ADVANCED MICROBIAL CHECK VALVE DEVELOPMENT

Final Report

By

G. V. Colombo
(D. R. Greenley)

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By

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Myrtle Creek, OR

For
LYNDON B. JOHNSON SPACE CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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INTRODUCTION

Under previous contract effort (NAS9-15616) a flight certified assembly identified as a Microbial Check Valve (MCV) was developed and tested. The MCV is a canister packed with an iodinated anionic exchange resin. The device is used to destroy organisms in a water stream as the water passes through the device. The device is equally effective for fluid flow in either direction and its primary method of organism removal is killing rather than filtering.

The Microbial Check Valve (MCV) has been successfully developed for the Space Shuttle to: 1) disinfect fuel cell water; and 2) prevent back contamination of the stored potable water supply. One version of the device consists of a "high residual" iodinated resin bed that imparts approximately 2 ppm of iodine to the fuel cell water as it flows to the potable water tanks. A second version of the device consists of a "low residual" iodinated resin bed. One of these "low residual" beds is located at each use port in the potable water system for the dual purpose of removing some iodine from the potable water as it is dispensed and also to prevent back contamination of the potable supply. A third version of the device, which contains the "high residual" resin, is used to disinfect water used in the EMU.

The Microbial Check Valve has potential space applications beyond the basic Space Shuttle mission. It appears to be also suited for use in advanced water reclamation systems that NASA has under development for the disinfection of humidity condensate, wash water and human urine.

So far, the only effective method for maintaining microbial control in development space-type water reclamation systems has been heat at pasteurization temperatures and an iodine system which requires an iodine monitor and injection system. It is recognized that these are relatively high penalty approaches, but no other completely satisfactory method has been developed.
Methods including the use of: microbial filters, U-V radiation, chlorination and silver ions have been evaluated and all have been found deficient. It is felt that the microbial check valve may be able to satisfactorily replace heat as a microbial control method in some or all of these reclamation systems and thereby effect a large savings in weight, power and cost.

**OBJECTIVES**

The objectives of this effort were:

a. To evaluate the high residual (2 ppm) iodinated resin developed for disinfecting Space Shuttle fuel cell water and define its limitations, if any, for use with space-type water reclamation systems.

b. To retrofit three flight prototype MCVs previously delivered to NASA to reflect the design of the flight units delivered under NAS9-15616.

c. To provide quantities of flight certified low residual and high residual resins to support the Space Shuttle Operation Flight Test program plus the first six operational flights.

d. To evaluate the reuse potential of resin and if economically practical to develop procedures to accomplish this.

e. To develop and demonstrate a more efficient container concept for the flight MCVs.
**Task 1. ADVANCED MCV APPLICATIONS**

Six potential advanced MCV applications are projected for water streams in space-type water reclamation systems. These six water streams are listed in Table 1 in what is felt to be an increasing order of difficulty for the MCV. The principal dissolved materials that must be dealt with, and their estimated concentrations, are listed after each of the water streams.

<table>
<thead>
<tr>
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<th>Major Contaminant</th>
<th>Amount (ppm)</th>
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<td>ammonia</td>
<td>18</td>
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<tr>
<td></td>
<td>ethanol</td>
<td>8</td>
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<tr>
<td></td>
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<tr>
<td>Urine distillate</td>
<td>ammonia</td>
<td>20</td>
</tr>
<tr>
<td>without carryover</td>
<td>ethanol</td>
<td>10</td>
</tr>
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<td></td>
<td>acetaldehyde</td>
<td>2</td>
</tr>
<tr>
<td>Wash water after reverse osmosis treatment</td>
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<td>40</td>
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<tr>
<td></td>
<td>sodium chloride</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>lactic acid</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>urea</td>
<td>7</td>
</tr>
<tr>
<td>Wash water after chemical addition/filtration</td>
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<td>20</td>
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<tr>
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<td></td>
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<td></td>
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<td>35</td>
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<tr>
<td></td>
<td>urea</td>
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<td>Urine distillate</td>
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<td>2</td>
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<td></td>
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<td>Water Stream, cont.</td>
<td>Major Contaminant</td>
<td>Amount (ppm)</td>
</tr>
<tr>
<td>----------------------------------------</td>
<td>------------------</td>
<td>--------------</td>
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<tr>
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<td>lactic acid</td>
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The approach to this task was to first challenge resin beds with solutions that contain known concentrations of the following contaminants: ammonia, ethanol, acetaldehyde, sodium chloride, urea, lactic acid, two cleansing agents, and two treatment chemicals.

