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Evaluation of RTV as a Moldable Matrix When Combined With Molecular Sieve and Organic Hydrogen Getter

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J. A. Knight

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Final Report

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Final Report
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Abstract

This work was undertaken in an effort to develop a combined RTV 615/3Å molecular sieve/DEB molded component. A molded RTV 615/3Å molecular sieve component is currently in production, and an RTV 615/DEB component was produced in the past. However, all three materials have never before been combined in a single production part, and this is an opportunity to create a new component capable of being molded to shape, performing desiccation, and hydrogen gettering. This analysis looked at weapons system parameters and how they might influence part design. It also looked at material processing and how it related to mixing, activating a dessicant, and hydrogen uptake testing.

Summary

Weapons systems have long used both desiccants and getters to scavenge moisture and undesirable gaseous species. These components are typically separate. Desiccants usually consist of either a loose molecular sieve powder contained within a bag or a combination RTV or molecular sieve molded to shape. Getters originally were granulated material in a cloth bag, but they are more recently contained in a tube or a combined RTV and DEB molded component.

From a design standpoint, the use of two separate components, getter and desiccant, poses an issue with placement. The designer has to accommodate an area within the system to house two separate components. Neither of these components provides any structural support; furthermore, their incorporation may reduce structural integrity. From a manufacturing perspective, the use of two components presents an issue from an inspection standpoint. Having two additional components increases opportunities for failed parts due to manufacturing defects or poor product performance.

Building upon previous work by Schicker[1] and multiple successful examples of combining an RTV and molecular sieve, an effort was undertaken to combine getter and dessicant within a single molded RTV component. If successful, this would require the incorporation of one less component into the system. Additionally, the moldable nature of the RTV material would give designers the flexibility to place the new component in places historically unavailable to tube getters and bagged desiccants. Finally, the elastomeric nature of the RTV material could potentially allow for a component that performed three specific functions: gettering, desiccating, and compression/shock absorbing For manufacturing, the combined part would reduce the total number of manufacturing operations previously required for two components. It would also reduce the number of dimensional inspections. With a combined part, the dimensional requirements would only need to be measured once instead of multiple times.

This report addresses many of the issues faced in combining these components. Desiccating requirements were addressed by looking at the total moisture that could be brought into a system by polyurethane foam. An empirical equation was determined to relate the surface area and molded density of a polyurethane part to the total amount of moisture it could adsorb. Simple proof-of-concept parts were molded with RTV 615, filler, getter and desiccant, at percentages of 50 wt. %. At this loading, parts were molded consisting of 45 wt. % desiccant and 5 wt. % getter as well as 45 wt. % getter and 5 wt. % desiccant. The final weight percentages selected were 45 wt. % desiccant and 5 wt. % getter. The selection of these ratios was based on legacy system requirements, o-ring leak rate experiments, and manufacturing considerations. RTV 615 with filler percentages of greater than 50% is difficult to mold

due to material viscosity, and the final molded parts are brittle. Initial design definitions consisted of a ring component with an outside diameter of 12 inches and an inside diameter of 10 inches. A sample part was successfully molded from an existing desiccant mold. This part met the formulation requirements and radiography showed only a single void. A stereolithographed mold was created to specifically meet the current component design definition. Sample parts were successfully made with this mold. In conjunction with the ring component, there was a proposed second component loosely based on a legacy component. Using existing tooling, a prototype was molded with the defined component ratios.

Material processing factors were also considered. It was shown that the three components, RTV 615, getter, and desiccant, could be homogeneously mixed within a Thinky planetary centrifugal mixer model ARE-250. Mixing was achieved solely by the Thinky mixer with no prior hand mixing required. The affect that post mixing material handling had on the moisture content of the desiccant was evaluated. It was determined that mixing directly after drying the desiccant and then freezing results in no appreciable moisture uptake out to seven days post mixing. Additionally, it was shown that at 60 °C and vacuum there is no detrimental effect to gettering capabilities. Minimal weight loss was observed. This was shown to be due to outgassing of various volatile organics. This appears to be linked almost exclusively to the RTV composition of the parts. In an effort to resolve this, modifying the resin to catalyst ratio was attempted, but resin to catalyst ratios above 11:1 caused incomplete curing of the part. An empirical equation was determined to link the surface area to volume ratio of a part to the rate at which it adsorbs moisture. This information will be useful in development of a final dimensional design definition.

This report is not conclusive, but it does provide a first pass at many of the design hurdles presented by this new component. Further testing will need to be conducted before this component is ready for manufacturing. However, it shows great promise as a potential replacement for the traditional tube getter assemblies and bagged desiccants.

Discussion

Scope and Purpose

This work was undertaken with the goal of developing a moldable component consisting of RTV 615, DEB getter, and 3 Å molecular sieve desiccant. The fact that the RTV can be molded to shape allows designers to place the desiccant and getter components in spaces previously unattainable to tube/pellet assemblies and bagged desiccant. It also eliminates the need to specifically design around these components. This allows the desiccant and getter to be designed around existing voids and cavities within the system. Ultimately, the assembly may serve a structural role. Manufacturing is simplified since a single component is built instead of a minimum of two, and dimensional inspection requirements are decreased.

This report addresses many of the manufacturing issues faced by the development of this product. System requirements are addressed. Part composition and geometric definition are also highlighted. Material processing factors such as mixing method, desiccant activation method, and material handling are explored. Mold design is briefly addressed as it relates to current part definition.

Prior Work

Schicker[1,2] has shown the feasibility of combining RTV 615 and DEB getter in a single component. Parts were successfully molded and getter activity was demonstrated. The combination of RTV 615 and 3 Å molecular sieve desiccant has been used previously. The W76-1 AF&F desiccant is currently manufactured using these two components. Additionally, palladium catalyst and molecular sieve have been incorporated into RTV 615 to produce a molded component with both moisture adsorption and hydrogen gettering capabilities[3]. However, the gettering capabilities of this component are reliant on the availability of oxygen, and as a result, are not suitable for applications in an anaerobic environment.

Activity

Foam Hydration Study

This study was aimed at determining the total amount of moisture structural polyurethane foams of varying surface area, molded density, and surface finish could adsorb. Additionally, moisture uptake rates were evaluated from a qualitative standpoint for foams of varying conditions mentioned above.

Experimental

All foam samples were molded from PMDI polyurethane foam with a free rise density of 10 lb/ft^3. Aluminum molds were manufactured to produce parts with right circular cylinder geometry of three different surface areas with a consistent volume. Table 1 shows the mold dimensions and the resultant part volume and surface area. The parts were molded to three different molded densities 10, 20, and 30 lb/ft^3. A picture of the test molds and the parts produced can be found in appendix 1, figures 26 and 27 respectively. The samples were all hand poured in triplicate and then post cured at 250 °F for two hours. After stripping from the mold, half of the samples had the skin machined off their respective surfaces. All samples were then allowed to stabilize for 14 days at 15% relative humidity (RH). The samples were then dried under vacuum at 250 °F until their weight stabilized and all adsorbed moisture was assumed to be driven off. After drying, the samples were placed into a humidity chamber kept at 98% RH. Sample weights were taken periodically until their weight stabilized. Microsoft Excel and MATLAB were used to analyze the data. The raw data was normalized and an average, standard deviation, confidence interval, and t test was performed. The normalized averages were plotted against time, and a curve fit was applied assuming the data fit the following equation, $w(t)=-x_o e^{-\alpha t}+x_o+x_i$, where x_i is the initial weight, x_o is the total amount of weight change at time $t = \infty$, and α is the rate at which the weight is changing (1/hr). Using Minitab and the average values for x_o and α, a statistical comparison was made between each group. An empirical equation was developed to relate the total moisture uptake to the foam surface area and molded density. A least squares regression analysis was performed with the data fit to the following equation, $x_o(\rho, SA) = a_o + a_1\rho + a_2 SA$, where x_o is as defined previously, ρ is the molded density, SA is the surface area, and the a_i are constants.

Table 1. Mold Dimensions and resultant part surface area and volume

Mold Depth (in.)	Mold Diameter (in.)	Part Surface Area (in.^{2})	Part Volume (in.^{3})
.500	2.000	9.42	1.57
.757	1.625	8.01	1.57
1.060	1.375	7.54	1.57

Results

The foam samples were organized into 18 different groups. They were grouped first by surface condition, molded or machined, then by molded density, and finally by surface area. Appendix 1, table 10 shows the corresponding groups and their descriptions. Appendix 1, tables 11 and 12 shows the raw weight data for each of these sample groups. Figures 1-3 show the normalized weight gain for each group. After analysis and curve fitting, the resultant values for x_o and α were determined for the average normalized values from each group. These are shown in appendix 1, table 13. A one-way ANOVA performed between the surface condition of as molded to machined showed no statistical difference for x_o and α with p values of 0.240 and 0.424 respectively. Subsequent calculations thus grouped the molded and machined samples into one group for each surface area and density. This was done to increase the sample size of each. Table 2 below shows the p values for comparisons amongst each group for x_o and α. Using the calculated values of x_o and α for each group, an equation was developed to relate both x_o and α to the surface area and molded density. The data was assumed linear and a multivariable least squares regression analysis was used. The values of the coefficients a_i are shown in table 3. The resultant equations are $x_o(\rho,SA)=2.6453-0.015932(\rho)+0.135360(SA)$ and $\alpha(\rho,SA)=0.013852-0.0016182(\rho)+0.0046262(SA)$. Figures 4 and 5 show surface plots of the measured values of x_o and α, respectively for varying foam molded densities and surface areas.

Table 2. P values for comparison between groups

X_o	
Comparison Group	**p Value**
7.54 $in.^2$ to 8.01 $in.^2$	0.035
7.54 $in.^2$ to 9.42 $in.^2$	0.000
8.01 $in.^2$ to 9.42 $in.^2$	0.004
10 lb/ft^3 to 20 lb/ft^3	0.000
10 lb/ft^3 to 30 lb/ft^3	0.000
20 lb/ft^3 to 30 lb/ft^3	0.002
α	
Comparison Group	**p Value**
7.54 $in.^2$ to 8.01 $in.^2$	0.738
7.54 $in.^2$ to 9.42 $in.^2$	0.097
8.01 $in.^2$ to 9.42 $in.^2$	0.167
10 lb/ft^3 to 20 lb/ft^3	0.000
10 lb/ft^3 to 30 lb/ft^3	0.000
20 lb/ft^3 to 30 lb/ft^3	0.000

Table 3. Coefficients for linear systems of equations

X_o	
a_o	2.6453
a_1	-0.015932
a_2	0.135360
R^2	0.96594
α	
a_o	0.013852
a_1	-0.0016182
a_2	0.0046262
R^2	0.96542

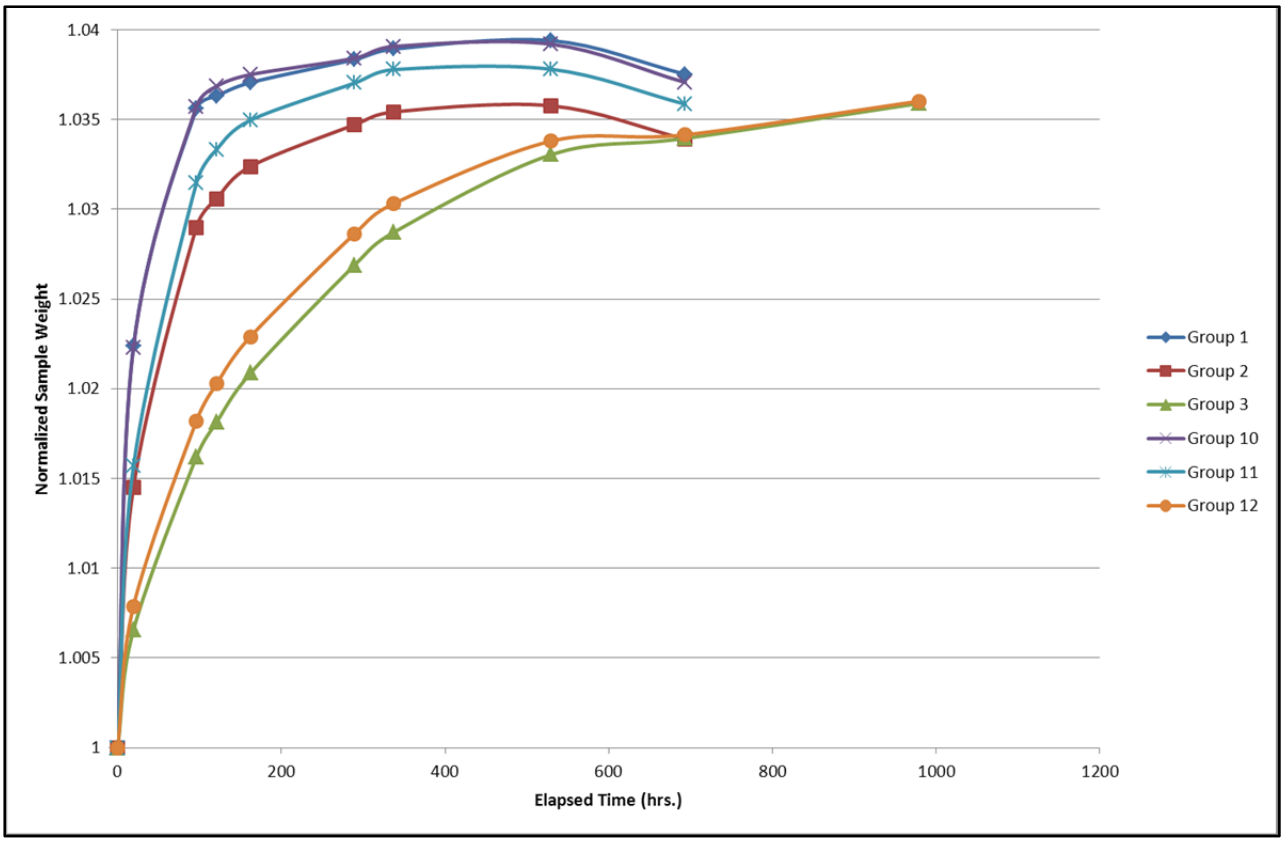

Figure 1. Normalized weight gain of foam samples with 9.42 in.^{2} surface area. Surface finish and density vary.

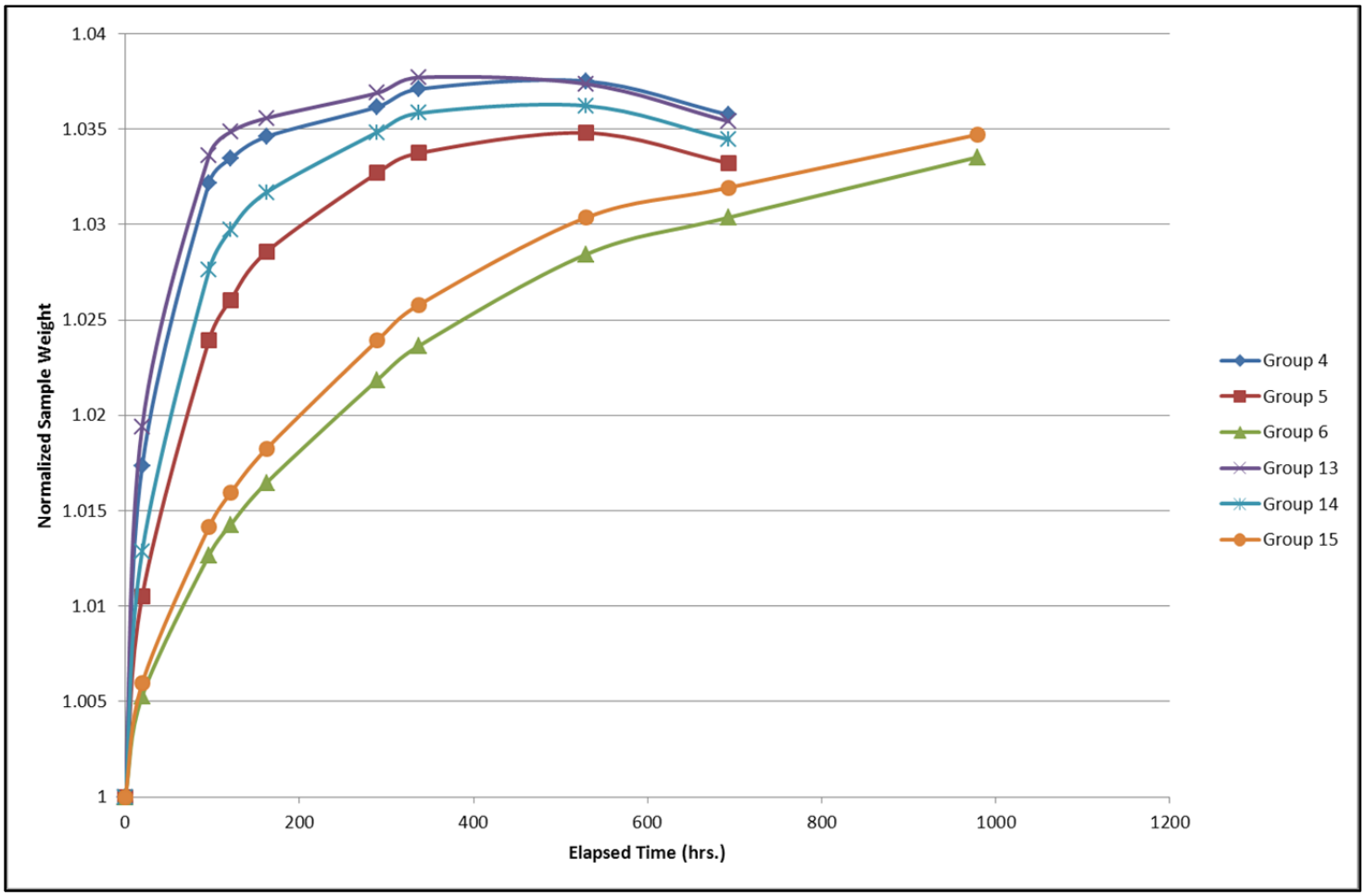

Figure 2. Normalized weight gain of foam samples at 8.01 in.^{2} surface area. Surface finish and density vary.

