

History of NASA's Determination of Offgassed Products (Test 7)

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NASA's Determination of Offgassed Products (Test 7) from materials and assembled articles for spaceflight has evolved since the Apollo program for over 50 years to meet various habitable spacecraft nonmetallic programmatic requirements. Now mandated by NASA-STD-6016A, *Standard Materials and Processes Requirements for Spacecraft*, all nonmetallic materials used in habitable flight compartments, with the exception of ceramics, metal oxides, inorganic glasses, and materials used in sealed containers, must meet the offgassing requirements in NASA-STD-6001B Test 7. This manuscript presents the history of Test 7, beginning with the Apollo spacecraft nonmetallic materials selection guidelines and test requirements in 1967, in which tests were performed in mostly oxygen atmospheres. It progresses through Skylab, Space Shuttle, International Space Station nonmetals testing, and acceptance requirements with milder test environments. This review of the history of Test 7 presents the reader with a perspective on the development and changes undergone since inception to the present. Related NASA standard tests (some now former, discontinued, combined, or supplemental) including Test 6, *Odor Assessment*, Test 16, *Determination of Offgassed Products from Assembled Articles*, and Test 12, *Total Spacecraft Cabin Offgassing*, are discussed in context.

Nomenclature

<i>3D</i>	=	<i>three-dimensional</i>
<i>ASPO</i>	=	Apollo Spacecraft Program Office
<i>BSI</i>	=	British Standards Institution
<i>cm³</i>	=	cubic centimeter
<i>ISO</i>	=	International Organization for Standardization
<i>CO</i>	=	carbon monoxide
<i>CVCM</i>	=	collected volatile condensable materials
<i>ECSS</i>	=	European Cooperation for Space Standardization
<i>ESA</i>	=	European Space Agency
<i>GC</i>	=	gas chromatography
<i>GC-MS</i>	=	gas chromatography-mass spectrometry
<i>FID</i>	=	flame ionization detector
<i>FTIR</i>	=	Fourier transform infrared

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<i>g</i>	=	gram
<i>HCl</i>	=	hydrogen chloride
<i>HCN</i>	=	hydrogen cyanide
<i>hr</i>	=	hour
<i>IR</i>	=	infrared spectrometry
<i>JSC</i>	=	Johnson Space Center
<i>L</i>	=	liter
<i>M&P</i>	=	materials and processes
<i>MAC</i>	=	maximum allowable concentration
<i>MAPTIS</i>	=	Materials and Processes Technical Information System
<i>MS</i>	=	mass spectrometry
<i>MSC</i>	=	Manned Spaceflight Center
<i>MSFC</i>	=	Marshall Space Flight Center
<i>NHB</i>	=	NASA handbook
<i>NTRS</i>	=	NASA Technical Reports Server
<i>ppm</i>	=	parts per million
<i>psia</i>	=	pounds per square inch absolute
<i>RQTD</i>	=	Reliability, Quality, and Test Division
<i>s</i>	=	second
<i>SMAC</i>	=	Spacecraft Maximum Allowable Concentration
<i>T</i>	=	Toxic Hazard Index
<i>TML</i>	=	total mass loss
μg	=	microgram
$^{\circ}\text{C}$	=	degrees Celsius
$^{\circ}\text{F}$	=	degrees Fahrenheit
<i>VCS</i>	=	voluntary consensus standard
<i>WVR</i>	=	water vapor regained
\pm	=	plus or minus

I. Background

Materials and processes (M&P) control specifications as they have existed for many years, such as NASA-STD-6016A,¹ *Standard Materials and Processes Requirements for Spacecraft*, are used to define minimum requirements for incorporation in NASA program/project hardware procurements and technical programs. For example, NASA-STD-6016A requires materials shall meet the requirements of NASA-STD-6001B,² *Flammability, Offgassing, and Compatibility Requirements and Test Procedures*. NASA-STD-6016A also addresses toxic offgassing, and requires that all nonmetallic materials used in habitable flight compartments, with the exception of ceramics, metal oxides, inorganic glasses, and materials used in sealed containers (maximum leak rate less than $1 \times 10^{-4} \text{ cm}^3/\text{s}$), shall meet the offgassing requirements of Test 7, *Determination of Offgassed Products*, of NASA-STD-6001B. These data are used to evaluate whether the offgassed compounds pose a threat to the health and safety of astronauts in spacecraft. The basis for this are Spacecraft Maximum Allowable Concentration (SMAC) values, which are obtained from JSC 20584 (2017),³ *Spacecraft Maximum Allowable Concentrations for Airborne Contaminants*, or from NASA's *Materials and Processes Technical Information System* (MAPTIS) for compounds for which no SMAC values are found in JSC 20584.

NASA-STD-6001B² Test 7 is the current NASA standard test for determining the offgassing characteristics under standardized conditions for materials and assembled articles to be located within habitable environments. Test 7's development, implementation, and improvement over the years since its inception in 1967 for the Apollo nonmetals evaluation program have required significant experience and efforts. Whereas the original tests were promulgated by NASA, the test method was transferred to the International Organization for Standardization (ISO)

ISO 14624-3:2005,⁴ *Space Systems—Safety and Compatibility of Materials—Part 3: Determination of Offgassed Products from Materials and Assembled Articles*, with a number of additions, exceptions, and clarifications for NASA use.

II. Objective

This manuscript examines the history of Test 7 and its enhancements from inception as a standard test for the evaluation of nonmetals for the Apollo program. Related NASA standard tests (some former, discontinued, combined, or supplemental) including Test 6 (*Odor Assessment*), Test 16 (*Determination of Offgassed Products from Assembled Articles*), and Test 12 (*Total Spacecraft Cabin Offgassing*) are discussed in context.⁸

III. Standard Test Development

Literature dated from the Apollo program era to the present relevant to Test 7 was reviewed for the development of this manuscript. Standard procedures and requirements for the evaluation of spacecraft nonmetallic materials for the Apollo program were developed and mandated in MSC-A-D-66-3,⁵ *Procedures and Requirements for the Evaluation of Apollo Crew Bay Materials*, MSC-PA-D-67-13,⁶ *Apollo Spacecraft Nonmetallic Materials Requirements*, and Addendum No. 1 to MSC-PA-D-67-13,⁷ *Apollo Spacecraft Nonmetallic Materials Requirements*, which were issued by the Manned Spaceflight Center (MSC).

