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Elaborated Odor Test for Extended Exposure

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Concerns were raised when incidental exposure to a proprietary bonding material revealed the material had an irritating odor. The NASA-STD-6001B document describes a supplemental test method option for programs to evaluate materials with odor concerns (Test 6, *Odor Assessment*). In addition to the supplemental standard odor assessment with less than 10 seconds of exposure, the NASA White Sands Test Facility (WSTF) Materials Flight Acceptance Testing section was requested to perform an odor test with an extended duration to evaluate effects of an extended exposure and to more closely simulate realistic exposure scenarios. With approval from the NASA Johnson Space Center Industrial Hygienist, WSTF developed a 15-minute odor test method. WSTF performed this extended-duration odor test to evaluate the odor and physical effects of the bonding material configured between two aluminum plates, after the safety of the gas was verified via toxicity analysis per NASA-STD-6001B Test 7, *Determination of Offgassed Products*. During extended-duration testing, odor panel members were arranged near the test material in a small room with the air handlers and doors closed to minimize dilution. The odor panel members wafted gas toward themselves and recorded their individual assessments of odor and physical effects at various intervals during the 15-minute exposure and posttest. A posttest interview was conducted to obtain further information. Testing was effective in providing data for comparison and selection of an optimal offgassing and odor containment configuration. The developed test method for extended exposure is proposed as a useful tool for further evaluating materials with identified odors of concern if continued use of the material is anticipated.

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Nomenclature

ACGIH = American Conference of Governmental Industrial Hygienists
CIH = Certified Industrial Hygienist
GC-FID = Gas Chromatography Flame Ionization Detection
GC-MSD = Gas Chromatography Mass Spectrometry Detection
JSC = NASA Johnson Space Center
MPH = Master of Public Health
NASA = National Aeronautics and Space Administration
OSHA = Occupational Safety and Health Administration
RTV = Room Temperature Vulcanization
STEL = Short-term Exposure Limit
WSTF = White Sands Test Facility

I. Introduction

THE NASA White Sands Test Facility (WSTF) Materials Flight Acceptance Testing section has conducted odor testing for several decades. The NASA-STD-6001B[1] document describes a supplemental test method option for programs to evaluate materials with odor concerns (Test 6, *Odor Assessment*). The standard method for this supplemental test is performed by exposing each odor panel volunteer to a 30 mL volume of test atmosphere for less than 10 s of total exposure. This test effectively identifies immediately objectionable odors; however, it does not provide data for any physical effects of the odor or odors that may become more intense and objectionable after prolonged exposure.

In 2014, WSTF performed an offgas test per NASA-STD-6001B Test 7, *Determination of Offgassed Products*, of a proprietary bonding material sandwiched between two aluminum plates with an exposed bondline. Incidental exposure while handling the bonding material before and after testing indicated that the bonding material's odor may become more objectionable after prolonged exposure as well as affect the individuals physically. Despite the fact that the material was established to have a clear odor, it continued to be the preferred recommended material because of its performance characteristics. Knowing that crew members would be exposed to the odor for an extended duration, a better understanding of the odor impression and effect over a longer exposure time was desired. In addition, a closer simulation of realistic exposure scenarios was needed than what was represented in the supplemental standard odor test method. To address these questions and concerns, WSTF developed an extended-duration (15-min) odor test method to evaluate the odor and physical effects of the bonding material in an exposure scenario more closely approximating realistic conditions. Various sample configurations were considered with the goal of selecting an optimal configuration that would best minimize offgassing toxicity (NASA-STD-6001 Test 7) and odor by means of containment. The supplemental standard (<10 s) and extended-duration (15-min) odor tests were both used as tools in comparing configurations for final selection and implementation.

II. Test Method

Offgas toxicity testing was completed first per NASA-STD-6001B Test 7 to evaluate the material for spacecraft offgassing acceptance and to ensure the specific test gas was safe for human exposure prior to odor testing. After offgas toxicity testing was complete, a comparative standard (<10 s) odor test of the two odor containment configurations of the bonding material was performed, followed by extended-duration (15-min) odor tests the next day. The objective of this test method was to determine which odor containment configuration performed best while simulating long-term exposures and realistic exposure scenarios.