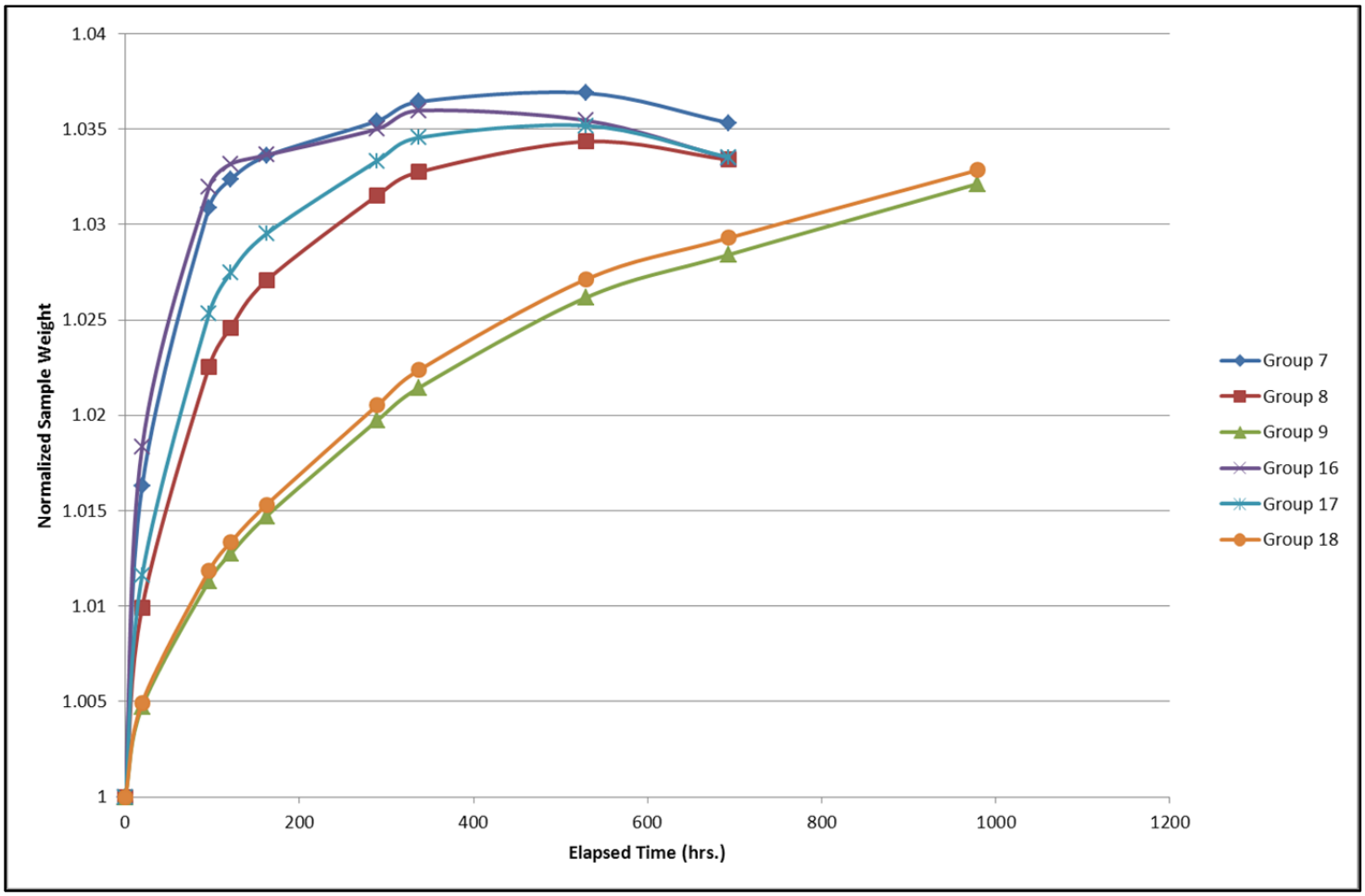

Figure 3. Normalized weight gain of foam samples at 7.54 in.^{2} surface area. Surface finish and density vary.

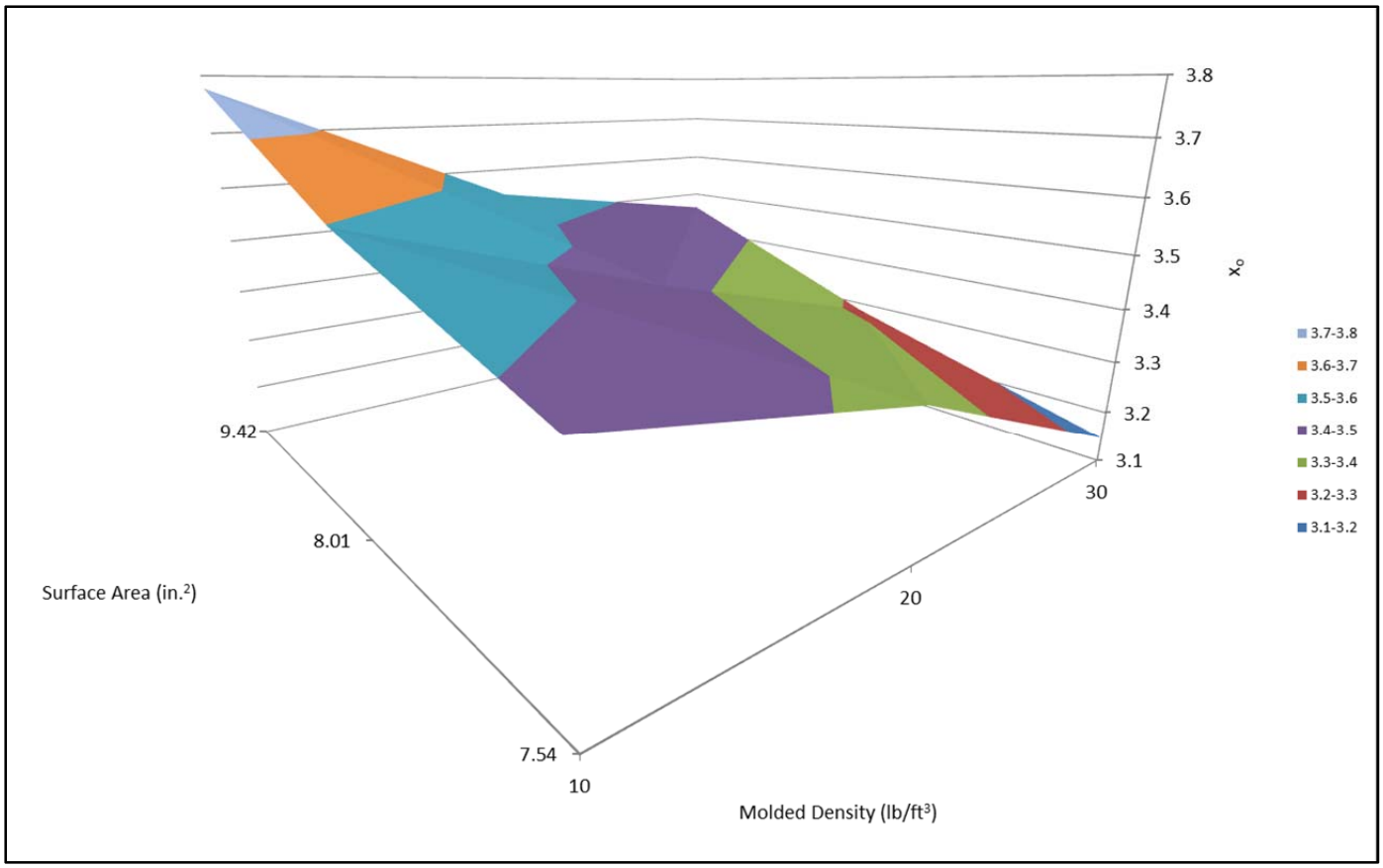

Figure 4. Surface plot of measured values of x_o

Foam surface area and molded densities vary.

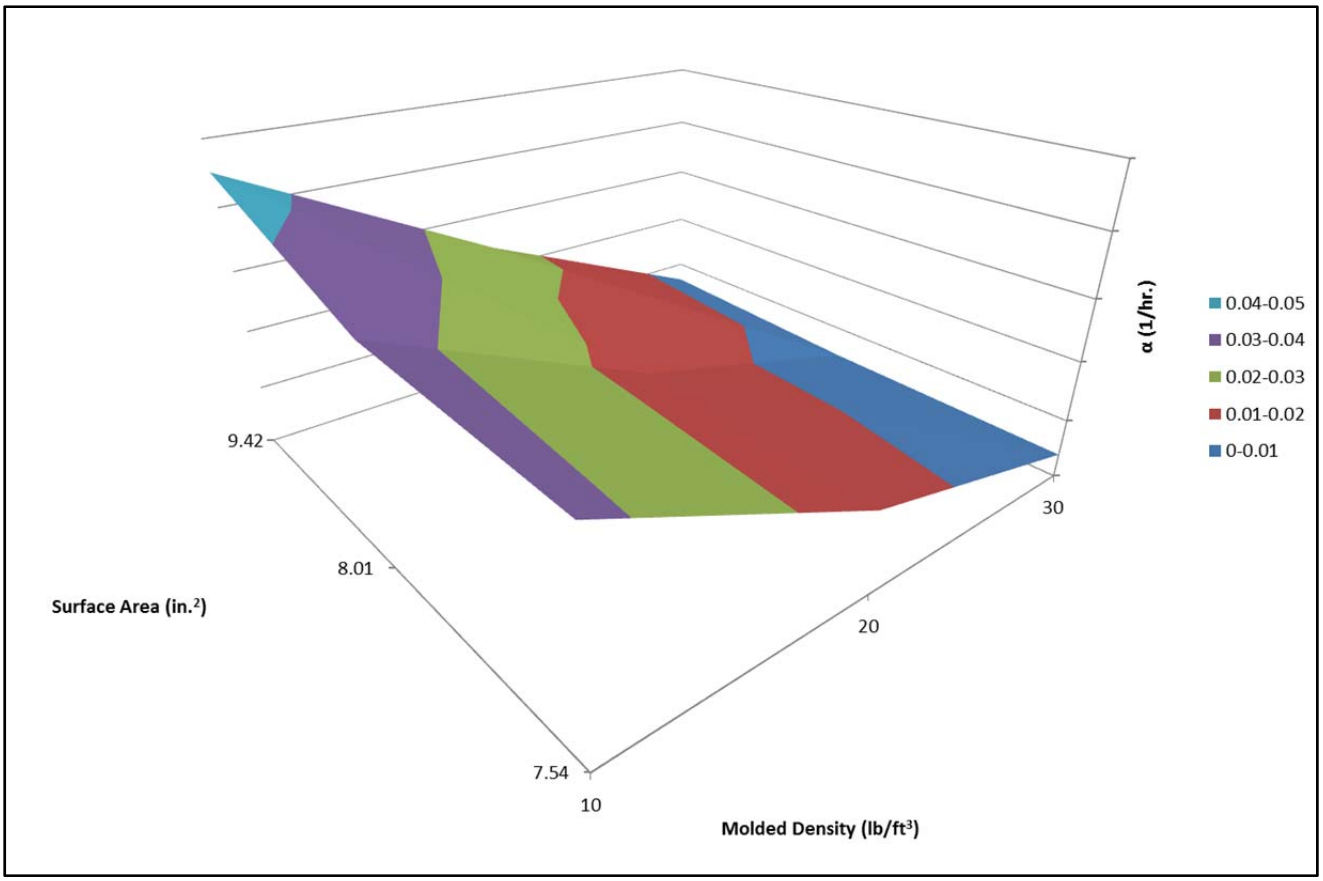

Figure 4. Surface plot of measured values of α.

Foam surface area and molded densities vary.

From this work, it can be seen that the surface condition of the foam, molded or machined, plays no role in either the total moisture uptake or the rate at which it occurs. This is contrary to what is expected. The potentially larger surface area of machined foam, due to exposed cell structure, would theoretically increase both total water capacity and uptake rate. Except for comparing the 7.54 $in.^2$ to 8.01 $in.^2$ surface areas, there were statistical differences in the total amount of moisture that was adsorbed by foams of varying surface areas and molded densities. All foams adsorbed between 3.1 and 3.8 wt. % water. There were also statistical differences noted between the uptake rates. All three molded densities showed statistical differences with the fastest uptake being shown by the 10 lb/ft^3 foam and the slowest by the 30 lb/ft^3 foam. Increased packing density reduces the rate of moisture diffusion throughout the foam. As well, statistical differences were noted between the various surface areas. As expected, the largest surface area showed the fastest uptake while the smallest surface area showed the slowest. The statistical differences were weaker than when comparing densities, but perhaps this can be attributed to the smaller percentage differences between each surface area. While the empirical equation for x_o can be used to quantify moisture loading values, the equation for α is strictly valuable from a qualitative standpoint. The impact that both surface area and molded density have on uptake rate can be seen from this equation, but the actual value of α should not be used from a design perspective. It is highly subjective to the environment, and this experiment was conducted at an extreme humidity of 98%.

O-Ring Leak Rate Experiment

See appendix B for attached memo from SNL employee.

Proof of Concept Preliminary Molding

Using existing tooling for the o- ring desiccant and the type 3 filler, sample parts were molded at the defined ratios of 50 wt. % RTV 615, 45 wt. % 3 Å molecular sieve desiccant, and 5 wt. % DEB getter. These parts were molded to determine if any serious design flaws or unexpected hurdles were present prior to beginning work on tooling to meet the new product definition.

Experimental

For both parts, material was mixed within a one liter Jelenko mixing cup. Initially, RTV 615 A and B components were weighed into the cup. These components were then mixed for 30 seconds with the Jelenko mixer. To this mixture, the previously dried and weighed desiccant was added, followed by the DEB getter. Both of these powdered materials were added a small amount at a time with stirring in between additions. Once all the dry powder had been added, the material was mixed again on the Jelenko. Mixing was performed for 30 seconds followed by 30 seconds rest, alternating until 2.5 minutes of total mix time passed. After mixing, the material was hand poured into a 20 oz. Semco cartridge. The material was degassed under vacuum and then either injected or frozen at -14 °F if not used immediately. Material was injected into the molds using a pneumatic ram operated at 80 psi. The lid for the o-ring mold was modified and made out of Lexan instead of aluminum. Dimensionally, it was identical; the material is the only thing that changed. The clear Lexan allowed visual inspection of material flow within the mold during injection. The molds were treated with Miller Stephenson mold release MS-122AD. The mold release was sprayed on with no subsequent buffing. After molding, samples were cured for two hours at 170 °F, followed by a minimum of 16 hours post cure at the same temperature. After post cure, samples were removed from the mold and deburred.

Results

Two examples of the ring were molded and one example of the filler was successfully molded. There were no major processing hurdles that had to be overcome. Observing the material flow in the ring mold, it was noted that the flow was retarded along the path containing the most features. The highly viscous nature of the RTV/3Å/DEB material created a large amount of friction between all the mold features slowing the flow. This was evidenced in the fact that the two halves of material flowing through the mold did not meet opposite the gate. Instead they met along the side with the most features. This indicates the flow that was least retarded moved faster, as is to be expected. Radiography of the ring components showed the presence of a single void. It was present in both samples, and such an occurrence can easily be remedied through venting. Figures 5 and 6 show radiographs of the complete ring and the void respectively.

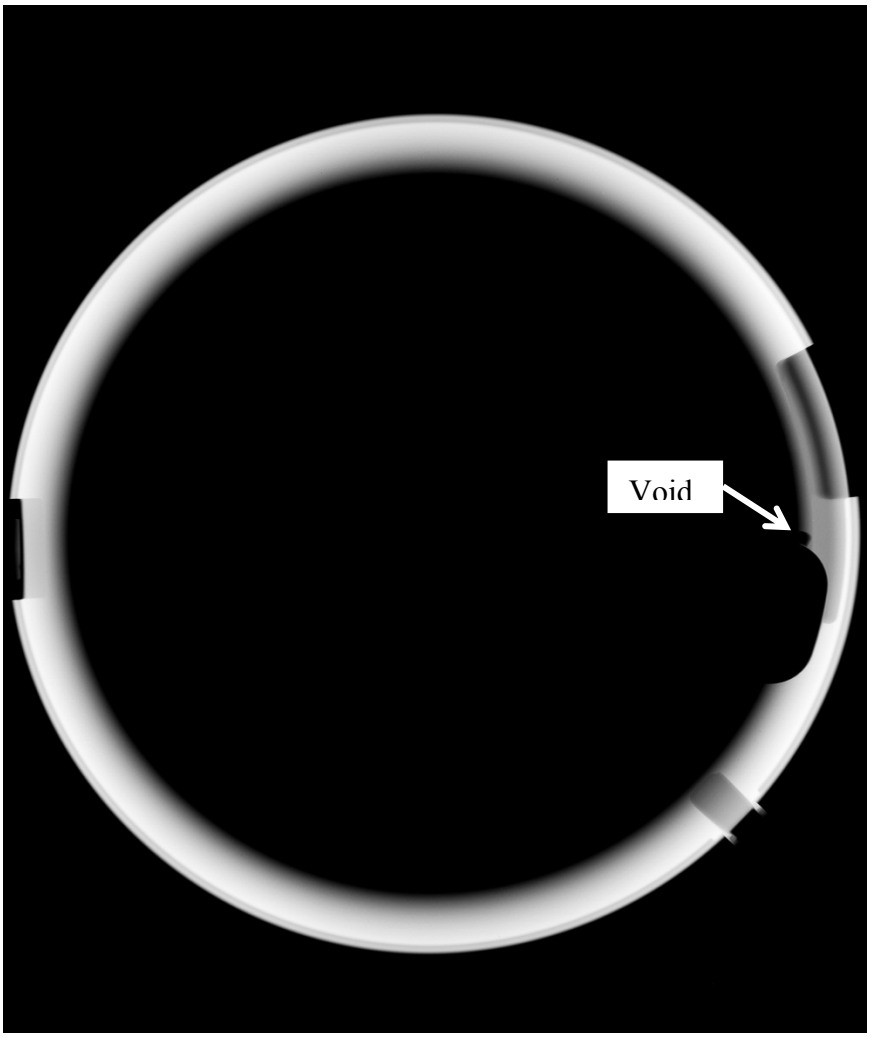

Figure 5. Complete Ring

Figure 6. Close up of void present ring

Evaluation of Feasibility to Mix Directly Within Thinky Mixer

Although successful mixing has been achieved using the Jelenko mixer, the ease of use and speed of the Thinky mixer is advantageous. The Thinky mixer was evaluated to determine if homogenous material mixing could be achieved by simply combining all ingredients within either a 55cc syringe or a 2.5 oz. Semco cartridge. Furthermore, it is possible to mix within a Thinky mixer under vacuum. If successful mixing could be achieved under vacuum, this would combine multiple operations required by the use of the Jelenko into a single, quick operation.

Experimental

All samples were composed of the standard mix ratio of 50 wt. % RTV 615, 45 wt. % 3 Å molecular sieve, and 5 wt. % DEB getter. The desiccant was weighed out "wet," and it was assumed the additional moisture mass would not adversely affect the material mixing. A Thinky model ARE-250 was employed with custom inserts to mix directly in either a 55cc syringe or a 2.5 oz. Semco cartridge. The materials were each weighed into either the cartridge or syringe and then mixed. If successful mixing occurred, the samples were dispensed into an aluminum weighing dish and cured for 18 hours at 170 °F. Table 4 shows the conditions tried. It should be noted that headspace refers to the empty volume left within the mixing container. It is based upon an assumed final material density of 1.28 g/ml. In reality, the actual headspace will be less due to the greater volume occupied by the uncompressed powder.