Requirements given in MSC-PD-66-3, *Procedures and Requirements for the Evaluation of Apollo Crew Bay Materials*, could not be assessed because the document could not be found. MSC-PD-66-3 was a pre-Apollo 1 (AS-204)** fire incident⁸ standard.

MSC-PD-66-3 Revision A contained 11 required (standard) tests and two supplementary tests, each numerically identified (e.g., Test 1, Test 2, etc.). This test numbering system is consistent, with some exceptions for Test 7, throughout the succession of revisions to NASA standard materials testing protocols. There have been some Test 7 title changes, addition and deletion of tests, and re-designation or addition of tests to “supplemental.” The Apollo-era program material test requirements in MSC-A-D-66-3,⁵ MSC-A-D-66-3A,⁹ MSC-PA-D-67-13,⁶ D-NA-0002,¹⁰ MSFC-SPEC-101,¹¹ MSC-PA-D-67-13 Addendum No. 1,⁷ MSFC-SPEC-101A,¹² NASA-TM-79493¹³ (draft prepared by the Skylab working group), and MSFC-SPEC-101B¹⁴ focused primarily on fire, flammability, ignition, combustion, offgassing, and odor testing, whereas the NASA Handbook (NHB) 8060.1,¹⁵ NHB 8060.1A,¹⁶ NHB 8060.1C,¹⁷ NASA-STD-6001,¹⁸ NASA-(I)-STD-6001A,¹⁹ NASA-STD-(I)-6001B,²⁰ and NASA-STD-6001B² standards retained similar focus. Test No. 7, or Test 7 as it is now commonly referred to, has had a continuous presence as a required standard test for offgassed products from nonmetallic materials in the spacecraft materials testing protocols and requirements identified in this manuscript.

A chronology of NASA Test 7 and its identified precursor documentation are outlined in order as follows in Table 1. European and international documents are not included in Table 1.

§ The language in this manuscript was adapted with minimal modification from the source documents to maintain technical continuity and may not reflect proper grammatical structure.

**During one of the final prelaunch tests for the first manned Apollo Flight on January 27, 1967, a fire aboard the Apollo Command Module, located on top of a Saturn launch vehicle on the launch pad of Complex 34 at the John F. Kennedy Space Center, resulted in the deaths of Astronauts Virgil I. Grissom, Edward H. White, and Roger B. Chaffee (Report to Congress, 1968). Marshall Space Flight Center (MSFC) issued MSFC-SPEC-101.¹¹

Table 1. Chronology of NASA Test 7

Document	Title	Date
MSC-A-D-66-3	<i>Procedures and Requirements for the Evaluation of Apollo Crew Bay Materials</i>	May 13, 1966
ASPO-RQTD-D67-5A	<i>Apollo Spacecraft Program Office Nonmetallic Materials Selection Guidelines</i>	May 3, 1967
MSC-A-D-66-3 Revision A	<i>Procedures and Requirements for the Evaluation of Spacecraft Nonmetallic Materials</i>	June 5, 1967
MSC-PA-D-67-13	<i>Apollo Spacecraft Nonmetallic Materials Requirements</i>	February 9, 1968
D-NA-0002	<i>Procedures and Requirements for the Flammability and Offgas Evaluation of Manned Spacecraft Nonmetallic Materials</i>	July 18, 1968
MSFC-SPEC-101	<i>(Specification) Flammability Requirements and Test Procedures for Materials in Gaseous Oxygen Environments</i>	August 20, 1968
MSC-PA-D-67-13 Memorandum	<i>Addendum No. 1, MSC-PA-D-67-13</i>	November 7, 1969
MSFC-SPEC-101A	<i>(Specification) Flammability, Odor, and Toxicity Requirements and Test Procedures for Materials in Gaseous Oxygen Environments</i>	January 12, 1970
NASA-TM-79493	<i>Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments which Support Combustion (Draft Copy)</i>	December 1970
MSFC-SPEC-101B	<i>(Specification) Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments which Support Combustion</i>	March 15, 1971
NHB 8060.1	<i>Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion</i>	November 1971
NHB 8060.1A	<i>Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion</i>	February 1974
NHB 8060.1B	<i>Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion</i>	September 1981
NHB 8060.1C	<i>Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion</i>	April 2, 1991
NASA-STD-6001	<i>Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion – Previously Published as NHB 8060.1C</i>	February 9, 1998
NASA-STD-(I)-6001A	<i>Flammability, Offgassing, and Compatibility Requirements and Test Procedures</i>	April 21, 2008
NASA-STD-(I)-6001B	<i>Flammability, Offgassing, and Compatibility Requirements and Test Procedures</i>	November 10, 2009
NASA-STD-6001B	<i>Flammability, Offgassing, and Compatibility Requirements and Test Procedures</i>	August 26, 2011
NASA-STD-6001B w/Change 2: Administrative/Editorial Changes	<i>Flammability, Offgassing, and Compatibility Requirements and Test Procedures</i>	April 21, 2016

IV. Guidelines, Requirements, and Description of Tests

Test materials were classified by surface (materials that are essentially two-dimensional), volume (materials having an indefinite volume but having a large surface area due to surface convolutions or matting including foams and other blown or foamed materials), weight (materials having a definite bulk and not conforming to the volume classification, including potting compounds, molding compounds, cast or formed objects, solid wires, thick plastics, and liquids not used or applied as coatings or thin films), or as specialized items (materials not meeting the other classification; most often non-homogeneous materials). Test materials such as coatings, finishes, primers, adhesives, and solid film lubricants were first applied to a chemically cleaned aluminum substrate of suitable thickness and in a coating thickness equal to production practice. A test material was placed in a cleaned, dry, leak-free/leak-checked test chamber with a minimum volume of 2 liters (L) and a certified background atmosphere, filled with a known and relevant test atmosphere, and thermally equilibrated for a specified time. For example, nearly pure oxygen atmospheres were used in the Apollo-era procedures, where the same oxygen atmosphere was used on the spacecraft. Following the designated exposure period, the chamber was brought to room temperature and samples of the test atmosphere were withdrawn and analyzed using instruments as required; in particular gas analyzers, gas chromatography (GC) with FID, mass spectrometry (MS), infrared spectrometry (IR), and their gas sampling interfaces as required. Suitable chromatographic columns were required for separating analytes. In early versions, a total hydrocarbon analyzer or an equivalent instrument could be used for determination of total organics. After analysis of the test atmosphere, the test specimen was removed from the chamber and weighed. The test protocols contained specific requirements for analyte quantitation and specific reporting requirements were indicated. The focus on toxicity calculations performed by the laboratory changed and increased as the test documents progressed to the present. Early iterations of Test 7 required characterization of condensable offgassed components. At some point, as indicated below, determination of condensable components became a separate test and requirements related to condensable material were eliminated from Test 7. The terms out-gassing, outgassing, and offgassing were used without apparent distinction and are used as they are called out in the specific test procedures discussed below.

