Sharpsville Container Corporation Stainless Steel Cylinder Stability White Paper

Purpose

The objective of the current study is to determine the stability of low-level gas mixtures of reactive gaseous materials when packaged in the new Sharpsville Container Corporation stainless steel disposable cylinder fitted with C-10 outlet connections. The number of cylinders used in the study is not meant to demonstrate consistency (cylinder to cylinder) but to provide an overall indication of material stability in the presence of reactive gas components. Additionally, data from an external study using aluminum cylinders is shown as a comparison to the Sharpsville stainless cylinder. There may be differing cylinder drying and passivation techniques proprietary to potential users of this product. Here, the preparation methods for both the stainless steel and aluminum cylinders were exactly the same. This was done to demonstrate stability in a well-defined and normalized comparison of the two cylinders.

Cylinder Preparation

One significant advantage in using stainless steel cylinders over other materials is the reduction in preparation and treatment time required. While other gas cylinders require longer passivation times (in many instances timeframes on the order of weeks), the stainless-steel cylinders used in the present study were passivated for 48 hours using the higher-level mixture of the target component. A follow-on study is planned to determine whether the passivation time can be further decreased by either increased preparation temperatures or gas concentrations.

The C-10 valve selected by Sharpsville Container Corporation uses an external pressure relief mechanism in lieu of a soldered pressure relief valve. This allows the stainless-steel package units to be heated to a significantly higher temperature than units using a soldered pressure relief valve. At this point, the maximum temperature that can be applied approaches 400^{o} F, which is the failure point of the Viton O-rings. The ultimate preparation temperature will be determined during a subsequent test regimen.

All stainless-steel cylinders used in this study were rinsed with acetone, dried thoroughly with heated air prior and, inspected for residual hydrocarbons prior to the installation of the outlet valve into the cylinder. After the cylinders were valved and leak-checked using 1000-1200 psig for duration of ~5 minutes, they were cycled through a heat/evacuation cycle in a cylinder treatment oven. While monitoring the vacuum level of the oven manifold systems (including the cylinders undergoing treatment), the temperature of the cylinders was controlled between 150^{o} – 200^{o} Fahrenheit until the vacuum indicated a value less than 50 microns. The end of the heating/evacuation treatment cycle was determined by the ultimate vacuum achieved at temperature. Therefore, more than one cycle of heating and evacuation may have been required depending on the bake-out characteristics of the specific cylinders in the run.

Prior to filling the cylinders with the reactive standard mixtures used to monitor stability, the cylinders were exposed to a pre-fill treatment gas mixture of the component to be packaged at a higher concentration. Here, the pre-fill treatment mixture was ~2.5 times the concentration of the final desired standard concentration. For a 10-ppm mixture of hydrogen sulfide in nitrogen, the pre-fill gas treatment consisted of a 25-ppm mixture of hydrogen sulfide in nitrogen. The pre-fill treatment gas was left in the cylinders as a passivation cycle for approximately 48 hours. Following the pre-fill treatment, the cylinders were vented, purged, and evacuated prior to final fill.

Testing Protocol

The reactive components used in this study are compounds commonly used for calibration or "bump-test" gas monitors. Four gas mixtures were selected for the initial testing regimen and included Hydrogen Sulfide, Nitrogen Dioxide, Ammonia and Chlorine. Nominal Test Value concentrations were 10-ppm. Fill pressures were ~1000 psi for the stainless units and ~500 psi for the aluminum cylinders. The cylinders were tested for concentration at 3 days, 1 week, 2 weeks, 4 weeks, 2 months, and 3 months post-fill using a range of instruments calibrated at each time of use. The cylinder performance results are shown below. Across all four test protocols, the results indicate similar or superior stability to aluminum packaging. In three out of four tests, the stainless-steel cylinder package outperformed the aluminum package. The analytical accuracy is also indicated on each chart for reference purposes.