Table 4. Mixing protocol for Thinky trials

Trial	Headspace	Mix Speed (rpms)	Mix Time (secs.)	Material Addition Order	Container
1	75%	1900	30	liquid then powder	55 cc Syringe
2	50%	1900	30	liquid then powder	55 cc Syringe
3	50%	1900	30	powder then liquid	55 cc Syringe
4	50%	1900	---	liquid then powder added in batches with 30 sec. mixing after each addition of powder	55 cc Syringe
5	50%	1900	60	liquid then powder	55 cc Syringe
6	50%	1000	60	liquid then powder	55 cc Syringe
7	50%	2000	60	liquid then powder	55 cc Syringe
8	75%	1900	30	liquid then powder	2.5 oz. Semco
9	50%	1900	30	liquid then powder	2.5 oz. Semco

Results

All samples mixed in the 55 cc syringes showed no mixing. Instead, there was definite stratification of the material. The mixing speed, time, and material addition order seemed to have no effect on the result. Figure 8 shows an example of the results obtained by mixing within the 55 cc syringe. Mixing within the 2.5 oz. Semco cartridge resulted in a homogeneous mixture of material. Upon dispensing of the material, there was a small amount of unmixed RTV located in the nipple at the bottom of the syringe. Initial samples that were not degassed showed some signs of unmixed desiccant and cellular structure upon sectioning. Figures 7, 8 and 9 show examples of this phenomenon. Subsequent samples that were degassed showed no evidence of lack of mixing. Figures 10 and 11 are representative of this.

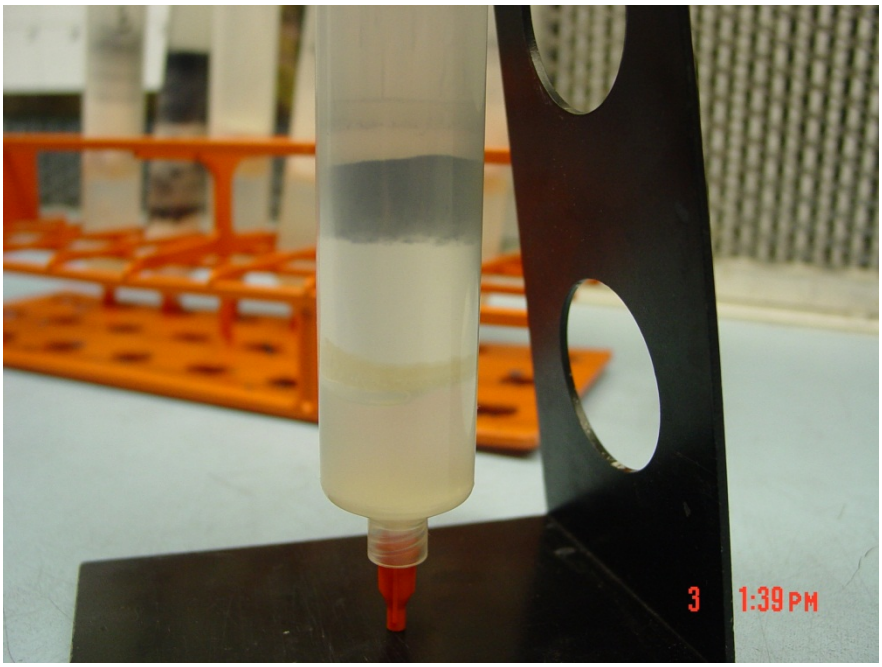

Figure 7. Material stratification within 55 cc syringe

Figure 8. Trial 9 bottom view, some inhomogeneity visible.

Figure 9. Section view of trial 9 samples, cell structure and inhomogeneity visible

Figure 10. Example of degassed trial 8 sample

Figure 11. Section view of degassed trial 8 sample

Conclusions

The inability of the material to mix within the 55 cc syringe is thought to be a function of the diameter of the container in relation to the depth of the material. There is simply not enough room for the material to move. Instead, the mixer behaves more like a centrifuge, simply compacting the layers. However, there was no evidence of separation by density, simply stratification without mixing. The mixing within the 2.5 oz. Semco was much more successful. Examples of inhomogeneity were evident when degassing was not performed. It is thought that the degassing removes the last amount of inhomogeneity by providing further mixing. As the material moves due to dissolved gasses moving through the material, some additional mixing occurs. Indeed as can be seen in figure 11, sectioning of the samples showed no inclusions or cell structure due to dissolved gasses. The use of the Thinky with the 2.5 oz. Semco cartridge shows promise to provide a rapid means of mixing. Future work will need to focus on whether Thinky mixers with vacuum capabilities can provide further step reduction by performing mixing and degassing all in one operation. It should be noted that Thinky does offer mixers capable of handling larger production sized volumes. These are available both with and without vacuum capabilities.

Effect of Material Processing on Desiccant Activation Time

Since the amount of desiccant in the part is based upon a weight percentage, it is critical that the desiccant weight be a dry weight. Since the desiccant starts dry, it would be advantageous for it to remain dry, thus avoiding a post molding activation of the part. One method to achieve this is to process the part dry, for example, in a glovebox. This complicates processing and if possible should be avoided. Since the desiccant is dried prior to weighing, an attempt was made to characterize how much moisture was picked up during mixing, molding, and storage while exposed to ambient conditions.

Experimental

Material was mixed with the aforementioned ratio of components using the Jelenko mixer following the protocol outlined in the experimental section of Proof of Concept Preliminary Molding. A single lot was manufactured, and all samples were produced from this one lot. Parts were molded in triplicate using a 2 inch diameter, .500 inch thick aluminum mold. Parts were injected using a pneumatic ram operated at 30 psi. Figure 12 shows an example of the injection device and mold.

Figure 12. Injection device and mold

After molding, parts were cured 2 hours at 170 °F followed by an additional 16 hours post cure at 170 °F. After post cure, the samples were stripped from the mold, weighed, and double bagged in vapor barrier bags. Material was kept at -14 °F between moldings. After all parts had been molded, the parts were placed into a vacuum oven and soaked under vacuum at 60 °C until part weights stabilized. Parts were weighed periodically throughout the soak. Table 5 shows the material mixing and subsequent molding dates and times.

Table 5. Material mixing and molding schedule

Date/Time	Action
5/11/2011 14:00	Material Mixed
5/11/2011 15:00	Initial Mold Cycle
5/12/2011 11:00	Mold Cycled
5/16/2011 14:35	Mold Cycled
5/17/2011 12:30	Mold Cycled
5/18/2011 12:30	Mold Cycled

Data was analyzed using Excel and Matlab. The data was analyzed statistically and a curve fit was applied using a Gauss Newton nonlinear fit to the following equation, , - , where x_i is the initial weight, x_o is the total amount of weight change at time , and α is the rate at which the weight is changing (1/hr).

Results

Appendix 3, table 19 shows the raw weight values for each sample. Figure 13 shows an overlay of the weight loss for each group. Table 6 shows the resultant values for x_o and α for each curve. It also shows the R^2 value. Figures 15 and 16 show the values of x_o and α with error bars. All samples lost an average of .38 to .41 wt. % over the course of 507 hours.

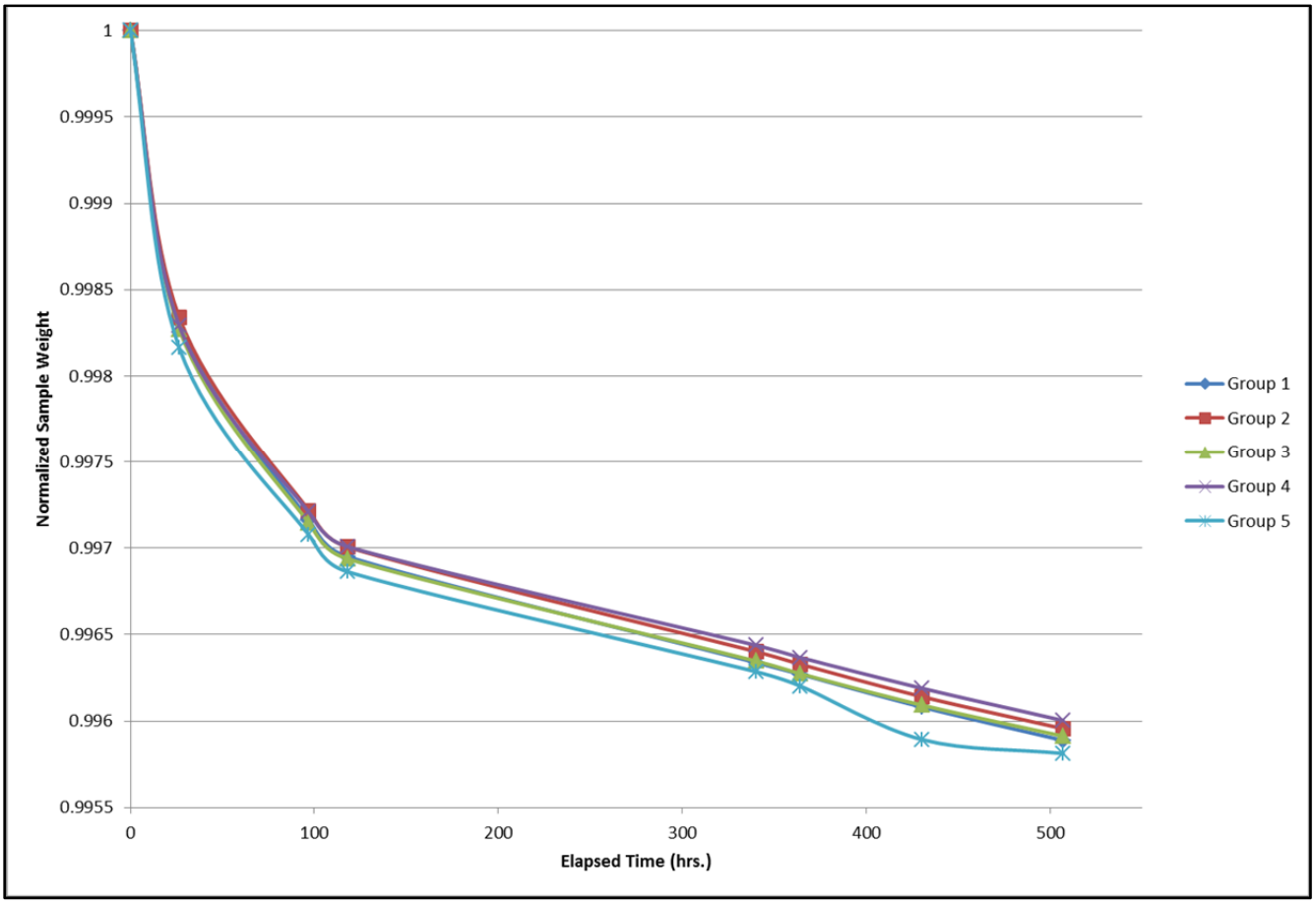

Figure 13. Overlay of sample drying

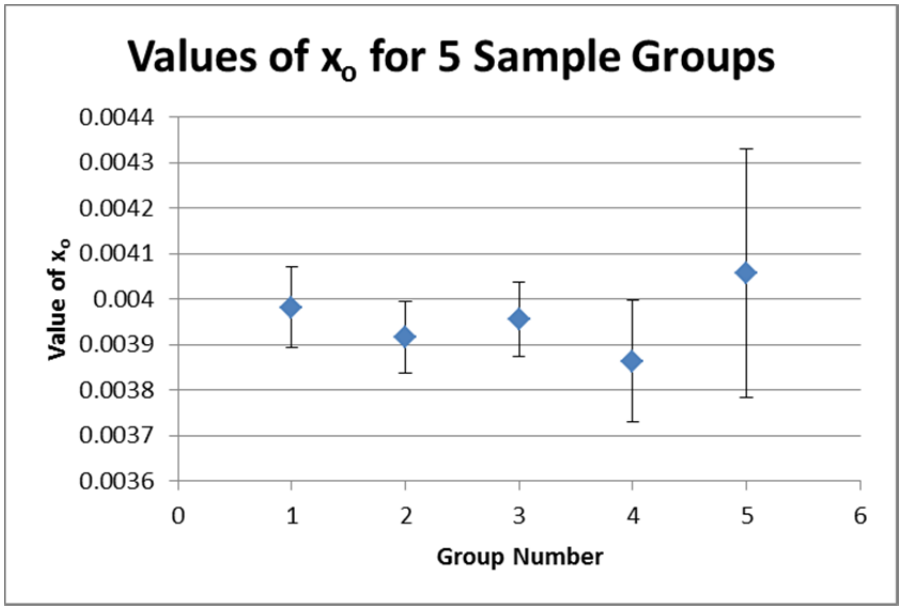

Figure 13. Values of x_o for each molding group

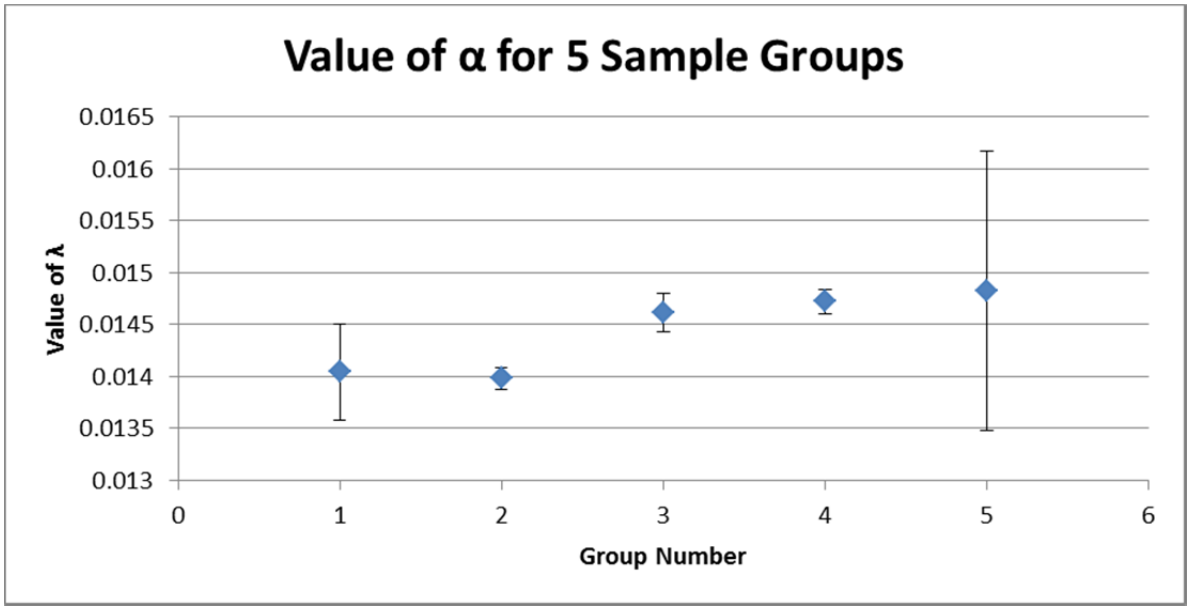

Figure 14. Values of α for each molding group

Table 6. Average values of x_o, α, and R^2 from curve fit of each group

Group	x_o	α (1/hr.)	R^2
1	0.00383178	0.0156272	0.976413
2	0.00377023	0.0155417	0.976049
3	0.00381294	0.0162294	0.975331
4	0.00371768	0.0164472	0.973811
5	0.00391333	0.0164744	0.968250

Conclusions

All of the samples lost some weight. The amount of weight is low enough and occurred over such a long time interval to seem to suggest that it is not moisture. Any moisture that the samples picked up during processing would likely be surface moisture that did not have time to diffuse within the part. As a result, it should appear as a rapid weight loss within the initial few hours to days instead of over the course of 21 days. Figure 13, coupled with applying a student's t test, shows that the weight loss by each group is statistically the same. This also seems to indicate this is not adsorbed moisture. The moisture on each sample should have increased from group 1 to 5 due to increased time exposed to the ambient environment. However, this is not the case. It can be seen that the drying rates for each group are the same except for groups 3 and 4. These two groups are different from groups 1 and 2. Group 5 is statistically the same as all groups, but that is only due to the large error associated with this measurement. It is unknown at this time what caused the difference in the rate of weight loss. All samples should have been geometrically the same; as well, their time and temperature exposure should have been the same. This study shows that the desiccant likely does not pick up any moisture during processing or after drying. However, it raises the question of where this weight loss is coming from. Two possible scenarios are sublimation of the organic DEB from the getter and outgassing of low molecular weight components from the RTV. Subsequent experiments will aim to address these questions.

Evaluation of RTV as Source of Material Weight Loss

One possible scenario for the low mass, long term weight loss observed previously is that it is related to the RTV itself. Samples of RTV alone were molded at the typical 9:1 resin to catalyst ratio. Additionally, samples of RTV alone were molded with a 10:1 and 11:1 resin to catalyst ratio. All samples were molded to the same geometry of a 2 inch diameter disc .500 inch thick just as was previously done with the RTV/3 Å molecular sieve/DEB getter parts. This was done to determine if the same weight loss is observed with RTV alone. Also it was done to see if altering the amount of catalyst changes the amount of weight loss observed. In conjunction with this, .010 inch thick sheet material was molded with RTV 615/3Å molecular sieve/DEB, RTV 615/3Å molecular sieve, and RTV 615 alone. Headspace analysis was performed on this material directly after molding and multiple days afterwards during a soak under vacuum at 60 °C to determine what, if any, species were outgassing.

Experimental

For the weight loss analysis of the RTV alone, all samples were molded using an aluminum mold, which produceda right circular cylinder part with a 2 inch diameter and .500 inch thick. The RTV was mixed in a 2.5 oz. Semco cartridge in a Thinky model ARE-250 mixer. Mix ratios of resin to catalyst were the standard 9:1 as well as 10:1 and 11:1. The material was degassed for 20 minutes after mixing and prior to injection. After degassing, samples were injected directly using a pneumatic ram. All were cured 2 hours at 170 °F followed by a 16 hour post cure at 170 °F. After molding, samples were placed into a vacuum oven and soaked under vacuum at 60 °C. Sample weights were monitored periodically until stabilized. Using Excel and MATLAB, the weight loss was analyzed using a Gauss-Newton nonlinear fit to the following equation, $w(t)=x_o e^{-\alpha t}-x_o+x_i$, where x_i is the initial weight, x_o is the total amount of weight change at time $t = \infty$, and α is the rate at which the weight is changing (1/hr).