ASPO-RQTD-D67-5A²¹ (1967), *Apollo Spacecraft Program Office Nonmetallic Materials Selection Guidelines*, issued from the Manned Spaceflight Center Apollo Spacecraft Program Office (ASPO) Reliability, Quality, and Test Division (RQTD) established acceptance guidelines and test requirements for nonmetallic materials in the Apollo Spacecraft. The guidelines applied to all man-rated^{††} test and flight spacecraft. Two test methods related to Test 7, *Odor and Carbon Monoxide*, and *Total Organics*, were called out and referenced in MSC-A-D-66-3⁵ for specific applications, but the tests were not numbered or given a numerical designation in this document. Materials usage categories where the tests were required were also established. Specific test requirements for usage categories included determination of nonmetallic material property requirements depending on the material applications with respect to quantity, proximity to ignition sources or exposure to oxygen, and use over specified areas of the spacecraft.

MSC-A-D-66-3⁵ (1966), *Procedures and Requirements for the Evaluation of Apollo Crew Bay Materials*, was not found for reasons unknown, despite search attempts using multiple resources including the Marshall Space Flight Center (MSFC) Document Repository, NASA Technical Reports Server (NTRS), the JSC Library, subject matter experts, and internet resources. According to references made in Apollo Accident Hearing before the Committee on Aeronautical and Space Sciences (1967),^{‡‡} MSC-A-D-66-3 contained two relevant tests: Odor and CO, and Total Offgassing.

MSC-A-D-66-3 Revision A⁹ (1967), *Procedures and Requirements for the Evaluation of Spacecraft Nonmetallic Materials*, issued by the MSC Reliability, Quality, and Test Division and the Apollo Spacecraft Program Office was the first document to contain specific Test 7 requirements and procedures. This specification consisted of revisions to the standardized test plans found in MSC-A-D-66-3 to conform to the new nonmetallic materials selection guidelines of ASPO-RQTD-067-5A, which imposed more requirements on the selection and performance of Apollo Spacecraft nonmetallic materials. Test 7 was identified as *Determination of Organic Offgassing Products and Carbon Monoxide*. The test procedure established the criteria for determining the suitability of nonmetallic materials for use in the space vehicle crew compartment environments with respect to production of potentially toxic or objectionable materials by outgassing. The test was designed to determine the amount, nature and rate at which potentially toxic and objectionable products outgas from crew bay materials in an effort to ensure physiological safety and optimum mission performance

^{††} “Man-” has been deprecated to “Human-“ or “Crewed-” in more recent times.

^{‡‡} The program office issued a document in May of 1966 that established uniform criteria for the selection of non-metallic materials in the crew compartment.

of the astronauts in manned space vehicles. Criteria specified that materials that did not offgas organic compounds under the test conditions could be used freely in the spacecraft crew bay area. The maximum allowable concentration of total organics in the as-configured spacecraft shall not exceed 100 parts per million (ppm) at 14.7 pounds per square inch absolute (psia) or 300 ppm at 5.0 psia, and the maximum allowable concentration of carbon monoxide (CO) in the as-configured spacecraft shall not exceed 25 ppm at 14.7 psia or 75 ppm at 5.0 psia.

Test material was exposed to 95 percent pure oxygen at 5.0 plus or minus (\pm) 0.1 psia and 155 \pm 5 degrees Fahrenheit ($^{\circ}$ F) for a total period of 72 hr. The offgassing rate was determined by analyzing samples at 8, 24, 48 and 72 hr then graphing the results and evaluating the resultant curve. Condensates in the cold trap were recovered as gas and likewise analyzed and plotted. Gases were analyzed for total organics and results expressed as methane equivalents. A complete analysis of individual outgassing components above 10 ppm or as specified was performed. The quantity of CO evolved was reported.

MSC-PA-D-67-13⁶, *Apollo Spacecraft Nonmetallic Materials Requirements* (February 9, 1968), issued by MSC Apollo Spacecraft Program Office established requirements for control of flammability and toxicity of nonmetallic materials used in the Apollo Spacecraft and in test equipment used inside the Apollo Spacecraft during closed hatch testing. It identified Test No. 7 as *Determination of Carbon Monoxide and Total Organics*. The volatiles to be determined by this test were limited to two categories: 1) Carbon Monoxide, and 2) Total Organics. Material acceptability was based on the concentration of out-gassed volatiles (total organics) relative to the weight of material in use aboard the spacecraft and also of the effective spacecraft crew bay volume. The document indicated the maximum allowable concentration of total organics in the tested configuration shall not exceed 200 μ g/g of total organics per gram of sample; the maximum allowable concentration of CO from the sample in the tested configuration shall not exceed 25 μ g/g.

Test samples were exposed to 95 percent pure oxygen at 5.0 \pm 0.1 psia and 155 \pm 5 $^{\circ}$ F for a total period of 72 hr. Total organics were expressed as pentane equivalents and the quantity of CO evolved was reported. The CO content of the evolved gases was determined using an appropriate separation and analytical technique.

D-NA-0002¹⁰, *Procedures and Requirements for the Flammability and Offgas Evaluation of Manned Spacecraft Nonmetallic Materials* (July 18, 1968), issued by the MSC Reliability and Quality Assurance Office provided standard test procedures for the evaluation of nonmetallic materials for flammability and offgassing. The minimum requirements for acceptance of the materials were included for each category of application within manned spacecraft. In an apparent reversal of Test No's., it identified Test No. 6 as *Determination of Organic Offgassing Products and Carbon Monoxide* and Test No. 7 as *Odor Test*. This part of this manuscript briefly describes the Test 6, *Determination of Organic Offgassing Products and Carbon Monoxide*, procedure because the focus is on offgassed products testing irrespective of Test No. This procedure established the criteria for a screening test for the suitability of nonmetallic materials for use in the space vehicle crew compartment environments. The criteria was established with respect to production by out-gassing of potentially toxic or objectionable volatiles. The volatiles were separated into two categories: Carbon Monoxide and Total Organics. The procedure required that samples meet applicable flammability criteria prior to offgas testing unless specifically requested otherwise. Also of note, Test No. 7 *Odor Test* of D-NA-0002¹⁰ stated, "All materials shall have been tested for CO content and total organics before being subjected to the odor test. If a material has over 25 μ g/g of CO, or over 100 μ g/g of total organics, no odor test shall be performed. If the offgassing products above 10 μ g/g are analyzed and are judged by the appropriate space medicine division to be noxious, this fact shall be noted on the test report, and no odor test need be performed."

