	98%

The water recovery rates are presented in Table **5**. While Brine 2 ran for a total of 213.5 hours, permeation was only active for 127.5 hours, as indicated by the temperature and power data previously discussed. The average permeation rate and accompanying flux was thus calculated using the total recovered water mass and active permeation time. The average permeation rates were less than predicted. The predicted peak permeation rate was 0.4 kg/hr, with a predicted average of 0.33 kg/hr. Because of the unreliable RH meter data, the peak permeation rate cannot be determined. The average rates were 27-37% of the predicted rate. As previously discussed, non-ideal flow distribution with poor airflow on the bottom of the bladder was observed during testing. Little air flow over the bottom half of the bladder may have decreased the permeation rate.

It is also suspected that the originally predicted permeation rate was unrealistically high. The conceptual system design used permeation rates coupled with urine/brine concentration from previously performed small-scale endurance testing. While the permeation rates corresponded to the concentration of urine from initial urine to concentrated brine, the rates were taken from the beginning of the endurance test when there were fewer total solids in the test rig to inhibit water access to the membranes.[5] Thus the solids-to-surface area ratio during that segment of time was much lower than it was during EDU brine testing. Additionally, the EDU testing average permeation rate includes the taper in permeation rate towards the end of drying which skews the average rate down. The lower permeation rate from Brine 2 than Brine 1 is believed to be a result of this taper. The permeation rate towards the end of the active permeation time may have been so slow that there was not a significant mass of water recovered during that time compared to the duration.

Although the achieved permeation rates were not has high as anticipated, water recovery was still high enough to process the brine produced by the ISS UPA. In 22 L of brine, there is approximately 17.6 kg of water. Recovering 17.6 kg of water over the 18-day cycle time requires an average recovery rate of 0.04 kg/hr, which is 2 to 3 times less than the average rate reached in both brine tests. This substantial margin over the target system requirement demonstrates opportunities to make the system more efficient and reduce mass and/or power. It also shows extensibility to next-generation systems outside of ISS which may have higher recovery rate requirements. For example, a one year transit mission to Mars with a crew of four is expected to produce 28L of brine over 21 day periods. Of that 28L of brine, 86.7% by volume is recoverable water, or 24.28L.[3] To recover this amount of water over 21 days requires a water production rate of 0.048 kg/hr. The sub-scale EDU designed for a different system already exceeds this recovery rate with an average permeation rate of ~0.1 kg/hr.

To directly compare EDU testing to previous performance during small-scale endurance testing, the average flux is also presented, normalized to small-scale testing flux. The average flux for small-scale testing was calculated in the same manner as EDU testing: total mass loss over test duration. In comparison to previous testing, the flux rate was approximately half that from small-scale testing. But small-scale testing was performed with urine as the starting fluid, not brine. Thus the near full-scale EDU processed brine that was 5 times more concentrated than urine at a rate of half that achieved during ideal scenario, small-scale urine testing. Permeation rate can be increased with better flow control and distribution in required by the system application.

Table 5: EDU Brine Testing Water Recovery Rates

	Brine 1	**Brine 2**	**Small-scale Endurance**
Recovered Water Mass	10.767 kg	11.351 kg	10.33 kg
Total Test Duration	91.5 hrs (5491 min)	213.5 hrs (12814 min)	416.7 hrs (25000 min)
Active Permeation Time Duration	91.5 hrs (5491 min)	127.5 hrs (7657 min)	416.7 hrs (25000 min)
Average Permeation Rate	0.12 kg/hr	0.089 kg/hr	0.025 kg/hr
Average Flux Normalized to Urine Testing	$0.65\,\frac{g/in2-hr}{g/in2-hr}$	$0.48\,\frac{g/in2-hr}{g/in2-hr}$	$1\,\frac{g/in2-hr}{g/in2-hr}$

3. Water and Air Quality

Water samples were condensed and analyzed throughout all four EDU tests. Air samples were also taken during both brine tests and the first air test to evaluate purge gas quality and trace contaminants. Figure 11 shows product water being condensed during brine testing.

A summary of water sample collection for analysis is presented in Table 6. At times, multiple samples were taken each day, but only one sample per day was analyzed. Thus the sample numbers as presented in the table are sequential but not fully inclusive. Samples were typically taken at either the beginning or end of the work day. The system ran 24 hours a day during a test run.

