Development of Ionomer-membrane Water Processor (IWP) technology for water recovery from urine

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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 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 being used onboard ISS distills water from pretreated urine, recovering ~75% of the water in the urine. However the system is complex and produces concentrated brine that requires further processing for water recovery. Paragon is developing the use of ionomer-microporous membrane technology known as Ionomer-membrane Water Processor (IWP) to improve the robustness and effectiveness of and to simplify water recovery processes for space applications. Through a NASA SBIR Phase 2 effort, Paragon developed IWP through membrane selection testing, a conceptual system analysis utilizing IWP as brine processor for ISS, and design of an engineering development unit (EDU). Results of that development and testing are presented, including achieved water recovery and product water purity.

Nomenclature

ARFTA	=	Advanced Recycle Filter Tank Assembly	*PP*	=	Polypropylene
BRIC	=	Brine Residual In-Containment	*PTFE*	=	Polytetrafluoroethylene
CCAA	=	Common Cabin Air Assembly	*PUP*	=	Paragon Urine Processor
CDRA	=	Carbon Dioxide Removal Assembly	*PVDF*	=	Polyvinylidene
CHX	=	Condensing Heat Exchanger	*RH*	=	Relative Humidity
EDU	=	Engineering Development Unit	*SBIR*	=	Small Business Innovative Research
ePTFE	=	expanded Polytetrafluoroethylene	*SLPM*	=	Standard Liters Per Minute
F.A.S.T.	=	Filtration and Separation Technology	*TC*	=	Thermocouple
ISS	=	International Space Station	*TDS*	=	Total Dissolved Solids
IWP	=	Ionomer-membrane Water Processor	*TIC*	=	Total Inorganic Carbons
JSC	=	Johnson Space Center	*TOC*	=	Total Organic Carbons
MDL	=	Minimum Detection Limit	*UPA*	=	Urine Processor Assembly
NASA	=	National Aeronautics and Space Administration	*VCD*	=	Vapor Compression Distillation
PMP	=	Polymethylpentene	*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,

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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 methods such as phosphoric acid, ~15% of the water is left behind in the brine. Paragon seeks to recover a higher percentage of water 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 is developing the use of ionomer-microporous membrane technology to simplify and improve the effectiveness of water recovery processes for space applications. Improved effectiveness will be evident through (1) reduced loading on the downstream post-processor due to the ionomer's unique property of selective permeability, (2) near complete removal of water from wastewater, and (3) inclusion of a backup barrier between the retentate and permeate. The technology offers simplification over existing technology through (1) a lower dependency on moving parts, and (2) integrated capture of wastewater solutes for disposal. Testing to date has demonstrated 99% removal of contaminants in pretreated urine and virtually complete dewatering of the brine in a configuration designed for microgravity application. The IWP system is being conceived to remove nearly all of the solids from a wastewater stream, while operating at low temperatures and allowing only minor contamination cross-over. As the technology is fully developed, it can be inserted into existing and/or developing water recovery system architectures to increase water recovery rates beyond those available to date.

A. Background

To date, little research has been done to incorporate cation exchange ionomer membranes such as Nafion® to separate water from wastewater for space applications. Nafion is a copolymer of tetrafluoroethylene (Teflon) and perfluro-3,6-dioxa-4-methyl-7-octene-sulfonic acid[i]. Like Teflon, Nafion is highly resistant to chemical attack, but the presence of its exposed sulfonic acid groups confers unusual properties. Of relevance, "the sulfonic acid group is immobilized within the bulk fluorocarbon matrix and cannot be removed, but unlike the fluorocarbon matrix the sulfonic acid groups do participate in chemical reactions. The presence of the sulfonic acid adds three important properties to Nafion:

1. Nafion functions as an acid catalyst due to the strongly acid properties of the sulfonic acid group.

2. Nafion functions as an ion exchange resin when exposed to liquid solutions.

3. 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. Nafion functions like a very selective, semi-permeable membrane to water vapor."[i]

Nafion's ability to selectively allow water to permeate suggests a possible role in water purification processes. This activity is accomplished by means of the sulfonic acid groups. The 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[i]. 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 by blocking it from liquid contact. The IWP membrane pairing and water transport is demonstrated in Figure 1.

As previously mentioned, Nafion is extremely resistant to chemical attack. In fact, strong acids are routinely used to regenerate Nafion if it has been exposed to solutions containing cations. The hydrogen ions in the acid exchange places with the cations that had supplanted the hydrogen ions of the sulfonic acid group, thus regenerating the ionomer. The membrane is inert and is safely disposed of in landfills. Nafion does not burn in ambient air and is one of the most flame resistant plastics, with a limiting oxygen index of 95%. Nafion starts to thermally degrade at temperatures above 250°C, evolving SO_2 at 280°C and HF beginning at 400°C. In fuel cell applications, it is believed that hydroxyl radicals can "unzip" the polymer backbone in Nafion, releasing fluoride ions in the product water.[ii] However, in the intended application of Nafion in the IWP, the ionomer will not be exposed to sufficient heat or hydroxyl radicals to cause said degradation.
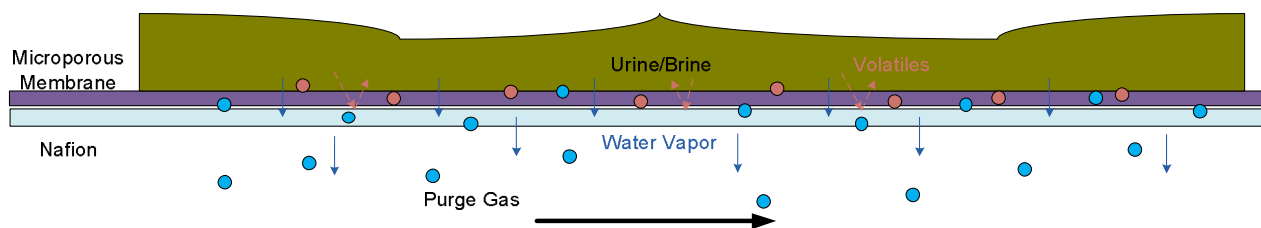
Figure 1. Water Transport across IWP Membranes (Solid blue arrows and blue circles represent water vapor. Red circles and dashed red arrows represent volatile gases. No liquid is in contact with the Nafion)

When active, Nafion's sulfonic acid groups allow some chemicals other than water to pass. Of note, alcohols, ammonium hydroxide, and primary and secondary amines are selectively passed through the membrane, driven by their vapor pressure gradients. Nafion is also a super-acid catalyst. The sulfonic acid groups are strong proton donors, capable of converting certain organic compounds into other compounds. Alkenes and alkynes, double and triple-bonded organic compounds, both undergo acid catalysis and may be transformed into different compounds by the Nafion. Organic compounds with single bonds or benzene rings do not undergo acid catalysis. In some instances the reaction products of acid catalysis are permeable and pass through the membrane. Organic carbons containing a carbonyl group undergo acid-catalyzed enolization to form an alcohol, which subsequently can permeate through Nafion.[i]

Nafion's ability to retain numerous contaminants could significantly reduce the load on downstream post processors, and when paired with a hydrophobic membrane, would serve to contain 100% of the solid mass in the wastewater.

Expanded polytetrafluoroethylene (ePTFE) is a hydrophobic microporous membrane paired with Nafion. The pores of the membrane are sufficiently small to prevent liquid water from passing through, but do allow gases and vapors to pass. The ePTFE membrane thus protects the Nafion membrane from liquid contamination, but allows gas to reach the Nafion, which then selectively permeates the water vapor.

II.   Membrane Selection Testing

A. Test description and Analysis Details

Membranes were tested in a planar test rig to evaluate membrane characteristics, operating conditions, and water recovery. The Nafion membranes varied in thickness and reinforcement. The microporous membranes varied in porosity and surface behavior (hydrophobic and oleophobic). Each of three microporous membranes tested were each paired with Nafion A. Once it was determined which membrane performed best with Nafion A, that membrane was then paired and tested with both Nafion B and C to determine which Nafion has the best performance. The last membrane tested was a composite microporous and Nafion membrane.

The membranes were laid on top of each other and placed in the test rig. The test rig was then sealed and the reservoir filled with 300-700 mL of sulfuric acid-chromium trioxide pretreated urine. Urine was collected from both male and female Paragon employees. The urine was augmented to increase calcium, phosphate, sulfate, and organics levels to those found in typical astronaut urine following Verostko's 2010 formula.[iii] The urine was then pretreated with the Russian ISS pretreatment of chromium trioxide and sulfuric acid.[iv]
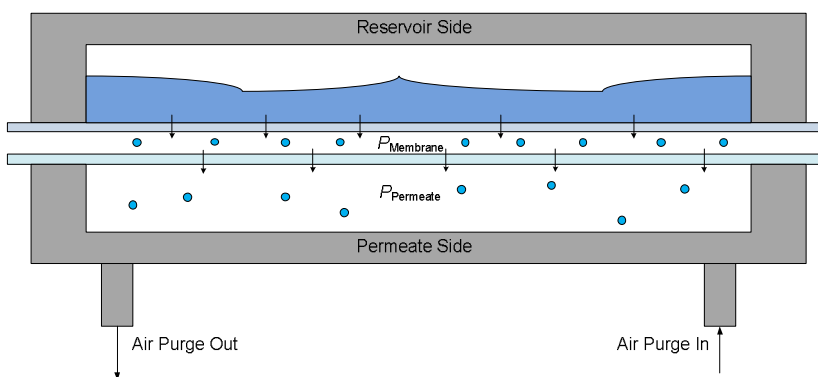

Figure 2. Test Bed Transport Illustration

Testing was conducted in two modes – liquid and vapor contact. In vapor contact mode, the reservoir was on the bottom and an air gap existed between the urine and the membranes. In liquid contact mode, pictured in Figure 2, the test rig was inverted so that the reservoir was on top and the urine resided directly on the microporous

membrane. Figure 2 illustrates a gap between the membranes to demonstrate the water phase contact of each membrane; however, there is no mechanism maintaining a gap between the membranes. The membranes are simply sandwiched together. The permeate side consisted of flow channels and an inlet and outlet manifold. The purge air flowed across the surface of the Nafion membrane, picking up permeated water vapor and transporting the water to a condenser.

