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Air Replacement Testing for the 9975 Primary Containment Vessel with Food Pack and Hex Can Configurations

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Air Replacement Testing for the 9975 Primary Containment Vessel (PCV) with Food Pack and Hex Can Configurations

Introduction

The 9975 Shipping Package is authorized to ship plutonium and uranium oxide materials per the Safety Analysis Report for Packaging[1] (SARP). The safety analysis and authorization require the PCV to contain an atmosphere > 75% carbon dioxide to prevent a flammable gas mixture for certain oxide contents. The substitution of carbon dioxide for most of the air in the PCV was proposed as a method for increasing detonation cell size, thus preventing the detonation reaction between hydrogen and oxygen.[2]

Previous work showed that for materials packaged in 3013 shipping cans and placed within the PCV, the PCV could be successfully inerted by replacement of more than 75% of the air with carbon dioxide.[3] This work demonstrates that the PCV can be inerted with food pack or hex cans permitted by the SARP and achieve the 75% carbon dioxide atmosphere.[4] As before, successful inerting requires that more than 75% of the air originally present in the PCV vapor space be replaced with carbon dioxide. The packaging configurations used for the PCV tests are shown in Figures 1 and 2.

Four different interior can configurations were tested in this study.

- Configuration 1, the Hex-Can configuration, shown in Figures 2 and 5, consisted of (from the bottom up) the PCV bottom (honeycomb) spacer, the PCV sleeve, a hex can spacer, the solid model hex can (cut out of solid plastic to have the same dimensions as a hex can), a second hex can spacer, and a tube spacer. The hex can is a hexagonal cylinder 4.25” in height and 4.25” at the widest diameter of the top.
- Configuration 2, the LANL configuration, is shown in Figures 1 and 6, and consisted of the PCV bottom spacer, the PCV sleeve, a sealed food-pack type product can, and a vented spacer can (dimensions 404x700). The can dimensions are given in inches, with the first digit representing inches, and the next two digits representing sixteenths of inches. For example, a 404x700 can has a diameter of 4.25 in. and a height of 7.00 in. The sealed product can dimensions were 401x509. This configuration was tested both with and without 32 grams of compressed aluminum foil the nominal amount that filled the void space. Up to 200 grams of Al foil is permitted per the SARP.[1]
- Configuration 3 (SRS-A) consisted of the PCV bottom spacer, the PCV sleeve, and two stacked product cans, size 404x700. This configuration was performed on food pack cans supplied by LLNL.
- Configuration 4 (SRS-B) consisted of the PCV bottom spacer, the PCV sleeve, and three stacked product cans, size 404x414. This three can arrangement

resulted in less than 1" axial void space between the top of the uppermost can and the vessel seal area. See figure 7. This configuration was judged by the Design Agency and Design Authority to be the most restrictive arrangement of food pack cans authorized per SARP Appendix 1.2 and bounds the inerting protocol for the other cans listed.

Discussion

The objective of these experiments was to demonstrate the capability to replace greater than 75% of the air with carbon dioxide in a PCV containing any of four sets of contents. The testing was designed to assure any of the food pack can configurations listed in the SARP Appendix 1.2 could be inerted with the established protocol and satisfy the >75 % carbon dioxide atmosphere requirement. The method tested for replacing the air was simple displacement by introducing carbon dioxide into the PCV using an insert tube. The experiments were conducted under an approved Task Technical and Quality Assurance Plan.[5]

Dilution and displacement facilitate replacement of the air by carbon dioxide. Displacement is enhanced by the density difference between carbon dioxide and air. At 25 °C, air has a density of 1.18 g/L and carbon dioxide has a density of 1.80 g/L; therefore, the density of carbon dioxide is 1.53 times greater than air. The gap between PCV and its contents is too small to allow a purge wand to be inserted to the bottom of the PCV. As a result, displacement is a critical mechanism for replacing air with carbon dioxide.