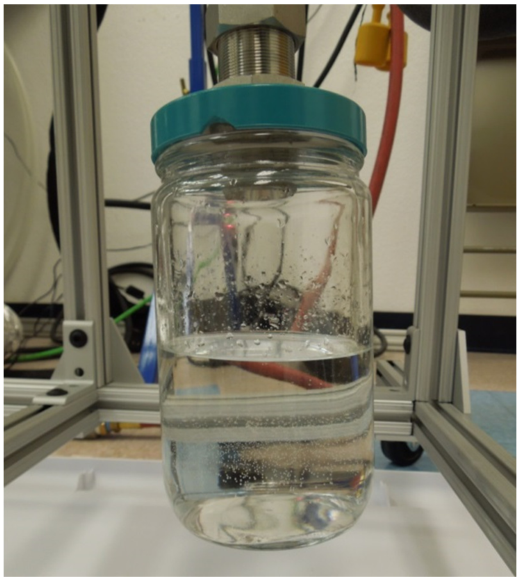

Figure 11: EDU Brine Testing Product Water Condensate

Table 6: EDU Testing Water Sample Summary

Test #	Sample	Date	Time	Sample ID
Water 1	1	5/4/2014	10:11	W1S1
Brine 1	Brine Feed 1	4/16/2014	N/A	B1FEED
	1	5/7/2014	8:25	B1S1
	3	5/8/2014	16:55	B1S3
	4	5/9/2014	16:36	B1S4
Brine 2	Brine Feed 2	4/23/2014	N/A	B2FEED
	1	5/11/2014	7:40	B2S1
	2	5/12/2014	9:01	B2S2
	3	5/12/2014	16:30	B2S3
	5	5/13/2014	16:19	B2S5
	7	5/14/2014	16:30	B2S7
	9	5/15/2014	16:05	B2S9
Water 2	1	5/20/2014	8:32	W2S1
	2	5/20/2014	14:50	W2S2

The results of water quality analysis are presented in Table 7. All water quality analyses were performed by NASA Ames Research Center, with the exception of the chromium analysis, which was performed by Accutest Laboratories in San Jose, CA. Overall recovered water quality was high with over 99% contaminant concentration reduction from the brine feed. There appears to be a slight trend towards increasing contaminant concentration over duration of a test, particularly with ammonium, although the end concentration is still extremely small compared to the brine feed and are low in concentration overall. There may have been some condensate cross-over between Brine 1 and Brine 2 given the slightly higher concentrations in the first sample from Brine 2. Contaminant concentrations dropped between Brine 2 Sample 1 and Sample 2. The trend towards increasing concentrations over the course of processing duration is apparent again with Sample 2.

Chromium analysis was only performed on the Brine Feeds and one water sample. A 99.996% reduction in total chromium was achieved in Brine Test 2. 96% of the chromium actually detected was in the form of trivalent chromium instead of hexavalent. The hexavalent chromium concentration was only 0.0068 ppm. Total Cr was 0.188 ppm, compared to the EPA safe drinking water level of $\leq$0.10 ppm.

There does not appear to be a significant contaminant accumulation between tests, as indicated by the Water 2 sample quality. At first glance, it appears that certain ion concentrations, such as ammonium and chloride, are higher in the Water 2 samples than Water 1; however, variation between compound detection and non-detect is present in the Brine tests, indicating the detected levels may be within the uncertainty of the analysis method or random variation of the test bed.

The overall water quality achieved during brine testing is very similar to the water quality results from lab-scale urine testing. Compared to UPA distillate quality, the recovered water from brine testing is on the same order of magnitude in the key areas of TOC, conductivity, and ammonium.[7] The product water can easily be processed by a downstream primary water processor to reach potable standards and not present risk or significant contaminant loading to the downstream processor.