For the headspace analysis, all samples were mixed directly in a 2.5 oz. Semco cartridge using a Thinky mixer. Three different samples were molded. The first contained the RTV 615/3 Å molecular sieve/DEB mix. The second sample contained only RTV 615 and 3 Å molecular sieve, and the third sample contained RTV 615 only. The samples were not degassed and were injected directly onto a flat slab mold using a pneumatic ram. The mold produced a part .010 inch thick. All samples were cured 2 hours at 170 °F, followed by a 16 hour post cure at 170 °F. Samples were collected immediately after molding. The remaining samples were placed in a vacuum oven and allowed to soak under vacuum at 60 °C. Samples were removed at 24, 48, and 72 hours post molding. The headspace of each of the samples was qualitatively analyzed for volatile organics using a GC-MS.

Results

The results of the headspace analysis are shown in appendix D. In general, there were several species present in all sample groups regardless of the components mixed together. There did not seem to be any pattern to the species present. In time, the amount of different compounds dropped off sharply.

Figure 15 shows an overlay of the measured normalized weight loss values for the RTV and filler, the RTV alone at a resin to catalyst ratio of 9:1, and the RTV alone at a resin to catalyst ratio of 10:1. appendix D, table 20 shows the average normalized weight loss data. The RTV only samples molded at a resin to catalyst ratio of 11:1 never fully cured even after a 72 hour post cure. They were omitted from this analysis. All samples showed a normalized weight loss of less than 1% with this weight loss occurring over the span of over a month. Statistical analysis showed a statistical difference between the

values of x_o and α comparing the RTV with filler to the RTV alone. There were not statistical differences between the values of x_o and α for the RTV alone. Plots of these values are shown in figures 15 and 16 respectively. Table 7 shows the values of x_o and α for each of these groups.

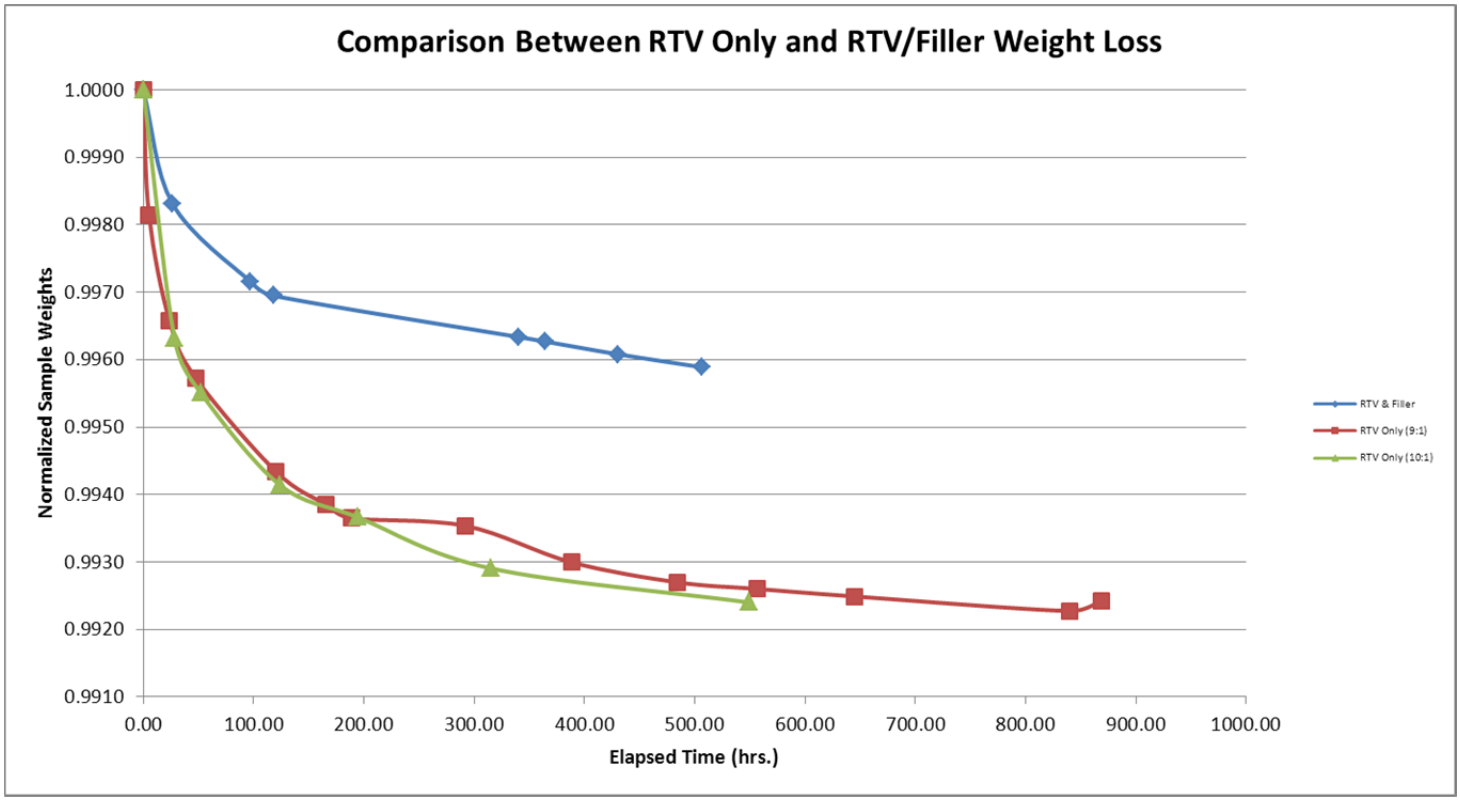

Figure 15. Overlay of weight loss by RTV and filler and RTV only

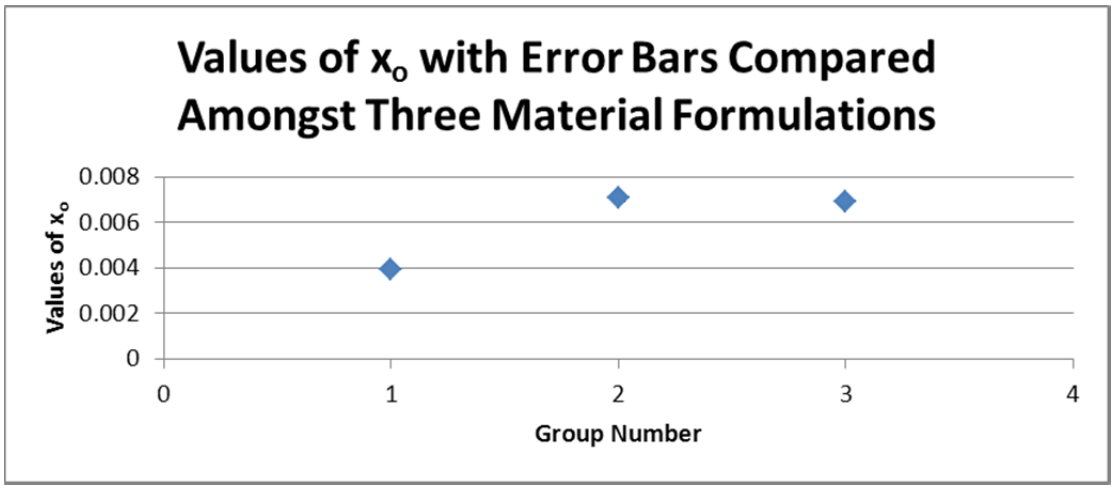

Figure 16. Values of x_o for each material formulation

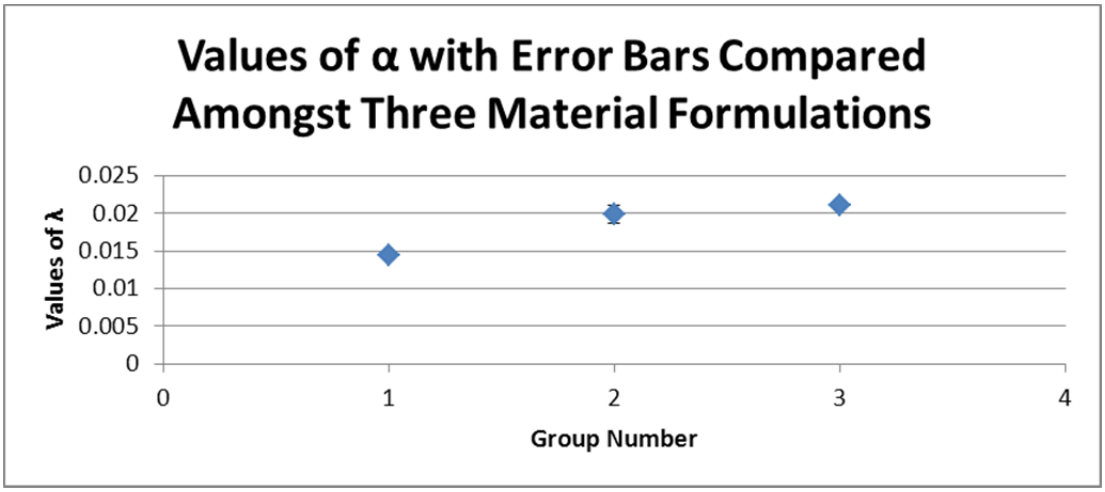

Figure 17. Values of α for each material formulation

Table 7. Values of x_o and α for each material formulation

Group	x_o	α
1	0.003954	0.014438
2	0.007070	0.019904
3	0.006936	0.021022

Conclusions

From the headspace analysis, it is reasonable to think that some of the weight loss could be due to volatile organics. This was not a quantitative analysis so it is difficult to make direct comparisons between the observed weight loss and the outgassing species. At 72 hours, there were only a handful of species left in the headspace analysis. However, weight loss was exhibited by the disc samples well out to 30 days. Again, since this was strictly qualitative, it is unknown if the species that dropped off after 72 hours would have contributed negligible mass relative to those that persisted after 72 hours. The weight loss analysis of the RTV alone and RTV filler seems to be more indicative of the fact that this weight loss is due to something from the RTV itself. The total weight loss for both ratios of RTV alone, 9:1 and 10:1, is much greater than that of the RTV with filler. If the weight loss is coming from the RTV this would be correct since the RTV and filler consist of the total RTV amount than would be in RTV alone. The differences seen in the rates of weight loss, between RTV and filler and RTV alone, could be attributed to different cell structure of the RTV itself resulting in different rates of diffusion. This work does seem to indicate that the small long term observed weight loss is due to some component of RTV.

Evaluation of Getter Effectivity Post Vacuum Oven Bake

To determine if the observed long term weight loss is due to sublimation of the organic DEB, the effectivity of the getter was evaluated before and after soaking under vacuum and 60 °C for 4 days. It is assumed that any sublimation of the getter will manifest itself as a reduction in the mean theoretical hydrogen capacity (MTHC).

Experimental

A sample of the RTV 615/3 Å molecular sieve/DEB getter, at the standard mix ratio, was molded into a .010 inch sheet. The material was mixed in a 2.5 oz Semco cartridge directly in a Thinky model ARE-250 mixer. The material was not degassed prior to injection into the mold with a pneumatic ram. The sample was cured for 2 hours at 170 °C, followed by a post cure at 170 °C for 16 hours. Samples were submitted for analysis of MTHC per LTM 3396 with samples taken at 2 hours and 16 hours. The remaining material, post molding, was placed into a vacuum oven under vacuum and 60 °C for 4 days. After this soak, samples were again analyzed per LTM 3396 with samples taken at 2 hours and 16 hours.

Results

Appendix E contains the raw data from the analysis. Figure 28 below shows a comparison between the samples that were exposed to the oven soak and those that were not.

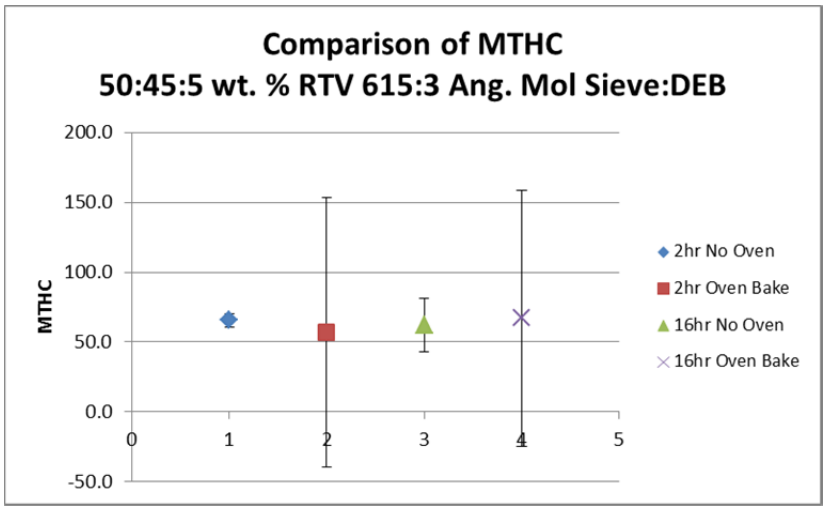

Figure 18. Comparison of MTHC for samples with and without an oven bake

Conclusions

From this analysis, there is no statistical difference between the getter that experienced the oven bake and the sample that did not. It should be noted that while the mean values were very close for each run the error associated with the measurements for the samples that experienced the oven bake is very high. These data points only represent an n=2 population, and the experiment should be duplicated to determine if this variability continues. However, at this point, it does not appear that the vacuum oven bake does anything to reduce the effectivity of the getter.

Proof of Concept Molding to Current Design Definition

A preliminary design was established for the new getter and desiccant ring component. It would be fabricated from RTV 615, 3 Å molecular sieve, and DEB getter. The formulation would be 50:45:5 wt. %. The tentative location of the ring is to sit positioned between Pad R, 321883, and a deck foam. A schematic of this is shown in figure 19.

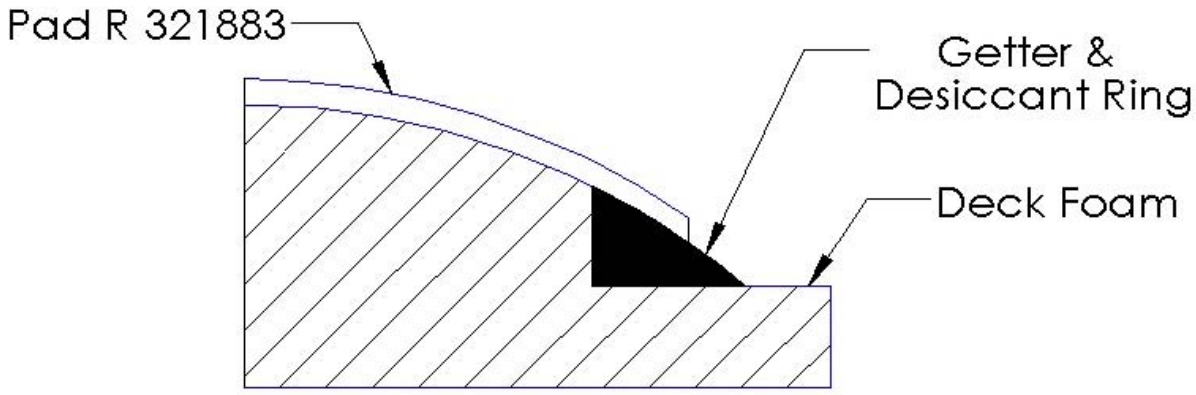

Figure 19. Cross-section view showing tentative location of getter and desiccant ring

Experimental

The new ring component consists of a simple ring structure with an inside diameter of 10 inches and an outside diameter of 12 inches It has a roughly triangular cross-section with a vertical leg of .500 in. and a horizontal leg of 1.000 in. The remaining side has a radius that matches the inner radius of Pad R, 12.655 inches. Appendix E figure 27 shows a dimensional drawing for the part. From this part definition, a mold was designed and fabricated using stereolithography. Appendix E, figures 28 and 29 show drawings for the mold. The material was mixed in a Jelenko mixer according to the protocol outlined in the experimental section of Proof of Concept Preliminary Molding. Samples were cured 2 hours at 170 °F followed by a 16 hour post cure at 170 °F. After molding, one of the samples was cut into two pieces, comprising approximately one quarter of the ring each and two pieces comprising approximately one eighth of the ring each. After approximately 72 hours, the two eighth pieces were placed into a humidity chamber maintained at 98 % RH. The samples were weighed periodically until their weight began to stabilize. The two quarter pieces were tested according to LTM 3396 for hydrogen uptake. One sample was exposed to hydrogen gas until the getter was completely reacted. The second sample was exposed for 24 hours, and then the test was ceased. The sample was then placed back into the reaction vessel and allowed to react to completion. The moisture uptake data was analyzed using Excel and MATLAB. The data, using a Gauss-Newton nonlinear method, was fit to the following equation, $w(t)=-x_o e^{-\alpha t}+x_o+x_i$, where x_i is the initial weight, x_o is the total amount of weight change at time $t = \infty$, and α is the rate at which the weight is changing (1/hr).

Results

As of writing this report, the hydrogenation results were not complete. Table 8 below shows the raw weights of the moisture uptake samples. The average value of x_o, from the curve fit, was .079348, and the average value of α, from the curve fit, was .009898 (1/hr). Figure 20 shows a plot of the moisture uptake with an overlay of the curve fit.