Initial tests examined the effects of each contaminant separately to establish contaminant concentration and resin life/effectiveness relationships. Later tests combined contaminants to simulate typical reclamation system use points. Finally, tests were conducted using real urine to simulate typical "carry over" conditions and real wash water to simulate the wash water application.

1.1 MCV CHALLENGES

Challenges of the resin were performed by pumping prepared solutions through a bed of resin and monitoring the influent and effluent for significant changes in the pH, specific conductivity and iodine residual. The tests were conducted with 8 mm dia x 76 mm test beds of 2 ppm resin and 10 ml/min flow rate at room temperature. The concentrations of the solutions were varied in an effort to identify contaminant levels that would not adversely affect the resin. The contaminant was assumed to have no effect on the resin if the effluent $I_2$ residual was within 10% of the control bed. The overall test direction was to start with single-contaminant challenges and work up to the multiple-contaminant test water streams. With each test contaminant or test stream, the concentration was either increased to the failure point of the resin, or decreased until there was no adverse affect on the resin. In some cases the test streams reached.
very high levels without significant effects or the test stream interfered with the Leuco-crystal violet test reagents, precluding any measurement of the I₂ residuals.

**CHALLENGE WITH (NH₄)₂SO₄** - Dilutions of (NH₄)₂SO₄ were prepared by adding 50, 5, 0.5 and 0 grams of (NH₄)₂SO₄ to four 5 l aliquots of deionized, (D.I.) water. This resulted in challenge solutions of 1% (10,000 ppm) 0.1% (1000 ppm) 0.01% (100 ppm) and a control deionized water. These solutions were added to feed tanks and individually pumped through 4 respective resin beds (Figure 1). The flow rates were adjusted to 10± 0.5 ml/min and maintained at that rate for the duration of the challenge. The effluents from the control bed and from the bed being fed to 0.01% solution had iodine residuals of a little less than 2 ppm. The effluents from the 0.1% and the 1% beds was about 2.2 ppm. The slight increase indicates that the (NH₄)₂SO₄ has the ability to strip iodine from the resin, but since it occurred only at high levels of (NH₄)₂SO₄, it was not considered to be a significant effect. One thousand ppm (NH₄)₂SO₄ is a much greater concentration than will be encountered by the resin in actual use.

**CHALLENGE WITH NH₄OH** - Challenges of the resin were conducted at 0.1, 1, 10, 100, 1000 and 10,000 ppm as NH₃. Iodine was stripped from the resin at greatly accelerated rates, approximately 100 times normal at 1% (10,000 ppm) NH₃ down to 2 times normal at 1 ppm NH₃. At 0.1 ppm there was no noticeable effect of the ammonia on the resin. The effect of NH₄OH on the resin is very marked. At the higher concentrations, the resin was visibly decolored towards the end of a 6 hour challenge. The stripping rate is readily evident at the 1% level. The 1 hour I₂ residual reading was about 150 ppm I₂ residual steadily decreased down to 25 ppm at the end of the run.
**Figure 1. Test Equipment**

- **Reservoir**
- **Pump**
- **Sample**

**Diagram Details:**
- **Polypropylene Caps + Screens**
- **8 mm ID x 76 mm Glass**
UREA - Urea challenge solutions were prepared at 0.01% (100 ppm) 0.1% (1000 ppm) and 1% (10,000 ppm). There was no significant effect of the urea on the resin with any of the levels tested, up to 10,000 ppm. It appears that urea to 10,000 ppm has no adverse effects on the resin.

CALSOFT L-40* (Sodium Sulfonate) - Challenge solutions were prepared at 0.01% (100 ppm), 0.025% (250 ppm), and 0.05% (500 ppm) active ingredients. Calsoft at 0.05% had an I₂ demand that exceeds the capacity of the bed, i.e., there was no I₂ residual in the effluent stream. Solutions of 0.025% and 0.01% had no noticeable effect on the I₂ residual of the resin. The I₂ demand of the sodium sulfonate was calculated to be about $10^{-2}$ mg I₂/mg sodium sulfonate. Sodium sulfonate would appear to be allowable in the water streams at levels not to exceed 250 ppm, active ingredients.