Carbon monoxide and organic offgassing products were the target analytes, although some inorganic species were analyzed (hydrogen cyanide (HCN), hydrochloric acid (HCl), ammonia, hydrogen fluoride, carbonyl fluoride, and silicon tetrafluoride). The test conditions were 155 \pm 5 $^{\circ}$ F for a total period of 72 hr. Test atmosphere composition and pressure varied depending on materials category designated by the requirements for each program. Total organic results were reported in units of μ g/g using methane as a standard.

Total Organics were expressed as methane equivalents. Outgassing components exceeding 10 μ g/g including but not limited to HCN, benzene, xylene, methyl ethyl ketone, chloroform, n-butanol, dichloromethane, 1,4-dioxane, formaldehyde, trichloroethylene, HCl, ammonia, hydrogen fluoride, carbonyl fluoride, and silicon tetrafluoride were reported. Condensates in the cold trap could be recovered as gas and likewise analyzed and reported. The CO content of the evolved gases was determined with an appropriate separation and analytical technique having the sensitivity to detect within 0.5 μ g/g CO.

MSFC-SPEC-101,¹¹ *Flammability Requirements and Test Procedures for Materials in Gaseous Oxygen Environments* (August 20, 1968), was a MSFC specification approved by the George C. Marshall Space Flight Center (MSFC) and available for use by MSFC and associated contractors. The specification established flammability

acceptance criteria for materials used in gaseous mixtures containing oxygen. It identified Test No. 7, *Materials in Closed (Unsealed) Containers*, which was not applicable to the offgassing Test 7 that is the focus of this manuscript. This MSFC specification contained only requirements related to flammability in oxygen environments, and none for offgassing or odor.

MSC-PA-D-67-13,²² *Apollo Spacecraft Nonmetallic Materials Requirements Addendum No. 1* (November 7, 1969), issued by the MSC Apollo Spacecraft Program Office did not change Test No. 7 from MSC-PA-D-67-13⁶ but made changes to other tests including the related Test No. 6 (*Odor Test*). The conditions of Test No. 7 exposure remained at 5.0 ±0.1 psia of 95 percent pure oxygen and 155 ±5 °F for a total period of 72 hr if required. Total organics were expressed as pentane equivalents. The quantity of CO evolved was reported. This addendum integrated Test 6 (*Odor Test*) with Test 7 as a toxicity screening test prior to the odor test.

MSFC-SPEC-101A,¹² *Flammability, Odor, and Toxicity Requirements and Test Procedures for Materials in Gaseous Oxygen Environments* (January 12, 1970), was a MSFC specification establishing flammability, odor, and toxicity acceptance criteria for materials used in gaseous mixtures containing oxygen. This specification was available for use by MSFC and associated contractors, and did not supersede any Manned Spacecraft Center (MSC) program documents. It identified Test No. 7, *Materials in Closed (Unsealed) Containers*, Test No. 8, *Determination of Organic Offgassing Products and Carbon Monoxide*, and Test No. 9, *Odor*. Of these, Test No. 8 corresponded to the Test 7 addressed in this manuscript. Test No. 8, *Determination of Organic Offgassing Products and Carbon Monoxide*, was a screening test to determine the suitability of nonmetallic materials for use in manned spacecraft. The criteria was established with respect to production by out-gassing of potentially toxic or objectionable volatiles. The volatiles were separated into two categories: CO and total organics. This test was not conducted prior to applicable flammability tests, nor was it conducted on materials that failed a flammability test unless specifically requested. The criteria of material acceptability was that total organics were reported as µg/g of sample material using methane as a standard. The maximum allowable level of total organics in the tested configuration could not exceed 100 µg/g. Carbon monoxide in the tested configuration could not exceed 25 µg/g. The test was conducted in an oxygen environment 95 percent by volume or the worst-case test pressure and gas mixture conditions designated for each program.

The conditions of exposure were 155 ±5 °F for a total period of 72 hr. After the final gas samples were taken for analysis, the test specimen was removed from the chamber and weighed. The gases were analyzed using methane as a calibration gas. Total organics were expressed as methane equivalents. Outgassing components exceeding 10 µg/g including but not limited to hydrogen cyanide (HCN), benzene, xylene, methyl ethyl ketone, chloroform, n-butanol, dichloromethane, 1,4-dioxane, formaldehyde, trichloroethylene, hydrochloric acid (HCl), ammonia, hydrogen fluoride, carbonyl fluoride, and silicon tetrafluoride were analyzed. If condensates occurred in the cold trap, they were recovered as gas and likewise analyzed. Outgassing components above that exceeded 10 µg/g were identified and reported. The CO content of the evolved gases was determined by an appropriate separation and analytical technique having the sensitivity to detect within 0.5 µg/g CO.

NASA-TM-79493,¹³ *Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments which Support Combustion* (Draft Copy) (December 1970), was a draft technical memorandum (TM) prepared by the Skylab Intercenter Working Group ostensibly for Skylab use, though no evidence of its use was identified. It described itself as a specification controlling the selection of materials with respect to flammability and offgassing to be used in and around manned spacecraft during flight and test operations. It identified Test No. 7 as *Determination of Offgassing Products and Carbon Monoxide Test*. NASA TM X-64900,²³ *Skylab Atmospheric Contamination Control*, was later published in 1974 and required all materials considered for use on Skylab, including stored materials and experiments, be evaluated for flammability and offgassing characteristics in accordance with MSFC-SPEC-101B¹⁴, D-NA-0002,¹⁰ and NHB 8060.1A.¹⁶

NASA-TM-79493¹³ (draft) described a procedure Test No. 7, *Determination of Offgassing Products and Carbon Monoxide Test*, that was not a copy of MSC-PA-D-67-13⁶, *Apollo Spacecraft Nonmetallic Materials Requirements Test 7*, or MSC-A-D-66-3.A⁹, *Procedures and Requirements for the Evaluation of Spacecraft Nonmetallic Materials Test 7*, though its Test 7 appeared derived and expanded from those documents. Test 7 was referred to as a specification in its introduction. The Test 7 criteria were established with respect to production of offgassing of potentially toxic or objectionable and condensable volatiles. The volatiles were separated into three categories: CO, total offgassing, and observable condensates. The test pressure and gas mixture conditions for the pertinent materials category were designated by the requirements for each program. Material usage categories, material usage types, and material groups and test requirements were established in this draft specification. Exposures were in 95 percent pure oxygen at 5.0 ±0.1 psia at 155 ±5 °F for a total period of 72 hr. Total organics were expressed as methane equivalents.