Raw data from each test was recorded using data acquisition software to record temperatures, flow rates, pressures and relative humidities. The data was transferred and analyzed in Excel. Water and gas samples collected during testing were sent to three separate labs. The water sample was tested for total ammonium, urea, methanol, ethanol, propylene glycol, total dissolved solids (TDS), acetate, formate, sulfate, total inorganic carbons, total organic carbons, pH and conductivity. The gas sample was analyzed for total trace contaminants; however, the results were inconclusive and are not discussed further.

After the membrane selection tests were completed, the best overall performing membrane configuration was chosen to perform an endurance test to determine the life expectancy of the membrane pair. The same analyses performed for the membrane selection tests were also done for the endurance test. Total urine processed and water recovered were also evaluated. A condenser captured much of the permeated water vapor. But not all of the water vapor condenses, as the purge air stream exits the condenser saturated at the condenser outlet temperature. For this reason, water vapor flow rate as calculated from the measured relative humidity was used as the primary measurement of permeated water, as opposed to amount of water collected. The total mass of water that permeated through the membranes was determined by calculating the area under the curve of the graphed permeation rates over time using the trapezoidal rule, Equation 1.

$$\int_a^b f(x) = \sum_m^n (b-a)\frac{f(a)+f(b)}{2} \qquad (1)$$

In this case f(a) and f(b) represent the permeation rate in kg/sec while (b – a) represents the time difference in seconds. Since the Labview data acquisition software was programed to record data points at set intervals, the length between each data point remained constant at 60 sec. When multiplying the height by the length the resulting solution shows the total kilograms of water that permeated in that 60 sec period. Therefore, adding each 60 sec segment shows the total amount of water that permeated for the duration of the test.

Once the total mass of permeated water was determined, the total mass of water in the pretreated urine was calculated so that the total percent water recovery could be found. To determine the total mass of water injected into the test bed, the solids mass fraction of the untreated urine was established. Based on urine composition data from Putnam[v], 3.5% was assumed as the mass fraction of untreated urine. Given 3.5% solids mass fraction, the density of urine is 1.010 g/ml[v] and thus the mass of 1 liter is 1010 g. Therefore the total mass of water in 1 liter of normal, untreated urine is approximately 973 g. The untreated urine is then augmented to match that of a crewmember of the International Space Station (ISS).[vi] During augmentation, 79.93 g of non-water compounds and 277.70 g of water are added. The total mass after augmentation is 1317.99 g. Dividing the total amount of non-water compounds by the total mass of the pretreated, augmented urine results in a mass fraction of 6.06% of non-water compounds and 93.94% water.

To calculate the total mass of water in the pretreated, augmented urine used in the endurance test, the total amount of urine added was multiplied by the mass fraction of water in the pretreated augmented urine. The percent water recovery was then calculated by dividing the mass of permeated water by the total mass of water in the pretreated, augmented urine added to the reservoir.

Table 1. Membrane Selection Test Designations

Test Designation	Date	Contact Mode	Membrane Pair
Water Baseline	05/14/2013	Liquid	Oleophobic and Nafion A
Urine Test 1	05/15/2013	Liquid	Oleophobic and Nafion A
Urine Test 2	05/30/2013	Vapor	Oleophobic and Nafion A
Urine Test 3	06/04/2013	Vapor	No Membrane
Urine Test 4	06/10/2013	Liquid	Oleophobic and Nafion A
Urine Test 5	06/20/2013	Liquid	Oleophobic and No Nafion
Urine Test 6	06/24/2013	Liquid	Hydro A and Nafion A
Urine Test 7	06/26/2013	Liquid	Hydro B and Nafion A
Urine Test 8	06/28/2013	Vapor	Hydro B and Nafion A
Urine Test 9	07/11/2013	Liquid	Oleophobic and Nafion B
Urine Test 10	07/18/2013	Liquid	Hydro A and Nafion B
Urine Test 11	07/22/2013	Liquid	Hydro A and Nafion C
Urine Test 12	07/24/2013	Liquid	Composite Membrane
Urine Test 13	07/31/2013	Vapor	Hydro A and Nafion B
Urine Test 14	08/05/2013	Liquid	Nafion B only
Urine Test 15	08/08/2013	Liquid	Hydro A only
Urine Test 16 (Endurance Test)	8/12/13 – 9/11/13	Liquid	Hydro A and Nafion B

Table 2. Membrane Description

Membrane	Description
Oleophobic	Oleophobic ePTFE membrane, small pores
Hydro A	Hydrophobic ePTFE membrane, small pores
Hydro B	Hydrophobic ePTFE membrane, large pores
Nafion A	thin Nafion membrane
Nafion B	thick Nafion membrane
Nafion C	reinforced Nafion membrane
Composite	Nafion Coated ePTFE membrane

B. Membrane Selection Test Results

Table 1 presents each test date, its designation and the membrane pair that was tested. Table 2 shows a brief description of each membrane. Figure 3 illustrates each membrane pair's performance when no heat is applied to the test bed at varying purge flow rates. Figure 4 shows the performance of each membrane pair when heat was applied and held steady at approximately 35°C.

Figure 5 shows the effect that temperature has on the permeation rate for some of the membrane pairs at 50 slpm. From the charts it can be seen that temperature has more of an effect on permeation rate than the purge flow rates. When the Nafion B was tested by itself it performed worse than it did when paired with a microporous membrane. This was expected due to ion exchange that would occur when in contact with the pretreated urine. The microporous membrane however performed just as well if not better as some of the membrane pairs. The membrane pair that had the best permeation rate was the Hydro B and Nafion A, but due to its dimensional instability during handling and overall low durability, it was eliminated from the microporous membrane choices. The next best membrane pair was the water baseline test that used the Oleophobic membrane and Nafion A. This was only shown on the water baseline however and not the urine test. Therefore the best pair, based on both handling and performance, was determined to be the Hydro A and Nafion B.
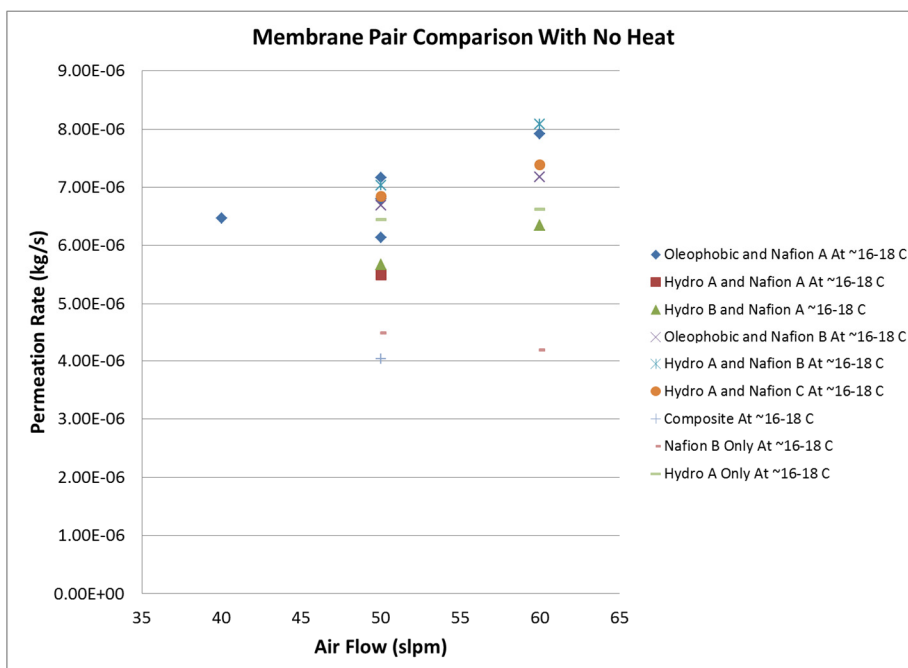
Figure 3. Membrane Pair Comparison Graph with No Heat Applied in Liquid Contact Mode
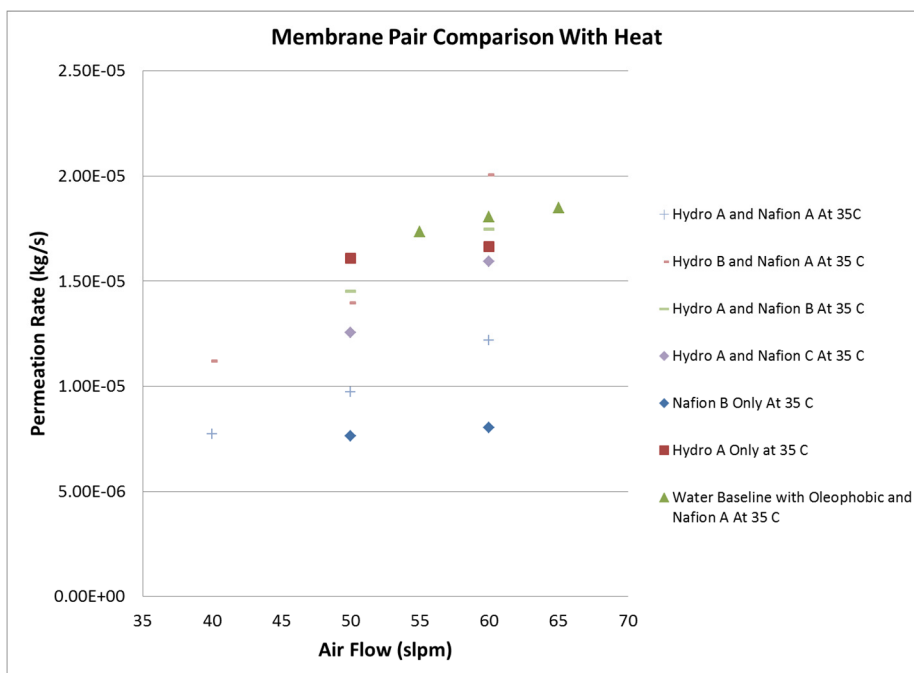

Figure 4. Membrane Pair Comparison Graph with Heat Applied in Liquid Contact Mode

Figure 6 shows the reservoir after Urine Test 7. The test started with 395.8 g of pretreated urine and ended with a final brine mass of 29.8 g, a 92.5% recovery of the total mass and a total *water* recovery of 98% by mass. Using the RH data to calculate total water processed results in 97% water recovery with 6% uncertainty, demonstrating good correlation between the two measurement methods (mass and RH). The best recovery was seen during Urine Test 4, which demonstrated 95% mass recovery and 98% total water recovery.