Table 7: EDU Brine Testing Water Quality Results (units in ppm)

Sample ID	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	Cl^-	NO_2^-	Br^-
W1S1	0.9	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
B1FEED	11022	1875	13817	592	1813	19764	LDL	LDL
B1S1	0.8	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
B1S3	1.2	0.9	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
B1S4	1.3	2.4	<0.5	<0.5	<0.5	0.97	<0.5	<0.5
B2FEED	13678	2173	16753	443	1430	21544	LDL	LDL

Sample ID	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	Cl^-	NO_2^-	Br^-
B2S1	1.3	3.3	<0.5	<0.5	<0.5	2.89	<0.5	<0.5
B2S2	0.8	<0.5	<0.5	<0.5	<0.5	0.6	<0.5	<0.5
B2S3	0.6	3.7	<0.5	<0.5	<0.5	3.1	<0.5	<0.5
B2S5	1.0	4.8	<0.5	<0.5	<0.5	2.6	<0.5	<0.5
B2S7	1.3	6.4	<0.5	<0.5	<0.5	2.6	<0.5	<0.5
B2S9	1.5	8.0	<0.5	<0.5	<0.5	1.8	<0.5	<0.5
W2S1	0.7	5.4	<0.5	<0.5	<0.5	1.3	<0.5	<0.5
W2S2	0.8	2.4	<0.5	<0.5	<0.5	0.7	<0.5	<0.5
Sample ID	**NO_3^-**	**PO_4^{3-}**	**SO_4^{2-}**	**TOC**	**pH**	**Cond (µS)**	**Cr, hexavalent**	**Cr, trivalent**
W1S1	<0.5	<0.5	<0.5	73.9	3.8	194	–	–
B1FEED	LDL	100739	15089	49500	2.6	225000	<0.50	5730
B1S1	<0.5	<0.5	<0.5	16.5	5.6	19.8	–	–
B1S3	<0.5	<0.5	<0.5	37.01	4.0	145	–	–
B1S4	<0.5	0.7	0.7	90.3	3.9	202	–	–
B2FEED	LDL	101083	14885	51760	2.6	229000	<0.50	4560
B2S1	<0.5	0.9	0.6	113	3.9	226	–	–
B2S2	<0.5	<0.5	<0.5	37.48	3.9	155	–	–
B2S3	<0.5	<0.5	0.8	107	3.6	210	0.0068	0.18
B2S5	<0.5	<0.5	<0.5	123	3.8	217	–	–
B2S7	<0.5	<0.5	<0.5	148	3.9	208	–	–
B2S9	<0.5	<0.5	<0.5	136	4.1	161	–	–
W2S1	<0.5	<0.5	<0.5	33.7	5.6	52.4	–	–
W2S2	<0.5	<0.5	<0.5	36.7	6.3	24.7	–	–

A quantitative purge gas analysis for specific trace contaminants was performed on gas samples taken during Water 1 and Brine 1. The purge gas analysis was performed by ALS Environmental in Simi Valley, CA. The trace contaminants list was selected by cross-referencing the volatiles found in the UPA purge gas[8] which vents to the cabin on ISS, with the driving contaminants list for the Trace Contaminant Control Assembly (TCCA)[9]. Water 1 gas analysis was performed to establish a baseline for the lab air used as the purge gas. The only significant contaminant increase from the water to brine test was acetone. Acetone is normally present in urine and is also known to pass through Nafion®,[10] so the presence of acetone in the purge gas is not surprising. The concentration is well below the 180 day Spacecraft Maximum Allowable Concentration (SMAC), as are all of the other compounds tested. The only other compound with a slight increase from water to brine testing was acetaldehyde, though it is difficult to determine if the increase is significant compared to atmospheric and indoor concentrations.[11]

Table 8: Quantitative Purge Gas Analysis Results and Corresponding SMAC[12]

Test	Ammonia (ppm)	Acet-aldehyde (ppb)	Methanol (ppm)	Acetone (ppb)	Carbonyl Sulfide (ppb)	Benzene (ppb)	Toluene (ppb)	Dichloro-methane (ppb)
Water 1	<2.0	5.6	<1.7	91	<6.9	<0.22	1.1	<0.20
Brine 1	<2.0	12	<1.4	2100	<7.0	<0.44	1.5	<0.40
180 d SMAC	3	2000	70	22000	N/A	70	4000	3000

4. Observations

Overall, the EDU functioned as designed. The bladders were installed into the EDU and filled in-place with minimal operator exposure to brine. The bladders remained intact and were not compromised during brine testing. Brine was fully contained in the bladders at all times. No brine leaked into the EDU housing or purge flow. However, pinholes were observed in the microporous membrane after testing where brine could be seen through the clear Nafion®. During Brine 2, brine actually leaked through the microporous ePTFE in three locations, but was fully contained by the Nafion® in all cases. Figure 12 through Figure 14 demonstrate the bladders after testing and the leak through ePTFE and Nafion® containment. Nafion® is the clear top layer and ePTFE is the white bottom layer. Manufacturing methods will be refined in follow-on development work to eliminate the small leaks through the ePTFE.