A. Sample Preparation

A sample of the bonding material with the exposed bondline was tested for NASA-STD-6001B Test 7 offgassing toxicity and resulted in high offgassing toxicity levels. Because of the high offgassing toxicity level of the exposed bondline configuration, odor testing was not performed. Two offgassing and odor containment sample configurations of the bonding material were considered with the goal of selecting an optimal configuration that would best minimize offgassing toxicity and odor. Sample A was configured with the bonding material sandwiched between two aluminum plates, using aluminum (Al) tape to seal the bondline to reduce the toxicity and odor of the bonding material. Sample B was configured identically, but a room temperature vulcanization (RTV) compound was used to seal the bondline. Exact material specifications are not provided due to the proprietary nature of the design. Figure 1 illustrates the sample configurations.

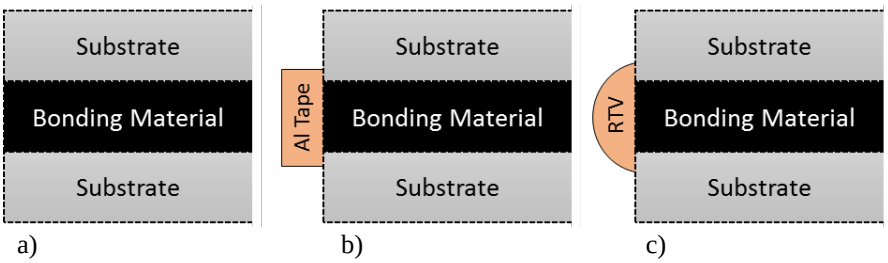


Figure 1. **Sample configurations.** *Cross-section of the initial bonding material sample a) without odor containment, b) Sample A with aluminum (Al) tape sealant as odor containment, and c) Sample B with RTV sealant as odor containment.*

B. Offgassing Toxicity Standard Procedure

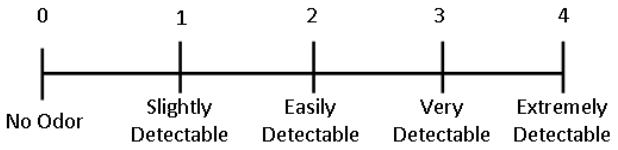
The samples were loaded into separate, identical containers with purified air as the test atmosphere for offgassing toxicity testing per NASA-STD-6001B Test 7. The sample containers were conditioned for 72 h at 120 °F (49 °C) and 11.9 psia (82.1 kPa). Upon removal from the oven, each sample was cooled to room temperature and analyzed by gas chromatography flame ionization detection (GC-FID) and gas chromatography mass spectrometry detection (GC-MSD) for a standard toxicity test. The toxicity data were used to determine if a dilution was necessary to safely perform an odor test. Sample A (Al tape sealant) did not require a dilution. Sample B (RTV sealant) required approximately a 4/5 dilution due to the higher toxicity rating.

C. Comparative Supplemental Standard (< 10 seconds) Odor Test Procedure

A supplemental standard comparative (<10 s) odor test was completed the day before the extended-duration (15-min) odor test. The same gas samples used for standard comparative odor tests were also used for extended-duration (15-min) odor tests. Procedurally, the NASA supplemental standard odor assessment is a blind test, and as such, associated human test subjects are unable to see the related samples. As part of this supplemental standard odor test, a glass syringe is used to draw headspace gas of the conditioned sample. The collected headspace gas is then injected into the odor panel member's mask (Figure 2) for a direct exposure, thereby minimizing ambient source air dilution. By the nature of the exposure method, exposures are brief, less than 3 s in length. In order to minimize variability due to inherent human subjectivity, the odor panel members evaluate each sample three times and provide potency ratings on a scale of 0 to 4 using the scale shown in Figure 3. In addition, the human test subject is asked to declare their overall impression of the odor as pleasant, irritating, revolting, or neutral. Members are also asked to further describe the odor and to list any effects they experience. A comparative odor test is a standard odor test where more than one material is tested with consecutive exposures using the identical human panel. For this comparative odor test, each human test subject was blindly administered three consecutive aliquots of Sample A (Al tape sealant), approximately 1 min apart, through a glass syringe. The test subject was then blindly administered a blank sample of purified air through a second glass syringe. Approximately 1 min later, the test subject was blindly administered three consecutive aliquots of Sample B (RTV sealant, 4/5 dilution), approximately 1 min apart, using a third glass syringe. Once all sample aliquots were administered, the test conductor repeated this test sequence for all additional test subjects.