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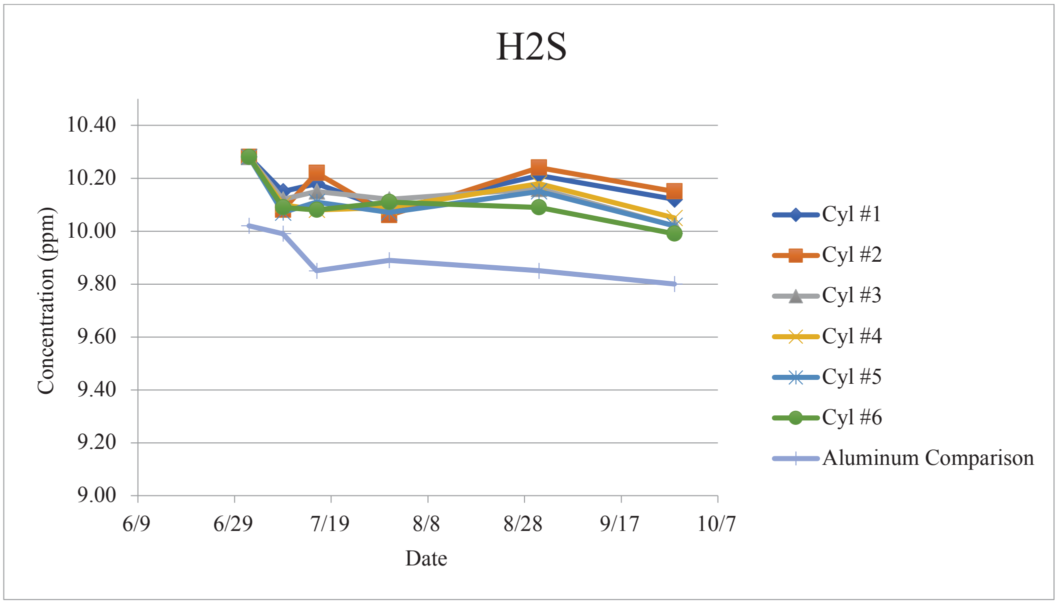
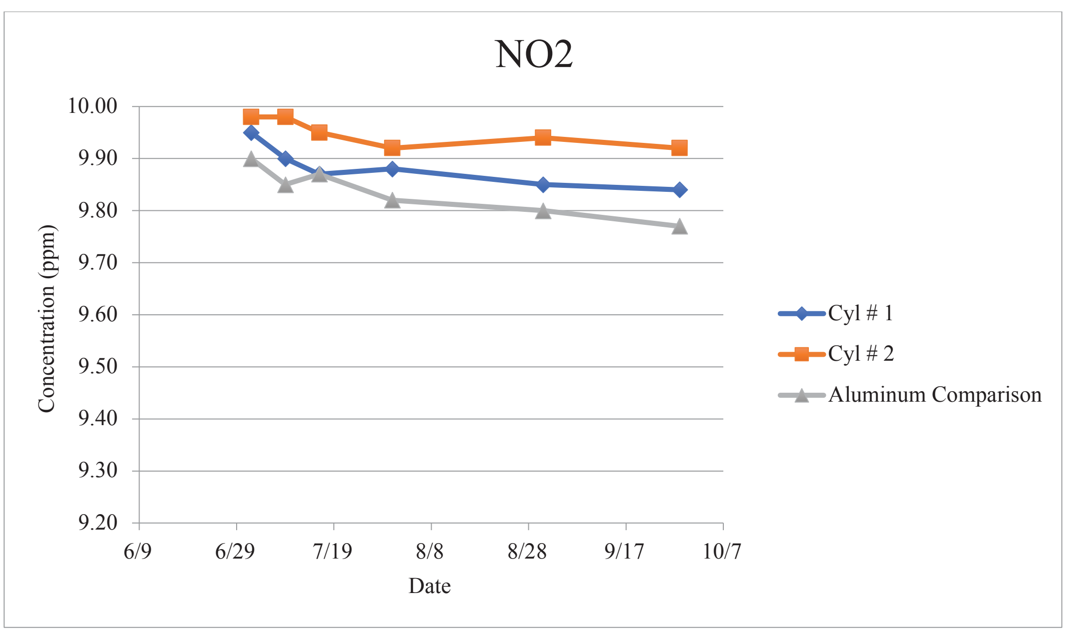
Hydrogen Sulfide Test Results