The criteria of acceptability was a maximum allowable concentration of total organics, excluding water, in the tested configuration not to exceed 100 µg/g; a maximum allowable concentration of CO in the tested configuration not to exceed 25 µg/g; and condensates determined as visible or invisible or as a function of any detection system selected by the testing agency in accordance with the requirements and criteria set by the program office.

The NASA-TM-79493¹³ draft test procedure had some similarities and differences with MSC-A-D-66-3 Revision A⁹ (1967) and MSC-PA-D-67-13,⁶ *Apollo Spacecraft Nonmetallic Materials Requirements* (February 9, 1968). The conditions of exposure were 155 ±5 °F for a total period of 72 hr. Following the designated exposure period, the evolved gases interfaced directly into the GC while being maintained at 155 ±5 °F. After the final gas samples were taken, the test specimen was removed from the chamber and weighed. The basic calibration gas was methane. The gases were analyzed for total organics expressed as methane equivalents; all offgassing components exceeding 10 µg/g were identified and quantified. These included but were not limited to the organics benzene, xylene, methyl ethyl ketone, n-butanol, chloroform, dichloromethane, 1,4-dioxane, formaldehyde, trichloroethylene, and carbonyl fluoride; non-organics HCN, HCl, ammonia, and silicon tetrafluoride; and CO. Outgassing components, including the target components, that exceeded 10 µg/g were identified and reported with estimated concentrations. The CO content of the evolved gases was determined using an appropriate separation and analytical technique having the sensitivity to detect within 0.5 µg/g CO.

MSFC-SPEC-101B¹⁴ (March 15, 1971), *Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments which Support Combustion*, was the next revision of MSFC-SPEC-101A¹², approved by MSFC, and available for use by MSFC and associated contractors, and did not supersede any MSC program documents. This MSFC specification identified Test No. 7 as *Determination of Organic Offgassing Products and Carbon Monoxide*. The purpose of the test was similarly to establish the criteria for a screening test, which would determine the suitability of nonmetallic materials for use in manned spacecraft crew compartment environments. The criteria was established with respect to production by offgassing of potentially toxic or objectionable and condensable volatiles. The volatiles were separated into three categories: CO, total offgassing, and observable condensates. The criteria of acceptability were total organics reported as µg/g using methane as a standard. The maximum allowable concentration of total organics in the tested configuration could not exceed 100 µg/g. Carbon monoxide in the tested configuration could not exceed 25 µg/g. Condensates were determined as visible or invisible, or as a function of any detection system selected by the testing agency in accordance with the requirements and criteria established by the program office. The test was conducted at the most hazardous test pressure and gas mixture conditions designated for the applicable program. The conditions of exposure were 155 ±5 °F for a total period of 72 hr. The gases were analyzed for total organic offgassing expressed as methane equivalents. Analysis for outgassing components exceeding 10 µg/g included but not limited to the organics benzene, xylene, methyl ethyl ketone, chloroform, n-butanol, dichloromethane, 1,4-dioxane, formaldehyde, trichloroethylene, and carbonyl fluoride; inorganics ammonia, HCN, HCl, and silicon tetrafluoride. If condensates occurred in the cold trap, they were recovered as gas, analyzed, and plotted. The quantity of CO evolved was determined. Outgassing components that exceeded 10 µg/g were identified and reported with their estimated concentrations. The CO content of the evolved gases was determined by an appropriate separation and analytical technique having the sensitivity to detect within 0.5 µg/g CO.

NHB 8060.1¹⁶ (November 1971), *Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion*, was issued as a NASA handbook (NHB) by the Office of Manned Space Flight. It was managed by the NASA Headquarters Reliability, Quality Assurance, and System Safety to establish uniform material selection, evaluation, and control criteria for all materials that are under consideration for use in and around manned spacecraft during flight and test operations. The contents of this NHB, based on the materials technology developed during the Apollo and Skylab Programs, was intended for implementation on all future NASA contracts and programs as applicable. The document provided standard requirements for control of flammability, odor and offgassing of materials to be used in the design and development of manned space vehicles; guidelines and directions for material selection; and testing procedures for the candidate materials used in and around manned space vehicles during flight and test operations. NHB 8060.1 Test 7 was a screening test for the suitability of nonmetallic materials for use in the space vehicle crew compartment environments. It identified Test No. 7, *Determination of Offgassing Products and Carbon Monoxide Test*. Criteria were established with respect to production of offgassing of potentially toxic or objectionable and condensable volatiles. The volatiles were separated into three categories: CO, total offgassing, and observable condensates.

The criteria for acceptability of a material were a maximum allowable concentration of total organics, excluding water, in the tested configuration not exceeding 100 µg/g, and the maximum allowable concentration of CO in the tested configuration not exceeding 25 µg/g.

The test pressure and gas mixture conditions for the pertinent materials category were designated by the requirements for each program. Test 7 exposures were in 95 percent of the test atmosphere (the test pressure and gas mixture conditions for the pertinent materials category were designated by the requirements for each program) at 155 ± 5 °F for a total period of 72 hr. Total offgassing (organic and nonorganic) was expressed as pentane equivalents. The identity of all products greater than $10 \mu\text{g/g}$ was reported. The identity of nonorganic species, and presence and quantity of condensables were among the required reporting results.

Following the designated exposure period, the evolved gases interfaced directly into the gas chromatograph while being maintained at 155 ± 5 °F. The basic calibration gas was methane. After the final gas samples were taken, the test specimen was removed from the chamber and weighed. The gases were then analyzed for total organic offgassing expressed as pentane equivalents and all outgassing components exceeding $10 \mu\text{g/g}$ were identified and quantified. These included but were not limited to the organics benzene, xylene, methyl ethyl ketone, n-butanol, chloroform, dichloromethane, 1,4-dioxane, formaldehyde, and trichloroethylene; and nonorganics HCN, HCl, ammonia, silicon tetrafluoride; and CO. Carbon monoxide was determined using an appropriate separation and analytical technique having the sensitivity to detect within $0.5 \mu\text{g/g}$ CO.