A test with the composite membrane was started (Urine Test 12), but stopped very early into the test due to a leak through the O-ring seal of the test bed. The composite membrane had a very thick microporous layer and a very thin Nafion layer. It was much thicker and firmer than the other

Figure 5. Membrane Comparison at Constant Flow in Liquid Contact Mode

membranes tested. It was determined that the composite membrane sample simply did not seal well enough in the designed test bed and testing with it could not be continued. It was therefore taken out of membrane test consideration.

Figure 7 shows the average steady state permeation rate results of each membrane pair at 50 slpm and different temperatures during vapor contact mode. When comparing vapor test mode to liquid it is shown that liquid contact mode permeation rates are an order of magnitude higher than in vapor mode. Vapor contact mode is significantly affected by heat because of the increased vapor pressure, but purge flow rate had less impact on permeation rates as illustrated in Figure 8.

The Hydro B and Nafion A performed better that the Hydro A and Nafion B during one run when the flow was set to 50 slpm. This is thought to be due to the larger pore sizes of the Hydro B allowing water vapor to travel across the microporous membrane more easily. The other run in which the Hydro B and Nafi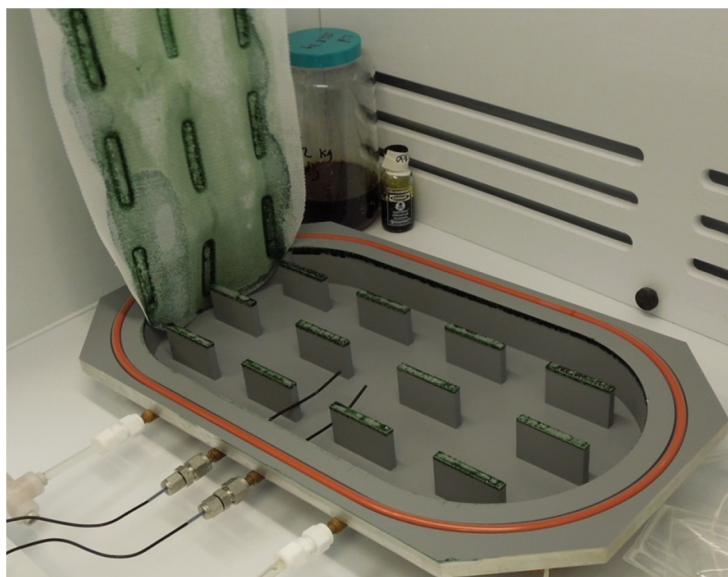on A is slightly lower than the Hydro A and Nafion B is thought to be due to not allowing enough time for the permeation rate to reach its actual steady state rate before changing set points. Fewer tests were done for vapor test mode after it was determined that an IWP assembly would primarily be used in liquid contact mode if used aboard ISS or long duration missions because surface tension would tend to keep the urine or brine in contact with the membrane. It was also determined that the only membrane pair needing vapor contact mode testing would be the best overall performing pair from the liquid contact mode tests, which was the Hydro A and Nafion B pair (Urine Test 10).
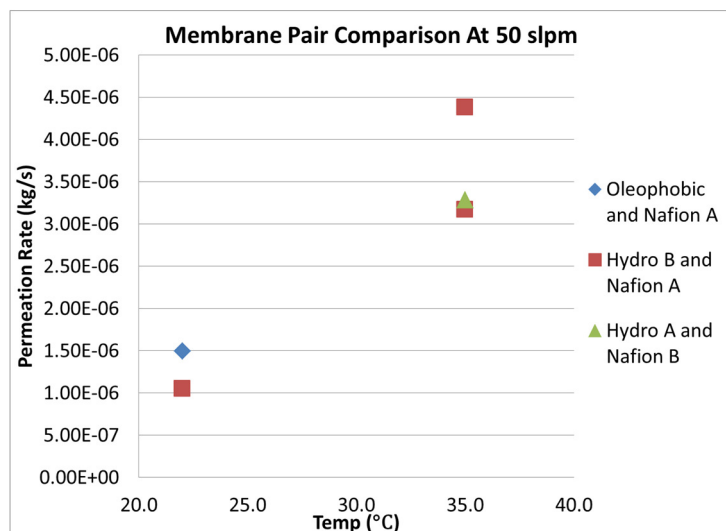

Figure 6. Urine Test 7 Post Test Reservoir

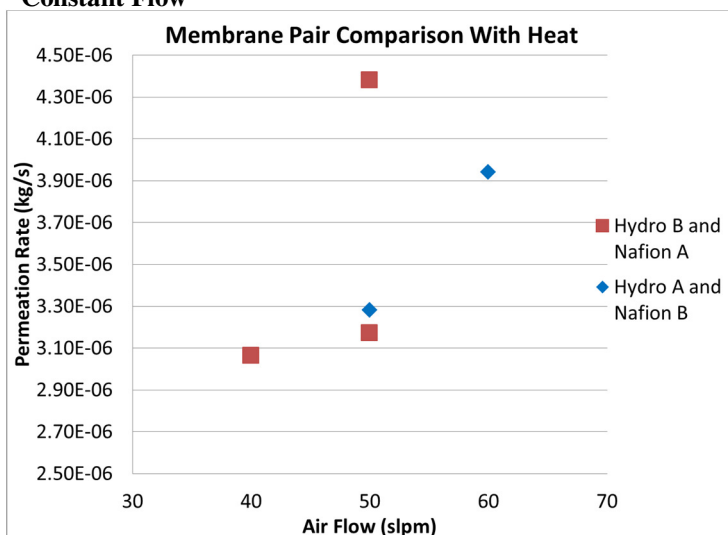**Figure 7. Membrane Comparison Vapor Contact Mode at Constant Flow**

Figure 8. Membrane Comparison Vapor Contact Mode Heated to 35°C

Table 3 shows the measured water quality of 1) pretreated urine, 2) permeate from the first water baseline run, and 3) permeate from Tests 1-15. The analyzed pretreated urine was from a batch that was made from a mixture of multiple urine donors. The permeate samples taken for analysis were from a collection jar that accumulated condensate for the duration of a test.

The pretreated urine was only analyzed for pH, conductivity and TDS. Pretreated urine was only analyzed for pH, conductivity and TDS due to the highly corrosive nature of the fluid and the potential to damage the analytical equipment.

For every test run, methanol, ethanol, and propylene glycol were below the minimum detection limit (MDL) of 1 ppm of the analysis. The ammonium concentration for each test run was also below the ammonium MDL of 0.5 ppm. All sulfate analyses came in below the MDL of 0.08, with the exception of test 10 which registered a value of 0.48 ppm. Urine Test 6 displayed a much higher TIC value than any other test. Since no other membranes showed a spike in TIC it is suspected that there was a contamination during the sample collection or lab analysis.

In addition to high permeation rates, Hydro A/Nafion B also had excellent product water quality compared to the other membrane configurations, though all combinations had the same order of magnitude reduction in contaminants from the pretreated urine levels. Hydro A/Nafion B had among the highest pH at 3.4. The lowest recovered water measured pH was 2.5 and the highest was 3.5, compared to the pretreated urine pH of 1.53. Over 99% reduction in TDS from pretreated urine was achieved. For comparison, water quality data from Brine Residual In-Containment (BRIC) testing is presented in Table 4.[vii] BRIC is an evaporative brine processing technology under development at NASA JSC that uses heat and vacuum to promote evaporation. The feed to BRIC was sulfuric acid/chromium trioxide pretreated, 70% concentrated brine. IWP achieved similar water quality to BRIC for TOC, conductivity, and total solids, though IWP water was more acidic.

Notably, the microporous membrane by itself (Hydro A) did not have significantly different water quality results, with nearly identical pH, TOC, and TDS to the Hydro A/Nafion B. A more significant difference in purge gas trace contaminants would have been expected without Nafion, but unfortunately the trace contaminants testing data was inconclusive. The Hydro A permeation rate was 8% lower than Hydro A/Nafion B, a somewhat surprising result since there should be less diffusion resistance when using only one membrane compared to two. The Nafion by itself did have a lower permeation rate than when paired with microporous, as expected. This is due to ion exchange between the Nafion and the urine reducing Nafion's effectiveness at transporting water. It is important to note that it did not completely fail, but the permeation rate was reduced to ~50% less than when paired with Hydro A.

Table 3. IWP Membrane Selection Testing Water Quality Results (best performing membrane pair highlighted)

Test	Contact Mode	Membrane Pair	pH	TOC (ppm)	TIC (ppm)	Cond. (mS/cm)	TDS (ppm)	Acetate (ppm)	Formate (ppm)	Urea (ppm)
Pretreated Urine	N/A	N/A	1.53	N/A	N/A	23.77	15213	N/A	N/A	N/A
Water Baseline	Liquid	Oleophobic and Nafion A	4.3	2.07	0.46	0.01	6.50	<0.11	<0.10	N/A
Urine Test 1 Permeate	Liquid	Oleophobic and Nafion A	2.5	66.13	<0.004	0.25	161.92	50.27	61.77	<0.03
Urine Test 2 Permeate	Vapor	Oleophobic and Nafion A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Urine Test 3 Permeate	Vapor	No Membrane	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Urine Test 4 Permeate	Liquid	Oleophobic and Nafion A	2.5	82.68	<0.004	0.31	196.48	84.35	100.30	<0.03
Urine Test 5 Permeate	Liquid	Oleophobic and No Nafion	2.5	86.46	<0.004	0.30	190.72	60.47	137.80	N/A
Urine Test 6 Permeate	Liquid	Hydro A and Nafion A	2.5	60.82	108.90	0.26	167.68	42.57	129.41	<0.03
Urine Test 7 Permeate	Liquid	Hydro B and Nafion A	2.6	52.45	<0.004	0.30	191.36	38.63	80.68	<0.03
Urine Test 8 Permeate	Vapor	Hydro B and Nafion A	2.6	73.58	<0.004	0.33	209.92	38.63	80.68	<0.03
Urine Test 9 Permeate	Liquid	Oleophobic and Nafion B	3.5	32.79	<0.004	0.17	107.78	21.27	45.26	0.03
Urine Test 10 Permeate	Liquid	Hydro A and Nafion B	3.4	36.58	0.15	0.16	102.98	23.75	45.74	<0.03
Urine Test 11 Permeate	Liquid	Hydro A and Nafion C	3.4	36.32	0.26	0.16	102.98	28.47	46.77	<0.03
Urine Test 12 Permeate	Liquid	Composite Membrane	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Urine Test 13 Permeate	Vapor	Hydro A and Nafion B	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Urine Test 14 Permeate	Liquid	Nafion B only	3.3	43.72	0.25	0.21	132.42	24.30	75.05	0.14
Urine Test 15 Permeate	Liquid	Hydro A only	3.46	34.85	0.22	0.16	103.55	32.06	56.48	0.02

Table 4. BRIC Water Quality Results[vii]

Brine Residual In-Containment (BRIC)			
	Feed	**Product**	**% Change**
pH	2.42	4.44	>95
K (mS/cm)	133.6	0.197	>99
TOC (mg/l)	31400	75	>99
TN (mg/l)	30200	26	>99
TS (g/l)	154.9	0.04	>99
Σ IONS (mg/l)	58377	29.2	>99
Σ Metals (g/l)	3950	0.59	>99

C. Endurance Test Results

The endurance test was performed with the Hydro A and Nafion B (Urine Test 10) because of its relatively high permeation rate, low water quality concentrations and better dimensional stability when compared to the Hydro B and Nafion A pair. The endurance test was performed at 60 slpm and 35°C. The test began on August 12th and ended September 11th.