Table 8. Raw moisture uptake values

Ring Weights		
Date/Time	Sample 3/g	Sample 4/g
9/12/2011 10:00	23.1120	23.6560
9/13/2011 15:00	23.6816	24.2905
9/14/2011 9:30	23.8670	24.4868
9/19/2011 10:45	24.3922	25.0308
9/23/2011 11:00	24.7327	25.3772
9/30/2011 6:20	24.9932	25.6787

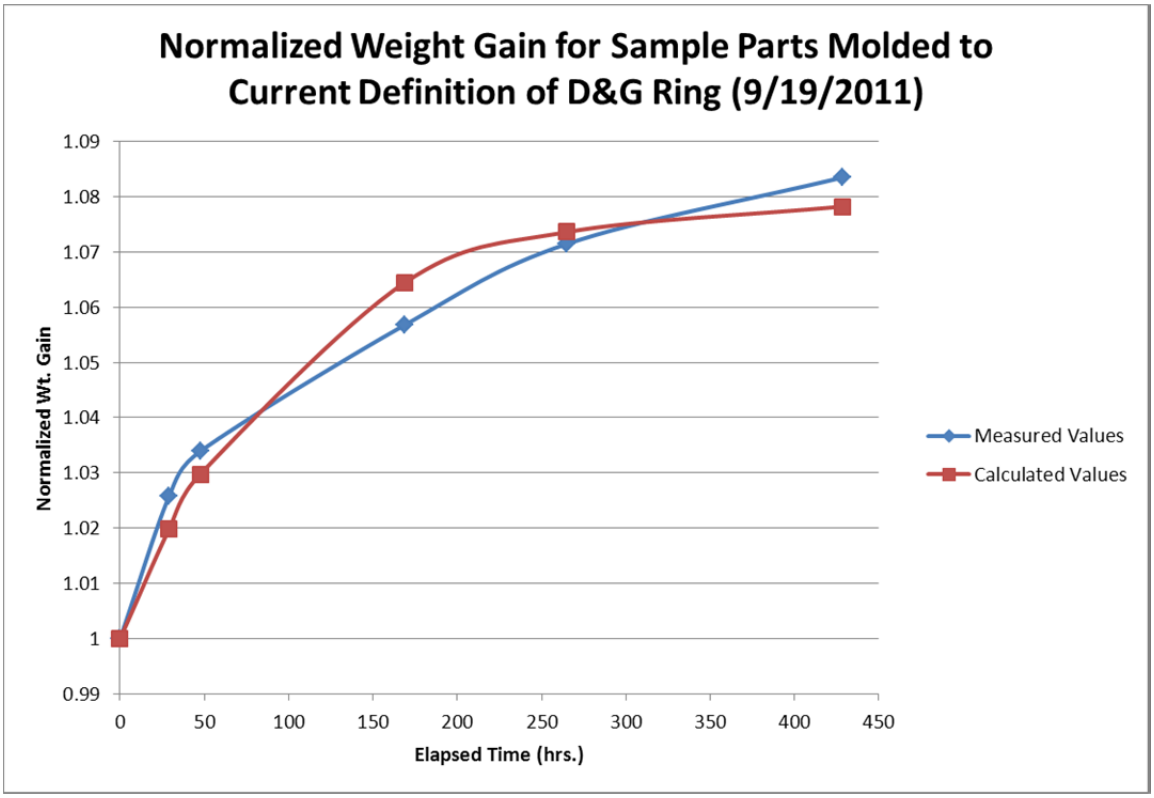

Figure 20. Plot of moisture uptake values for molded to definition part

Conclusions

As of this time, no conclusions can be derived from the hydrogenation testing. The moisture uptake data is utilized in a subsequent experiment looking at hydration rates as related to a part's surface area to volume ratio. It should be noted that the value of x_o will likely be lower than that for samples born dry. The portions of the molded ring sat exposed to the ambient environment for approximately 72 hours before being placed into the humidity chamber. Some of the moisture uptake capacity would have been compromised during this time. However, this previous exposure should not affect the uptake rate determined from the curve fit. The value of α should remain the same.

Impact of Surface Area to Volume Ratio on Moisture Uptake Rate

Increasing the surface area of the desiccant part improves the rate at which moisture diffuses through the part. Increasing this rate is important from a manufacturing standpoint because it can help to decrease activation times. Sample parts of the RTV 615/3Å molecular sieve/DEB material were molded at a fixed volume with varying surface areas. These parts were allowed to saturate within a humidity chamber, and the resulting rehydration rates were monitored as they related to the surface area to volume of the components.

Experimental

All molding was done within a dry nitrogen glovebox kept at less than a -40 °C frost point. The 3Å molecular sieve was dried, under nitrogen purge, at 428 °F and then placed within the glovebox. The samples were molded at the standard ratio of 50:45:5 wt. % RTV to molecular sieve to DEB. Samples were mixed in a 2.5 oz. Semco cartridge using a Thinky model ARE-250 mixer. Samples were then degassed and pneumatically injected into aluminum molds. The molds were designed to produce discs of varying surface area to volume ratios. Appendix F, figure 30 shows the drawing for the mold insert. All samples were molded in triplicate. The surface area to volume ratios produced were 20.0, 24.0, 31.4, and 41.3 1/inch. After the samples were molded, they were cured for 18 hours at 170 °F. After curing, the samples were removed from the mold and glovebox. They were then placed in a humidity chamber kept at 98 % RH. The samples were weighed periodically until their weight stabilized. The moisture uptake data was analyzed using Excel and MATLAB. The data, using a Gauss-Newton nonlinear method, was fit to the following equation, $w(t)=-x_o e^{-\alpha t}+x_o+x_i$, where x_i is the initial weight, x_o is the total amount of weight change at time $t = \infty$, and α is the rate at which the weight is changing (1/hr).

Results

Appendix F, table 21 shows the raw weight values for the uptake analysis. Table 9 below shows the values of x_o and α obtained from the curve fit. Included in this table are the values from the moisture uptake analysis on the molded to shape part. Figure 21 shows an overlay of the rehydration for each surface area to volume ratio. Additionally, figures 22 and 23 show plots of x_o and α respectively for each surface area to volume ratio. A curve fit has been applied to the plot of α versus surface area to volume ratio yielding the following equation,

$\alpha\left(\frac{SA}{V}\right) = 0.0002\left(\frac{SA}{V}\right)^{1.7427}$ with an R^2 value of .9988.

Table 9. Results from curve fit for surface area to volume of rehydrated samples

Surface Area to Volume Ratio (1/in.)	x_o	α (1/hr.)	x_i	R^2
10.3	0.0794	0.00989	1	0.9704
20.0	0.1036	0.03290	1	0.9851
24.0	0.1040	0.04295	1	0.9921
31.4	0.1058	0.07279	1	0.9987
41.3	0.1074	0.10922	1	0.9983

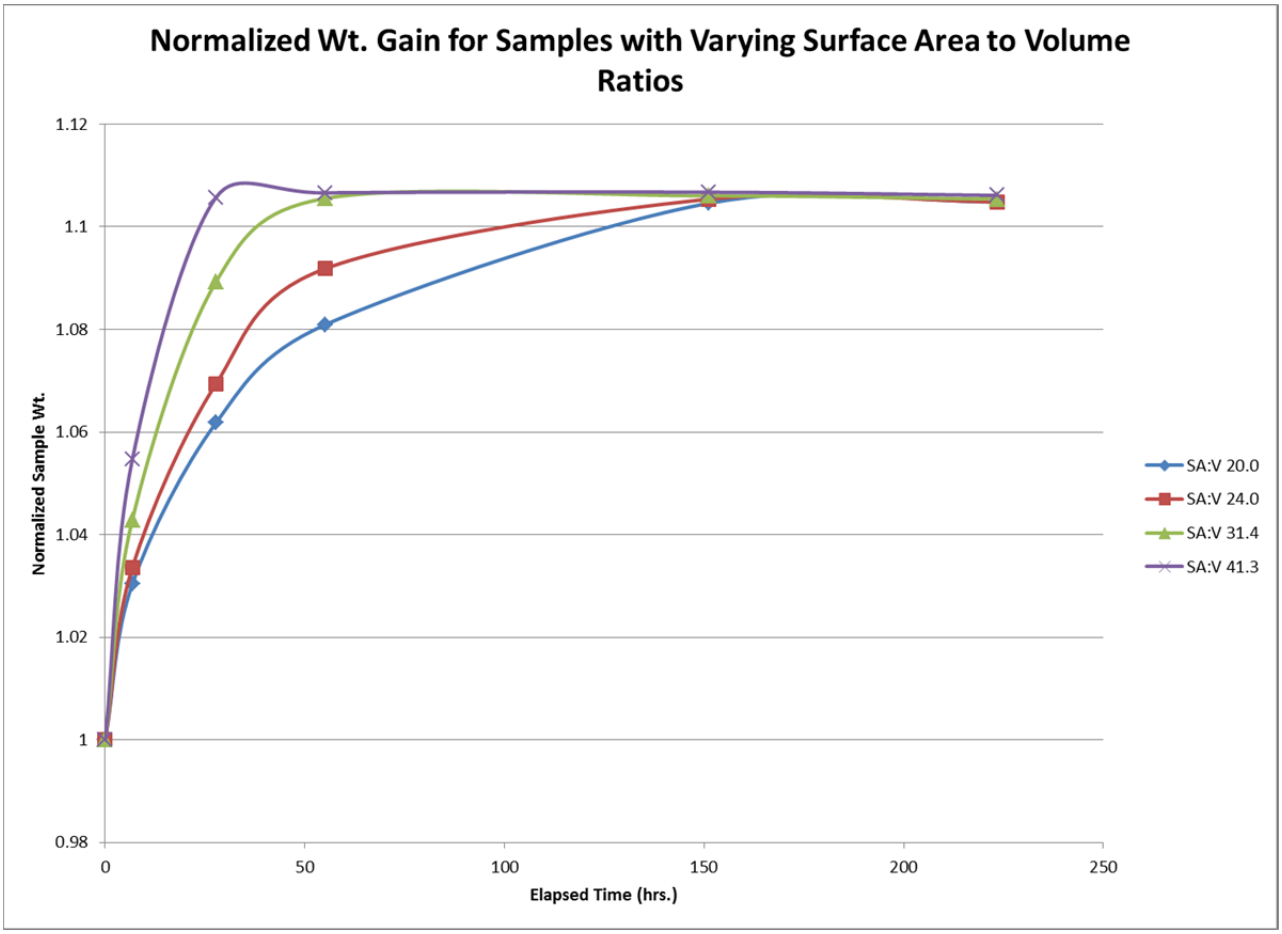

Figure 21. Overlay of rehydration of samples with varying surface area to volume ratios

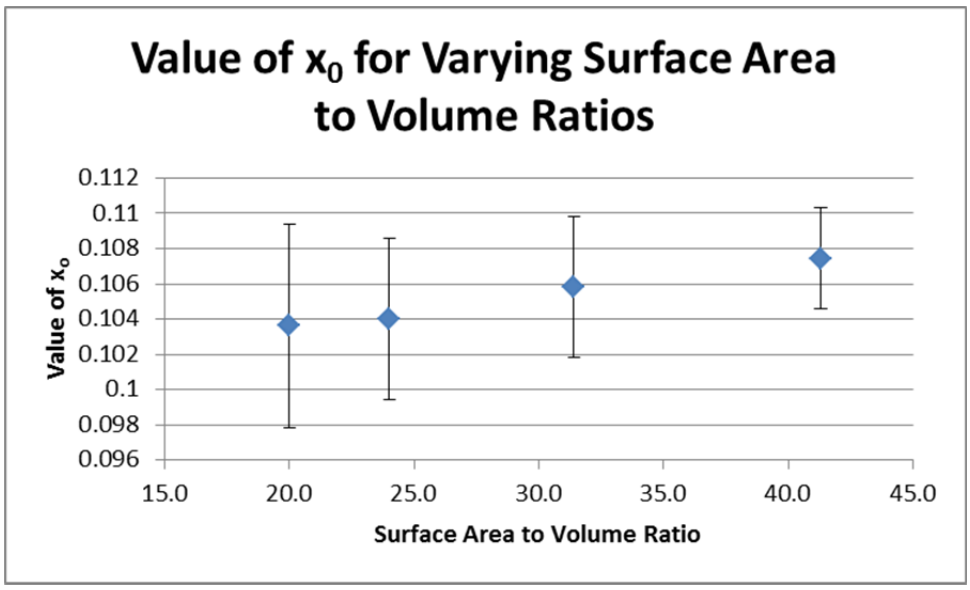

Figure 22. Values of x_o for varying surface area to volume ratios

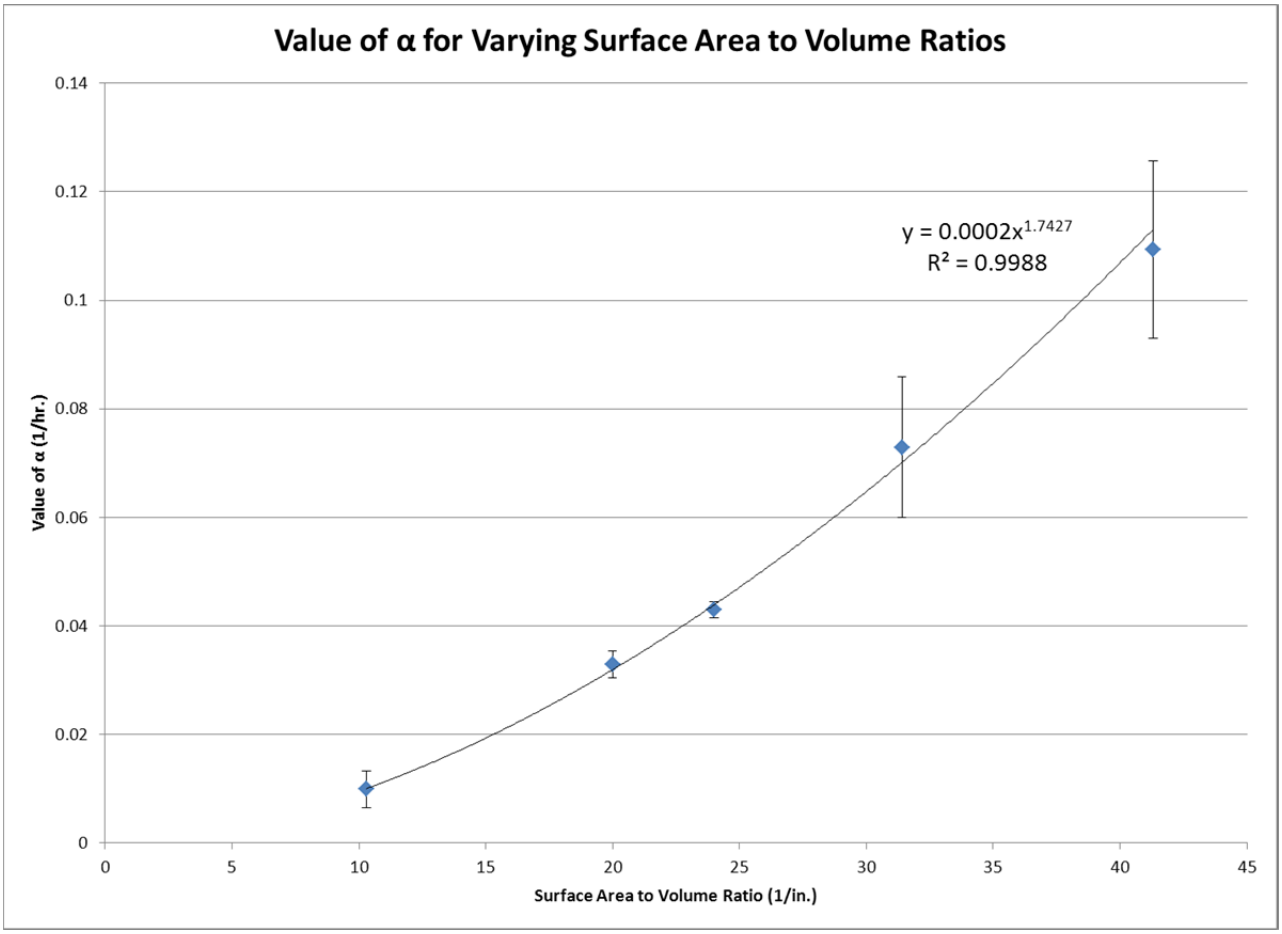

Figure 23. Values of α for varying surface area to volume ratios

Conclusions

All samples, except for the partially hydrated sample at a 10.3 (1/inch) surface area to volume ratio, showed a total moisture uptake of slightly greater than 10% by weight. These values all showed to be statistically the same. The values of α, however, are all statistically different. The measured values of α give good agreement with the power fit. Although the value for α for the 10.3 (1/inch) surface area to volume point was arrived at from a different experiment, it provided good agreement with the data from this experiment. This seems to indicate that this analysis will be a good predictive tool for design. It should be noted that comparisons amongst various values of α can only be done qualitatively. The numerical values of this experiment cannot be applied to other situations in which the rehydration or drying follows a different temperature profile or is in a different environment.

Accomplishments

This work has been able to show that for polyurethane foams, density plays the largest role in the rate of moisture adsorption. It has shown that the surface finish of a foam part, machined or as molded, does not have a statistical impact on the moisture uptake rate. Although statistically significant differences were observed between the total moisture uptake of foam parts, all densities and surface areas adsorbed between 3 and 4 wt. % moisture. Equations were developed to relate foam surface area and molded density to the total moisture uptake and the rate at which this would occur. These equations are $x_o(\rho, SA) = 2.6453 - 0.015932(\rho) + 0.135360(SA)$ and $\alpha(\rho,SA)=0.013852-0.0016182(\rho)+0.0046262(SA)$.

Proof-of-concept molded parts were produced using a desiccant mold. These parts yielded good results with a single void that could easily be remedied through the use of appropriate venting. Additionally, proof-of-concept parts were molded to the most recent design definition of the ring component with an inside diameter of 10 inches, an outside diameter of 12 inches, and an approximately triangular cross section. In both instances Miller Stephenson mold release MS-122AD was used successfully. There were no difficulties associated with this molding and further design development can begin.

It was shown that the three components, RTV 615, 3 Å molecular sieve, and DEB can be mixed directly in a 2.5 oz. Semco cartridge using the Thinky model ARE-250 planetary centrifugal mixer. It was shown that while processing within the glovebox is possible, it is not necessary. Material mixed, molded, and stored up to 7 days showed no appreciable moisture adsorption. As a result, parts can be produced dry within the ambient environment so long as proper post molding storage is considered. Preliminary work indicates that any activation or oven cycles post molding does not adversely affect the getter. Finally, the impact that the surface area to volume ratio has on the moisture uptake of parts was analyzed. The moisture uptake rate followed an exponential path given by the equation $\alpha\left(\frac{SA}{V}\right) = 0.0002\left(\frac{SA}{V}\right)^{1.7427}$. This data is for qualitative comparisons only since the exact values apply only to parts rehydrated within a 98 % RH environment. However, the relative behavior of one surface area to volume ratio to the next should remain constant regardless of the environment. This information can be used to further refine the design as it relates to the surface area of the component.

Future Work

With respect to processing, one important area for future work is in relation to mixing. It was shown that the material can be successfully mixed directly in a Semco cartridge using the Thinky model ARE-250 mixer. Thinky makes models capable of mixing under vacuum and models capable of handling production sized quantities. These mixers should be evaluated for their ability to combine multiple manufacturing operations into a single step. A continuation of the surface area to volume analysis and how it relates to part drying needs to be pursued. This plays a critical role in final part design, and the behavior of these parameters needs to be better characterized. As work is being focused on a final design, the molds themselves need to be considered. Things such as vent locations, gate size, and even mold material should be considered. Since the material is so viscous, these molds might perform better with a larger diameter gate. Also, since the part is relatively large, the question arises as to whether a steel or aluminum mold with a hardened coating would be better. One final area of focus should be on product testing both in relation to initial part inspection and surveillance of fielded components. Product acceptance could be based on test coupons or an entire part analysis. It would be performed lot to lot, but how should a lot be defined? Also, since the getter is intimately incorporated with the RTV, traditional surveillance techniques to look at percent hydrogenation will not work. Other methods such as reacting a part to completion and then estimating the level of hydrogenation based off a theoretical value could be considered. These are not insurmountable design problems, but they are questions that remain.