Figure 12: Bladder after Brine Test 1

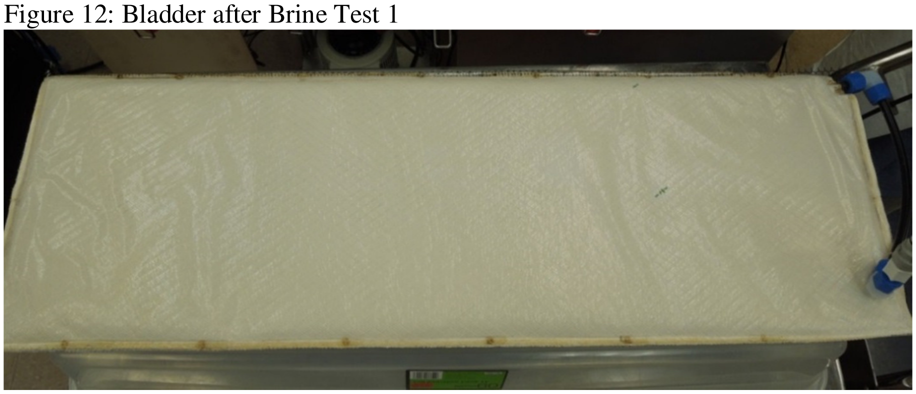

Figure 13: Bladder after Brine Test 2

Figure 14: Brine leak through ePTFE contained by Nafion®

III. IWP EDU Testing at Ames Research Center

The IWP EDU was delivered to Ames Research Center in June 2014 for evaluation. Four test runs having various objectives were conducted. Table 9 gives a description of the runs with objectives, and a summary of analysis and results. **Table 10** gives a summary of performance data. The primary objective of Brine Test 1 (BT1) was to verify the IWP performance as reported by Paragon. The IWP was run under similar conditions of heater power and temperature as Paragon. A summary of the results as compared to Paragon testing results is given in Table 10.

The IWP was run as an open system and vented to the fume hood using lab environment air as the carrier gas for evaporation of water from the membrane bag. (Succeeding tests were conducted as a closed system with the air being recycled back into the IWP in order to control dew point and obtain a more accurate mass balance and production rates). Open system venting was basically the same operational test procedure as Paragon, however, humidity control as measured by the dew point could not be replicated at Ames. The Ames lab environment air had a dew point temperature of about 12.5°C whereas at Paragon the dew point was 7°C to 8°C.

Water recovery was based on the measured density of the initial brine of 1.139 g/ml and 0.256 g/ml total solids; the water available for recovery was calculated to be 77.3% by mass.[5] This method of calculated water available for recovery differs from the method utilized for Paragon testing. In order to directly compare Ames and Paragon testing, the Paragon water recovery test results were recalculated using the brine density and total solids measurements utilized by Ames; results are presented in Table 9.

[5] This corresponds to approximately 2% solids by mass in the urine with alternate pre-treat before processing in the WFRD. This urine pre-treat (with 98% water) is processed by the WFRD which then removes 85% of the total urine mass as water through distillation. The 2% solids in the pre-treated urine is less than the generally accepted concentration of 3-4% solids in untreated urine. This difference in solids concentration may be due to the pretreat process. The calculation also takes into consideration volume of solids. These values correspond to the measured initial density of the brine of 1.139 g/ml.