The test was stopped periodically to refill the reservoir. To do so, the permeate air flow rate was stopped and pressure relieved on the reservoir side of the test bed. The pressure drop through the purge gas side of the test bed increased the total pressure on the permeate side of the test rig to ~2 psig. To counteract this force on the membranes, the reservoir was also pressurized with air to 2 psig. Once the pressure was relieved, the reservoir was then filled in the same manner as the start of a test. The endurance test was also stopped at the end of the work week and resumed at the beginning of the next work week. This was done to ensure that the Nafion would not dry out due to all or a majority of the water permeating out of the reservoir side of the test bed. With the exception of the periodic refills, the endurance test was run and recorded in the same manner as the pervious membrane selection tests.

The permeation rate of the membrane declined seemingly exponentially during the endurance test. The highest recorded steady state permeation rate was 1.98×10^{-5} kg/s, while the lowest rate was 5.0×10^{-6} kg/s. These steady state permeation rates are at varying brine concentrations. Figure 9 illustrates the decline of permeation rate over the duration of the endurance test. In the figure, "Purge Water Flow Rate" is the total water vapor flow rate exiting the test rig as indicated by the relative humidity. The inlet water vapor flow rate was then subtracted from the "Purge Water Flow Rate" to determine the "Permeation Rate." The spikes in the chart indicate times when the test was stopped and refilled. The approximate steady state times after a refill are indicated by the green triangles in the graph.

Between each refill, the permeation rate declines because of the decrease in vapor pressure as water is removed from the urine. As soon as the reservoir is replenished, the permeation rate jumps back up. But over time, the permeation rate does not recover as well and there is an overall decrease in permeation. The decline in permeation is thought to be the result of a few different factors. The main factor reducing the permeation rate is the increased concentration of non-water compounds in the concentrated mixture. According to Raoult's law, shown in Equation 2, the vapor pressure of a mixture is directly affected by the mole fraction and vapor pressure of each component.

$$p = \sum_{1}^{i} p_i^* x_i \qquad\qquad (2)$$

p represents the vapor pressure of the mixture, while p_i^* represents the pure vapor pressure of each component i. x_i represents the mole fraction of each component.

At the start of the test the main component in the brine mixture is water, meaning the mixture vapor pressure is close to that of water, 2.3 kPa at 20°C. As more water permeates through the membrane, the vapor pressure becomes more dependent on the other components in the mixture. Urea, which has a high concentration in urine, has a vapor pressure of 80 Pa at 20°C, which would greatly decrease the vapor pressure of a mixture if it were to increase in concentration. There are also a number of non-volatile compounds and salts which severely reduce the vapor pressure. The decrease in vapor pressure would decrease the partial pressure of water vapor, which in turn would decrease the amount of water passing through the membranes.

Another factor affecting the permeation rate could be the buildup of solids. As more pretreated urine is added to the test bed during the endurance test, the amount of solids in contact with the membrane also increases. These solids could be blocking and fouling the pores on the microporous membrane, decreasing the amount of water passing through to the Nafion, which would decrease the overall permeation rate, though a point at which the membranes simply stopped working because of blockage was not observed. Figure 10 illustrates the effect that the non-water compounds have on the steady state permeation rate within the test bed and membrane area.

Both factors stated above would lead to a third factor in the decline in permeation.

Figure 9. Endurance Test Flow Rates

Figure 10. Steady State Permeation Rate Decay

With both factors above leading to less water vapor reaching the Nafion, the Nafion could dry out. Nafion permeates water best when it is fully hydrated. As Nafion dries, the permeation rate through the membrane would decrease.

Figure 11 represents permeation rates throughout the test in relation to the concentration of non-water compounds. Each data set represents a week of testing, which includes multiple stops and restarts in each week. As can be seen, the permeation rate decreases directly with increasing solids concentration, but also decreases over time. This supports a decrease in effectiveness of the membrane pair itself possibly due to fouling or solids buildup. The dotted line in the graph at 30% mass fraction represents the point during the endurance test when the test bed has permeated 85% of the water in the pretreated augmented urine. Therefore all the data on and to the right of the line is the permeation data for a concentrated brine process. This was calculated assuming that all the pretreated
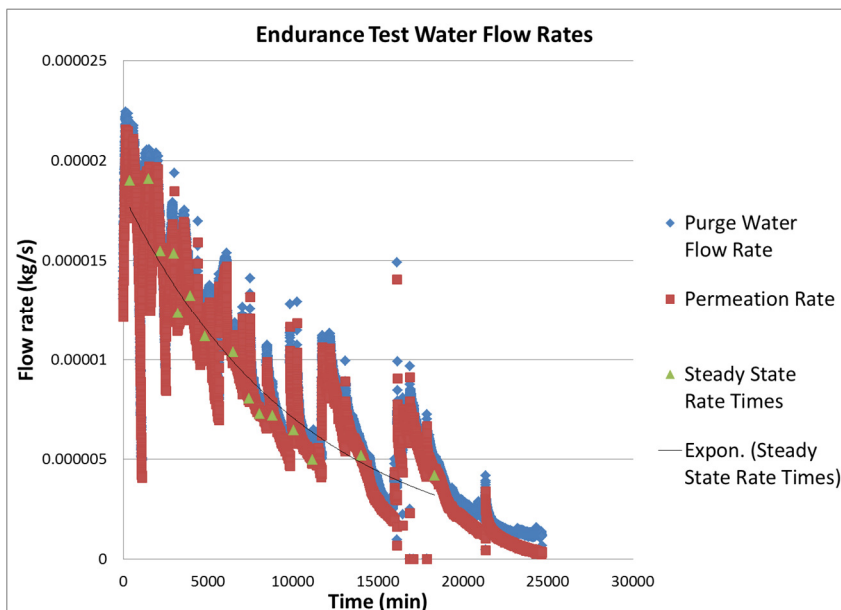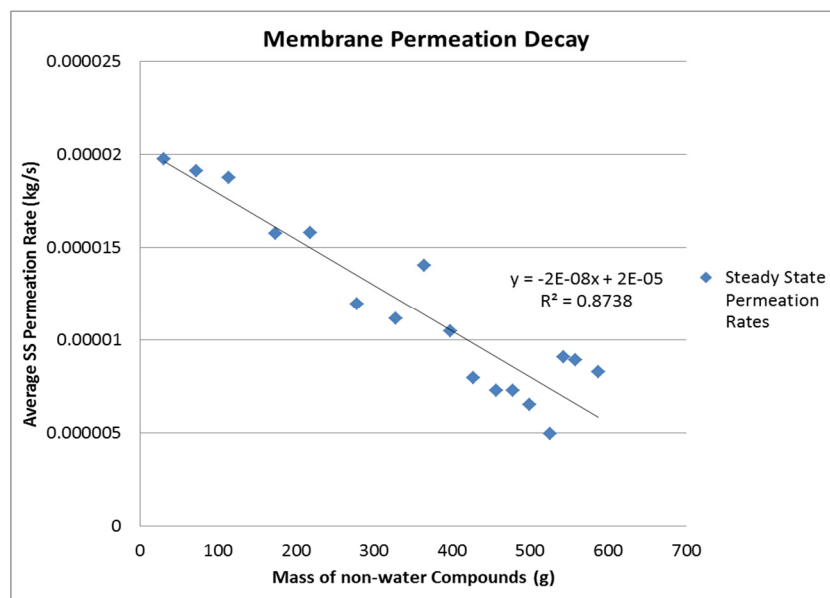
urine added contained the same mass fraction of non-water compounds of 6.06% and based on the measured quantity of the water removed.

If the membrane pair was to be used as a concentrated brine processor on ISS instead of a primary urine processor, the IWP assembly would be in contact with brine produced by 85% water recovery from urine. From the endurance test data the permeation rate for 85% dry brine ranged from 1.89×10^{-5} kg/s at the beginning of the test and dropped down to 8.16×10^{-6} kg/s after a week of testing. As the brine became more concentrated the permeation rates decreased as can be seen on Figure 11.

Table 5 shows the water quality results of the endurance test. Permeate water was sampled on multiple days during the test. Water was not sampled past August due to lower permeation rates and the condenser being unable to condense the lowered water concentration in the purge gas. Similar to the previous tests methanol, ethanol, propylene glycol and sulfate were all below their minimum detection limits. The ammonium concentrations were also below its minimum detection limit with the exception of the first analysis which registered a value of 0.09 ppm.