Figure 2. Supplemental Standard odor test. *Administration of supplemental standard odor test sample using a mask and glass syringe.* Photo ID wstf0807e06127

Each human test subject was also required to pass a three-bottle test by correctly identifying a water blank from two odor standards chosen at random. This three-bottle test is used to objectively validate the sense of smell of each test subject on the day of the test.



Odor Potency Rating	Description
Slightly Detectable	A barely detectable odor other than that from the mask and tube. If you walked into a room with this odor, you would vaguely notice odor.
Easily Detectable	An easily detectable odor. If you walked into a room with this odor, you would immediately and clearly notice its presence.
Very Detectable	A strong odor that you may still consider tolerable. If you walked into a room with this odor, you would immediately be struck by its strength.
Extremely Detectable	An overpowering odor. It is possible that the odor may cause any of the following: a burning sensation, tearing, pain, coughing, nausea, aftertaste, or headache. If you walked into a room with this odor, you would feel the need to evacuate.

Figure 3. Odor test potency rating scale and descriptions.

D. Extended-duration (15-minute) Odor Test Procedure

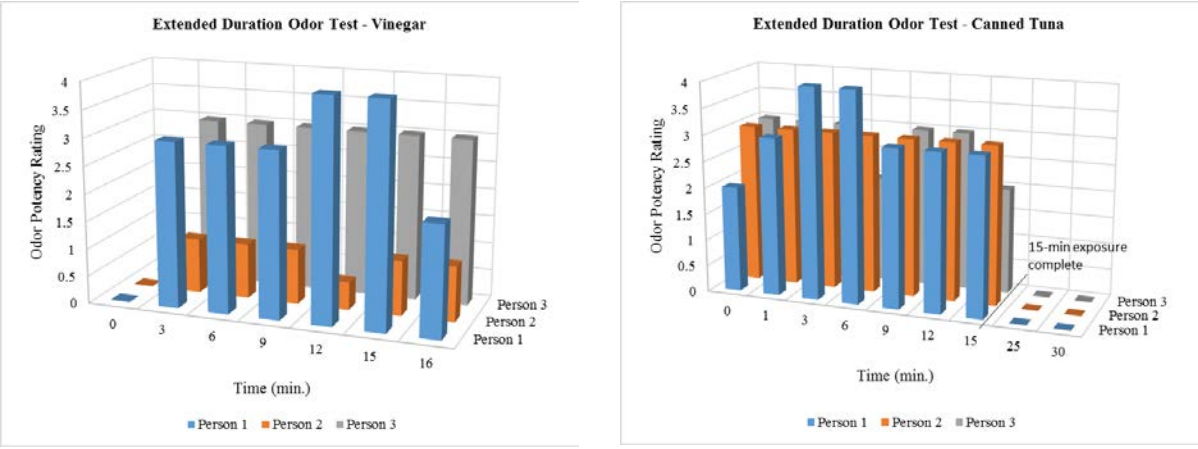
1. Initial Method Development and Findings

In the development of the odor test for extended exposure, a method change was desired to more closely simulate realistic exposure scenarios than what was represented in the supplemental standard odor test. Instead of injecting samples into a mask for direct inhalation, samples were placed in the center of small room (approximately 12 x 13 ft) with the doors closed to allow for natural diffusion and dilution of odor to occur, thus more realistically simulating odor behavior on a spacecraft. Nonetheless, to ensure excessive dilution did not occur, air handlers were disabled.

It was also the intent to develop a method that could adequately assess long-term and physical effects that may arise out of odor exposure and how these effects vary after exposure is ended. A maximum test exposure length of 15 min was established to comply with the most stringent requirements for ACGIH excursion factors as well as with ACGIH and OSHA short-term exposure limit (STEL) restrictions of no more than 30 min of exposure during a workday. Exposure limit calculations are documented in a WSTF memo titled *Odor Safe Dosage Calculations*, which was revised in June 2014 to capture the extended-duration odor test. The *Odor Safe Dosage Calculations* memo was reviewed and approved by the NASA Johnson Space Center (JSC) Certified Industrial Hygienists Manager Occupational Health and NASA Occupational Health Branch Acting Chief Master of Public Health prior to testing. Human test subjects were asked to assess odor and its effects during various time intervals within the maximum 15-min exposure as well as at various points post-exposure.

Food samples of tuna and balsamic vinegar were used in the developmental trials of the extended-duration (15-min) odor test procedure. These samples were chosen because they are commonly known as food items possessing strong odors yet safe for exposure during developmental trials. A team of three odor test conductors participated in the preliminary test and recorded their assessments and findings.