NHB 8060.1A¹⁶ (February 1974), *Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion*, was issued as an NHB by the Office of Manned Space Flight and managed by the NASA Headquarters Reliability, Quality Assurance, and System Safety. This was the next revision to NHB 8060.1 and established the criteria for selection of materials for use in future manned space vehicles and in equipment involving personnel in ground test facilities with environments that support combustion. The handbook noted that the designer and materials engineer must exercise care in evaluating the data from the tests specified herein since final acceptance of materials depends on their application in the spacecraft. Generally, materials were tested in their final configuration or a thorough analysis was performed of the test data in relation to the applications of the materials in the spacecraft to assure that no hazards existed. It identified Test No. 7, *Determination of Offgassing Products and Carbon Monoxide Test*. Exposures were at 95 percent of the test atmosphere at maximum use temperature, when designated, or 120 ± 5 °F for a total period of 72 hr. Total organic offgassing was expressed as pentane equivalents. The identity and quantity of all offgassing components exceeding $10 \mu\text{g/g}$, excluding water vapor and carbon dioxide, were reported. Condensables (organic and inorganic), were determined at room temperature after flushing from the test cell by solvent rinse or an equivalent method, and were among the required reporting results. The test pressure and gas mixture conditions for the specific materials category were designated by the program office, and these conditions represented the most hazardous atmosphere anticipated in the spacecraft.

Test 7 established the criteria for a screening test, which will determine the suitability of nonmetallic materials for use in the space vehicle crew compartment environments. The criteria were established with respect to production of offgassing of potentially toxic, or objectionable, and condensable volatiles. The products were separated into three categories: CO, organics and inorganics, and condensates. Definitions and terms were used in this test procedure:

- Offgassing: The evolution of gaseous products from a liquid or solid material.
- Condensables: The portions of offgassed products which condense on a cooled surface.
- Total Organic Offgassing: The total of the organic gaseous and condensed products which offgassed from a material. This does not include CO or carbon dioxide.
- Total Inorganic Offgassing: The total of the inorganic gaseous products and condensed products which offgassed from a material.

The criteria for acceptability of a material were a maximum allowable concentration of total organics, excluding water, in the tested configuration not exceeding $100 \mu\text{g/g}$. The maximum allowable concentration of CO in the tested material not exceeding $25 \mu\text{g/g}$. Inorganic gases such as HCN, ammonia, and HCl were recorded and evaluated by a toxicologist for potential toxicity levels.

The test pressure and gas mixture conditions for the pertinent materials were designated by the program office. These conditions represented the most hazardous atmosphere anticipated in the spacecraft. The conditions of exposure were the maximum use temperature, when designated, or 120 ± 5 °F for a total period of 72 hr.

The basic calibration gas was methane. Total organic offgassing was expressed as pentane equivalents. All offgassing components exceeding $10 \mu\text{g/g}$, excluding water vapor and carbon dioxide, were identified and quantified. These included but were not limited to the organics benzene, xylene, methyl ethyl ketone, n-butanol, chloroform, dichloromethane, 1,4-dioxane, formaldehyde, and trichloroethylene; and inorganics hydrogen cyanide, hydrogen chloride, and ammonia. Condensables (organic and inorganic) were determined at room temperature. The condensed products were flushed from the test cell by solvent rinse or an equivalent method. Offgassing components that exceeded $10 \mu\text{g/g}$ were identified and reported. The CO content or the evolved gases was determined using an

appropriate separation and analytical technique having the sensitivity to detect within 0.5 µg/g CO. The following special data and pertinent information were reported: CO in µg/g, total gas phase organic offgassing in µg/g as pentane equivalents, the identity of all gas phase organic products greater than 10 µg/g, total inorganic gas species in µg/g, and the identity and quantity of condensables.

NHB 8060.1B²⁴ (1981), *Flammability, Odor, and Offgassing Requirements and Test Procedures for Materials in Environments that Support Combustion*, was issued by the NASA Office of Space Transportation Systems and managed by the Reliability, Quality, and Safety Office at NASA Headquarters. This NHB was the next revision to supersede NHB 8060.1A.¹⁶ It was applicable to NASA installations responsible for hardware design, development, and testing. NHB 8060.1B²⁴ established uniform material selection, evaluation, and control criteria for all materials that are under consideration for use in or around space vehicles, ground support equipment, and facilities during assembly, test, and flight operations. The provisions of this handbook NHB 8060.1B was similar in intent and applicability as its predecessor NHB 8060.1A, though it contained changes to existing tests, including Test 7, and an additional related test, Test 16, *Determination of Offgassed Products from Assembled Articles*. It identified Test 7, *Determination of Offgassed Products Test*. Test 7 established the criteria for a screening test to determine the suitability of nonmetallic materials for use in the space vehicle crew compartment environments. The criteria were established with respect to production of potentially toxic volatile offgassed products. Definitions applying to the terms as used in the test procedure were:

- Offgassing. The evolution of gaseous products from a liquid or solid material.
- Offgassed Product. An organic or inorganic compound evolved from a liquid or solid material.
- Maximum Allowable Concentration (MAC). The maximum concentration of an offgassed product that is allowed in the spacecraft for a specified flight duration. MAC values for manned spacecraft are contained in Appendix D.

The criteria for acceptability of a material included the quantity of each offgassed product, as determined from either a standard quantity of a material or the actual quantity used in the spacecraft, would not result in a projected spacecraft concentration in excess of the MAC value for that product; the evaluation of the toxicological summation of the total offgassed products of a material for potential toxicity in accordance with the MAC guidelines by the responsible NASA toxicology group; and determination of the final acceptance of material by the responsible NASA medical office from an assessment of the potential toxicity of the total quantity of offgassed products from all contaminant generating items for a given mission.

Exposures were at a test pressure, temperature ± 3 °C for a period 72 ± 1 hr, and gas mixture conditions for the pertinent materials type designated by the cognizant center program office. These conditions represented the most hazardous atmosphere anticipated in the spacecraft. The test atmosphere was the worst-case atmosphere as defined by the applicable program office except that the pressure was slightly below ambient at the test laboratory. Test chamber atmospheres were blended from military specification grade nitrogen and oxygen. The identity and quantity of each analyzable offgassed product, excluding water vapor and carbon dioxide were reported. The calibration gas used with flame ionization detectors was propane and working standards were referenced to propane standards from the National Bureau of Standards. Results for organic and inorganic offgassed products and their quantities are reported in µg/g.