The endurance test processed approximately 11.15 kg of pretreated augmented urine, which equates to approximately 10.5 kg of water. The total amount of water that permeated through the membrane was calculated to be 10.33 kg from the relative
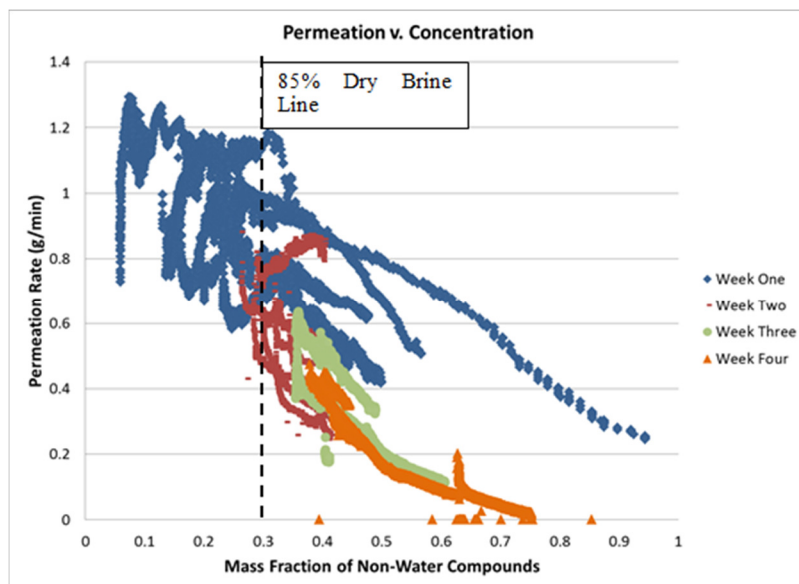

Figure 11. Overall Permeation Rates Dependence on Concentration

humidity (RH) sensor data. Therefore the membrane separated 98% of the water from the pretreated augmented urine.

Table 5. Endurance Test Results (Hydro A and Nafion B in Liquid Contact Mode)

Test	Date	pH	TOC (ppm)	TIC (ppm)	Conductivity (mS/cm)	TDS (ppm)	Acetate (ppm)	Formate (ppm)	Urea (ppm)
Pretreated Urine	N/A	1.53	N/A	N/A	23.77	15213	N/A	N/A	N/A
Urine Test Day 1 Permeate	08/12/13	3.55	28.07	0.14	0.14	87.36	26.11	36.86	<0.03
Urine Test Day 6 Permeate	08/15/13	3.22	74.67	0.14	0.26	164.42	72.16	101.78	<0.03
Urine Test Day 10 Permeate	08/23/13	2.93	218.58	0.14	0.54	346.24	219.22	306.13	
Urine Test Day 12 Permeate	08/27/13	2.98	173.58	0.26	0.45	290.56	112.72	186.22	

At the start of the endurance test, the empty mass of the test rig was 26.466 kg. At the end of the test, the test rig mass with the highly concentrated brine was 27.140 kg. From the starting and ending masses, the residual brine mass was 674 g. During the test, some brine had to be removed from the test bed because it entered the reservoir vent line. The brine that was removed was disposed of immediately and not weighed because of its hazardous nature, but was observed to be approximately 50 ml of concentrated brine. Table 6 shows a summary of the endurance test data.

Figure 12 represents three of the four liquid stages during the test process. The stages from left to right are untreated urine, pretreated & augmented urine, and condensed permeated water. Figure 13 shows the fourth stage, the viscous, concentrated brine left in the reservoir after the endurance test.

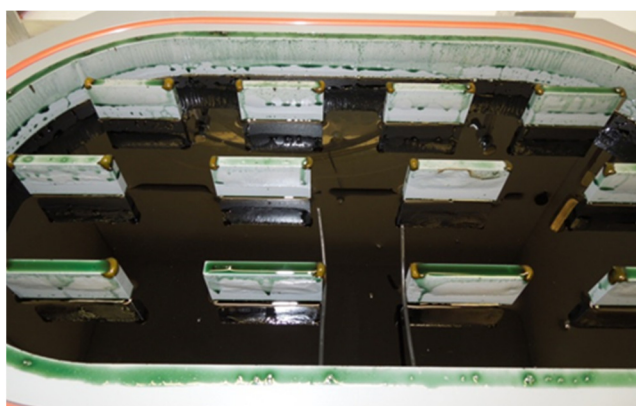**Figure 12. Three Liquid Stages of IWP Process (L-R, Raw Urine, Pretreated Augmented Urine, Permeate)**

Table 6. Endurance Test Data Summary

Test Parameter	Value
Total Pretreated urine processed	11.15 kg
Total mass of water in Pretreated urine	10.5 kg
Total mass of permeated water	10.33 kg
Processing Time	416.7 hr
Percent Water Recovery	98%
Amount of Brine left in Test Bed	674 g
Approximate amount removed from Test Bed	50 ml

III. Conceptual System Design

A. ISS Integration Design

A Conceptual System Design was performed utilizing IWP as a brine processor on ISS, separate from the Urine Processor Assembly (UPA). The UPA would utilize the same process and setup it currently uses and would incur no changes with the addition of the IWP assembly. As a brine processor, the IWP assembly was assumed to be processing 22 liters of ~85% dry brine in 18 day cycles. It is assumed that the brine concentration in the IWP bladder reaches 95% non-water compounds at the end of the drying process, the recovery achieved during small-scale testing.

The UPA would run normally until the Advanced Recycle Filter Tank Assembly (ARFTA) filled to capacity with concentrated brine. Once filled, the ARFTA would be emptied according to the current process and procedures directly into IWP. IWP would then process the brine until no more water can be recovered.

It was determined during small scale membrane testing that the Hydro A membrane and the Nafion B would be

Figure 13. Brine in Reservoir Side of Test Bed after Endurance Test

used in IWP design.[viii] Since both the microporous and Nafion membranes are Teflon based membranes, they are compatible with the concentrated brine, and the dual membranes provide two layers of protection. A third layer of protection is found in the IWP outer housing, complete with hydrophobic filters on the purge gas inlet and outlet. A simple diagram of the IWP bladder and housing is illustrated in Figure 14.

The purge stream originates from the Common Cabin Air Assemblies (CCAA) Condensing Heat Exchanger (CHX) with the assistance of a blower. The purge stream could use the same type of blower as the Carbon Dioxide Removal Assembly (CDRA) Blower, PN 2365514, to deliver the air to the IWP Assembly.[ix] In this analysis, the humid purge stream exits to the cabin. The permeated water is recovered by the CCAA CHX and sent to the Water Processor Assembly (WPA) for any additional treatment. A humidity sensor is located on the purge outlet to determine when the IWP bladder has dried out.

The air exiting the CHX was chosen as the purge stream because it is the air source aboard ISS with the lowest average water content. The air coming out of the CHX has a high average dew point of 7.2°C (45°F). It is assumed that the air has sufficient time to heat up to room temperature before reaching the IWP assembly. Therefore, the air entering the IWP preheater is assumed to be at 24°C with a relative humidity of 33%, corresponding to a 7.2°C (45°F) dewpoint. [x] The cabin air of ISS is maintained between 35% and 45% RH. [x, xi] Table 7 shows the IWP process assumptions.
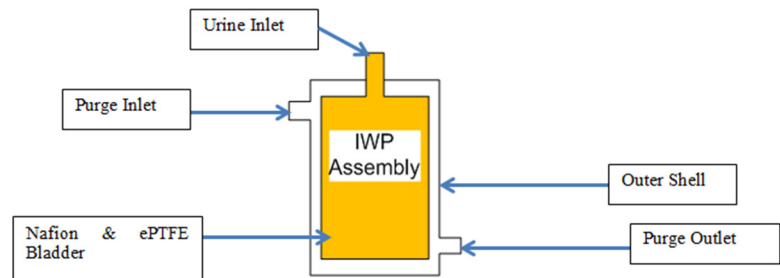

Figure 14. IWP Bladder and Housing Diagram

B. Design Configuration

Three different configurations were considered for the conceptual system design. The first was a single cycle process in which an intermediate transfer container such as a Russian EDV is used as a feed tank for the IWP bladder. A pump or compressor meters brine into IWP at a set rate until the process is complete. The bladder is then disposed of and a new one is used for the next cycle. While the single-cycle process is relatively simple, it requires a pump and controller to meter the brine, as well as an additional consumable of the transfer container (assuming it could be used for multiple cycles but not indefinitely).

Table 7. IWP Process Assumptions

Process Assumption	Value
Water recovery fraction of initial brine	85%
Purge Air Temperature	24°C
Relative Humidity	33%
End Concentration of Brine Within Bladder	95%
Duty Cycle	75%

The second configuration considered was identical to the first, except that the bladder would be re-used for a second cycle. The size of the bladder and housing would also be slightly larger to ensure that the any and all brine left could be contained after two cycles. Reusing a bladder for a second cycle had the potential to reduce consumable mass, however the added complexity quickly outweighed the consumable mass savings. The two-cycle process still had the complexity of a pump and controller, with little benefit over the single-cycle.

To minimize the use of on board consumables, specifically the brine storage tank, a third conceptual design was created in which the IWP Assembly was sized to fit all of the concentrated brine within its bladder. In the Direct Transfer configuration, the UPA would run normally until the ARFTA filled to capacity with concentrated brine.
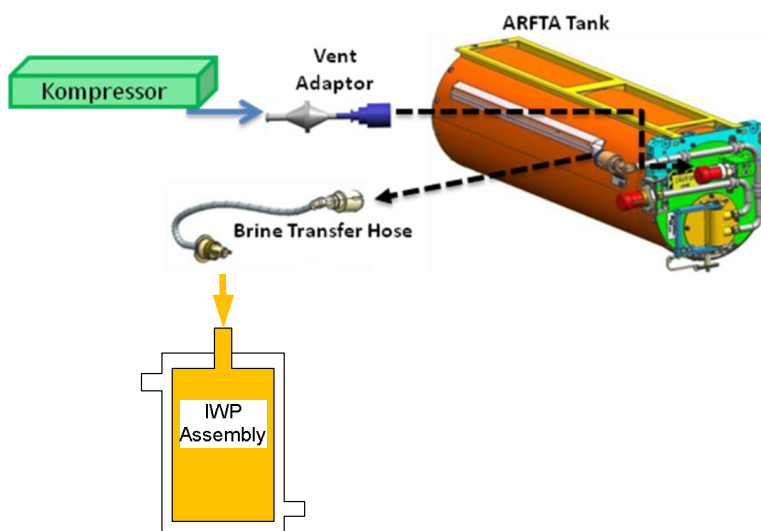

Figure 15. IWP Fill from ARFTA Diagram[xii]

Once filled, the ARFTA would be emptied according to the current process and procedures into the IWP bladder inside the outer housing directly instead of a brine storage tank. The IWP bladder and housing would then be taken off the ARFTA and reattached to the IWP Assembly. Figure 15 and Figure 16 illustrates the IWP brine fill from the ARFTA and the IWP brine processing schematic respectively.