Modifications Made to the PCV

An existing PCV (used previously for testing, WSRC-TR-2001-00304) was used for these experiments. To allow for the removal of gas samples from a sealed PCV, modifications were made to the PCV. Three ¼"-National Pipe Thread (NPT) holes were drilled and tapped in the side of the PCV. The top hole was drilled as close to the top as possible without interfering with the top weld. Similarly, the bottom hole was drilled as close to the bottom as possible without interfering with the bottom weld. The middle hole was situated approximately midway between the top and bottom. Threaded fittings were secured in the holes and equipped with airtight septa to permit the removal of gas samples. An 8"x 8" temporary cover was fabricated from plexiglass with a ¾" diameter hole drilled in the center to place the wand into and permit exhaust of excess gas flow.

Experimental Work

The inerting test procedure was based upon the procedure given in S-SARP-G-0003, Rev. 0 "Safety Analysis Report for Packaging Model 9975, Appendix 7.2" PCV CO_2 Dilution Procedure for Plutonium and/or Uranium Oxide Content Envelope C.4 in the 9975 Package Primary Containment Vessel. A list of the procedure steps follows.

1. Connect a purge wand (⅛-inch diameter metal tube of sufficient length) to a CO_2 supply. Place the purge wand within the top spacer cavity.
2. Using the finger holes, lift the top spacer and position the tip of the wand under the bottom edge of the spacer and against the PCV wall.
3. Cover the open PCV with a temporary purge lid to minimize mixing of ambient air with the PCV atmosphere. The temporary purge lid should:
    a. Cover the opening, but not create a seal.
    b. Be easy to remove.
    c. Be of a shape and size that will not agitate (induce mixing of) the air at the PCV opening when removed.
4. Set the CO_2 purge rate at 10-16 scfh and purge for 12 minutes.
5. Reduce the CO_2 flow to 3 to 5 scfh indicated flow for 3 more minutes of purging (to prevent turbulence in the vessel when removing the wand), then, slowly remove the wand.
    a. **NOTE**: *Minimize movement of the PCV as the cone-seal closure is installed.*
6. Within 5 minutes of completing the purging, slowly remove purge lid and lower the cone-seal closure assembly into position.
7. Follow the PCV closure procedure in Section 7.2.2.

The above procedure was modified to create the following test procedure:

- The PCV and its contents were configured for the test.
- A temporary cover, in this case an 8" x 8" plexiglass cover with centered 3/4"diameter hole for the wand, was placed on top of the PCV to minimize mixing with air from the atmosphere while allowing gas to escape the PCV.
- The purge wand was placed in open space above the uppermost can in the PCV. In test configuration 2, the wand was inserted through the top vent hole and inside the vented spacer can.
- Carbon dioxide was injected into the PCV at 10 cfh for 12 minutes
- Carbon dioxide flow was reduced to 3 scfh for an additional 3 minutes.
- The purge wand was carefully removed from the PCV, then flow was discontinued.
- The temporary cover was then replaced with the cap assembly by sliding off the temporary cover while simultaneously sliding on the cap assembly. The cap assembly was screwed down and secured.
- When the cap was secured, the time was recorded. The PCV was allowed to sit undisturbed for a period of hours (usually overnight) to allow the air/CO_2 mixture to diffuse throughout the PCV vapor space.
- Gas samples were then withdrawn from all three sample ports starting at the top and finishing at the bottom.
- The test apparatus was disassembled and tipped on its side for a few hours to allow the air/CO_2 mixture to be replaced with air.
- The gas samples were submitted for O_2 and N_2 analysis via gas chromatography.

The test procedure differs from that given in the SARP. Due to the different contents tested, the test procedure did not require that top spacer be lifted and the tip of the wand positioned under the bottom edge of the spacer and against the PCV wall as is required in step 2 of the SARP procedure. In addition, the CO_2 flow rate was conservatively set at the low end of the range allowed by steps 4 and 5 of the SARP procedure. The low flow rate is conservative, as higher flow rates have been shown to displace more air.[3]

The flow meter used in these tests to measure the carbon dioxide flow rate was a King Instrument Co, Model 75303222CO9, which had been calibrated in accordance with Savannah River Site procedures.