BT1 testing results were similar to the Paragon results. BT1 testing was composed of two parts. Part 1 was run for 147.5 h (6.12 days) which was thought to be of sufficient time to collect a majority amount of recoverable water. The amount of mass reduction of the initial brine for part 1 testing was 53.6%. This amount of time was comparable to Paragon's Brine Test 1 (91.5 h). The BT1 part 2 test was a continuation of BT1 part 1 with the objective of recovering as much water as possible within a reasonable amount of time. Part 2 continued for an additional 267 h. The combined time for Parts 1 and 2 was 480 hrs (20 days), for a combined mass reduction of the initial brine of 61.3% which corresponds to a water recovery ratio of 79.3%. This was comparable to the 60.4% mass reduction and 78.2% water recovery ratio of Paragon's Brine Test 2 where the objective was to also recover the maximum amount of water. Refer to Table 10 for a tabulated listing of all test run results.

Table 9: Summary of tests

Test Title	Brine Tested	Test Objectives	Summary of results and observations	Analysis
Brine Test 1: Part 1	Alternate Pre-treat	Verify performance reported by Paragon	Results generally confirmed the performance reported by Paragon. Mass reduction from initial brine is 53.58% over 147.5 hrs with total heater energy of 37kWh, and total blower energy of 38.74 for a total energy of 77kWh. This is comparable to Paragon's Brine Test 1. Time of operation is 55% longer than Paragon Brine Test 1	Longer run time than Paragon - possibly due to higher lab environment air dew point at 12.5 °C while Paragon ran a dew point of between 7 and 8 °C. The lower the dew point, the dryer the air. Also total heater energy was about same, but time 55% longer meaning ΔT lower for BT1.
Brine Test 1: Part 2	Alternate Pre-treat	Continue Brine Test 1 to maximum water recover; test closed – system recycle flow	Achieved 60.2% mass reduction from initial brine over 230 hrs of additional operation after BT1: part 1. Closed-system recycle flow was demonstrated, with control over inlet dew point and better overall mass balance of water production rate by measuring collected condensate over time.	Maximum mass reduction of 60.2% corresponding to a water recovery of 79.3% is comparable to Paragon Brine Test 2 maximum mass reduction test of 78.2%
Brine Test 2	Alternate Pre-treat	Repeat to confirm results of BT1; use recycle flow	Intent to run a brine alt pre-treat completely with recycle. Could not complete test because brine started seeping through the ePTFE membrane after overnight operation. Run was stopped, though it may have been useful to continue because Nafion® may have kept the brine contained.	It was surmised that the brine may have been contaminated with trace amounts of hygiene water. A hygiene pretreat was produced before this batch of brine, and even though the WFRD was thoroughly flushed out and cleaned, there could have been locations in the WFRD where a small amount of hygiene water may have remained.
Brine Test 3	Alternate Pre-treat	Retest BT2 to see whether seeping was due to bag or due to contamination with hygiene water	Seeping was observed in the same manner as BT2. The run continued until there was notice of a small amount of brine leaking out from the bag and into the housing near the neck of the fill tube. Seeping through the ePTFE was concentrated at the front of the bag where the warmed air entered the housing and where temperatures were highest. Less seeping was observed along the length of the bag as the temperatures decreased (see Fig. 4)	It appeared that brine seeped through the ePTFE membrane but was contained by the Nafion®. Seeping at the front of the bag where the temperatures are highest suggest that possibly the brine possibly contained trace hygiene solution since surface tension decreases with increasing temperature.
Brine Hygiene 1	Alt Pre-treat Solution 2^3 with hygiene water	Test to determine whether the membrane can retain Alt-pretreat with hygiene water	The system was run without heating using only recycle air. After two hours a small amount of seeping was observed as streaks on the ePTFE. The system was run over night and by the next morning the ePTFE was completely covered in brine. The system was stopped due to a brine film on the bottom of the housing. .	A detailed examination was conducted to determine whether the brine seeped through the Nafion®. There was no indication that there was seeping of the brine through the Nafion®, however there was a leak at the neck of the seam at the fill tube. It appears that Nafion® can retain the hygiene solution brine, but the ePTFE cannot. Sealing the Nafion® as part of the bag near the neck of the tube may prove difficult.