Sizing the IWP Assembly to fit all of the concentrated brine would eliminate the need for a brine storage tank. This would also reduce the overall size and needs for the IWP system. Eliminating the brine storage tank from the brine processing would also eliminate the need for a pump or

compressor to assist in the feed from the tank to the IWP, along with any plumbing material associated with the feed process. Similar to the single and two cycle designs a liquid or RH sensor would be placed on the purge outlet to determine when the IWP bladder has dried out.
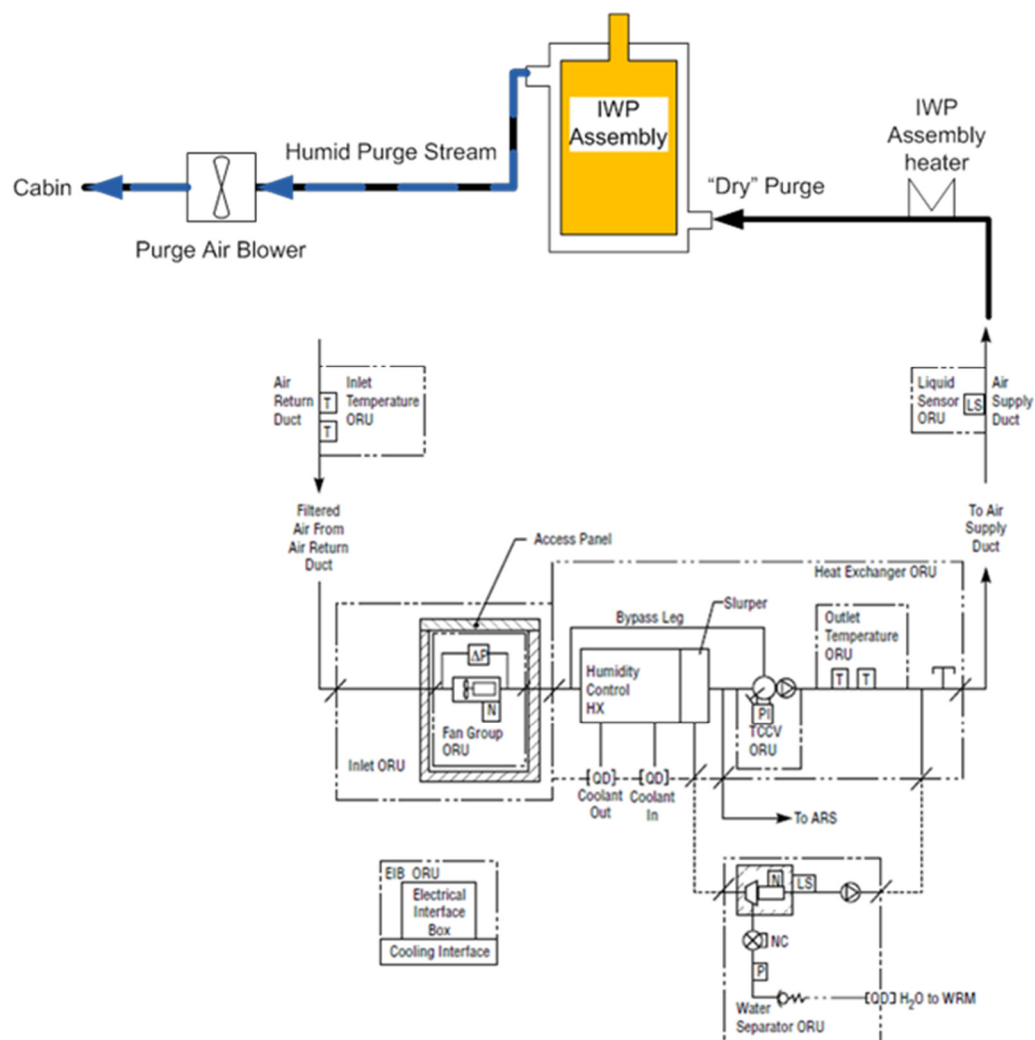

Figure 16. IWP Brine Processing Diagram[xiii]

The volume of the bladder for the direct transfer design was sized to be slightly larger than 22 L, the amount of brine being emptied out of the ARFTA. Since there is no longer a need for a feed tank, pump/compressor and plumbing, the amount of space needed for the whole system assembly would not be significantly increased over the alternate configurations. To maintain the maximum flux of the membrane pair, the purge flow rate would need to increase to 500 slpm, which is well within the capabilities of the CDRA blower. However, a purge flow rate as low as 300 slpm could be used and would still dry the brine in the allotted amount of time. At 500 slpm, the brine is predicted to dry out in ~60 hours. Table 8 shows the process requirements for a full size IWP assembly.

Transferring the brine from UPA directly into the IWP bladder and housing saves crew time, relative to the other design

Table 8. Direct Transfer Assembly Process Requirements

Process Requirement	Value
Purge Flow Rate (slpm)	300-500
Total Bladder Volume (L)	23
Initial Fill Volume (L)	22
Brine Temperature (°C)	35
Drying time at 500 slpm purge flow (hrs)	60

configurations, as it reduces set up and operation of the IWP brine processor assembly. The IWP bladder and housing could simply be attached to the support equipment and started, unlike the one or two cycle continuous processes, which require an initial fill and changing of operating set points. The operation process for the direct transfer IWP Assembly is simply to turn on the heater and blower.

Table 9. Direct Transfer Power Requirements

Component	Power
Blower (kWh)	7.0
Heater (kWh)	16.7
Total (kWh)	23.7

Table 10. IWP Direct Transfer Initial Supply and Resupply Weights

	Initial Supply	Resupply
IWP Bladders (kg)	2.0	2.0
Transfer tank (kg)	Not Needed	Not Needed
Brine Processing Pump (kg)	Not Needed	Not Needed
Purge Blower (kg)	0.8	Not Needed
Brine Heater (kg)	2.8	Not Needed
IWP Outer Housing (kg)	10.2	Not Needed
Total (kg)	15.8	2.0

Table 9 shows the power requirements for a direct transfer IWP for a full drying cycle.

The initial supply and resupply for a direct transfer IWP would not require an intermediate transfer or brine feed container. It also does not require a pump or compressor. This would reduce the supply weight of the IWP system. The weight of the IWP bladder and outer housing itself would increase, but there is still an overall mass savings. Therefore, the initial direct transfer system supply is 15.8 kg, with each subsequent resupply at 2.0 kg. Table 10 shows the weights of each piece of equipment for the Direct Transfer design along with the total weight for the initial supply and resupply.

C. Trade Study

A trade study was performed to compare the total system designs of each conceptual system. The trade study compares the volume and figures of merit (FOM) of each system.

The external dimensions of each piece of equipment were determined to calculate the space required for each conceptual system design. The volume analysis does not take into account the space required for hoses and tubes. The volume analysis does include the space needed to store the IWP bladders that have not yet been used. The pump dimensions were determined by using the dimensions of an off-the-shelf low flow peristaltic pump.[xiv] The blower dimensions were found from the specification drawing from the ISS CDRA blower.[ix] The total volume of the entire system was then calculated for each system by adding the volume of each piece of equipment used in the system to determine the space needed for each design. Table 11 shows the total volume requirements for each design.

An ESM analysis was also performed to compare every aspect of the design in terms of mass. Equation 2 shows the ESM equation that converts each system's parameters and requirements to mass. [xv]

Table 11. Conceptual Design Volume Requirements

Configuration	Total Volume
Single Cycle	3926 in^3 (2.3 ft^3)
Two Cycle	5815 in^3 (3.4 ft^3)
Direct Transfer	5017 in^3 (2.9 ft^3)

$$ESM = M + \left(V \times V_{eq}\right) + \left(P \times P_{eq}\right) + \left(C \times C_{eq}\right) + \left(CT \times D \times CT_{eq}\right) \qquad (2)$$

- ESM = the equivalent system mass of the entire IWP system (kg)
- M = the total supply and resupply masses of the system
- V = the total process volume of the IWP Housing
- V_{eq} = the mass equivalency factor for the process volume infrastructure (kg/m^3)
- P = the total power requirements of the system (kW)
- P_{eq} = the mass equivalency factor for the power generation infrastructure (kg/kW)
- C = the cooling requirement for system (SLPM, ml/hr)
- C_{eq} = the mass equivalency factor for the cooling requirement for the system (kg/SLPM, kg/ml/hr)
- CT = the total crew time requirement of the system (CM-h/y)
- D = the duration of the mission segment of interest (y)

- CT_{eq} = the mass equivalency factor for the crew time support (kg/CM-h)

The mass equivalency factors calculated for ISS were used to convert all of the non-mass values to their respective mass equivalents.[xvi] Table 12 presents all of the process parameters and mass equivalency values used for each system in the ESM analysis. M_1 includes all necessary equipment for IWP brine processing and enough consumables for a full year of processing. M_2 includes only replacement consumables for a year of brine processing.