The calibration gas used with FIDs was propane; working standards were referenced to propane standards from the National Bureau of Standards. Any other instruments the test laboratory required to assure accuracy and precision in the offgassed products analyses were allowed.

The chamber containing the specimen was heated to test temperature ± 3 °C for a period of 72 ± 1 hr. Sampling and analysis of offgassed products was performed at room temperature and was initiated within 72 ± 1 hr of the conditioning period. The identity and quantity of each analyzable offgassed product, excluding water vapor and carbon dioxide, was reported. It was noted that some offgassed components may be present at concentrations too minute for identification. These were reported as “unidentified component” and the quantities expressed in µg/g.

NHB 8060.1C¹⁷ (April 2, 1991), *Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion*, was issued by the NASA Office of Safety and Mission Quality. This NHB superseded NHB 8060.1B²⁴ and established requirements for evaluation, testing, and selection of materials intended for use in space vehicles, associated ground support equipment, and facilities used during assembly, test, and flight operations. Included were requirements, criteria, and test methods for evaluating the flammability, odor, offgassing, and compatibility of materials. The provisions of this handbook were applicable to NASA installations. Application of this handbook was appropriate to the needs of specific programs. It identified *Determination of Offgassed Products* (Test 7). NHB 8060.1C identified *Determination of Offgassed Products* (Test 7).

Exposures of materials and assembled articles in Test 7 were in a test atmosphere of at least 20.9 percent oxygen with the balance nitrogen or argon (with specified maximum impurity limits), and the test pressure should be at ± 0.5 psi (3.5 kPa) of the ambient pressure of the test facility. The sample was subjected to a thermal exposure for 72 ± 1 hr at 120 ± 5 °F (49 ± 3 °C). The test report included sample identification, configuration, test conditions, and observations from the test. For each offgassed product, the quantities were reported as mg/g of material or mg/100 cm² of material or mg/assembled article. The methods of quantitative analysis must be traceable to a primary gas standard. Any standards used to quantify specific compounds must be traceable to the National Institute of Standards and Technology (nothing said about hydrocarbon equivalents). Test chambers were no longer constrained to 2 L, but containers with a volume greater than 100 L must have the capability of forced convection inside the container during offgassing and analysis.

NHB 8060.1C¹⁷ introduced new content, including specific good laboratory practices and required precision of measurements. The purpose of the test was to determine the identity and quantity of volatile offgassed products from materials and assembled articles. Definitions included:

- Assembled article. An assembled article could be any component or assembly of components that is not a single material.
- Offgassed product. An organic or inorganic compound evolved as a gas from a material or assembled article.
- Offgassing. The evolution of gaseous products from a liquid or solid material into an atmosphere.
- Spacecraft Maximum Allowable Concentration (SMAC). The maximum concentration of an offgassed product that is allowed in the habitable area of the spacecraft for a specified flight duration. SMAC values for manned spacecraft are listed in MAPTIS.
- Toxic Hazard Index (T). The T value is determined by calculating the ratio of the projected concentration of each offgassed product to its SMAC value and summing the ratios for all offgassed products without separation into toxicological categories. [Note: These are now also listed in JSC 20584 and their full content (derivation) was published in five volumes by the National Research Council (NRC). JSC Toxicology now publishes SMACs in the peer-reviewed literature. As they continue to be developed, the newer SMAC values will also be periodically incorporated into JSC 20584.]

If a sample was an assembled article, it was inspected for parts that are not designated for flight, such as dust covers, tape, or test leads, and these items were removed before testing. The absence of such items as batteries or photographic film, which will be included during flight but are not included with the sample, must be recorded. The ratio of sample volume to sample container volume should be approximately 1:3.

The test system was a sample container, test chamber with controlled temperature, and analytical instrumentation. The analytical instrumentation was not specified; however, the instrumentation should allow separation, quantification, and identification of all offgassed products at or below their SMAC concentrations when tested at 5 g of sample per liter of container volume. If the instrumentation cannot achieve this sensitivity, the minimum reportable concentration for those offgassed products must be reported. The recommended analytical instruments included a GC primarily using an FID, gas chromatography-mass spectrometry (GC-MS), and IR.

The pretest procedure included cleaning of sample containers, certification of container cleanliness, and calibration of the quantitative analytical instruments. Before loading the sample into the container, the container was filled with the test atmosphere or nitrogen and then conditioned for at least 72 hr at 120 °F (49 °C). Alternatively, the sample container was conditioned for at least 24 hr at 155 °F (69 °C). The sample container atmosphere was analyzed for residual contamination and was clean for use if the concentrations of residual gases were sufficiently low that they would not interfere with interpretation of results of the offgas analysis. The methods of quantitative analysis were traceable to a primary gas standard. Any standards used to quantify specific compounds was traceable to the National Institute of Standards and Technology.

The sample was weighed and placed in the sample container. The room atmosphere in the sample container was replaced with the test atmosphere; however, exposure of the sample to a vacuum must be less than 3 minutes. The sample container was then pressurized to a pressure that will be the requested test pressure when the test temperature is achieved. For samples that cannot withstand the evacuation procedure, the containers were purged with the test atmosphere, then brought to the pressure as required. After thermally conditioning, the sampling and analysis of the offgassed products was initiated within 24 hr from the time the container cooled to 72 °F (22 °C). The identity and quantity of each analyzable offgassed product was determined and reported. The test report included sample identification, configuration, test conditions, and observations from the test. For each offgassed product, the quantities were reported as mg/g of material or mg/100 cm² of material or mg/assembled article. The test report was submitted

to the NASA MAPTIS. When there was a deviation from standard test parameters, such as nonstandard sample preparation or test conditions, the test was identified as nonstandard.

NASA-STD-6001¹⁸ (February 9, 1998), *Reactivity of Materials in Aerospace Fluids* (Test 15) (Previously Published as NHB 8060.1C¹⁷), was issued as a NASA technical standard by Office of the Chief Engineer and identified *Determination of Offgassed Products* (Test 7). Test 7, along with other NASA standard tests from NHB 8060.1C, transitioned verbatim to the NASA Technical Standard, NASA-STD-6001, *Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion* on February 9, 1998. NASA-STD-6001 was previously published as NHB 8060.1C. With the exception of the cover and administrative information, NASA-STD-6001 was essentially a verbatim copy of NHB 8060.1C but had transitioned from the NASA Office of Safety and Mission Quality to the Office of the Chief Engineer (now a technical standard versus a safety standard). The paragraph numbering system, table numbers, and figure numbers remained unchanged.