ML11** - ML11 soap was diluted to 0.001% (10 ppm), 0.01% (100 ppm) and 0.1% (1000 ppm), calculated as active ingredients. The soap solutions formed a flocculent precipitate upon addition of the iodine test reagents, so spectrophotometric measurement was precluded. Standards were prepared by adding known quantities of iodine to the soap solutions and then used as a basis for visual comparison to estimate the I₂ present in the effluents. The ML11 doesn't have a significant immediate I₂ demand, but after a 10 minute exposure, the I₂ level was significantly reduced in the 100 and 1000 ppm soap solutions.

HUMIDITY CONDENSATE - A simulated humidity condensate water was prepared with 18 ppm ammonia, 16 ppm ethanol and 1 ppm acetaldehyde. The simulated humidity condensate was supposed to have only 8 ppm ethanol but a post-experiment review of calculations and procedures revealed a miscalculation that resulted in twice as much ethanol being used as was called for. Since this error made the experiment even more conservative, no retest was performed. There were no adverse effects of this solution on the I₂ residual of the resin.

*40% Sodiumdodecylbenzenesulfonate - Pilot Chemical Co., Los Angeles, CA
**ML11 Soap, Rochester Germicide Inc., Rochester, N.Y.
**NaOH** - Challenges were made from 0.04 to 1 ppm as OH\(^-\). At 1 ppm, the NaOH stripped all of the I\(_2\) from the resin within 4 hours. At 30 minutes into the challenge, the iodine residual in the effluent was about 160 ppm. At 90 minutes the resin had been rinsed down to 32 ppm and by 4 hours, essentially all the iodine had been stripped from the resin. At 0.04 ppm OH\(^-\) the resin was being stripped of I\(_2\) at about 15 times the normal rate. It is evident that even at very low levels (pH>7) OH\(^-\) is quite deleterious to the resin.

**NaCl** - Challenge solutions were prepared at 50,000, 25,000, 10,000, 10, 1 and 0.1 ppm NaCl. Slightly elevated I\(_2\) residuals were noted at concentrations from 50,000 to 10 ppm, but not high enough to significantly deplete the resin. One and 0.1 ppm concentrations of NaCl had virtually no effect. There should be no problem with the resin up to 5% NaCl or higher.

**LACTIC ACID** - Lactic acid challenge dilutions were prepared at 1% (10,000 ppm) 0.1% (1000 ppm) and 0.01% (100 ppm). The 1% lactic acid solution had a slight inhibitory effect with I\(_2\) residual. The I\(_2\) residual ranged between 1.8 and 2.0. This challenge was not taken to the point of resin failure because 1% lactic acid is a much higher concentration than the resin will be subjected to in actual use. There appeared to be no significant adverse effects of lactic acid on the resin.

**ACETALDEHYDE** - Acetaldehyde challenge solutions were prepared at 0.01% (100 ppm) 0.1% (1000 ppm) and 1% (10,000 ppm). There was no significant change in the I\(_2\) residual even at 1% concentration.

**URINE DISTILLATE WITHOUT CARRYOVER** - This water stream was simulated by preparing a D.I. H\(_2\)O solution with 20 ppm ammonia, 20 ppm ethanol and 2 ppm acetaldehyde. A more conservative test stream was prepared containing twice the concentration of all ingredients. The simulated water stream was supposed to have only 20 ppm ethanol but a post-experiment review of calculations and
procedures revealed a miscalculation that resulted in twice as much ethanol being used as was required. The miscalculation also carried over into the second, more conservative water stream, so it has 4 times the ethanol originally needed. There were no adverse effects on the I₂ residual of the resin.

**WASH WATER AFTER REVERSE OSMOSIS TREATMENT** - This water stream was simulated by adding 40 ppm active ingredients of ML11 soap, 20 ppm NaCl, 10 ppm lactic acid and 7 ppm urea. This water stream had no adverse effect on the I₂ residual in the effluent water.

**SIMULATED URINE DISTILLATE WITH CARRYOVER** - A simulated water stream was prepared in D.I. H₂O by the addition of: 75 ppm urea, 45 ppm NaCl, 15 ppm potassium sulfate, 9 ppm potassium chloride, 8 ppm creatinine, 8 ppm ammonium hippurate, 4 ppm magnesium sulfate, 2 ppm phenol, 1 ppm potassium phosphate. This water stream had an immediate I₂ demand that exceeded the capacity of the resin bed to produce an I₂ residual.