Table 12. ESM Process Parameters and Mass Equivalency Values

First Year					
Single Cycle System		**Two Cycle System**		**Direct Transfer System**	
M_1 (kg)	21.9	M_1 (kg)	25.3	M_1 (kg)	12.7
V (m3)	0.007	V (m3)	16.5	V (m3)	23.0
V_{eq} (kg/m3)	66.7	V_{eq} (kg/m3)	66.7	V_{eq} (kg/m3)	66.7
P_{Blower} (kW)	0.0794	P_{Blower} (kW)	0. 0976	P_{Blower} (kW)	0.1158
P_{eq} (kg/kW)	166	P_{eq} (kg/kW)	166	P_{eq} (kg/kW)	166
P_{Heater} (kW)	0.154	P_{Heater} (kW)	0.222	P_{Heater} (kW)	0.278
P_{eq} (kg/kW)	166	P_{eq} (kg/kW)	166	P_{eq} (kg/kW)	166
P_{pump} (kW)	0.1	P_{pump} (kW)	0.1	P_{pump} (kW)	NA
P_{eq} (kg/kW)	166	P_{eq} (kg/kW)	166	P_{eq} (kg/kW)	166
C (kW)	0.508	C (kW)	0.678	C (kW)	0.799
C_{eq} (kg/kW)	40	C_{eq} (kg/kW)	40	C_{eq} (kg/kW)	40
CT (CM-h/y)	7.3	CT (CM-h/y)	5.5	CT (CM-h/y)	7.0
D (year)	1	D (year)	1	D (year)	1
CT_{eq} (kg/CM-h)	0.3	CT_{eq} (kg/CM-h)	0.3	CT_{eq} (kg/CM-h)	0.3
ESM (kg)	96.3	ESM (kg)	120.9	ESM (kg)	118.2
Each Subsequent Year					
Single Cycle System		**Two Cycle System**		**Direct Transfer System**	
M_2 (kg)	12.5	M_2 (kg)	12.0	M_2 (kg)	4.0
CT (CM-h/y)	7.3	CT (CM-h/y)	5.5	CT (CM-h/y)	7.0
D (year)	1	D (year)	1	D (year)	1
CT_{eq} (kg/CM-h)	0.3	CT_{eq} (kg/CM-h)	0.3	CT_{eq} (kg/CM-h)	0.3
ESM (kg)	14.9	ESM (kg)	13.8	ESM (kg)	6.3

The results of the ESM analysis are presented in Table 13. The analysis shows that over a 10 year period the Direct Transfer System would have the lowest mass equivalence of the three system configurations.

The key FOM's that were analyzed in this trade study are shown in Table 14 along with their associated units, weights and description. Each FOM is a ratio between the desirable attribute, water recovery, and an undesirable

Table 13. ESM Analysis Results

	Single Cycle System	Two Cycle System	Direct Transfer System
First Year ESM (kg)	96.6	120.9	118.2
Subsequent Years ESM (kg)	14.9	13.8	6.3
10 year Total	230.7	245.0	174.7

attribute. The FOM trade study is based on a 10 year system operation.

The points of failure associated with the system were determined by looking at the equipment and mechanisms of the process and determining points at which a leak, break or malfunction could occur resulting in a process stoppage. The failure points were then assigned a number. If the point of failure could lead to a brine leak it was assigned a 3. If the failure could be categorized as an electrical failure it was assigned a 2. If it were a mechanical failure that occurred, it was assigned a 1. The numbers were then tallied for each to determine the weighted points of failure for each conceptual design. Table 15 shows the raw data ratios between the water recovered and the undesirable attributes.

Table 14. Trade Study Figures of Merit

Parameters	Units	Weight	Description
Supply Mass	kg water recovered/ kg supply mass	30%	The supply mass for 10 years has the most weight due to the cost to transport material to the ISS costing approximately $22,000 per kg. The supply mass was calculated by adding the supply mass over a 10 year period.
Crew Time	kg water recovered/ CM-h/y	20%	Crew time is the second highest weighted, along with system volume and points of failure, due to limited crew time for system operations. The crew time was calculated by estimating the crew time need for each process cycle and adding it for 10 years of operation.
System Volume	kg water recovered/ in^3	20%	The space required for the system in also at 20% because of the limited space aboard ISS. The system volume was calculated using off-the-shelf and current ISS equipment along with preliminary IWP Housing sizes.
Power Consumption	kg water recovered/ kWh	10%	It is assumed that there is power readily available which is why it is the lowest weighted attribute. Power consumption was determined by calculating the power needs for a single cycle and converting that to a 10 year period.
Points of process Failure	kg water recovered/ Points of Failure	20%	Points of process failure are weighted at 20% due to the hazardous nature of the concentrated brine and the potential dangers associated with a process failure.

The raw data ratios were then normalized around the highest value in each parameter and multiplied by 10 to obtain a normalized value between 0 and 10. The normalized FOM values were then multiplied by their respected weighted percent and added together. Table 16 shows the normalized weighted FOM values. The FOM trade study shows that the Direct Transfer system has the highest value, once again indicating it would be the best option according to the attributes considered.

Table 15. FOM Raw Data Ratios

Parameters	Units	Single Cycle	Two Cycle	Direct Transfer
Supply Mass	kg water recovered/ kg supply mass	26.1	26.3	66.1
Crew Time	kg water recovered/ CM-h/y	21.0	24.6	21.1
System Volume	kg water recovered/ in^3	0.9	0.6	0.7
Power Consumption	kg water recovered/ kWh	0.2	0.2	0.1
Points of process Failure	kg water recovered/ Points of Failure	89.8	89.7	130.4

Table 16. Normalized Weighted FOM Values

Parameters	Single Cycle	Two Cycle	Direct Transfer
Supply Mass	1.2	1.2	3.0
Crew Time	1.7	2.0	1.7
System Volume	2	1.3	1.6
Power Consumption	1	0.8	0.7
Points of process Failure	1.4	1.4	2.0
Total	**7.3**	**6.7**	**9.0**

IV. EDU Design & Development

A. EDU Design

A 75% scale EDU based on the conceptual system design was developed. The bladder design was refined using the conceptual design as the basis. The structure of the bladder is based on urine bags developed and internally funded by Paragon for the Paragon Urine Processor (PUP). The bags are made by folding a single sheet of membrane over onto itself and sealing up the sides and top to create a flat, 4-sided envelope. See Figure 17. The discoloration in the seams is due to heating of the Nafion during the sealing process. Nafion discolors over time and under heat, but performance and integrity are not affected.

A vendor has been identified to manufacture the bladders. Filtration and Separation Technology LLC (F.A.S.T. LLC, La Porte, IN) specializes in developing and manufacturing filtration technology with a variety of membranes/materials. They commonly work with ePTFE and have a thermal welding process to seal ePTFE both to itself and other thermoplastics. F.A.S.T. LLC manufactured the urine bags for the Paragon Urine Processor, which is based on IWP technology. They have successfully manufactured ePTFE and Nafion bags in various configurations and sizes.

Due to the hazardous nature of the concentrated brine, substantial consideration was given to the selection of the materials at risk of brine contact. The main hazardous compounds in the brine are chromic acid and sulfuric acid or phosphoric acid, depending on the pretreatment used. The concentrations of chromic and sulfuric acid that could accumulate in the bladder are approximately 50% and 80% respectively. The phosphoric acid concentration that could accumulate is not known because testing of IWP with phosphoric acid has not yet been performed, so 100% phosphoric acid was assumed for chemical compatibility confirmations. The bladder material, which will be in constant contact with the brine, must be chemically compatible with the brine as chemical corrosion of the bladder could lead to a catastrophic failure. The IWP membranes are tetrafluoroethylene (PTFE) based fluoropolymers, which are some of the highest rated polymers for chemical inertness. It has also been proven during small scale

testing that both membranes are able to handle heated, highly concentrated brine without any chemical corrosion.[xvii] Chemical resistance charts also show that PTFE has excellent resistance to phosphoric acid.[xviii]

Figure 17. Paragon Urine Processor Nafion/ePTFE bag

To facilitate the drying process, the IWP bladders are placed inside an Outer Housing. The Housing provides containment for the bladder and purge air flow across the membranes, which controls permeation. An image of the SolidWorks model is shown in Figure 18. The Housing is a box with feedthroughs for purge air, brine fill, thermocouples, and a door to insert and remove the bladder. An inner cage holds the bladder in place and controls flow over the bladder. In operation, an empty bladder is placed inside the cage and connected to the quick-disconnect fill port. The door is closed and latched. Slotted hinges make for easy opening and closing of the door while still allowing for uniform compression for sealing. Feet on the bottom of the Housing raise the box to provide clearance for the hinges and door to open. The bladder is filled with brine by connecting the outside of the fill port bulkhead to the brine supply and pump. Self-closing quick-disconnects allow for safe removal of the bladder and supply line. The door is semi-transparent (PMP) for visibility into the Housing to monitor the bladder. A flap mechanism diverts the flow to maintain constant flow channels while the bladder deflates during drying. The flow is pre-heated prior to entering the Housing. The hot, dry air provides the energy to heat the brine to 35°C and overcome evaporative cooling. A flow plenum transitions the flow from a pipe to nearly the full cross-section of the Housing. Hydrophobic filters between the plenum and Housing proper provide containment in case

of a brine leak. The hydrophobic filters at the inlet also provide pressure drop for adequate flow distribution.

The overall outer dimensions of the Housing assembly are 3.8ft L x 1ft H x 1.3ft W. The width and height are tightly controlled dimensions because of flow velocity requirements. Length is the one less critical dimension. For this reason, ports, fittings, and any supporting structure were accommodated by increasing the length only.

Figure 18. EDU Outer Housing

A tube fitting PP bulkhead serves as a thermocouple feedthrough for temperature control and monitoring. Fine-wire thermocouples are potted into a short section of PP tubing using epoxy compatible with the brine. The tube is inserted into the outside of the bulkhead and thermocouples threaded through the fitting into the box. One thermocouple will be placed on the bottom of the bladder and used as feed-back control for the purge air heater to maintain the bladder at 35°C. Additional thermocouples monitor bladder and Housing temperature for the data acquisition system.

Hydrophobic filters are located at the purge flow inlet and outlet to the housing. The filters prevent brine from escaping the housing in the event of a leak. The inlet filter also provides a pressure drop to sufficiently distribute the flow entering the housing.