This technology shows promise for many different applications. The formulation in which a palladium catalyst is used in place of DEB could be employed in a weapons system where oxygen was available, either supplied or within the ambient environment. From a manufacturing perspective, this formulation would provide new possibilities for desiccant activation since sublimation of the organic getter, under vacuum and elevated temperature, would no longer be a concern. Additionally, continuing to use RTV as the binder and broadening the scope of desiccation and gettering from just water and hydrogen gas presents even more possibilities for component design. It is possible that fillers capable of scavenging other gaseous species could be incorporated into the RTV matrix. These possibilities still need to be explored.

References

[1]Schicker, J.R., Evaluation of Polyethylene and Other Polymers as a Moldable Matrix When Combined with Organic Hydrogen Getters (Final Report). UNCLASSIFIED. FM&T: KCP-613-5541, May 1995.

[2]Schicker, J.R., M59 Aiming Post Light Getter Tritium (Hydrogen) Confinement System Study (Final Report). UNCLASSIFIED. W15BW930478, September 1993.

[3]Schicker, J.R., Smith, H.M., Development of Inorganic Hydrogen and Moisture Getter for Sealed Devices – HMC Getter (Final Report). UNCLASSIFIED. FM&T: KCP-613-6278, January 2000.

Appendix A

Foam Hydration Study

Table 10. Sample groups with corresponding descriptions

Sample Group	Condition	Density (lb/ft^3)	Surface Area (in^2)
1	As Molded	10	9.42
2	As Molded	20	9.42
3	As Molded	30	9.42
4	As Molded	10	8.01
5	As Molded	20	8.01
6	As Molded	30	8.01
7	As Molded	10	7.17
8	As Molded	20	7.17
9	As Molded	30	7.17
10	Machined	10	9.42
11	Machined	20	9.42
12	Machined	30	9.42
13	Machined	10	8.01
14	Machined	20	8.01
15	Machined	30	8.01
16	Machined	10	7.17
17	Machined	20	7.17
18	Machined	30	7.17

Table 11. Raw, as-molded sample weights

Condition	As Molded Weight (g)											
Density (lb/ft^3)	10				20				30			
Diameter (in.)	2				2				2			
Replicate	1	2	3	Avg.	1	2	3	Avg.	1	2	3	Avg.
Sample Date & Time	AM10SA	AM10SB	AM10SC		AM20SA	AM20SB	AM20SC		AM30SA	AM30SB	AM30SC	
3/3/2011 13:30	6.2067	6.2589	6.0963	6.1873	8.0425	7.6580	7.9928	7.8978	11.8956	11.1883	11.7780	11.6206
3/4/2011 8:50	6.3470	6.3812	6.2484	6.3255	8.1446	7.7802	8.1115	8.0121	11.9702	11.2668	11.8530	11.6967
3/7/2011 13:35	6.4297	6.4700	6.3224	6.4074	8.2597	7.8924	8.2275	8.1265	12.0809	11.3814	11.9630	11.8084
3/8/2011 14:25	6.4339	6.4768	6.3247	6.4118	8.2743	7.9037	8.2393	8.1391	12.1034	11.4044	11.9853	11.8310
3/10/2011 7:45	6.4374	6.4837	6.3285	6.4165	8.2918	7.9154	8.2530	8.1534	12.1351	11.4363	12.0170	11.8628
3/15/2011 14:45	6.4508	6.4917	6.3310	6.4245	8.3175	7.9290	8.2689	8.1718	12.2064	11.5038	12.0879	11.9327
3/17/2011 15:10	6.4473	6.4978	6.3392	6.4281	8.3240	7.9338	8.2746	8.1775	12.2281	11.5240	12.1103	11.9541
3/25/2011 15:00	6.4507	6.5011	6.3413	6.4310	8.3299	7.9351	8.2756	8.1802	12.2818	11.5686	12.1627	12.0044
4/1/2011 11:00	6.4386	6.4906	6.3291	6.4194	8.3174	7.9186	8.2606	8.1655	12.2966	11.5749	12.1736	12.0150
4/13/2011 8:30									12.3228	11.5920	12.1983	12.0377
Condition	As Molded Weight (g)											
Density (lb/ft^3)	10				20				30			
Diameter (in.)	1.625				1.625				1.625			
Replicate	1	2	3	Avg.	1	2	3	Avg.	1	2	3	Avg.
Sample Date & Time	AM10MA	AM10MB	AM10MC		AM20MA	AM20MB	AM20MC		AM30MA	AM30MB	AM30MC	
3/3/2011 13:30	6.1267	6.3666	6.0694	6.1876	7.9889	8.1853	8.0657	8.0800	12.0869	12.0413	11.7124	11.9469
3/4/2011 8:50	6.2380	6.4745	6.1718	6.2948	8.0694	8.2797	8.1457	8.1649	12.1471	12.1036	11.7788	12.0098
3/7/2011 13:35	6.3270	6.5714	6.2616	6.3867	8.1812	8.3832	8.2559	8.2734	12.2325	12.1911	11.8706	12.0981
3/8/2011 14:25	6.3343	6.5795	6.2696	6.3945	8.1990	8.3985	8.2735	8.2903	12.2512	12.2099	11.8901	12.1171
3/10/2011 7:45	6.3408	6.5875	6.2768	6.4017	8.2212	8.4164	8.2945	8.3107	12.2774	12.2363	11.9168	12.1435
3/15/2011 14:45	6.3487	6.5983	6.2870	6.4113	8.2567	8.4461	8.3295	8.3441	12.3412	12.3008	11.9811	12.2077
3/17/2011 15:10	6.3544	6.6037	6.2933	6.4171	8.2646	8.4547	8.3383	8.3525	12.3630	12.3221	12.0027	12.2293
3/25/2011 15:00	6.3568	6.6061	6.2961	6.4197	8.2734	8.4635	8.3464	8.3611	12.4219	12.3794	12.0582	12.2865
4/1/2011 11:00	6.3454	6.5949	6.2859	6.4087	8.2589	8.4510	8.3348	8.3482	12.4469	12.4021	12.0798	12.3096
4/13/2011 8:30									12.4879	12.4405	12.1136	12.3473
Condition	As Molded Weight (g)											
Density (lb/ft^3)	10				20				30			
Diameter (in.)	1.375				1.375				1.375			
Replicate	1	2	3	Avg.	1	2	3	Avg.	1	2	3	Avg.
Sample Date & Time	AM10LA	AM10LB	AM10LC		AM20LA	AM20LB	AM20LC		AM30LA	AM30LB	AM30LC	
3/3/2011 13:30	6.1302	6.0977	6.2370	6.1550	8.1064	8.1225	8.1300	8.1196	12.1372	12.0423	12.0431	12.0742
3/4/2011 8:50	6.2312	6.1959	6.3387	6.2553	8.1875	8.1993	8.2141	8.2003	12.1947	12.0995	12.0996	12.1313
3/7/2011 13:35	6.3222	6.2840	6.4291	6.3451	8.2895	8.3025	8.3159	8.3026	12.2737	12.1796	12.1791	12.2108
3/8/2011 14:25	6.3308	6.2932	6.4382	6.3541	8.3063	8.3193	8.3321	8.3192	12.2910	12.1970	12.1962	12.2281
3/10/2011 7:45	6.3380	6.3012	6.4459	6.3617	8.3268	8.3398	8.3516	8.3394	12.3148	12.2209	12.2202	12.2520
3/15/2011 14:45	6.3490	6.3129	6.4571	6.3730	8.3638	8.3765	8.3862	8.3755	12.3750	12.2813	12.2807	12.3123
3/17/2011 15:10	6.3555	6.3192	6.4629	6.3792	8.3738	8.3863	8.3965	8.3855	12.3958	12.3023	12.3016	12.3332
3/25/2011 15:00	6.3577	6.3221	6.4662	6.3820	8.3870	8.4001	8.4084	8.3985	12.4524	12.3595	12.3588	12.3902
4/1/2011 11:00	6.3484	6.3133	6.4554	6.3724	8.3792	8.3931	8.4000	8.3908	12.4802	12.3866	12.3852	12.4173
4/13/2011 8:30									12.5258	12.4316	12.4291	12.4622

Table 12. Raw, as-molded sample weights

Condition	Machined Weight (g)											
Density (lb/ft^3)	10				20				30			
Diameter (in.)	2				2				2			
Replicate	1	2	3	Avg.	1	2	3	Avg.	1	2	3	Avg.
Sample Date & Time	MC10SA	MC10SB	MC10SC		MC20SA	MC20SB	MC20SC		MC30SA	MC30SB	MC30SC	
3/3/2011 13:30	5.6492	5.8143	5.3434	5.6023	7.2425	7.4958	7.5047	7.4143	11.4403	9.9644	11.3655	10.9234
3/4/2011 8:50	5.7797	5.9401	5.4619	5.7272	7.3630	7.6054	7.6230	7.5305	11.5260	10.0558	11.4447	11.0088
3/7/2011 13:35	5.8525	6.0178	5.5367	5.8023	7.4784	7.7242	7.7395	7.6474	11.6364	10.1767	11.5487	11.1206
3/8/2011 14:25	5.8591	6.0256	5.5414	5.8087	7.4907	7.7401	7.7532	7.6613	11.6589	10.1997	11.5709	11.1432
3/10/2011 7:45	5.8626	6.0299	5.5446	5.8124	7.5012	7.7544	7.7649	7.6735	11.6881	10.2278	11.5998	11.1719
3/15/2011 14:45	5.8670	6.0364	5.5490	5.8175	7.5143	7.7729	7.7793	7.6888	11.7530	10.2842	11.6661	11.2344
3/17/2011 15:10	5.8697	6.0404	5.5534	5.8212	7.5186	7.7789	7.7857	7.6944	11.7731	10.2989	11.6872	11.2531
3/25/2011 15:00	5.8708	6.0412	5.5536	5.8219	7.5183	7.7808	7.7847	7.6946	11.8189	10.3224	11.7334	11.2916
4/1/2011 11:00	5.8587	6.028	5.543	5.8099	7.5039	7.7659	7.7706	7.68	11.8271	10.3157	11.745	11.296
4/13/2011 8:30									11.8521	10.3276	11.7703	11.3167
Condition	Machined Weight (g)											
Density (lb/ft^3)	10				20				30			
Diameter (in.)	1.625				1.625				1.625			
Replicate	1	2	3	Avg.	1	2	3	Avg.	1	2	3	Avg.
Sample Date & Time	MC10MA	MC10MB	MC10MC		MC20MA	MC20MB	MC20MC		MC30MA	MC30MB	MC30MC	
3/3/2011 13:30	5.8997	5.7735	5.7267	5.8000	7.5950	7.5195	7.3401	7.4849	11.1799	10.4948	11.3304	11.0017
3/4/2011 8:50	6.0127	5.8833	5.8413	5.9124	7.6916	7.6132	7.4384	7.5811	11.2436	10.5637	11.3936	11.0670
3/7/2011 13:35	6.0996	5.9657	5.9195	5.9949	7.8017	7.7242	7.5483	7.6914	11.3313	10.6585	11.4809	11.1569
3/8/2011 14:25	6.1074	5.9734	5.9255	6.0021	7.8170	7.7407	7.5637	7.7071	11.3506	10.6785	11.5004	11.1765
3/10/2011 7:45	6.1116	5.9784	5.9290	6.0063	7.8318	7.7569	7.5772	7.7220	11.3764	10.7043	11.5255	11.2021
3/15/2011 14:45	6.1191	5.9869	5.9358	6.0139	7.8562	7.7821	7.5983	7.7455	11.4397	10.7647	11.5885	11.2643
3/17/2011 15:10	6.1236	5.9920	5.9404	6.0187	7.8639	7.7896	7.6056	7.7530	11.4607	10.7846	11.6098	11.2850
3/25/2011 15:00	6.1222	5.9907	5.9372	6.0167	7.8666	7.7933	7.6079	7.7559	11.5127	10.8292	11.6642	11.3354
4/1/2011 11:00	6.1098	5.9791	5.9269	6.0053	7.8545	7.7801	7.5935	7.743	11.5329	10.8413	11.6842	11.353
4/13/2011 8:30									11.5648	10.8647	11.7208	11.3834
Condition	Machined Weight (g)											
Density (lb/ft^3)	10				20				30			
Diameter (in.)	1.375				1.375				1.375			
Replicate	1	2	3	Avg.	1	2	3	Avg.	1	2	3	Avg.
Sample Date & Time	MC10LA	MC10LB	MC10LC		MC20LA	MC20LB	MC20LC		MC30LA	MC30LB	MC30LC	
3/3/2011 13:30	5.7129	5.6108	5.8821	5.7353	7.6934	7.7621	7.7614	7.7390	11.7096	11.6369	11.2536	11.5334
3/4/2011 8:50	5.8215	5.7078	5.9922	5.8405	7.7949	7.8468	7.8449	7.8289	11.7663	11.6918	11.3125	11.5902
3/7/2011 13:35	5.8972	5.7891	6.0693	5.9185	7.8968	7.9546	7.9532	7.9349	11.8440	11.7693	11.3965	11.6699
3/8/2011 14:25	5.9045	5.7970	6.0749	5.9255	7.9101	7.9730	7.9711	7.9514	11.8612	11.7861	11.4139	11.6871
3/10/2011 7:45	5.9063	5.8010	6.0772	5.9282	7.9226	7.9907	7.9889	7.9674	11.8839	11.8086	11.4371	11.7099
3/15/2011 14:45	5.9130	5.8100	6.0849	5.9360	7.9468	8.0227	8.0213	7.9969	11.9434	11.8681	11.4980	11.7698
3/17/2011 15:10	5.9187	5.8154	6.0904	5.9415	7.9555	8.0323	8.0313	8.0064	11.9648	11.8895	11.5195	11.7913
3/25/2011 15:00	5.9158	5.8137	6.0860	5.9385	7.9568	8.0385	8.0384	8.0112	12.0207	11.9446	11.5730	11.8461
4/1/2011 11:00	5.9035	5.8023	6.0756	5.9271	7.944	8.0256	8.0255	7.998	12.0464	11.9714	11.5956	11.871
4/13/2011 8:30									12.0900	12.0140	11.6323	11.9121

Table 13. Values of x_o, α, and R^2 from curve fitting

Group	x_o	α (1/hr.)	R^2
1	0.037749	0.045326	0.99289
2	0.034367	0.023060	0.98767
3	0.034658	0.0058697	0.98828
4	0.035790	0.030697	0.99008
5	0.033633	0.013268	0.98918
6	0.032349	0.0043528	0.98388
7	0.035154	0.027419	0.98801
8	0.033264	0.011720	0.98759
9	0.031140	0.0038434	0.98201
10	0.037817	0.045095	0.99425
11	0.036509	0.024461	0.99062
12	0.034613	0.0072076	0.98486
13	0.036166	0.038024	0.99289
14	0.034956	0.017470	0.98862
15	0.033334	0.0049673	0.98420
16	0.034347	0.037863	0.99249
17	0.033946	0.015236	0.98692
18	0.031810	0.0039925	0.98265

Figure 24. Examples of foam test samples

Figure 25. Example of foam test mold

Appendix B

Memo from S. Thornberg

Operated for the U.S. Department of Energy by
Sandia Corporation
Albuquerque, New Mexico
87185-0889

date: January 25, 2011

to: B. Moore

from: S. Thornberg

subject: Desiccant sizing for unit center case

Executive Summary

Given the preliminary estimates of lengths and compressions of butyl o-rings, and a leak rate requirement of 1x10^{-6} atm cc/s helium, the total water ingress from both permeation and leakage, the internal frost point of the center case can be maintained for 30 years using approximately 300 grams of molded desiccant (40% zeolite 3A by weight) to a level of -60°C frost point (~10 ppmv water) or lower. That value results from the assumption of 95% relative humidity environment around the center case. A fast-acting portion of desiccant can be added to bring the initial frost point down quickly during the first year or so.

Background

One of the first measures taken to ensure the reliability and predictable aging of materials is to maintain these materials in a known, benign gaseous atmosphere. Many anomalies found have been the result of moisture, oxygen, or some other harmful gas interacting with materials causing materials failures. To control the moisture, desiccants have been routinely used in the stockpile, and for the unit center case, a desiccant will be used, also.

The source of moisture inside a weapon comes from three sources:

1) the inherent moisture that is adsorbed and absorbed on and in most materials that are assembled into the center case,

2) moisture permeating through all polymer seals (e.g., o-rings) from the outside environment, and

3) leakage directly through a conductance, or opening.

In the following models for permeation and leakage, an external environment of 95% relative humidity at 35°C was used. While it is unlikely that any weapon will be in such a humid environment for extended periods, these conditions can be considered to be a reasonable worst case.

Discussion

Moisture ingress into the center case can be estimated using standard permeation and leak rate equations. In this section, moisture ingress will be examined as a function of leak rate to test if the proposed leak rate criteria of $1x10^{-6}$ atm cc/s for helium is valid.

Permeation

The permeation of moisture through o-rings can be determined using the following equation:

$$C_{permeation} = \frac{4}{\pi} DL(1 - S)^2$$

where the D is the diffusion coefficient of water through the o-ring, L is the length of the o-ring, and S is the fraction squeeze imposed on the o-ring. Table 14 contains a list of the specifications and locations of the o-rings that seal the center case from the outside environment.

Table 14. List of the specifications and locations of the o-rings used

Inner diameter (in)	Cross section diameter (in)	Center axis arc length (in)	Center axis arc length (cm)	Compressed standoff (in)	% Squeeze
1.114	0.070	3.720	9.448	0.052	25.71%
1.114	0.070	3.720	9.448	0.052	25.71%
1.864	0.070	6.076	15.433	0.052	25.71%
2.714	0.202	9.161	23.269	0.162	19.80%
2.714	0.202	9.161	23.269	0.162	19.80%
11.950	0.210	38.202	97.032	0.184	12.38%
1.114	0.070	3.720	9.448	0.052	25.71%

By substituting the information in Table 14 into the previous equation, an estimate of the amount of water that will permeate into the center case can be calculated (Table 15).

Table 15. Contribution of permeation to the total amount of water in the center case

Leak Rate (atm cc/s)	Permeation mass of H2O (g/year)	Permeation mass of H2O (g/30 years)
5E-04	0.030	0.90
1E-05	0.030	0.90
1E-06	0.030	0.90

From the table, it is obvious that the leak rate has no bearing on the moisture permeation since the leakage is a different process than permeation. So, the amount of moisture that permeates into the center case is fixed and will remain constant (given the assumption of external conditions of 95% RH and 35°C).