In the test method development, the vinegar test was performed first. During the initial procedure, the vinegar sample was removed from the room at 15 min. It was noted that despite removing the odor source, the odor of the vinegar remained in the room, thus exceeding the 15-min exposure-duration limit. To ensure a defined end of exposure, the procedure was updated requiring the human test subjects to leave the test room at the 15-min time limit and move to a non-adjacent room. Having the human test subjects use a cupped hand to waft the sample toward them prior to recording the odor assessment was added to the procedure to maximize the odor detection. In addition, the procedure was updated to include a rotation of the human subjects around the sample after each odor assessment to minimize positional effects. The tuna sample was tested with the updated procedure, which was then also used for the final bonding material test samples. The tuna and vinegar odor potency ratings by each human test subject are displayed in Figure 4. Human subjects were asked to record impression and physical effects of exposure at the various time intervals. Test subjects reported burning eyes, headache, and frustration throughout the 15-min exposure. These effects were, however, not reported during the posttest analysis points after the odor exposure had ended. The collected data verified that the designed test methodology effectively met its desired intent, adequately assessing long-term and physical effects that may arise out of odor exposure and how these effects vary after exposure has ended.



a) b)

Figure 4. Extended-exposure individual food odor potency results. *Results for the a) vinegar and b) canned tuna practice food tests for developing the extended-duration odor test.*

2. Bonding Material Extended-duration (15-minute) Odor Test.

The day after the comparative supplemental standard odor test (<10 s) was completed on the two bonded samples with alternate sealing material configurations, the same five human test subjects were used to complete the extended-duration (15-min) odor test. The extended-duration odor test was performed in a small room (approximately 12 x 13 ft) with the doors closed and the air handlers disabled to minimize ambient air dilution. The five odor panel members were positioned in a circle within 2 ft of the sample container. Time 0 was recorded when the sample container lid was opened. Each odor panel member rated their odor assessment at 0, 1, 3, 6, 9, 12 and 15 min, gently wafting the sample toward them immediately before measuring odor potency. After each recorded assessment, each odor panel member rotated their position in the circle around the sample to minimize potential effects due to their position in the room. At 15 min, all odor panel members promptly departed to a non-adjacent room in order to be removed from the odor immediately. Each test subject continued to record their reaction and physical effect assessments at both 10 and 15 min posttest. Posttest interviews by the test conductors were used to capture any additional data.

3. Comparative Extended-duration (15-minute) Odor Test

Extended-duration odor tests on the two candidate odor containment configurations of the bonding material were performed on the same day with a 60-min break between samples. In addition to comparing data from the individual tests, the human subjects were asked during the posttest interview to provide their impressions of how the two samples compared.

III. Results

A. Comparative Supplemental Standard (< 10 seconds) Odor Test Results

The individual odor assessments for the comparative supplemental standard (<10 s) odor tests for Sample A (Al tape sealant) and Sample B (RTV sealant) were recorded by the human test subjects, and the results are displayed in Figure 5. Sample B (RTV sealant) required a 4/5 dilution to be odor-tested safely, meaning that only 20 percent of the original gas remained in the tested sample. Linear associations would indicate that the odor could be as much as five times more potent in the full concentration gas; however, the maximum odor potency rating is 4. Considering the diluted Sample B (RTV sealant) already received a relatively high initial odor potency rating, if full-strength samples were to be tested, it is anticipated that potency ratings would likely be one or two levels higher. Figure 5 displays the results from the diluted sample as well as an extrapolated representation of a full-strength Sample B (RTV sealant). Using extrapolated Sample B (RTV sealant) potency data, it is clear that Sample B (RTV sealant) generates higher potency ratings over Sample A (Al tape sealent).