Age	Cyl # 1	Cyl # 2	Cyl # 3	Cyl # 4	Cyl # 5	Cyl # 6	Al Cyl
2 days	10.15	10.18	10.08	10.21	10.12	9.95	10.02
1 week	10.08	10.22	10.06	10.24	10.15	9.88	9.99
2 weeks	10.12	10.15	10.12	10.16	10.02	9.85	9.85
1 month	10.10	10.08	10.09	10.18	10.05	9.66	9.89
2 months	10.07	10.11	10.07	10.15	10.02	9.51	9.85
3 months	10.09	10.08	10.11	10.09	9.99	9.39	9.80
change	-1.85%	-1.95%	-1.65%	-1.85%	-2.82%	-8.66%	-4.67%



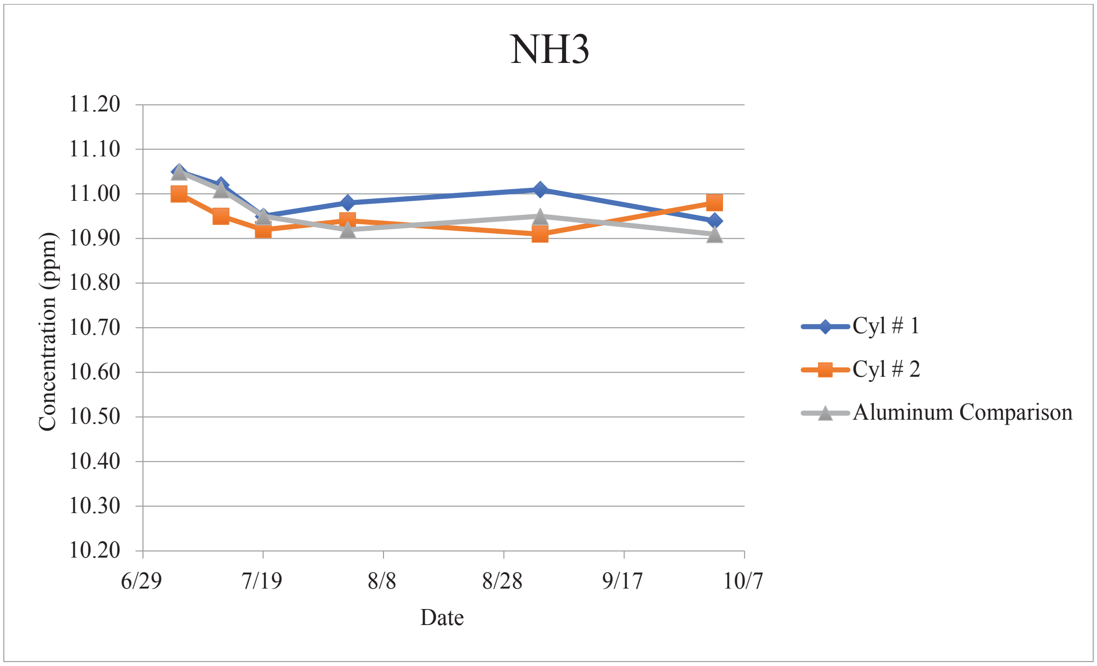
Nitrogen Dioxide Test Results

Age	Cyl # 1	Cyl # 2	Al Cyl
2 days	9.95	9.98	9.90
1 week	9.90	9.98	9.85
2 weeks	9.87	9.95	9.87
1 month	9.88	9.92	9.82
2 months	9.85	9.94	9.80
3 months	9.84	9.92	9.77
change	-1.99%	-1.20%	-2.69%