**RAW WASH WATER WITH FILTRATION** - A simulated water stream was prepared in D.I. H₂O by the addition of: 110 ppm ML11 cleansing agent, 40 ppm sodium chloride, 35 ppm urea and 13 ppm lactic acid. This water stream turned turbid with the addition of the iodine test reagents, precluding spectrophotomic measurement. Approximate I₂ levels were estimated by visual comparison with iodine standards prepared in the test stream water. This test stream has an immediate I₂ demand of about 1 ppm I₂.

**URINE DISTILLATE WITH CARRYOVER - BREAKDOWN** - In an attempt to identify which components created the I₂ demand, they were run separately in the concentration used for the simulated test stream formulation. The creatinine and hippuric acid had no significant effect on the I₂ residual,
but the phenol had an $I_2$ demand that exceeded the capacity of the resin bed.

**PHENOL** - To determine potentially acceptable phenol levels, solutions were made up at 0.1 ppm, 0.5 ppm and 1 ppm in D.I. H$_2$O and pumped through the resin beds. At 0.1 ppm the phenol has about 0.5 ppm immediate $I_2$ demand. At 0.5 ppm the demand is about 1 ppm and at 1 ppm phenol the demand is about 1.5 ppm $I_2$. $I_2$ residual tests taken on samples after one-half hour exposure showed no more $I_2$ loss than the control. So it appears that the $I_2$ demand is immediate.

**PRETREATMENT CHEMICALS** - Most urine recovery systems use pretreatment chemicals to minimize the decomposition of urea to ammonia which carries over into the reclaimed water. The pretreatments usually contain acid and a strong oxidizing agent to minimize thermal degradation, and a biocide to minimize microbial decomposition. The two pretreatment chemicals selected for testing were:

1. McDonnell Douglas (MDAC) suspension containing 4.12 grams potassium dichromate ($K_2Cr_2O_7$), 11.3 grams H$_2$SO$_4$, 1.4 grams CuSO$_4$5H$_2$O, 2.4 grams Dow H-10 antifoam and 13 grams H$_2$O.
2. Life Systems, Inc. treatment suspension containing 11.3 grams H$_2$SO$_4$, 11.7 grams Poviodine, 2.4 grams Dow H-10 antifoam and 25 grams H$_2$O.

Both treatment chemicals, when added to the water in the prescribed concentrations, so grossly discolored the water that $I_2$ determination was not possible. It was decided to dilute the MDAC treatment chemical down to where the chrome concentration is at the drinking water limit. The logic is that the chrome concentration will exceed the drinking water limits before the treatment chemical itself will prove deleterious to the resin. The MDAC treatment chemical was diluted to give chrome concentrations of 0.05, 0.5 and 5 ppm.
During the actual challenge it was noted that the iodine residual in the effluent seemed to increase with the exposure time. This is explained by the fact that potassium dichromate is a strong oxidant and thus is a positive interference in the iodine test. Since the co3 changed rather rapidly, it was not possible to make any I2 determinations. The Life Systems suspension contains free I2, thus has no I2 demand on the resin.

RAW URINE - Assuming the urine to be 5.5 to 7% solids, approximately 50% of which is urea, the urine was diluted out to the following concentrations (given in % urine and approximated ppm urea) in D.I. H2O: 1% urine (150 ppm urea); 0.5% urine (75 ppm urea); 0.25% (38 ppm urea); 0.1% urine (15 ppm urea); 0.05% urine (7.5 ppm urea); 0.02% urine (3 ppm urea). All the urine dilutions down to 0.25% (38 ppm urea) had an I2 demand that exceeded the capacity of the resin. The immediate iodine demand of the 0.1% (15 ppm urea) urine solution was about 1 ppm. The iodine demand of the 0.02% urine (3 ppm urea) was about 0.1 or 0.2 ppm I2.

WASH WATER AFTER FLOCCULATION/FILTRATION - A simulated solution was prepared in D.I. H2O by adding 50 ppm ML11 soap (active ingredients) 20 ppm FeCl₃ as the treatment chemical, 40 ppm NaCl, 35 ppm lactic acid and 13 ppm urea. This water stream has an immediate iodine demand, about 0.5 ppm at this composition.

RAW, TREATED URINE - To test the pretreatment chemicals under more realistic conditions 0.1% urine samples were treated with 5 ml/l of the treatment chemicals. These pretreated raw urine test streams were then pumped through the resin beds. The resulting effluents showed no adverse effects, very little difference between the control I2 residuals and the test stream residuals.
1.2 MCV CHALLENGE SUMMARY

The results of the challenges with individual chemicals, simulated waste streams and real waste streams are summarized in Table 2.