Leakage

For leakage, the equation describing the moisture conductance into the volume is

$$C_{leakage} = \frac{Q}{Pu - Pd}$$

where Q is the leak rate, and Pu is the upstream pressure, and Pd is the downstream pressure. Note that if the leak rate is in units of atm cc/s and if the differential pressure across the leak is 1 atmosphere, the conductance is numerically the same value as the leak rate. Using the same assumptions as previously (95% RH at 35°C), the amount of moisture that will leak into the center case can be calculated assuming the moisture concentration in the center case is zero (the maximum amount of moisture with that scenario). The results of this calculation are shown in Table 16.

Table 16. Contribution of leakage to the total amount of water in the center case

Leak Rate (atm cc/s)	Leakage mass of H2O (g/year)	Leakage mass of H2O (g/30 years)
5E-04	4.7	142
1E-05	0.094	2.8
1E-06	0.009	0.28

Total H_2O mass flux

Using the previous equations, the total flux of moisture into the center case is found by simply summing the leakage and permeation. Results from these calculations are shown in Table 17.

Table 17. Total amount of water in the center case for permeation and leakage

Leak Rate (atm cc/s)	Mass of H2O (g/year)	Mass of H2O (g/30 years)
5E-04	4.7	142
1E-05	0.12	3.72
1E-06	0.039	1.18

Thus, given the o-rings shown in table 17 and a leak rate of $1x10^{-6}$ atm cc/s, the total mass of water that will enter the center case in 30 years will be 1.18 grams.

Desiccant sizing

Zeolite desiccant

Desiccants are typically used to reduce the moisture content in the gas inside of a volume. Equilibrium is established between the moisture in the gas phase and the moisture in the adsorbed phase in the desiccant. The stronger the binding force for the adsorbed water molecules to the desiccant, the lower the moisture level in the gas. Zeolites have very good moisture binding properties and provide low moisture content in the gas phase. The relationship between the moisture in the gas phase and adsorbed phase is called an isotherm and a plot of the isotherm for zeolite 3A is shown in figure 26 for various temperatures.

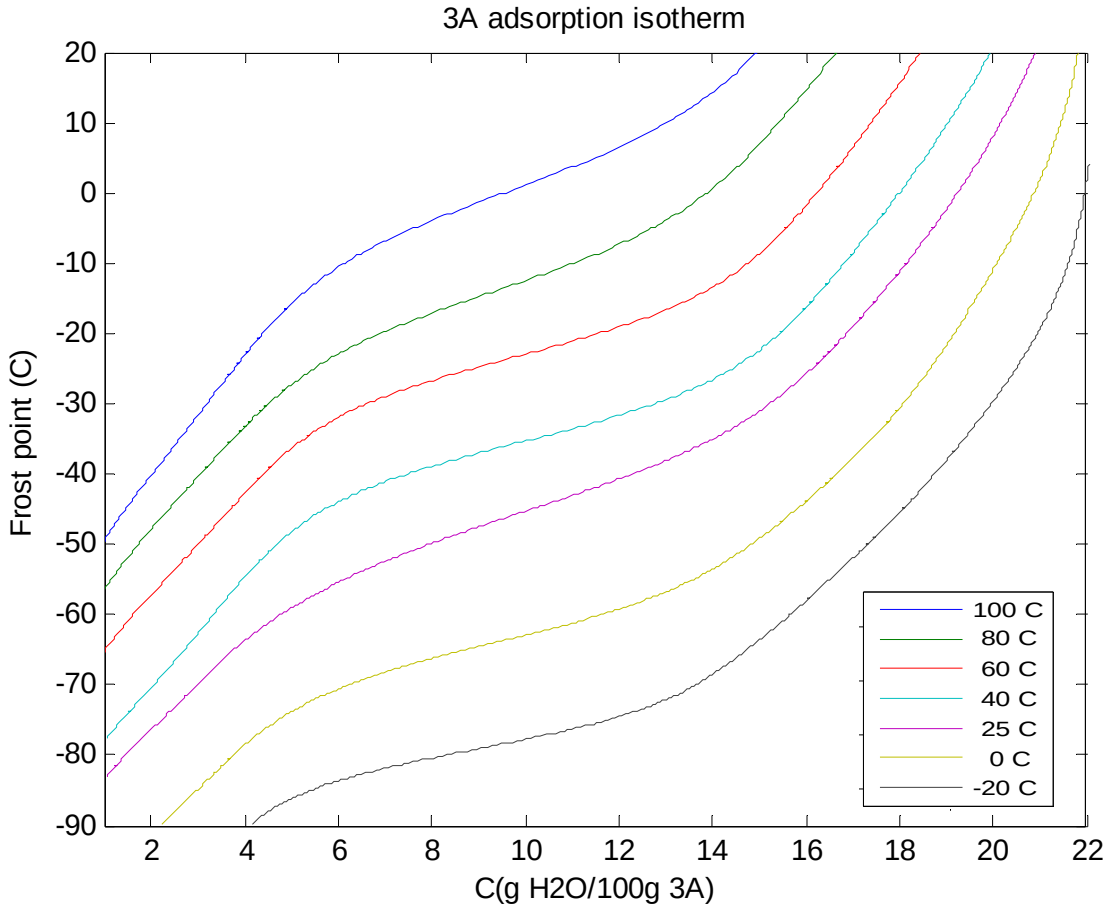

Figure 26. Adsorption isotherms for zeolite 3A

Each line in Figure 1 represents an isotherm at a specific temperature. So, if a moisture level of -60°C frost point (FP) at 25°C is desired, the moisture loading of the desiccant would have to be below about 5 grams of water per 100 grams of 3A molecular sieve (zeolite). In order for this chart to have use in a particular situation, the total mass of water that is expected to enter into the center case must be determined.

Using the loading values for a desiccant temperature of +60°C (a conservative assumption), the maximum loading for the zeolite 3A would be only 2 g H_2O per 100 g 3A in order for the moisture level to stay below -60°C FP. In the table below, the amount of zeolite 3A that is calculated is for 30x2 years, or 60 years, to provide a factor of 2 of margin. The mass of the molded desiccant is calculated by dividing the mass of zeolite 3A by 0.4 since the maximum amount of zeolite 3A that can be put into a molding binder (e.g., silicone, epoxy) is about 40% zeolite/60% binder.

Table 18. Mass of zeolite 3A and molded desiccants

Leak Rate:	5.E-04	atm cc/s	1.E-05	atm cc/s	1.E-06	atm cc/s
Time:	30 years x 2		30 years x 2		30 years x 2	
FP (C)	Mass 3A (g)	Mass molded (g)	Mass 3A (g)	Mass molded (g)	Mass 3A (g)	Mass molded (g)
-20	2182	5455	57	143	18	46
-40	7092	17729	186	465	59	148
-50	9456	23639	248	620	79	197
-60	14183	35459	372	930	118	296

Table 18 shows that a leak rate of $1x10^{-6}$ atm cc/s for helium provides a reasonable size of desiccant of ~300 grams in order to maintain the internal moisture content below -60°C FP.

Fast-acting desiccant

Molded desiccants have a much slower uptake of moisture than pure zeolite powder because the moisture has to diffuse through the binder material in molded desiccants before reaching the zeolite. According to stockpile gas data, most of the internal equilibration of gases is realized in approximately two years. An option has been discussed to provide a more rapid uptake of moisture immediately after assembly so that the two year dry-down can be shortened. In order for a fast acting desiccant to be most effective, it must be installed as part of the last of the assembly operations immediately prior to the center case being sealed. Then the purge and backfill operation is needed to set the initial conditions of the internal gas as dry as possible and to check for any leaks in the seals.

The prime advantage to this approach is the ability to dry down the internal gas of the center case rapidly and keep it dry during the first year or two. An additional advantage is that the fast-acting desiccant can count towards the mass of zeolite 3A that is required, so the molded desiccant size can be reduced. Challenges include the requirement for the case to be sealed quickly after the fast-acting desiccant is installed, the need to find the internal volume in which to place it, additional hardware for installing and holding the desiccant, and the need to replace the desiccant if assembly operations take longer than the time window allowed.

Summary

The proposed desiccant sizing of ~300 grams of molded desiccant is meant to keep the center case moisture level to less than -60°C FP. The goal is to keep the internal atmosphere dry in order to keep deleterious processes (e.g., corrosion, oxidation, polymer degradation) from occurring. The size estimate is conservative in the sense that the modeled external environment is moist (95% RH at 35°C), and that the time period modeled is 60 years. A fast-acting desiccant can be implemented to reduce the moisture level during the first 2 years.

Memo from S. Thornberg, Attachment

Matlab routine used to model water permeation through o-rings and moisture ingress through a leak.

```
% oringperm_keenanmodel.m
% o-ring permeation modeling
%
% based on Keenan's Matlab script
clear
C_leakarray = [1 .1 .01 .001 5e-4]; % Leak conductance in cm^3/sec
C_leakarray = [5e-4 1E-5 5E-6]; % Leak conductance in cm^3/sec
%C_leak = 0;  %Leak conductance in cm^3/sec (set to zero for only permeation)
%
Permeability = 3.8e-8 * 76; % Permeability of EPDM in cm^2/sec;
oring_material='EPDM';
%
Permeability = 1.8e-9 * 76; % Permeability of butyl in cm^2/sec;
oring_material='Butyl';
%
Squeeze = 0.1238;    % O-ring squeeze
Length = 149.6;   % Total O-ring length (cm)
Length = 97.032;    % Total O-ring length (cm)
C_perm = (4/pi)*Permeability*Length*(1-Squeeze)^2; %Permeation conductance
%
T0 = 273.16;
T = 35 + T0;    % Temperature, K
P = 40.28;   % External water vapor pressure, Torr, ~95% RH at 35 C
P0 = 0;     % Internal WVP (Torr)
%
R = 62363;   % Gas Constant (cm^3 torr)/(mole K)
M = 18;     % Molecular weight of water, g/mole
sec_per_year = 3600*24*365;
%
for i=1:length(C_leakarray)
  C_leak=C_leakarray(i);
  Fleak = (C_leak)*(P-P0) *M/(R*T0) * sec_per_year;
  Fperm = (C_perm)*(P-P0) *M/(R*T0) * sec_per_year;
  disp(['_____________________________________________________'])
  disp(['Leakage conductance = ' num2str(C_leak,'%.2e') ' cm^3/sec'])
  disp(['Mass flow (leakage) = ' num2str(Fleak) ' g/yr'])
  disp(['------'])
  disp(['O-ring material: ' oring_material])
  disp(['O-ring squeeze: ' num2str(Squeeze)])
  disp(['O-ring length: ' num2str(Length) ' cm, ' num2str(Length/2.54) ' in. '])
  disp(['Permeation conductance = ' num2str(C_perm,'%.2e') ' cm^3/sec'])
  disp(['Mass flow (permeation) = ' num2str(Fperm) ' g/yr'])
  disp(['------'])
  disp(['Total mass flow = ' num2str(Fperm+Fleak) ' g/yr'])
end
```

Appendix C

Effect of Material Processing on Desiccant Activation Time

Table 19. Raw data from sample drying

	Sample Wt. (g)														
Mold Group	1			2			3			4			5		
Replicate	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
Sample Date & Time															
5/19/2011 10:30	31.8633	31.7458	32.0162	31.9418	31.9024	31.6815	31.9444	31.7372	31.9295	32.0019	31.9516	31.7488	32.0217	31.8877	31.8267
5/20/2011 13:00	31.8092	31.6917	31.9631	31.8887	31.8490	31.6295	31.8887	31.6825	31.8746	31.9480	31.8964	31.6944	31.9639	31.8290	31.7674
5/23/2011 11:30	31.7724	31.6552	31.9263	31.8530	31.8128	31.5939	31.8525	31.6468	31.8387	31.9139	31.8614	31.6596	31.9296	31.7940	31.7325
5/24/2011 8:30	31.7656	31.6485	31.9196	31.8463	31.8061	31.5873	31.8459	31.6404	31.8320	31.9080	31.8548	31.6532	31.9228	31.7871	31.7259
6/2/2011 14:30	31.7458	31.6292	31.9001	31.8271	31.7864	31.5684	31.8265	31.6218	31.8135	31.8901	31.8364	31.6349	31.9050	31.7681	31.7074
6/3/2011 14:45	31.7437	31.6271	31.8978	31.8247	31.7840	31.5662	31.8241	31.6195	31.8112	31.8880	31.8341	31.6325	31.9025	31.7655	31.7045
6/6/2011 9:00	31.7374	31.6212	31.8919	31.8188	31.7782	31.5600	31.8183	31.6137	31.8054	31.8818	31.8287	31.6271	31.8965	31.7590	31.6874
6/9/2011 13:30	31.7311	31.6152	31.8858	31.8129	31.7723	31.5542	31.8126	31.6080	31.7997	31.8758	31.8226	31.6214	31.8907	31.7532	31.6914
6/17/2011 13:00	31.7184	31.6032	31.8737	31.8010	31.7604	31.5424	31.8010	31.5966	31.7876	31.8636	31.8105	31.6098	31.8793	31.7414	31.6788

Appendix D

Evaluation of RTV as Source of Material Weight Loss

MEMO

To: J. Knight

From: L. Watts, C. Dowdy

CC: E. Kheyfets, J. Schicker

Date: August 1, 2011

Re: Headspace Analysis of RTV samples

Work Order 203 was submitted to the Analytical Sciences Laboratory to analyze the headspace of provided samples to qualitatively determine what was outgassing. There were three types of samples:

Group 1: RTV 615 (components A&B), 3 Angstrom Zeolite Molecular Sieve, and DEB Getter
Group 2: RTV 615 (components A&B) and 3 Angstrom Zeolite Molecular Sieve
Group 3: RTV 615 (components A&B)

Each of these samples was provided 4 times for a total of 12 samples. The following are the compounds detected in the headspace of each bottle.

1st Sampling	Group 1	Group 2	Group 3
Trimethyl Silane		x	x
Trimethylsily Fluoride			x
Tetramethyl Silane		x	x
Ethanol			x
Acetone	x		x
IPA	x		x
Trichlorotrifluoroethane	x	x	
Trimethyl Silanol	x		x
Pentamethyl Disiloxane		x	
Benzene	x	x	
Hexamethyl Disiloxane	x	x	x
Heptane	x	x	
Toluene	x	x	x
Hexamethyl Cyclotrisiloxane	x		
Ethylbenzene	x	x	x
1,3-Dimethyl Benzene	x	x	
1,2-Dimethyl Benzene	x	x	
1-Methylethyl Bezene	x		
Octamethyl Cyclotetrasiloxane	x	x	x

The second sampling found fewer materials in the headspace and at much lower quantities. See table 2.

Table 2

2nd Sampling	Group 1	Group 2	Group 3
Trimethyl Silane		x	x
Tetramethyl Silane		x	x
Trichlorotrifluoroethane	x	x	x
Benzene	x		
Hexamethyl Disiloxane	x		
Toluene	x		

The third and fourth samplings found trace amounts of the following compounds listed in Table 3 and Table 4.

Table 3

3rd Sampling	Group 1	Group 2	Group 3
Trimethyl Silane		x	x
Trimethylsilyl Fluride			x
Tetramethyl Silane		x	
Trichlorotrifluoroethane			x
IPA	x		

Table 4

4th Sampling	Group 1	Group 2	Group 3
Trimethyl Silane		x	x
Trimethylsilyl Fluride			x

The IPA found in the testing could be from cleaning the bottles before use for this experiment.

Table 20 Average Normalized Weight Loss for Different Material Formulations

RTV & Filler		RTV Only (9:1)		RTV Only (10:1)	
Time (hrs.)	Avg. Normalized Wt.	Time (hrs.)	Avg. Normalized Wt.	Time (hrs.)	Avg. Normalized Wt.
0.00	1.0000	0.00	1.0000	0.00	1.0000
26.50	0.998313	5.18	0.998146	28.25	0.99632
97.00	0.997162	23.75	0.996579	52.25	0.99551
118.00	0.996951	47.92	0.995720	123.50	0.99415
340.00	0.996338	120.50	0.994339	194.50	0.99366
364.25	0.996270	165.25	0.993857	315.25	0.99291
430.50	0.996080	188.50	0.993652	549.00	0.99240
507.00	0.995888	292.00	0.993538		
		388.33	0.992996		
		484.58	0.992699		
		556.83	0.992601		
		645.08	0.992490		
		840.33	0.992274		
		868.58	0.992424		

Appendix E

Evaluation of Getter Effectivity Post Oven Bake

	Sample 1				
	Unit	System Volume	Ballast Volume	Reactor Volume	Ratio
P_I (initial rxn vessel pressure, torr)$=P_B \times R_E$	1	2587.7	2097.3	490.5	0.8105
Capacity (gas uptake, cc-atm/g material = $[(P_I-P_F) \times V_T]/(760 \times W_S)$	2	2632.6	2135.6	497	0.8112
Capacity-organic (capacity/g organic) = capacity/w	3	2606.2	2118.9	487.3	0.813
2 hours					
File name = RTV615+DEB_Sample 2					
		Unit 1	Unit 3		
Time (hours)					
W_S (Weight of sample)		4.5141	4.5179		
P_B (ballast pressure, torr)		649	645		
R_E (expansion ratio)		0.8105	0.813		
P_I (Initial rxn vessel pressure, (torr)		526	524.4		
P_F (Final test gas pressure, torr)		514.8	515.8		
V_T (Volume for test system, cc)		2587.7	2606.2		
Capacity (gas uptake, cc-atm/g material)		8.5	6.5		
Average Capacity		7.5			
Std dev Capacity		1.4			
%RSD Capacity		18.9			
Organic content (weight fraction, w)		0.0375	0.0375		
Capacity-organic (Capacity cc-atm/g organic, C/w)		226.7	173.3		
Average Capacity-organic		200			
Std dev Capacity-organic		37.7			
%RSD Capacity, organic		18.9			
% Total capacity = (C org/352)*100		**64.4**	**49.2**		
Average % Total Capacity		**56.8**			
StdDev % Total Capacity		**10.7**			
16 hours					
File name = RTV615+DEB_Sample 2					
		Unit 1	Unit 3		
Time (hours)					
W_S (Weight of sample)		4.5141	4.5179		
P_B (ballast pressure, torr)		649	645		
R_E (expansion ratio)		0.8105	0.813		
P_I (Initial rxn vessel pressure, (torr)		526	524.4		
P_F (Final test gas pressure, torr)		513	514		
V_T (Volume for test system, cc)		2587.7	2606.2		
Capacity (gas uptake, cc-atm/g material)		9.8	7.9		
Average Capacity		8.9			
Std dev Capacity		1.3			
%RSD Capacity		15.2			
Organic content (weight fraction, w)		0.0375	0.0375		
Capacity-organic (Capacity cc-atm/g organic, C/w)		261.3	210.7		
Average Capacity-organic		236			
Std dev Capacity-organic		35.8			
%RSD Capacity, organic		15.2			
% Total capacity = (C org/352)*100		**74.2**	**59.8**		
Average % Total Capacity		**67**			
StdDev % Total Capacity		**10.2**			