The CO_2 supply tank was certified to have 99.0% purity by Air Liquide America Specialty Gases LLC, specification sheet attached in Appendix 1.

Results

A summary of the data from the experiments is shown in Table 1. Problems were encountered during the analysis of several samples. These problems included a broken tip on a gas sampling syringe, gas sampling syringes that apparently developed slight leaks, and a "sealed" product can that apparently was not completely sealed. The complete set of data for all the experiments is shown in Table 2 at the end of the report, including the data that were rejected as a result of the various problems encountered. The air concentration was calculated by adding the measured oxygen and nitrogen concentrations and dividing by 0.99 to account for the argon present in air.

A one-sided, upper confidence level on the mean air concentration after inerting is calculated for Configurations 1, 2, and 4 from

$$\mu \leq \overline{X} + \frac{t_{\alpha,n-1}S}{\sqrt{n}}$$

where, μ = the mean air concentration after inerting
X = the calculated average of the data
$t_{\alpha,n-1}$ = the upper α percentage point of a t distribution with n-1 degrees of freedom
S = the calculated standard deviation of the data
n = the number of data points.

The averages and standard deviations were calculated using the air concentrations from Table 1. The air concentrations that were used in the statistical analysis are the average of the air concentrations from the three samples taken from the top, middle and bottom of the PCV.

Table 1. PCV Inerting Test Data Summary

Test	Content Configuration	Air Concentration Average (vol %)	Air Replacement (Carbon dioxide Concentration) Average (vol %)
1	1	6.6	93.4
2	1	8.9	91.1
3	1	10.9	89.1
4	2	25.2*	74.8*
5	2	2.9	97.1
6	2	3.0	97.0
7	2	3.2	96.8
8	3	7.2	92.8
9	4	31.6**	68.4**
10	4	17.1	82.9
11	4	13.4	86.6

*Data not used due to air leakage from a poorly sealed product can.
** Data not used due to air leak.

The data show that for Configuration 1 (hex can) the average volume percent of air remaining after inerting was 9.1% with a standard deviation of 3.5%. Since the balance of gas is essentially all carbon dioxide, tests for Configuration 1 produced an average carbon dioxide concentration of 91%. This shows that the spacers above and below the hex can do not prevent the displacement of air by carbon dioxide and that Configuration 1 can be successfully inerted using the suggested procedure. The upper 95% confidence level on the mean air concentration for Configuration 1 after inerting is 12.7%.

The first experiment run with Configuration 2 showed a much higher than expected concentration of air remained after inerting. Further investigation showed that the product can that was part of the Configuration 2 contents had been poorly sealed, allowing air to diffuse into the PCV vapor space after the PCV had been inerted and closed and before the vapor space was sampled. As shown in Figure 6, the product can was sealed with two layers of Parafilm and a layer thick black tape. This adequately sealed the product can, and subsequent experiments were run with the taped product can. After the product can had been taped, the average volume percent of air remaining after inerting was 3.1%, with a standard deviation of 1.1%, for Configuration 2, giving an average carbon dioxide concentration of 97%. The upper 95% confidence level on the mean air concentration for after inerting is 3.3%. The wand is placed inside the vented spacer can, which allows the successful inerting of PCV vapor space with very little air remaining.

Only one experimental run was made with Configuration 3 as it is very similar to Configuration 4. The contents of both of these configurations fill most of the space inside the PCV. These configurations were expected to have the highest fraction of air present after inerting, since some air will enter the PCV when the purge lid is removed and the cone-seal closure is installed. Since the two cans that make up Configuration 3 are the same diameter but with a lower total height than the three cans used in Configuration 4, it was expected that Configuration 4 would have a higher air concentration after inerting. The one experiment conducted with Configuration 3 had 7.2 volume % of air remaining after inerting, or a carbon dioxide concentration of 93%.