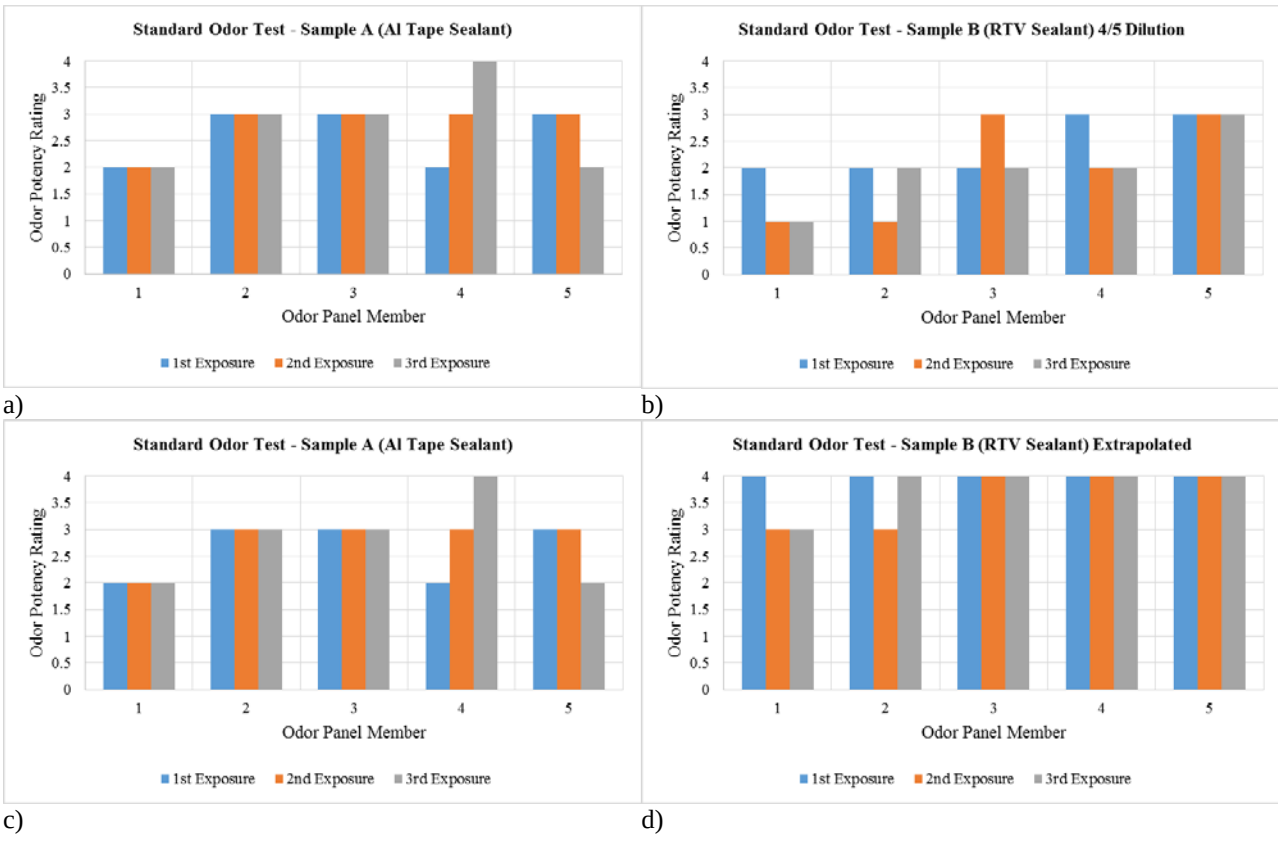
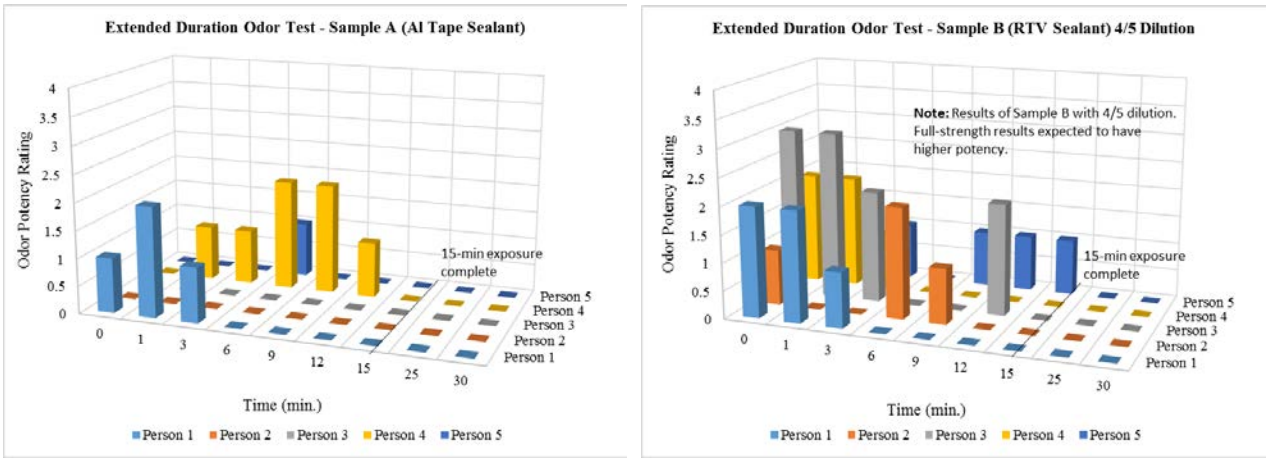