Table 10: Summary of Water Recovery Data and Averaged Results

Test ID	Initial Brine Mass	Final Brine Mass	Final - Initial= water recoved	% mass reduction	% water in Initial Brine based on measured brine density of 1.139 g/ml and 0.256 g/ml total solids	Amount of "recoverable water"	% water recovery	Run Time	Run to completion?
Paragon Brine Test 1	18.79	8.02	10.77	57.3%	77.30%	14.521	74.1%	91.5	not quite
Paragon Brine Test 2	18.79	7.43	11.35	60.4%	77.30%	14.521	78.2%	213.5	nearly
ARC BT1 parts 1&2	17.77	6.88	10.90	61.3%	77.30%	13.739	79.3%	480.77	probably
ARC BT2	18.75	13.59	5.17	27.5%	77.30%	14.494	35.6%	41.72	no
ARC BT3*	17.17	8.47	8.70	50.6%	89.02%	15.285	56.9%	147.12	no

* Measured density dissolved solids initial brine = 0.125 g/ml

BT3 was run in recycle flow configuration for a total of 147 h before stopping the test due to leakage. Seeping was observed similar to BT2. After 21 hrs there appeared green streaks on the bag surface which gradually increased over time. The distribution of the streaks was concentrated at the front end of the bag where the warm heated air entered the EDU housing, and gradually diminished along the length of the bag in the direction of flow. Because of this observation, we determined that it was probable that surfactant did contaminate the brine, since surface tension decreases with increasing temperature, hence the higher number of streaks (brine breakthrough) at the warmer part of the bag, as shown in Figure 15. The run continued as long as there was no leakage into the housing. At 147 hrs we observed a small drop of brine below the bag's fill tube. Subsequently the run was stopped. Later examination of the bag showed that there was not any seeping of brine through the Nafion® as determined by wiping a white tissue on the Nafion® surface and not seeing any pickup of green brine. It was also discovered that there was a small leak at the seam joining the fill tube with the bag.

The objective of the brine hygiene test (BHT) was to determine whether the membrane bag could operate if surfactants (from the hygiene water) were a part of the brine. The results showed that the brine seeped through the ePTFE membrane but was contained by the Nafion® ionomer membrane.

The system was run without heating using only recycled air that exited from the condenser. After two hours some seeping was observed as streaks on the bag. The system was run over night and by the next morning the ePTFE was completely covered in brine as shown in Figure 15. The system was paused at 17.5 h and opened to see if there were any leaks. A brine film on the bottom of the housing was observed and so the run was stopped. A detailed test was conducted to determine whether the brine leaked through the Nafion® using a dry tissue and wiping it on the Nafion® bag surface. There was no indication that there was seeping of the brine through the Nafion®. The leaking brine was found to be coming from the seal between the membranes and the fill tube as shown in Figure 15. This defect in the sealing allowed brine that seeped through the ePTFE to then leak into the housing. It appears that Nafion® can retain the hygiene solution brine, but the ePTFE cannot. Because of the leak, the test was stopped and because no heater power was applied, no relevant performance data was collected.

It is interesting to note that even though there was zero heat input into the IWP, that is, the inlet conditions to the housing were those of ambient ISS cabin air conditions at a dew point of about 7.5°C, there was still permeation and evaporation of water from the brine. A subsequent calculation of the electronics inverse specific energy (water produced per unit energy of operating electronics) was highest at 1.56 l/kWh vs. the other runs, which were less than 0.47 l/kWh. In addition, a production rate of 24 g/hr of condensate was obtained. It appears that the efficiency of the IWP is much better at lower production rates, hence, in designing a system one might design to the minimum production rate as required in order to achieve the highest specific energy.

After the brine bag was removed from the housing it was examined to determine whether there was any seeping through the Nafion®. A tissue was wiped on the surface of the Nafion®. There was no indication of brine seeping, as the tissue remained clean. See Figure 15. Leaking of the brine occurred at the neck of the fill tube connected to the bag. It is surmised that even though the ePTFE appears to be sealed to the fill tube, the Nafion® was not properly sealed to the tube. This would allow leaking of brine at the tube neck after seeping through the ePTFE membrane. That is, trapped brine between the ePTFE and Nafion® membranes leaked at the seal between the fill tube and Nafion®.