The first experimental run made with Configuration 4 had a much higher remaining air concentration of 31.6 volume % than was expected. The PCV and sampling system were inspected and no obvious leaks were found. Two of the three sampling septa were replaced and the o-rings on the sampling fittings were inspected and found to have no apparent defects. The sampling fittings with the o-rings were carefully tightened and the experiment was repeated. The experimental run led to an air concentration after inerting of 17.1 volume %. The sampling septa were again replaced and another experimental run was made. It was discovered before sampling the last experiment that the tips of the sample syringes were slightly loose. The PCV vapor space was sampled using the newly-tightened syringes and the air concentration of the third and last experimental run was found to be 13.4 volume %. Based on the lower air concentrations found in the last two experimental runs made with Configuration 4, it was concluded that the high air concentration in the first run was due to leaking septa and/or leaking syringes. The data from the first run were therefore not used in the analysis. The average volume percent of air remaining after inerting was 15.3% for Configuration 4, with a standard deviation of 2.6%, giving an average carbon dioxide concentration of 85%. The upper 90% confidence level on the mean air concentration for Configuration 4 after inerting is 21.0%. This high value for the upper 90% confidence level on the mean air concentration is due to the high uncertainty that results from only having two data points for the average air concentration.

Conclusions

The replacement of air with carbon dioxide in the PCV can be reliably performed using gas injection through a purge wand. The data show average carbon dioxide concentrations of at least 85% for all tested PCV contents, and that there is a high confidence that air replacement in excess of 75%, as required by the SARP, can be consistently achieved in the PCV. The air replacement was demonstrated using a procedure and equipment that are similar to those that will be used on a production basis. The experimental results for the amount of air remaining in the PCV are biased high due to leakage of air into the test syringes. Even with this conservative bias, the test results show that 85% or more of the air is replaced.

References

[1] Safety Analysis Report for Packaging, Model 9975-85, WSRC-SA-2002-00008, Revision 0, December 2003 and Model 9975-96, S-SARP-G-00003, Revision 0, January 2008.

[2] S. J. Hensel, "Combustion Analysis of Flammable Gas Mixtures in the 9975 Package," Calculation Note M-CLC-F-00499, July 1999.

[3] R. A. Pierce and N. M. Askew, "Air Replacement Testing for the 9975 Container Vessel," Technical Report WSRC-TR-2001-00304 Rev. 2, August 25, 2004.

[4] N- TTR-G-00007, 9975 PCV CO_2 Inerting Test for Food Pack and Hex Can Configurations, June 29, 2009.

[5] Task Technical and Quality Assurance Plan: TT & QA Plan SRNS-RP-2009-01027, Inerting of a 9975 PCV with Specific Content Cans.

Figure 1. Typical Single Food-Pack Can Configuration with Perforated Spacer Can.