The tests with the individual chemicals which are the major constituents of waste streams that will be subjected to water reclamation show no effect at very high levels. Tests with NH₄OH and NaOH resulted in I₂ stripping by OH⁻ ions which is to be expected from the strong base resin. Ammonium ion has very little if any effect when the pH is held below 7. The only chemical which showed a high I₂ demand was phenol, however at levels well above those expected or allowed in reclaimed water.

Raw urine has a fairly high I₂ demand which is significantly reduced by the addition of the two pretreatment chemicals currently being used in water reclamation systems. When these systems are operating normally and producing potable water, the MCV will iodinate the effluent with no difficulty. Upsets or malfunctioning systems could produce water with up to 1000 ppm pretreated urine content, and the MCV would still maintain an I₂ residual.

Tentative wash water standards are shown in Table 3. Depending on the iodine demand of the allowable 200 ppm TOC, and assuming the pH can be kept below 7, the MCV should promise to be an attractive method of microbial control. Results reported in Reference 1* on the Springborn system show finished water with organic levels well below the tentative standards, thus the MCV should have no problems with this water. Reported soap residuals of approximately 50 ppm ML11 after the flocculation step indicates that the MCV could be used at this point in

<table>
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<tr>
<th>Contaminant</th>
<th>Maximum Expected ppm</th>
<th>Maximum Tested with no adverse effect ppm</th>
<th>I&lt;sub&gt;2&lt;/sub&gt; Demand** ppm I&lt;sub&gt;2&lt;/sub&gt;/ppm Contaminant or composition</th>
<th>I&lt;sub&gt;2&lt;/sub&gt; Stripping** ppm I&lt;sub&gt;2&lt;/sub&gt;/ppm Cont.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>NH&lt;sub&gt;4&lt;/sub&gt;OH</strong></td>
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<tr>
<td><strong>NH&lt;sub&gt;4&lt;/sub&gt;</strong> in (NH&lt;sub&gt;4&lt;/sub&gt;) SO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>20</td>
<td>1,000</td>
<td></td>
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<td>NaOH</td>
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<td>40</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acetaldehyde</td>
<td>2</td>
<td>4</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* See challenge section for composition page 10.

** I<sub>2</sub> demand is a reduced I<sub>2</sub> residual compared to a control bed with DI water, I<sub>2</sub> stripping is an increased I<sub>2</sub> residual.
<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Maximum Expected ppm</th>
<th>Maximum Tested with no adverse effect ppm</th>
<th>I₂ Demand ppm</th>
<th>I₂ Stripping ppm I₂/ppm Cont.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Simulated Urine Distillate with Carryover</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urea</td>
<td>75</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NaCl</td>
<td>45</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>K₂SO₄</td>
<td>15</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>KCl</td>
<td>9</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Creatinine</td>
<td>8</td>
<td></td>
<td></td>
<td>&gt;2/composition</td>
</tr>
<tr>
<td>NH₄Hippurate</td>
<td>8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MgSO₄</td>
<td>4</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Potassium phosphate 1 (K₂PO₄)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phenol</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Simulated Wash Water after RO</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MLI</td>
<td>40</td>
<td>40</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NaCl</td>
<td>20</td>
<td>20</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lactic acid</td>
<td>10</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urea</td>
<td>7</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Raw Urine</strong></td>
<td>5000</td>
<td>200</td>
<td>1/1000</td>
<td></td>
</tr>
<tr>
<td>Urine treated with MDAC</td>
<td>1000</td>
<td>&gt;2/5000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urine treated with LS</td>
<td>1000</td>
<td>&gt;2/5000</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Simulated Wash Water with Filtration</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MLI</td>
<td>110</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NaCl</td>
<td>40</td>
<td></td>
<td></td>
<td>approx. 1.0/composition</td>
</tr>
<tr>
<td>Urea</td>
<td>35</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lactic acid</td>
<td>13</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
### Table 2. cont.