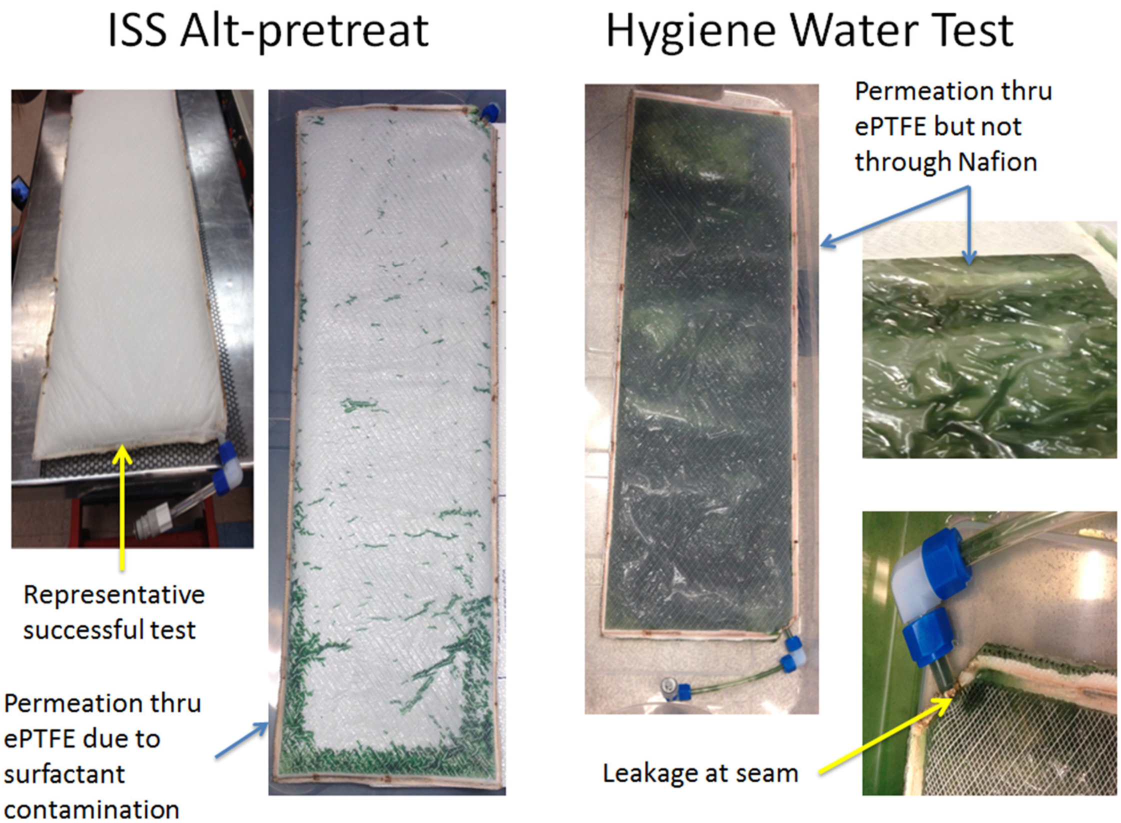

Figure 15: Patent-pending IWP brine bags post-testing at NASA ARC. From left to right bags are shown from Brine Test 1 (BT1), Brine Test 3 (BT3), and Brine Hygiene Test (BHT).

Table 11 lists the results of the water analysis. Water quality analysis was performed by NASA ARC. Overall water condensate quality was good, though not drinking water quality. TOCs in the condensate generally ranged from 10-100 ppm at the initial stages of condensate collection and increased to > 465ppm for BT1 at the later stages of condensate collection. A qualitative GC-MS analysis was performed on the purge gas by Lance Delzeit of NASA ARC and is reported separately in ICES-2015-69 *Results of the GCMS effluent gas analysis for the Brine Processing Test*.[13]

Table 11: Ames IWP EDU Testing Water Quality Results (all units in ppm)

Sample ID	**Na^+**	**NH_4^+**	**K^+**	**Mg^{2+}**	**Ca^{2+}**	**Cl^-**	**NO_2^-**	**Br^-**
IWP Brine Test 1 Condensate Jar 9	3006	0.6	<0.5	<0.5	<0.5	<0.5	0.8	<0.5
IWP Brine Test 1 Condensate Jar 10	3007	<0.5	<0.5	<0.5	<0.5	0.8	3.6	<0.5
Sample ID	**NO_3^-**	**PO_4^{3-}**	**SO_4^{2-}**	**TOC**	**pH**	**Cond (µS)**	**%TSS**	**%TDS**
IWP Brine Test 1 Condensate Jar 9	2.5	225	3.5	154	*	<0.5	*	<0.5
IWP Brine Test 1 Condensate Jar 10	1.8	465	3.5	179	*	<0.5	*	<0.5