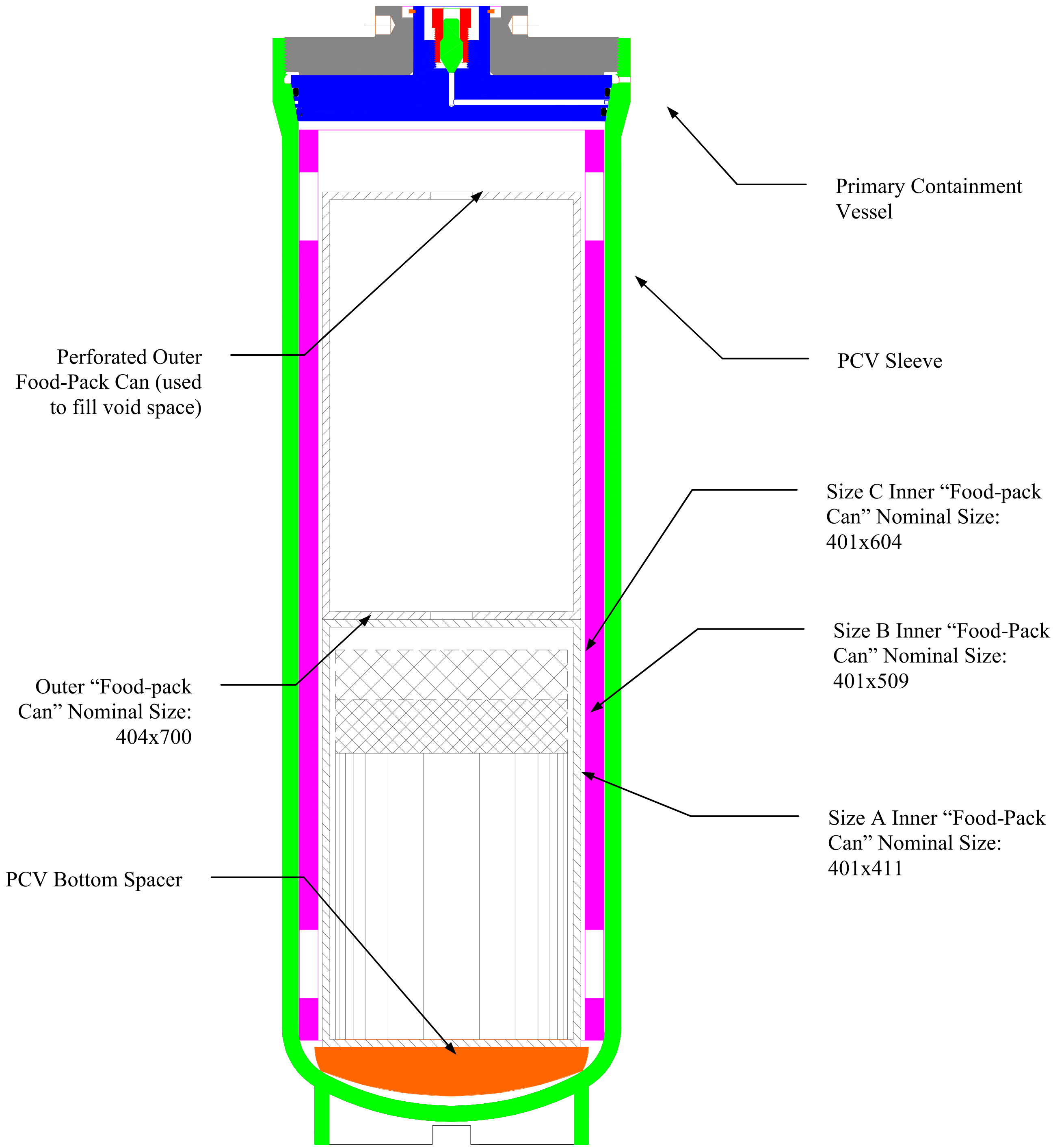

Figure 2. LLNL Hex-Can Configuration (Illustration is representative of LLNL Drawing AAA02-108850).