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Maximum Expected ppm</th>
<th>Maximum Tested with no adverse effect ppm</th>
<th>I$_2$ Demand ppm / ppm Contaminant or composition</th>
<th>I$_2$ Stripping ppm I$_2$/ ppm Cont.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Simulated Wash Water after Flocculation and Filtration</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ML11</td>
<td>50</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NaCl</td>
<td>40</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lactic acid</td>
<td>35</td>
<td></td>
<td>0.5/composition</td>
<td></td>
</tr>
<tr>
<td>Urea</td>
<td>13</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FeCl$_3$</td>
<td>20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Filtered Shower Water</strong></td>
<td></td>
<td></td>
<td></td>
<td>&gt;2/composition</td>
</tr>
<tr>
<td>ML11</td>
<td>212</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Flocculated &amp; Filtered Shower Water</strong></td>
<td></td>
<td></td>
<td></td>
<td>&gt;2/composition</td>
</tr>
<tr>
<td>FeCl$_3$</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
the process, however the subsequent charcoal and ion exchange columns would necessitate another unit for the final product.

In summary, the MCV has been shown to be able to process reclaimed water streams without degradation of the resin as long as the streams are within acceptable specifications. In the case of malfunctioning systems, excessive stripping of iodine from the bed would be the most damaging effect, which can be replaced as will be discussed later.

TABLE 3.
Tentative Wash Water Standards

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Organic Carbon (TOC), mg/l</td>
<td>200</td>
</tr>
<tr>
<td>Specific Conductivity, μmho-cm⁻¹</td>
<td>2000</td>
</tr>
<tr>
<td>pH</td>
<td>5 to 7.5</td>
</tr>
<tr>
<td>Ammonia, mg/l</td>
<td>5</td>
</tr>
<tr>
<td>Turbidity, ppm SiO₂</td>
<td>10</td>
</tr>
<tr>
<td>Color, Pt-Co Units</td>
<td>15</td>
</tr>
<tr>
<td>Foaming</td>
<td>Nonpersistent more than 15 seconds</td>
</tr>
<tr>
<td>Odor</td>
<td>Nonobjectionable</td>
</tr>
<tr>
<td>Total Dissolved Solids (TDS), mg/l</td>
<td>1500</td>
</tr>
<tr>
<td>Urea, mg/l</td>
<td>50</td>
</tr>
<tr>
<td>Lactic acid, mg/l</td>
<td>50</td>
</tr>
<tr>
<td>NaCl, mg/l</td>
<td>1000</td>
</tr>
<tr>
<td>Microorganisms, number per ml</td>
<td>10</td>
</tr>
</tbody>
</table>
Task 2. **RETOFIT FLIGHT PROTOTYPE MCV UNITS**

GFE prototype units serial numbers P2, P3, and P7 were modified to make them identical in function and operation to the Flight MCV and delivered to JSC. (See DD250 in Appendix)

Task 3. **MANUFACTURE FLIGHT CERTIFIED RESIN**

Sufficient resin for 30 low residual MCV recharges and 16 high residual MCV recharges was manufactured, certified, and delivered to JSC. (See DD250 in Appendix)
The MCV Resin has been shown to hold iodine in the $I_2^-$ form. The high residual resins, i.e. those that produce measurable iodine residuals have been found to release iodine primarily as $I_2^-$, with a very small amount of $I^-$ (approximately 0.2 ppm) accompanying the $I_2^-$. It should be possible, therefore, to regenerate a depleted high residual resin by simply replacing the $I_2^-$. If this can be accomplished without disassembling the MCV, a considerable savings in materials, but more importantly time is possible. In light of the previously discussed favorable results with reclaimed water, a MCV which could be regenerated in flight may provide a very attractive biocide dispensing system for long term missions involving closed systems.

Several tests were conducted with an 8 x 76 mm bed at a flow of 10 cc/min. Rather than try to exhaust a high residual bed and then regenerate it, a bed of low residual resin, 0.2 ppm, was used. A bed containing iodine crystals of approximately the same size as the resin bed was placed upstream and deionized water was pumped through. At room temperature, the saturation solubility of $I_2^-$ in water is about 200 ppm. Experience has shown, however that a bed of fresh $I_2$ crystals (under steady flow conditions) will initially produce water containing about 100 ppm, which will taper off after several hours to a steady value in the neighborhood of 50 ppm.

The first regeneration (actually $I_2$ loading) attempt was conducted with a fresh low residual bed. The $I_2$ crystal bed which was initially producing 115 ppm was removed when the effluent from the resin bed exceeded 2 ppm. After 100 hours the residual from the resin bed was still 5 ppm and showed no signs of tapering off; the test was terminated.
Figure 2 shows another test with a fresh low residual bed. This time the I₂ bed was removed when the effluent residual reached 0.7 ppm, however no significant long term effect on the bed I₂ residual was observed. The I₂ bed was replaced in the feed stream until the effluent reached 1.5 ppm, when it was removed. The I₂ residual continued to increase to about 4 ppm where it stabilized and then showed a rather erratic washout.