	Sample 2				
	Unit	System Volume	Ballast Volume	Reactor Volume	Ratio
P_I (initial rxn vessel pressure, torr)$_=P_B$ x R_E	1	2587.7	2097.3	490.5	0.8105
Capacity (gas uptake, cc-atm/g material = $[(P_I-P_F) \times V_T]/(760 \times W_S)$	2	2632.6	2135.6	497	0.8112
Capacity-organic (capacity/g organic) = capacity/w	3	2606.2	2118.9	487.3	0.713
2 hours					
File name = RTV615+DEB_Sample 2					
		Unit 1	Unit 3		
Time (hours)					
W_S (Weight of sample)		4.5141	4.5179		
P_B (ballast pressure, torr)					
R_E (expansion ratio)					
P_I (Initial rxn vessel pressure, (torr)					
P_F (Final test gas pressure, torr)					
V_T (Volume for test system, cc)					
Capacity (gas uptake, cc-atm/g material)					
Average Capacity					
Std dev Capacity					
%RSD Capacity					
Organic content (weight fraction, w)					
Capacity-organic (Capacity cc-atm/g organic, C/w)					
Average Capacity-organic					
Std dev Capacity-organic					
%RSD Capacity, organic					
% Total capacity = (C org/352)*100					
Average % Total Capacity					
StdDev % Total Capacity					
16 hours					
File name = RTV615+DEB_Sample 2					
		Unit 1	Unit 3		
Time (hours)					
W_S (Weight of sample)		4.5141	4.5179		
P_B (ballast pressure, torr)					
R_E (expansion ratio)					
P_I (Initial rxn vessel pressure, (torr)					
P_F (Final test gas pressure, torr)					
V_T (Volume for test system, cc)					
Capacity (gas uptake, cc-atm/g material)					
Average Capacity					
Std dev Capacity					
%RSD Capacity					
Organic content (weight fraction, w)					
Capacity-organic (Capacity cc-atm/g organic, C/w)					
Average Capacity-organic					
Std dev Capacity-organic					
%RSD Capacity, organic					

Appendix E

Proof of Concept Molding to Current Design Definition

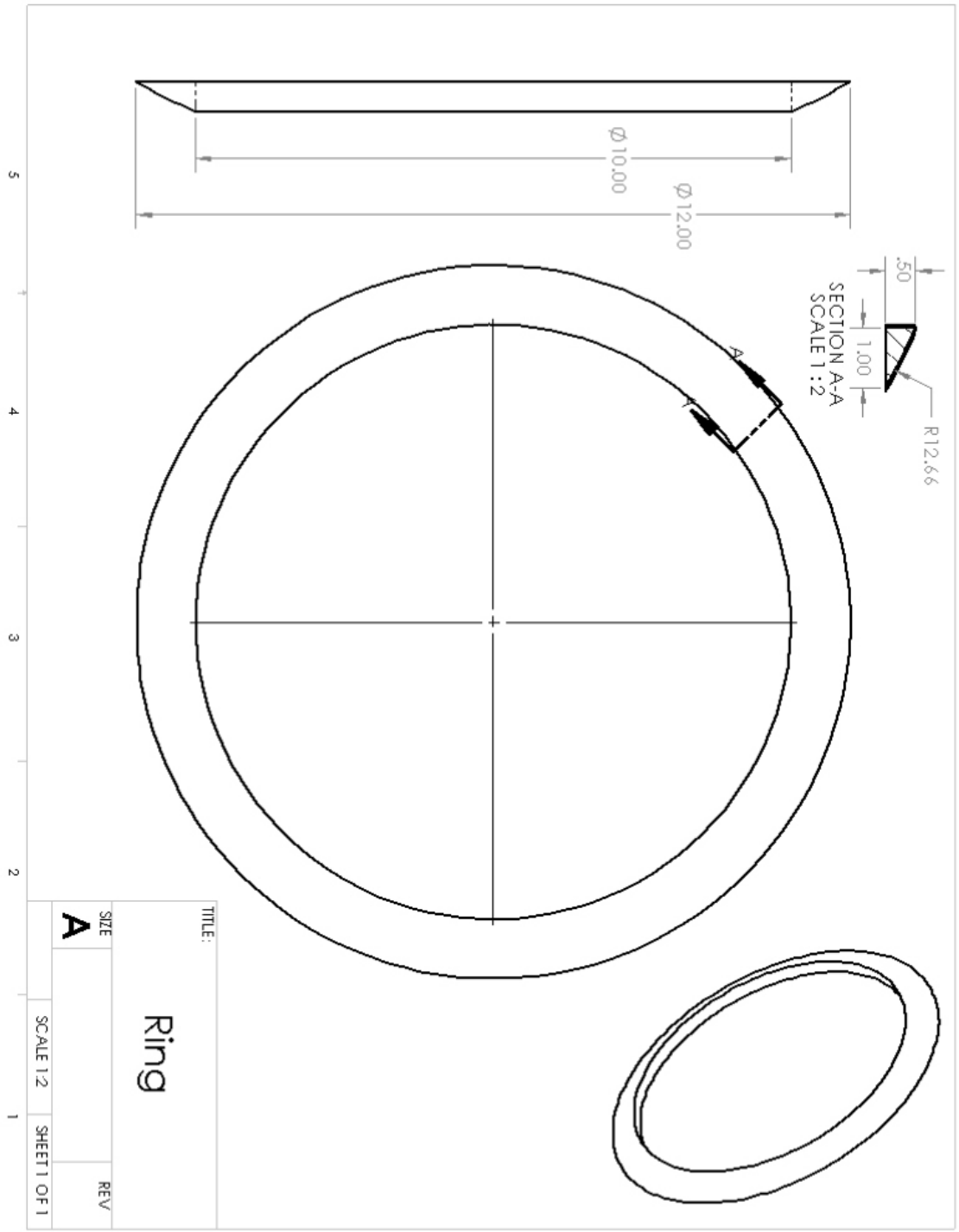

Figure 27. New desiccant and getter ring component

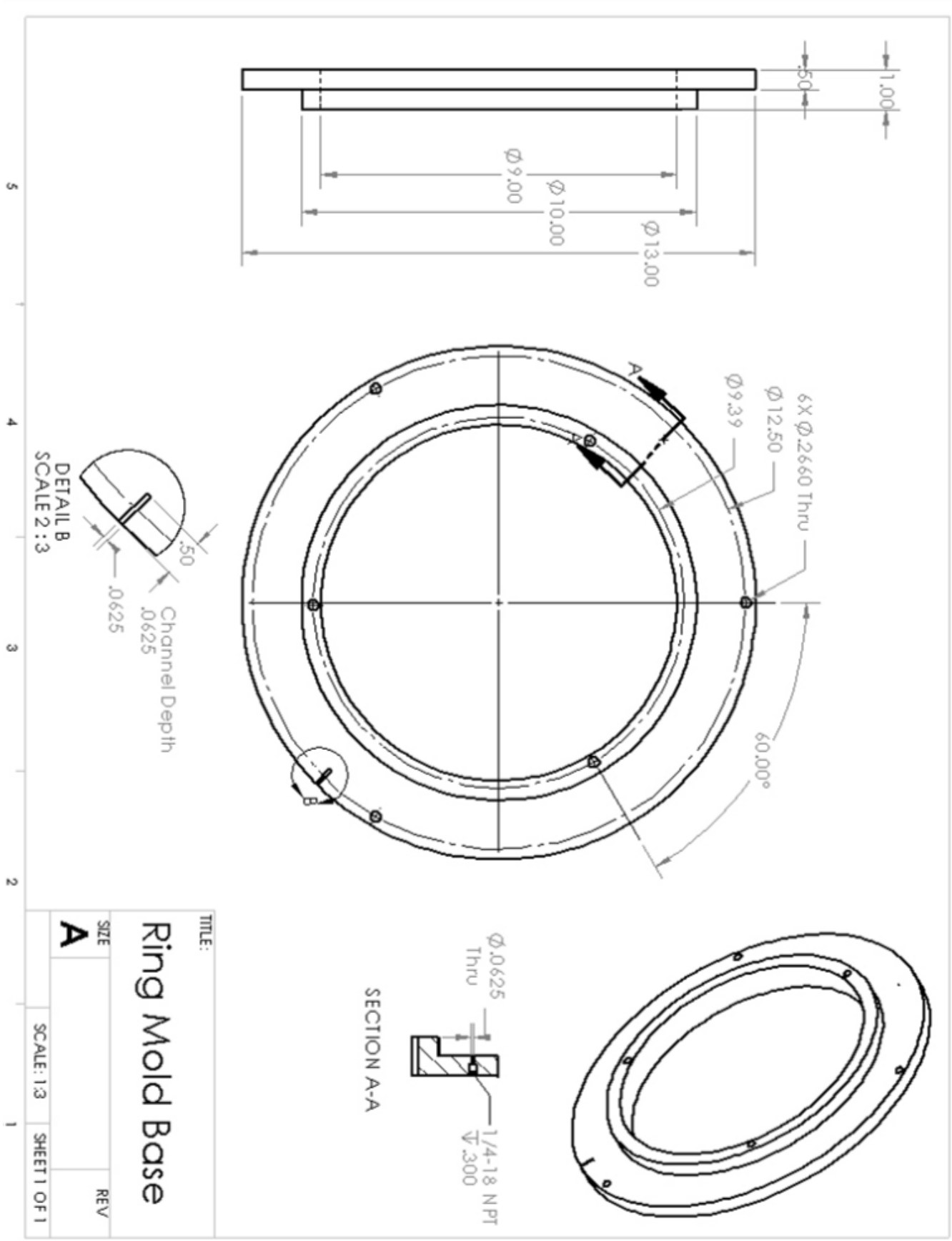

Figure 28. Mold base for stereolithographed ring mold

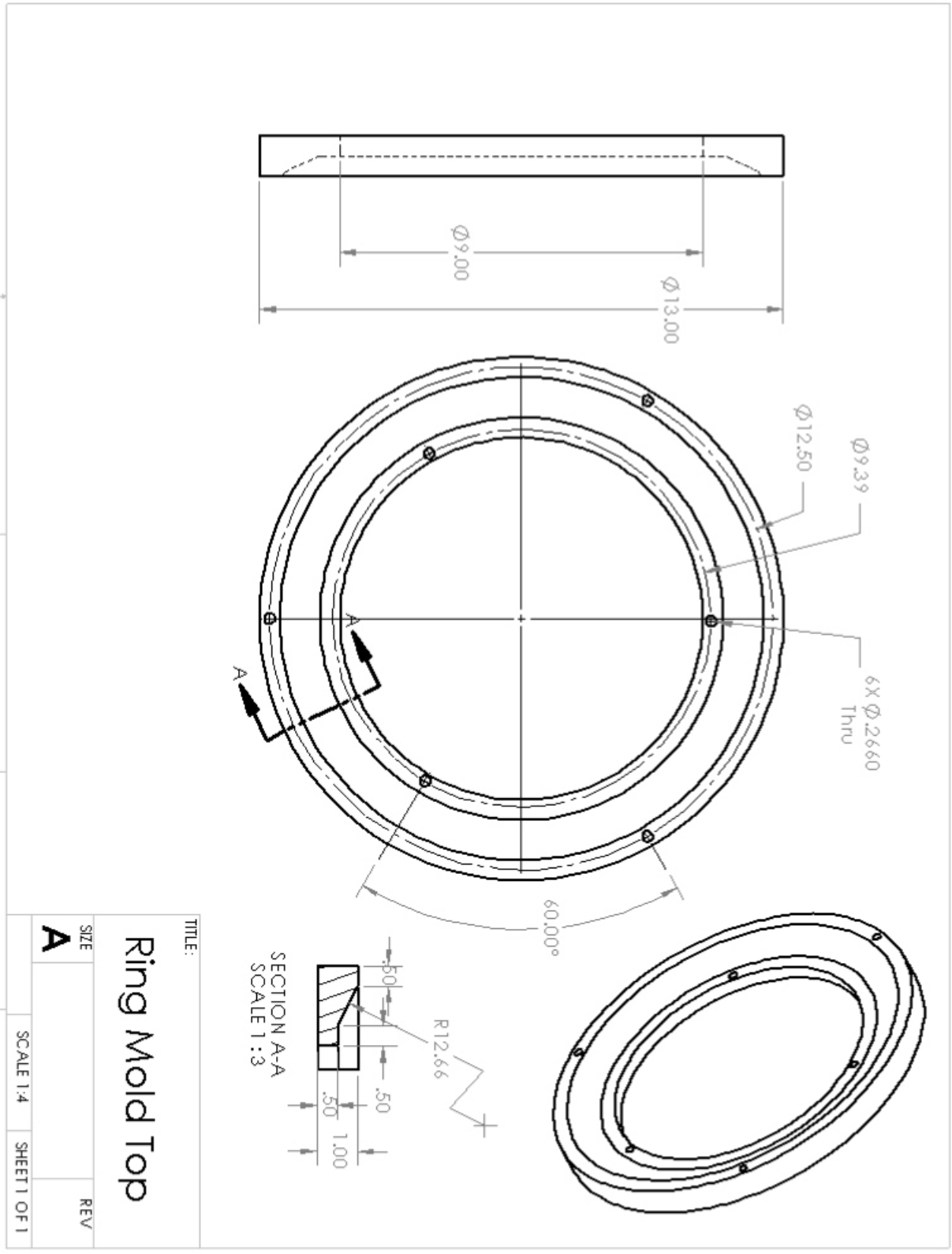

Figure 29. Mold top for stereolithographed ring mold

Appendix F

Impact of Surface Area to Volume Ratio on Moisture Uptake Rate

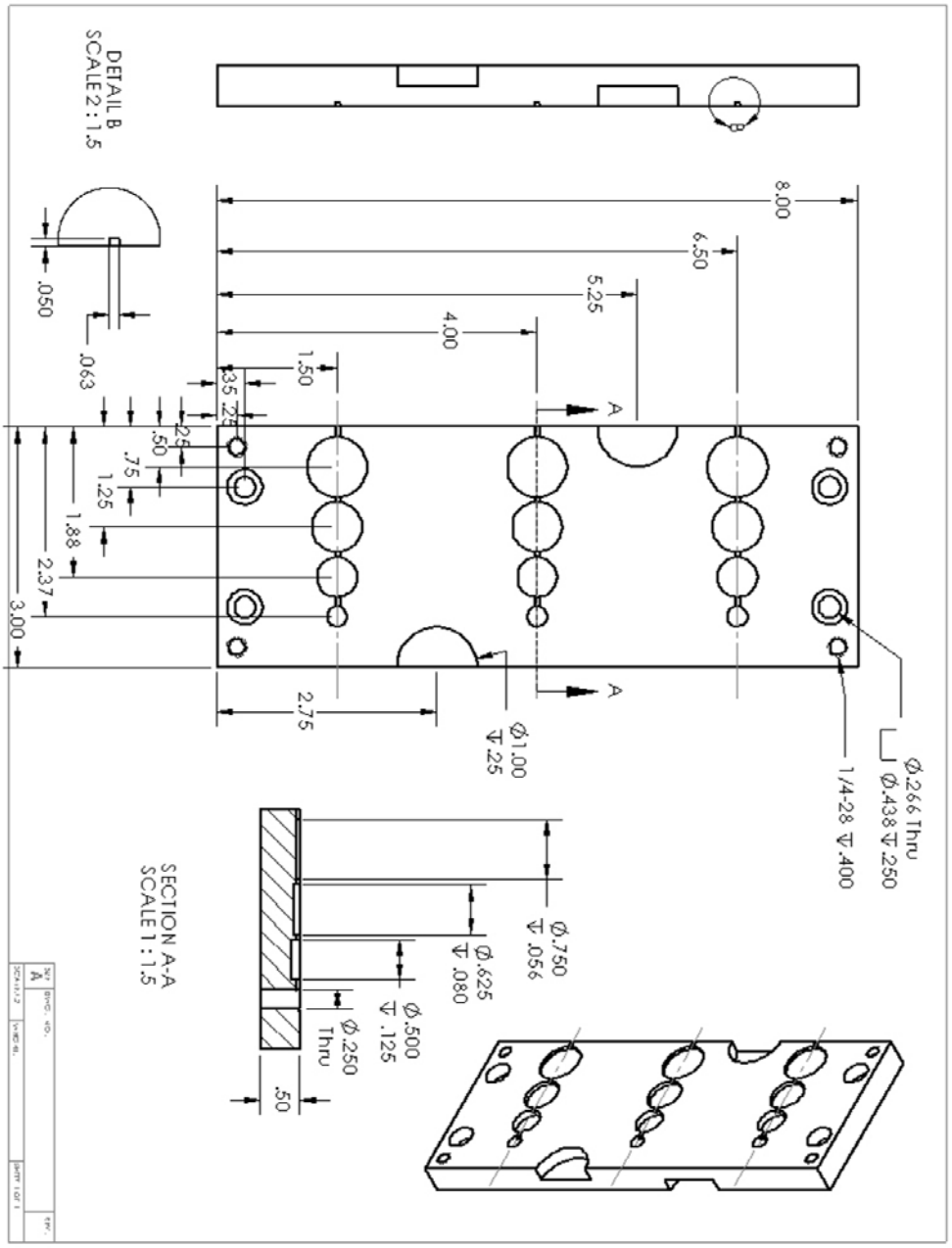

Figure 30. Test mold insert for surface area to volume experiment

Table 21. Raw weight data from uptake analysis

	Sample Weights (g)											
	SA/V Ratio 20.0 (1/in.)			SA/V Ratio 24.0 (1/in.)			SA/V Ratio 31.4 (1/in.)			SA/V Ratio 41.3 (1/in.)		
Replicate	1	2	3	1	2	3	1	2	3	1	2	3
Sample Date & Time												
8/17/2011 7:30	0.4903	0.4948	0.4985	0.5153	0.5115	0.5143	0.5235	0.5135	0.5219	0.5402	0.5292	0.5550
8/17/2011 14:15	0.5052	0.5104	0.5130	0.5327	0.5284	0.5317	0.5449	0.5367	0.5441	0.5678	0.5593	0.5860
8/18/2011 11:15	0.5202	0.5261	0.5290	0.5506	0.5470	0.5505	0.5690	0.5608	0.5683	0.5965	0.5853	0.6144
8/19/2011 14:30	0.5294	0.5357	0.5385	0.5619	0.5586	0.5621	0.5778	0.5679	0.5777	0.5971	0.5856	0.6149
8/23/2011 14:45	0.5405	0.5477	0.5506	0.5688	0.5653	0.5694	0.5782	0.5681	0.5779	0.5973	0.5857	0.6148
8/26/2011 15:00	0.5405	0.5478	0.5509	0.5684	0.5650	0.5693	0.5776	0.5678	0.5778	0.5968	0.5853	0.6147