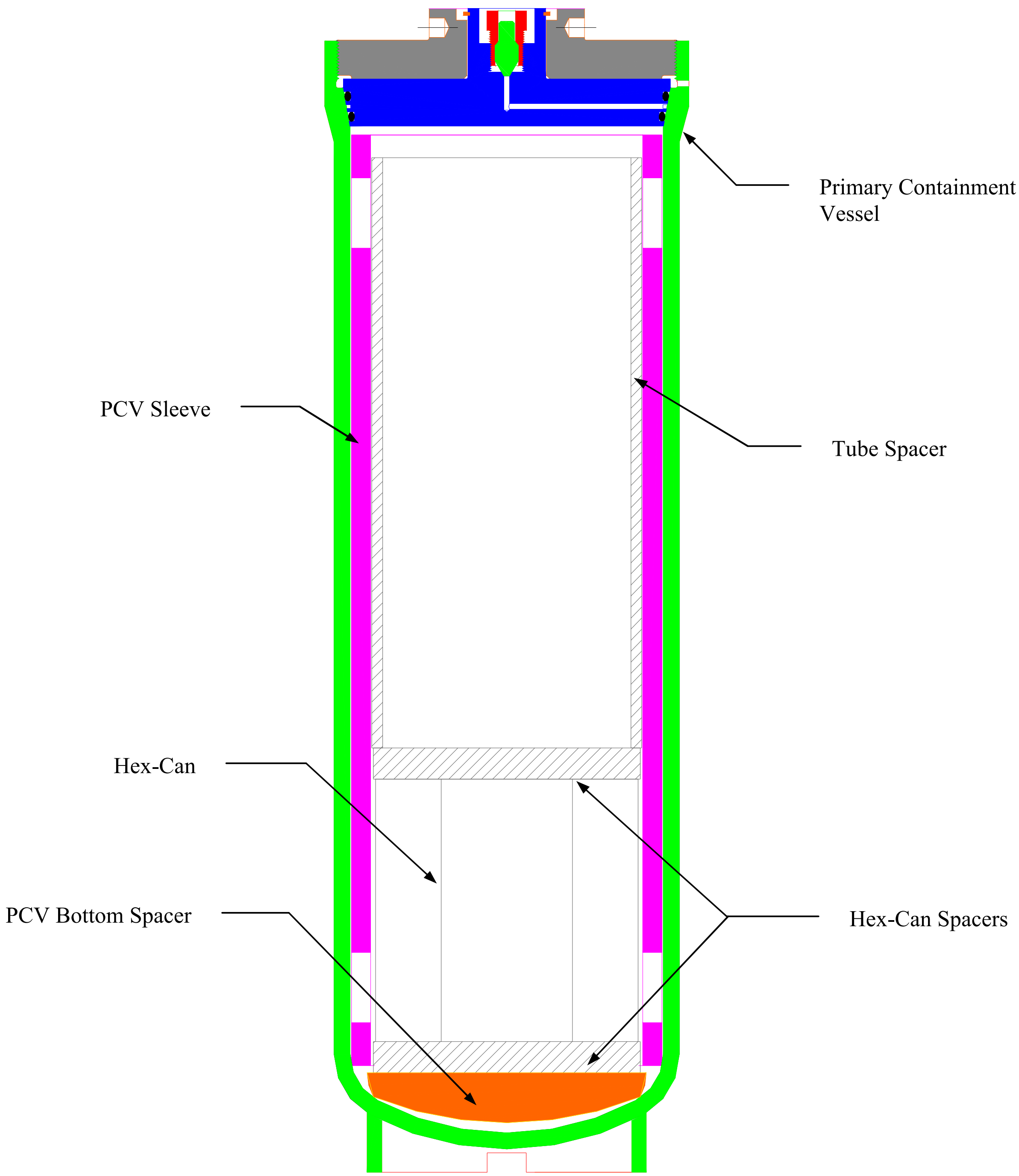

Figure 3. Inerting configuration: CO_2 supply, connected to flowmeter, PCV with acrylic cover plate, PCV lid (on table).

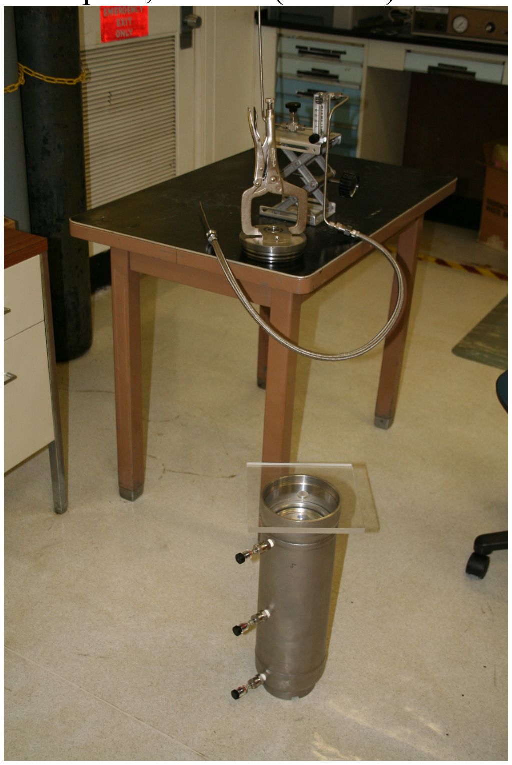

Figure 4. Top view of PCVwith SRNL Configuration 1.