The bladder rests on a screen atop flow channels in the bottom of the box. The screen is removable and can be slid out to help with handling of a used bladder. The top of the Cage assembly rests on the top of the bag and slides down as the bag deflates. A Teflon flap connected to the top of the box diverts the air to a set channel height above the bag to maintain velocity for mass transfer as the bag height decreases. The Cage assembly is shown in Figure 19.
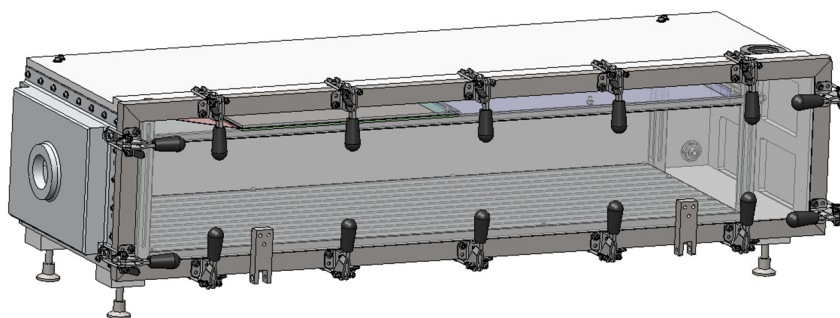
The diverting flap is made of flexible Teflon. The flap slides between two cantilevered Teflon sheets, fastened at the exit end of the cage. The sheets are cantilevered to allow the flap to extend all the way to the sides so that there is no gap to allow flow to bypass above the sheets instead of being kept against the bladder. A screen (not shown) rests on top of the bladder. The bottom Teflon sheet is offset from the screen to create the flow channel. As the bladder deflates, the screen and attached top cage structure slides down, maintaining the constant flow channel. The Teflon flap is long enough such that it is still between the cantilevered sheets at minimum bag height, thus forcing the flow against the bladder. A rendering of the Outer Housing assembly is also shown in Figure 20.
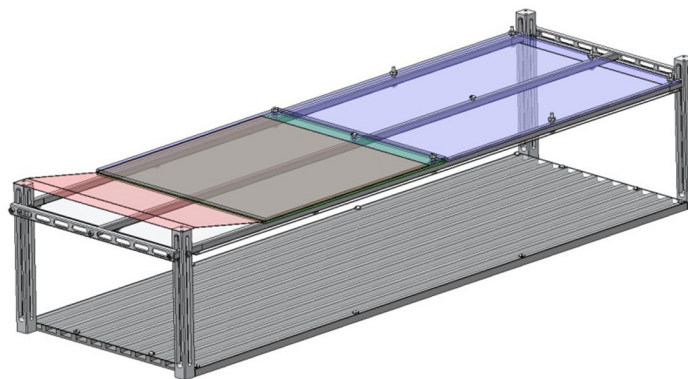

Figure 19. EDU Cage Assembly

The cage is mainly made up of aluminum, steel and PTFE. PTFE parts will not be affected by the concentrated brine if a spill were to occur. The aluminum and steel are not compatible with the concentrated brine and therefore must be coated with a chemically resistant coating to prevent corrosion. Making the entire cage assembly out of PTFE was considered due to its excellent chemical resistance, but due to operational weight and stiffness requirements of the cage, most components were changed to metal.
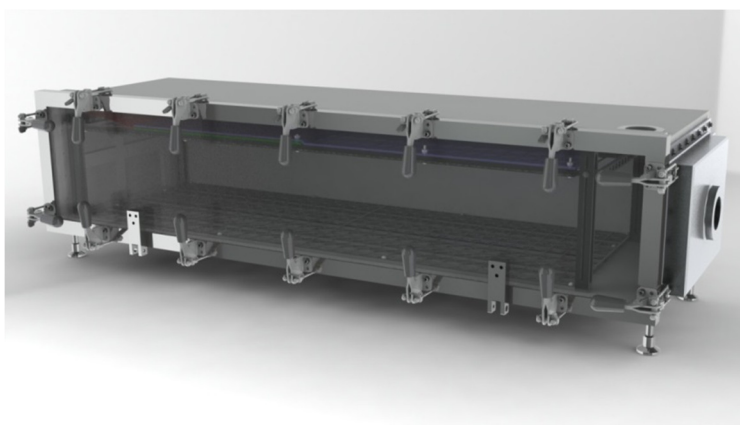
The housing that encompasses the entire assembly is made of several different materials. The main body which encloses the cage and bladder is comprised primarily of Polyvinylidene (PVDF) and

Figure 20. Rendering of SolidWorks EDU

Polymethylpentene (PMP, name brand TPX). PVDF is also known as the brand name Kynar. Like PTFE, PVDF is a fluoropolymer, but it is also categorized as a thermoplastic that could be welded, which led to its selection as one of the main components of the EDU body. Other materials such as Polyethylene terephthalate (PET) and PTFE were also looked at as possible materials for the housing. PET has good chemical resistance but lacks a high enough operating temperature. PTFE contains excellent chemical resistance and high operating temperatures, but lacks the weldability possessed by PVDF. Since PVDF is a fluoropolymer, it displays a wide range of chemical resistance to a wide range of compounds, including chromic, sulfuric and phosphoric acid.[xix] The door of the main body will be made out of PMP due to its semi-transparent characteristic. PMP is a thermoplastic that has similar chemical resistance to polypropylene, but with better resistance to chromic acid and sulfuric acid at higher concentrations.[xx] The other parts of the box will be made out of PP, PVDF, PTFE sealant, Viton, and hydrophobic PTFE filters.

B. Technology Challenges

During membrane selection testing there were instances of leakage through the membranes due to tearing. Figure 21 shows one such tear. When air was flowing through the test rig, the pressure of the permeate side was higher than the reservoir, pushing the membranes against the corners of the heating fins in the reservoir. The ePTFE stretched around the fin and eventually tore. The Nafion was much stronger than the ePTFE and often was not compromised when the microporous membrane tore.

Figure 22 shows a leak between the ePTFE and Nafion from the endurance test. The ePTFE again tore on the heater fins while the Nafion stayed largely intact. Upon close inspection of the test bed, it was also found that a small portion of the brine had leaked to the permeate side of the test rig. It is believed that the physical leak of brine onto the purge side of the test bed was the cause of the increase of certain contaminants over time in Table 5. The

membranes were visually inspected to determine if the cause of the brine transfer was due to a structural issue in the membrane pair as opposed to brine permeation. On visual inspection of the Nafion membrane, a rough area was found close to where the test rig o-ring seals against the membrane. This rough area was found to include tiny punctures which are suspected to be the cause of the brine crossover. After the test bed was cleaned out, corrosion was found on the edges and reservoir fins which likely caused the punctures in the Nafion membrane and microporous membrane. The leaks during membrane selection testing were primarily caused by failures of the test rig, which does not reflect a flight configuration. Steps were taken during EDU design to address and improve membrane integrity.

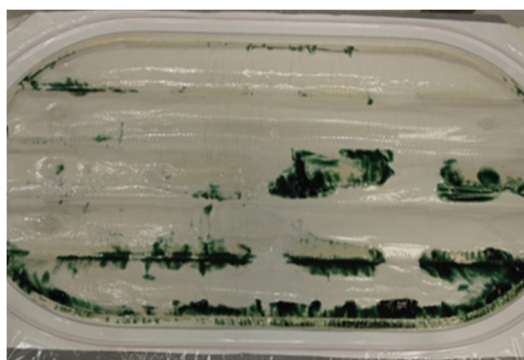
Figure 21. Membrane Tear During Small-Scale Testing

Figure 22. Microporous Membrane Tear Turing Endurance Test

V. Discussion

Membrane selection testing demonstrated that the membrane pairs have fairly similar permeation rates at each set flow rate when the reservoir is not being heated, but have less parity when heat is applied. The Hydro A and Nafion B membrane pair had the best performance and was selected for follow-on development.

Studies have shown that thinner Nafion membranes permeate water at a higher rate than thicker membranes. [xxi] So it was expected that the membrane pairs comprised of the Nafion A would have higher permeation rates than Nafion B and C. This was not found to be the case during membrane selection testing. The membrane with the largest thickness, Nafion B, displayed the highest permeation rate when paired with either of the smaller porosity membranes. The reason for the better performance with the thicker membrane is not fully understood. No other variables were identified that could have been responsible for the unexpected rates. Variations in urine temperature, purge gas temperature, humidity, flow rate, and even urine batch were all examined and ruled out. The Nafion itself is the only identified variable.

The permeation of water through Nafion is a function of multiple factors. The water vapor partial pressure differential across the membrane is the driving force for permeation. The larger the differential is, the higher the potential for permeation. But other factors influence permeation, including level of hydration of the Nafion (water activity), phase of water on the "wet" side (liquid vs. water vapor), purge gas flow rate, interfacial transport, and membrane thickness.[xxi]

Membrane thickness presents a resistance to diffusion. The thicker the membrane is, the more diffusion resistance there is. But Majsztrik et al. found that permeation rate did not scale linearly with membrane thickness as predicted, indicating that other factors play a significant role in permeation. It was also found that with increasing purge gas flow rate, interfacial transport became the limiting factor to permeation. Majsztrik hypothesized that an impermeable skin would form if the Nafion "dried out" too much on the purge gas side and permeation actually started to decline, even though increasing the purge gas flow typically increases permeation rate. This phenomenon only occurred when the wet side was water vapor at saturation as opposed to liquid water. The impermeable skin did not form when liquid water was in contact with the wet side. In the IWP system, the Nafion is actually exposed to water vapor because of the microporous membrane barrier.

There is still much to learn about water transport in in the IWP system. The experiments performed by Majsztrik were mostly done with thicker membranes than used in IWP testing. It is not clear if the diffusion resistance due to thickness is as apparent with the thinnest of membranes. More research into the behavior of thin membranes is needed to better understand performance observed during testing. It is possible that the permeation rate difference between Nafion A and B was due to an unknown variable in the Nafion membranes and not simply the thickness (the only known difference). Perhaps the Nafion A was simply a "bad batch" and another membrane roll would produce different results. More testing would be required to attempt to reproduce the results.

VI. Conclusions

Membrane selection and endurance testing demonstrated excellent water quality, with over 99% reduction in contaminants from pretreated urine. IWP water quality was also comparable to similar brine processing technology. Endurance testing of one membrane pair over 3 weeks achieved 98% water recovery. Results of testing, including lessons learned and membrane integrity challenges, were used to develop the conceptual system design utilizing IWP as a brine processor for ISS. An EDU consisting of a brine bladder and housing was designed based on the conceptual system design. The bladder is constructed of the IWP membranes and provides complete containment of the brine during the entire drying process. The bladder is disposable; after each cycle, the used bladder with dried brine solids is removed from the housing and replaced with a new one. This containment reduces crew interaction with the hazardous brine and risks of exposure. IWP brine processor has the capability to significantly increase water recovery from urine in a simple process, while at the same time providing containment of the processed brine to improve crew safety.

VII. Future Work

Future technical activities include:
- Manufacture of IWP EDU
- Testing of EDU with phosphoric acid/chromium trioxide pretreated brine
- Bladder construction development/improvements
- Testing with additional brines such as a combined humidity condensate/urine brine and alternative pretreatments.
- Microgravity sensitivities should be investigated through a microgravity experiment

Acknowledgments

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