IV. Conclusion

A 75% scale EDU of an IWP brine processor for ISS was developed that mimicked the functionality of a flight unit. The large-scale EDU was successfully designed, manufactured, and tested. The EDU was tested with brine produced by removing 85% of the water in ISS Alternative pretreated urine. The brine was produced by the Wiped-film Rotating Disc urine processor at NASA Ames Research Center and accurately represents the brine a processor on ISS would be treating. The IWP EDU successfully recovered 80-86% of the water from a 16 L batch of brine in 5.3 days. Because the starting brine retained 15% of the water from the initial urine, 86% water recovery from brine translates to 98% total water recovery from urine. The 75% of full-scale EDU achieved the same total water recovery from urine accomplished in previous small-scale testing. Independent testing of the IWP EDU by NASA Ames Research Center corroborated Paragon test results, with similarly-calculated water recovery of 80% achieved by both Ames and Paragon testing and comparable recovered water quality. Some membrane sealing issues were encountered during testing at both Ames and Paragon. An effort is currently underway to improve the manufacturing process to produce more reliable and robust seals.

Water recovery rates achieved during testing were high enough to process the brine produced by the ISS UPA. Recovering the 17.6 kg of water in 22 L of brine over the 18-day cycle time requires an average recovery rate of 0.04 kg/hr. The average rate reached in both brine tests was ~0.1 kg/hr, 2.5 times the water recovery rate required for ISS. This substantial margin over the target system requirement demonstrates opportunities to make the system more efficient and reduce mass and/or power. It also shows extensibility to next-generation systems outside of ISS which may have higher recovery rate requirements, such as a 4 person crew mission to Mars, which requires a water production rate of 0.048 kg/hr. The sub-scale EDU already exceeds this recovery rate with an average permeation rate of ~0.1 kg/hr.

The water quality from IWP water recovered from brine was high and could easily be polished to potable standards in a main water processor such as the WPA on ISS. The recovered water quality from brine was on the same level or better than urine distillate quality from the UPA. The pH was also high enough such that it would not be damaging to downstream processors. Purge air quality testing demonstrated no significant contaminants in the purge air, with all compounds tested having concentrations well below their corresponding SMAC. The low volatile concentrations reinforce Nafion®'s property of selective permeability of water vapor and few other gases. Preliminary purge gas testing indicates that the IWP purge gas is safe and would not contribute significant trace contaminants to cabin atmosphere or significantly increase the loading on a trace contaminant removal system. However comprehensive, quantitative purge gas analysis is needed and planned in future work to better characterize the volatile permeation through IWP.

IWP can be easily scaled up to full-scale systems, as demonstrated by the total water recovery and permeation rates achieved in EDU testing. There is high confidence for operating the IWP in micro-g since there are no free-floating phase change boundaries. The membrane bag of the IWP both constrains the brine and acts as a phase separator due to the dominant force of surface tension, thereby allowing for zero-g operation. Once the brine is transferred into the bladder, no human interaction is required with the brine, as the bladder provides the containment for the brine through all stages of processing and disposal. IWP is flexible enough to process not only urine brine, but can also directly process urine to the same levels of total water recovery. Thus IWP could function as a brine processor or primary urine processor. EDU testing demonstrated the batch-processing mode for an IWP brine processor that can be readily employed on ISS as an operational brine processor or as a flight experiment. Future work is planned to develop IWP as a continuous feed brine processor, such as would be ideal on next-generation exploration space craft. The ability of Nafion® to retain the urine/hygiene brine with surfactants, allowing only water through, also shows potential for IWP to process hygiene wastewater, even though it was only designed for urine brine, which would allow for greater water usage during long duration missions such as a mission to Mars. The IWP EDU fulfilled all of its design objectives, and demonstrated its performance as a simple, effective, and safe brine processor.

Acknowledgments

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