Figure 5. Components of hex can configuration for testing: PCV sleeve (left), tube spacer (center) and a solid hex can model made of plastic stacked with top and bottom acrylic hex can spacers (right).

Figure 6. Components of single food pack can configuration for testing: PCV spacer sleeve (left), empty perforated spacer can with foil discs (center), and sealed food pack can (right)

Figure 7. PCV axial void space above the most restrictive three food pack can configuration.

Table 2. Complete PCV Inerting Test Data

Test #	Test Description	Configuration	Position	O2 (vol%)	N2 (vol%)	N2/O2	Air (vol%)
1	Hex can 1	1	Top	12	48	4.00	60.61[1]
			Middle	1.4	5.7	4.07	7.17
			Bottom	1.2	4.8	4.00	6.06
2	Hex can 2	1	Top	1.6	6.8	4.25	8.48
			Middle	2.3	9.4	4.09	11.82
			Bottom	1.2	5.1	4.25	6.36
3	Hex can 3	1	Top	1.6	6.3	3.94	7.98
			Middle	1.9	6.4	3.37	8.38
			Bottom	3.3	13	3.94	16.46
Configuration 1 (Hex can) Air vol% Average							9.09
4	LANL 1	2	Top	4.9	20	4.08	25.15[2]
			Middle	4.9	20	4.08	25.15[2]
			Bottom	4.9	20	4.08	25.15[2]
5	LANL 2	2	Top	0.35	1.7	4.86	2.07
			Middle	0.45	2.1	4.67	2.58
			Bottom	0.83	3.3	3.98	4.17
6	LANL 3	2	Top	0.31	1.5	4.84	1.83
			Middle	0.7	3.2	4.57	3.94
			Bottom	0.62	2.7	4.35	3.35
7	LANL 4	2	Top	0.38	1.9	5.00	2.30
			Middle	0.39	1.9	4.87	2.31
			Bottom	0.97	4.1	4.23	5.12
Configuration 2 (LANL) Air vol% Average (LANL 1 not included)							3.08
8	SRS A 1	3	Top	1.2	5.1	4.25	6.36
			Middle	1.6	6.8	4.25	8.48
			Bottom	1.3	5.5	4.23	6.87
Configuration 3 (SRS A) Air vol% Average							7.24
9	SRS B 1	4	Top	7.1	28	3.94	35.45[3]
			Middle	5	20	4.00	25.25[3]
			Bottom	6.8	27	3.97	34.14[3]
10	SRS B 2	4	Top	3.7	15	4.05	18.89
			Middle	3.1	13	4.19	16.26
			Bottom	3.1	13	4.19	16.26
11	SRS B 3	4	Top	2.7	11	4.07	13.84
			Middle	2.7	11	4.07	13.84
			Bottom	2.4	10	4.17	12.53
Configuration 4 (SRS B) Air vol% Average SRS B 1 not included)							15.27

[1] Test 1 Top sample was not used in calculations due to air contamination from syringe failure.
[2] Test 4 samples were not used due to air contamination from product can.
[3] Test 9 samples were not used due to air leak.

Appendix 1. Scan of CO_2 Specification Sheet.

AIR LIQUIDE

CERTIFICATES FOR INDUSTRIAL GASES

2B-ALPCYL-OPS-0035-F
Rev.0
Effective Date: 07/31/2008

SPECIFICATION SHEET

Carbon Dioxide Industrial (gas)

Major Component	Specification
Carbon Dioxide Industrial (gas)	99.0 %

Typical Properties	
Moisture	<32 ppm
Oxygen	<50 ppm
Total Hydrocarbons	<50 ppm

Air Liquide America Specialty Gases LLC

1311 New Savannah Rd. Augusta, GA 30901-3843
Phone: 706-724-8725

WESTINGHOUSE SAVANNAH RIVER COMPANY
SAVANNAH RIVER TECHNOLOGY CENTER

Air Replacement Testing for the 9975 Primary Containment Vessel with Food Pack and Hex Can Configurations (U)

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