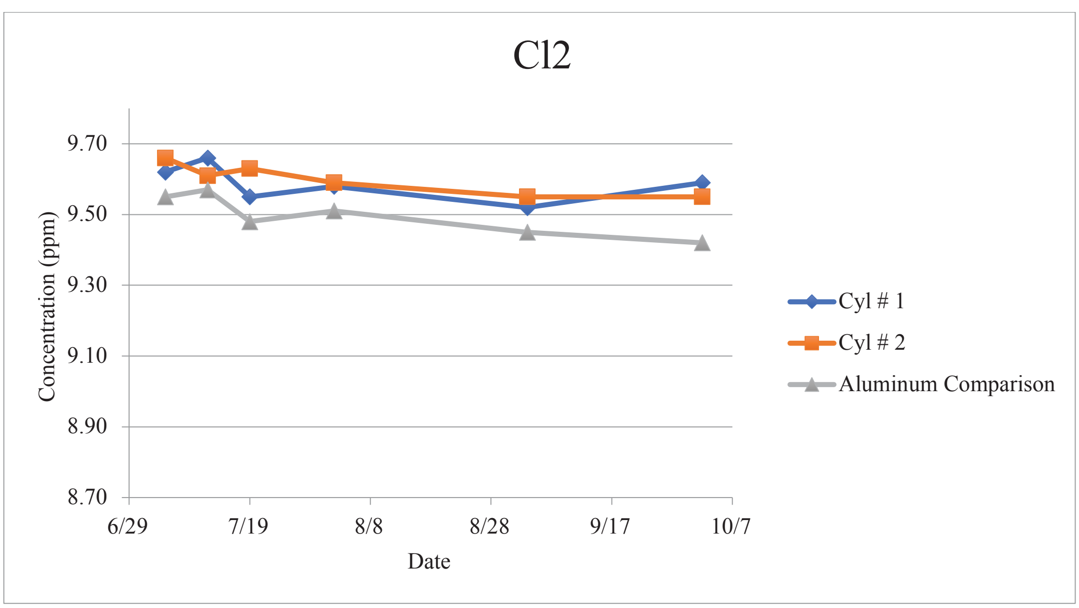
Ammonia Test Results

Age	Cyl # 1	Cyl # 2	Al Cyl
2 days	11.05	11.00	11.05
1 week	11.02	10.95	11.01
2 weeks	10.95	10.92	10.95
1 month	10.98	10.94	10.92
2 months	11.01	10.91	10.95
3 months	10.94	10.98	10.91
change	-1.88%	-1.52%	-2.15%



Chlorine Test Results

Age	Cyl # 1	Cyl # 2	Al Cyl
2 days	9.62	9.66	9.55
1 week	9.66	9.61	9.57
2 weeks	9.55	9.63	9.48
1 month	9.58	9.59	9.51
2 months	9.52	9.55	9.45
3 months	9.59	9.55	9.42
change	-1.54%	-1.95%	-3.29%



Conclusions

Based on average degradation values of the stainless steel and aluminum cylinders, the following can be extrapolated:

- Hydrogen Sulfide - Stainless Steel Cylinders outperformed the Aluminum unit by 56.6%(excluding Cylinder #6)
- Chlorine - Stainless Steel Cylinders outperformed the Aluminum unit by 46.9%
- Nitrogen Dioxide - Stainless Steel Cylinders outperformed the Aluminum unit by 40.7%
- Ammonia - Stainless Steel Cylinders outperformed the Aluminum unit by 21%

The test results indicate that the stainless-steel cylinder package developed by Sharpsville Container Corporation achieves increased maximum allowable operating pressures (MAOP) of ~1000-psig v. ~500-psig for aluminum cylinders, reduced preparation cycle time and similar to greater stability for reactive component gas standards.

With typical burst pressures of 4000-psi, the MAOP of these units may be increased to maximum allowable DOT standards. The method of construction allows internal volumes to be customized to meet specific customer criteria. The cause of the variation indicated on Cylinder #6 in the Hydrogen Sulfide testing may be explained by interstitial moisture, which had an adverse effect on the gaseous stability in this particular unit.

It is therefore considered an outlier for the purposes of this study. Data recording will be conducted on a regular schedule until such time that each cylinder is empty or until the gaseous concentrations degrade below minimum acceptable commercial standards. The next phase of the current work will include more aggressive materials used in specialized applications, increased preparation temperatures and increased passivation gas concentrations.

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