The most promising results are shown in Figure 3. A fresh bed was again installed, and an I₂ bed placed in the line which had been washed so it was only producing 40 ppm I₂. It remained in the line until the resin bed was producing 1.8 ppm and then removed. The effluent from the resin bed immediately dropped below 1 ppm and gradually washed out to 0.2 ppm. The I₂ bed which was conditioned until it was producing 63 ppm was reinstalled, and the I₂ from the resin bed rapidly approached 3 ppm at which time the I₂ bed was removed and the resin effluent immediately dropped to 2 ppm. The bed was then allowed to wash down to 1 ppm, and a bed of fresh I₂ crystals installed. It was producing 98 ppm and the resin effluent went to 4 ppm in 1 hour; thus the I₂ bed was removed. The resin effluent immediately dropped to 1.2 ppm and was allowed to return to 1 ppm when a bed of I₂ which was producing 55 ppm was reinstalled. The resin effluent went immediately to 3 ppm but quickly returned to 2 ppm when it remained for 4 hours. The I₂ bed was removed, and the next morning the effluent had washed to 1.6 ppm. A detail of the four hours the I₂ bed was in line is shown in the inset on Figure 3.
The two four hour regeneration cycles shown on Figure 3 were in effect conducted with beds of high residual resin that had been exhausted. They clearly demonstrate that resin can be regenerated without removal from the cartridge and suggest that a system for inflight regeneration is feasible.

The sharp spike which occurs when the I₂ bed is put on line would not be a problem in a flight system, since the resin bed would undoubtedly be feeding a storage tank where the spike would be lost by dilution.

It appears that a simple and reliable system could be developed for long term missions. Enough crystalline iodine for a mission could be stored in a single cartridge which could be inserted in the main or various water lines in the potable or wash water systems upon a signal from an I₂ monitor. Additional work is necessary to investigate the behavior of I₂ beds as well as the extensive cycling of a resin bed over a long period of time.
FIGURE 2, RESIN REGENERATION

I$_2$ FEED 7 Hrs @ 40 ppm

I$_2$ FEED 24 hr @ 58 ppm

TIME (hours)

EFFLUENT IODINE RESIDUAL

I$_2$ OUT ppm

0 5

1 4

2 3

3 2

4 1

5 0
FIGURE 3, REPEATED RESIN REGENERATION

EFFLUENT IODINE RESIDUAL

$I_2$ FEED  $I_2$ Conc.
40 ppm  63 ppm

$100$ $200$ $300$ $350$ $400$

TIME (hours)

$I_2$ OUT ppm

$I_2$ CONC.:
1 ppm $55$ ppm
Task 5. **ADVANCED MCV PROTOTYPE**

An advanced MCV has been designed using lighter materials and fabrication procedures. The design is shown in Figure 4. The major weight savings is realized by the body being manufactured by spinning 300 series stainless steel sheet. The end cap is also 300 series sheet stock. The snap ring collar is replaced with a sheet metal clamp which is similar to a commercially available design. It is estimated that this design will save approximately 90 grams per unit or 0.2 lbs (present design 500 gm).

A prototype unit was fabricated according to this design concept and delivered (See DD250 in Appendix). The delivered unit is shown in Figure 5. This unit was designed around an off-the-shelf clamp, consequently all dimensions are not within the constraints of the 2x5 envelope. The prototype was subjected to all of the flight acceptance tests required for the previously certified units and passed satisfactorily. Consequently, flight certification of the new design by similarity with limited testing should be possible.

A resin cartridge which will simplify recharging both old and new units was designed and built, see Figure 6. The prototype was machined out of a block of polypropylene, thus the wall thickness is somewhat heavier (and rougher) than would be possible with an injection molded part. This cartridge will make recharging the MCV on site a very simple procedure. The cartridges are designed so they can also be reloaded and stored for reuse.
COST ESTIMATES

Due to the similarity between the Advanced MCV design and the Flight Qualified MCV, it is estimated that it would cost in the neighborhood of $10,000 to flight certify the Advanced MCV.