Figure 5. Supplemental standard exposure individual odor potency results. *Results for the supplemental standard (<10 s) odor tests for a) Sample A (Al tape sealant) and b) Sample B (RTV sealant, 4/5 dilution). A comparison is shown for c) Sample A to d) an extrapolated Sample B as a representative reading for a sample without dilution.*

B. Comparative Extended-duration (15-minute) Odor Test Results

For extended-duration testing using a realistic exposure scenario on Sample A (Al tape sealant) and Sample B (RTV sealant), individual odor assessments were recorded by the human test subjects and the results are displayed in Figure 6. Sample B (RTV sealant) required a 4/5 dilution in order to be odor-tested safely and was diluted for the supplemental standard odor assessment the day before the extended-duration test. Despite significant dilution, Sample B (RTV sealant) still resulted in higher potency ratings over Sample A (Al tape sealent) during extended-duration testing. If full-strength samples were to be tested, it is anticipated that potency ratings would likely be one or two levels higher than reported for the 4/5 dilution test. Results for the extended-duration odor testing agreed with initial supplemental standard testing in that Sample A (Al tape sealent) was a superior performer in minimization and containment of offgassing and odor.



a) b)

Figure 6. Extended-exposure individual odor potency results. *Results for the extended-duration (15-min) odor tests for a) Sample A (Al tape sealant) and b) Sample B (RTV sealant, 4/5 dilution).*

C. Overview of All Odor Test Potency, General Assessment, Description, and Effects Results

An average was calculated using all odor potency ratings for the supplemental standard odor test and an average at each time interval of the extended-duration odor test. As an overall summary and overview of all data, the results are displayed in Figure 7 and Tables 1 and 2. For the supplemental standard (<10 s) odor test, despite dilution, Sample B (RTV sealant, 4/5 dilution) received very similar potency ratings as for Sample A, indicating that even the diluted odor was highly potent. Full-strength sample potency could be extrapolated to be between ratings of 3 and 4, indicating that Sample A (Al tape sealant) odor containment performance was superior. These findings were reiterated for the 15-min odor test, and even without extrapolation of 4/5 dilution data ratings of Sample B (RTV sealant, 4/5 dilution), stronger potency was reported. In an attempt to further compare candidate configurations, human subjects were asked during the posttest interview to provide their impressions of how the two samples correlated. Test subjects reported that the odor of Sample B (RTV sealant, 4/5 dilution), despite its dilution, was clearly stronger than the odor of the Sample A (Al tape sealant) configuration. No immediate or residual effects were noted. The Al tape containment configuration was selected as the preferred configuration for use on the space vehicle.

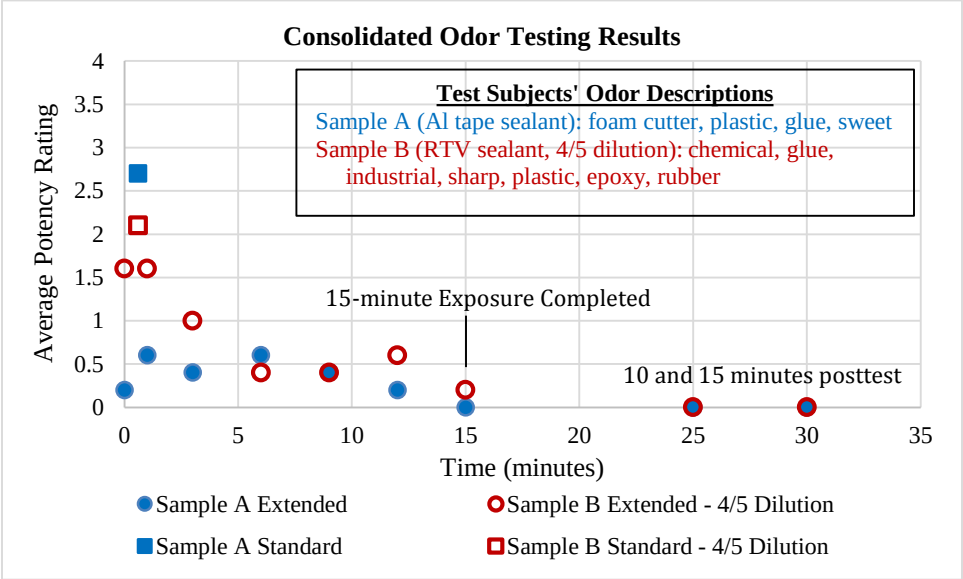


Figure 7. Test subject average individual odor potency results. *Average odor assessments for supplemental standard (<10 s) and extended (15-min) odor tests for Sample A (Al tape sealant) and Sample B (RTV sealant, 4/5 dilution).*

Table 1. Compiled Supplemental Standard Odor Test Data.

Configuration	Average Potency	General Assessment	Odor Descriptions	Effects Descriptions
Sample A	2.7	N, I	Styrofoam, chemical, model glue, paint, airplane glue, solvent, adhesive, sharp, citrus	None, tingling
Sample B (~4/5 Dil. ~30-min prior)	2	N, NI, I	Styrofoam, chemical, model glue, paint, airplane glue, solvent, adhesive, tangy, fragrant, citrus	None, tingling, burning

General Assessment: Non-irritating (NI), Neutral (N), Pleasant (P), Irritating (I), Revolting (R)
Potency Scale: No Odor (0), Slightly Detectable (1), Easily Detectable (2), Very Detectable (3), Extremely Detectable (4)

Table 2. Compiled Extended-duration Odor Test Data.

Exposure Duration	Average Potency	General Assessment	Odor Descriptions	Effects Descriptions
Sample A (Al tape sealant)				
0 min	0.2	NI, N	Foam cutter	None
1 min	0.6	NI, N	Foam cutter, plastic	None
3 min	0.4	NI, N	Plastic	None
6 min	0.6	NI, N	Glue	None
9 min	0.4	NI, N	Sweet, glue	None
12 min	0.2	NI, N	Sweet	None
15 min	0	NI, N	None	None
10 min post	0	NI, N	None	None
15 min post	0	NI, N	None	None
Sample B (RTV sealant, 4/5 dilution)				
0 min	1.6	NI, N, I	Chemical, model airplane glue, glue, industrial, sharp	None
1 min	1.6	NI, N, I	Chemical, model airplane glue, ball pit, glue, sharp	None
3 min	1	NI, N, I	Chemical, model airplane glue, glue, plastic	None
6 min	0.4	NI, N	Industrial, epoxy	None
9 min	0.4	NI, N	Industrial, epoxy, rubber mat	None
12 min	0.6	NI, N, I	Glue	None
15 min	0.2	NI, N	None	None
10 min post	0	NI, N	None	None
15 min post	0	NI, N	None	None

General Assessment: Non-irritating (NI), Neutral (N), Pleasant (P), Irritating (I), Revolting (R)
Potency Scale: No Odor (0), Slightly Detectable (1), Easily Detectable (2), Very Detectable (3), Extremely Detectable (4)

IV. Conclusion

Extended-duration odor testing with 15 min exposures proved to be an effective tool for realistically simulating odor exposures and assessing extended-duration exposure effects of materials with known odor concerns. The developed test method was verified to have effectively met its desired intent, adequately assessing long-term and physical effects that may arise out of odor exposure and how these effects vary after the exposure has ended. Comparing data from the extended-duration (15-min) odor test with the supplemental standard duration (<10 s) NASA-STD-6001B Test 6 for odor assessment validated that the supplemental NASA-STD-6001 Test 6 is effective and conservative in its assessment of material odors. Nonetheless, if a preferred material is confirmed to have an odor of concern, the test methodology for extended exposure is recommended as an additional tool for evaluating longer-term exposure effects in a realistic exposure scenario. Both extended-duration (15-min) and supplemental standard duration (<10 s) exposure tests are test method options that can be useful in comparing various odor containment configurations for ranking and selection of an optimal design.

Acknowledgments

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Reference

[1]NASA-STD-6001B, *Flammability, Offgassing, and Compatibility Requirements and Test Procedures,* Test 6, "Odor Assessment" and Test 7, "Determination of Offgassed Products," National Aeronautics and Space Administration, August 2011.