Fabrication costs for the Advanced MCV's should be approximately $2,000 per unit in lots of 14 units. The resin cartridge feature of the Advanced MCV will mean that fewer units will be required. Recharge time will be reduced to a few minutes between flights, and a unit may be recharged with a new cartridge and re-installed rather than replaced with a recharged unit.
**MATERIAL INSPECTION AND RECEIPT REPORT**

Contract Number NAS9-15854

<table>
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<tr>
<th>SHIPMENT NO.</th>
<th>B/L</th>
<th>R/C</th>
<th>1. PRICE INSTRUMENT IDENT (CONTRACT)</th>
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<td>Contract Number NAS9-15854</td>
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</tbody>
</table>

**PRIME CONTRACTOR**

UMPOQUA RESEARCH COMPANY  
P.O. Box 791 
Myrtle Creek, OR 97457

**SHIPPED FROM (IF OTHER THAN 9) CODE**

O

**SHIP TO**

Transportation Officer, Bldg. 420  
NASA-Lyndon B. Johnson Space Center  
Houston, TX 77058

Accountable Property Officer 807402  
For reissue to: Richard Sauer, SE3  
Bldg. 36, Rm 136

**ITEM**  
**STOCK/FRAG NO.**  
**DESCRIPTION**  
**QUANTITY**  
**UNIT PRICE**  
**AMOUNT**

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</thead>
</table>
| ru 3 | Microbial Check Valve Prototype  
Part No. MCV-02  
Serial No's P2, P3, P7  
Retrofitted to Flight Configuration | 3 | 2000 | 6000 |

---

**ORIGINAL PAGE IS OF POOR QUALITY.**

---

**RECEIVER'S USE**

Quantities shown in column 17 were received in apparent good condition except as noted.

**A. ORIGIN**

☐ POA ☐ ACCETPANCE OF LISTED ITEM HAS BEEN MADE BY ME OR UNDER MY SUPERVISION AND THEY CONFORM TO CONTRACT, EXCEPT AS NOTED HEREIN OR ON SUPPORTING DOCUMENTS.

**B. DESTINATION**

☐ POA ☐ ACCEPTANCE OF LISTED ITEM HAS BEEN MADE BY ME OR UNDER MY SUPERVISION AND THEY CONFORM TO CONTRACT, EXCEPT AS NOTED HEREIN OR ON SUPPORTING DOCUMENTS.
<table>
<thead>
<tr>
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<th>Quantity Ship'd/Rec'd</th>
<th>Unit Price</th>
<th>Amount</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td></td>
<td>Bulk high residual resin</td>
<td>1</td>
<td>2000.00</td>
<td>2000.00</td>
</tr>
<tr>
<td>1</td>
<td></td>
<td>Bulk low residual resin</td>
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<td>2000.00</td>
<td>2000.00</td>
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These are flight items
**Material Inspection and Receiving Report**

**1. PROCEDURE** IDENTIFICATION (CONTRACT) ORDER NO.

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<th>Amount</th>
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</thead>
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<tr>
<td>1</td>
<td>Advanced lightweight MCV prototype PB</td>
<td>THIS ITEM IS NOT FOR FLIGHT</td>
<td></td>
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**2. SHIPMENT NO.** 30June80

**3. DATE SHIPPED**

**4. B/L** TCN

**5. DISCOUNT TERMS**

**9. PRIME CONTRACTOR**

UMPUQA RESEARCH COMPANY
P.O. Box 791
Myrtle Creek, OR 97457

**10. ADMINISTERED BY**

**11. SHIPPED FROM (if other than 9) CODE**

**12. PAYMENT WILL BE MADE BY**

**13. SHIPPED TO CODE**

Transportation Officer, Bldg. 420
NASA-Lyndon B. Johnson Space Center
Houston, TX 77058

Accountable Property Officer 807402
For reissue to: Richard Sauer, SE3
Bldg. 36, Rm 136

**14. MARKED FOR CODE**

**15. ITEM NUMBER**

**16. STOCK, PART NO.** (Indicate number of shipping container-type of container/container number)

**17. QUANTITY SHIPPED/REC'D**

**18. UNIT**

**19. UNIT PRICE**

**20. AMOUNT**

**21. PROCUREMENT QUALITY ASSURANCE**

**22. RECEIVER'S USE**

Quantities shown in column 17 were received in apparent good condition except as noted.

**DATE RECEIVED**

**SIGNATURE OF AUTH GOVT REP**

**TYPED NAME AND OFFICE**

**DATE**

**SIGNATURE OF AUTH GOVT REP**

**TYPED NAME AND TITLE**

**23. CONTRACTOR USE ONLY**

**DD FORM 250** REPLACES EDITION OF 1 AUG 67 WHICH MAY BE USED