NASA-STD-(I)-6001A¹⁹ (April 21, 2008), *Flammability, Offgassing, and Compatibility Requirements and Test Procedures, Determination of Offgassed Products* (Test 7), and NASA-STD-(I)-6001B²⁰ (April 21, 2008) *Flammability, Offgassing, and Compatibility Requirements and Test Procedures* was issued as a NASA technical standard by the Office of the Chief Engineer. It identified *Determination of Offgassed Products* (Test 7)^{§§}. Test 7 determined the offgassing characteristics under standardized conditions for materials and assembled articles to be located within habitable environments with no significant changes from NASA-STD-6001.¹⁸ Specimens were thermally conditioned for 72 ± 1 hr at 50 ± 3 °C (122 ± 5) °F).^{***} The test atmosphere should be at least a volume fraction of 20.9 ± 2 percent for oxygen with the balance nitrogen or argon, and the test pressure should be ± 15 kPa ± 2.18 psi of the ambient pressure of the test facility.

The next update to Test 7 after NHB 8060.1C¹⁷/NASA-STD-6001¹⁸ was NASA-STD-(I)-6001A¹⁹, *Flammability, Offgassing, and Compatibility Requirements and Test Procedures*, an interim NASA technical standard issued by the Office of the Chief Engineer that was approved April 21, 2008 and identified *Determination of Offgassed Products* (Test 7). It remained mostly unchanged with respect to Test 7 in the next interim standard update, NASA-STD-(I)-6001B,²⁰ *Flammability, Offgassing, and Compatibility Requirements and Test Procedures*, which was approved November 11, 2009 (though the cover page retained the April 21, 2008 approval date). It also incorporated ISO 14624-3.⁴ Because of the number of exceptions, clarifications, and additions, both the NASA and the ISO documents were required in order to perform the test.

NASA-STD-6001A was never issued. The test methods described in both NASA-STD-(I)-6001A¹⁹ and NASA-STD-(I)-6001B²⁰ were identical, incorporating ISO 14624-3,⁴ with a number of exceptions, clarifications, and additions. An interim NASA standard is given an “-(I)-” designation and represents the technical consensus of the developing group but does not have final NASA approval. Interim NASA standards provide uniform engineering and technical requirements for processes, procedures, practices, and methods to meet urgent program and project technical needs, have the consensus of the developing group (Technical Working Group), but do not have the Agency-wide concurrence required for a NASA Technical Standard. The interim NASA-STD-(I)-6001A and NASA-STD-(I)-6001B standards were approved for use by NASA Headquarters and NASA Centers, including Component Facilities and Technical and Service Support Centers. The interim standards established requirements for evaluation, testing, and selection of materials that are intended for use in space vehicles, associated Ground Support Equipment (GSE), and facilities used during assembly, test, and flight operations. Included are requirements, criteria, and test methods for evaluating the flammability, offgassing, and compatibility of materials. The provisions of the standard, as with other NASA standards, will be included, as applicable, in all future contracts and programs involving space vehicles, payloads, and associated support equipment.

Interim standards NASA-STD-(I)-6001A¹⁹ and NASA-STD-(I)-6001B,²⁰ *Flammability, Offgassing, and Compatibility Requirements and Test Procedures*, were issued by the NASA Office of the Chief Engineer and were developed to provide the most recent standard test methods, including Test 7. NASA-STD-(I)-6001A was approved April 21, 2008, expired April 20, 2009, and, after the correction of a typographical error in units in an unrelated test (Test 6) and several administrative updates, NASA-STD-(I)-6001B was approved November 10, 2009, expired April

^{§§} The test method described in the baseline ISO 14624-3,⁴ *Space Systems—Safety and Compatibility of Materials—Part 3: Determination of Offgassed Products from Materials and Assembled Articles*, was adopted for this test, and in NASA-STD-6001 revisions thereafter with defined multiple exceptions, clarifications, and additions.

^{***} The standard test temperature was raised 1 °C (2 °F) from NHB 8060.1C/NASA-STD-6001 was a concession to European preference. 50 °C was specified by ESA-PSS-01-729-ISSUE-1³² (1989) specification. ECSS-Q-ST-70-29C³³ (2008) also specifies 50 °C.

21, 2010, and was given the same April 21, 2008 approval date as NASA-STD-(I)-6001A. Both NASA-STD-(I)-6001A and NASA-STD-(I)-6001B superseded NASA-STD-6001.¹⁸ NASA-STD-(I)-6001A and NASA-STD-(I)-6001B were both developed with the joint cooperation of a technical working group consisting of members of several NASA Centers, most of which had direct experience with the tests.

As in NHB-8060.1C¹⁷/NASA-STD-6001,¹⁸ the interim NASA-STD-(I)-6001A¹⁹ and NASA-STD-(I)-6001B²⁰ standards included Test 7, *Determination of Offgassed Products (Test 7)*, as the required test for material materials and components offgassing (Test 16 still applied to assembled articles, and Test 12 still applied to spacecraft). The purpose of this test is to determine the identity and quantity of volatile offgassed products from materials and assembled articles under standardized conditions for materials and assembled articles to be located within habitable environments. Perhaps the most noteworthy change was that Test 7 now adopted and tailored the voluntary consensus standard (VCS) ISO 14624-3,⁴ *Space Systems—Safety and Compatibility of Materials—Part 3: Determination of Offgassed Products from Materials and Assembled Articles*, but with numerous exceptions, clarifications, and additions required to make the test conform with NASA requirements. In order to perform Test 7, two documents were now required: ISO 14624-3 and NASA-STD-(I)-6001A, where ISO 14624-3 provided the basic procedure and NASA-STD-(I)-6001A provided the exceptions, clarifications, and additions. The toxicity calculations were greatly elaborated compared to earlier versions of Test 7. Calculating toxicity values was formerly the function of a specialized toxicologist at JSC. The conditions of exposure were 72 ± 1 hr at 50 ± 3 °C (122 ± 5 °F), rather than 120 ± 5 °F (49 ± 3 °C). The change in temperature was believed to be a concession to the ISO 13624-3 working group.

After thermal conditioning, the atmosphere inside the specimen container was analyzed to determine the offgassed compounds. Using the SMAC for each offgassed compound, the overall toxicity rating was determined. The overall rating of each material or assembled article determined the quantity of each material or the number of assembled article units that can be flown. For a material or assembled article, the total T values for all volatile offgassed products shall be less than 0.5.

All materials were evaluated individually using the ratings in the MAPTIS database (<http://maptis.nasa.gov/>). MAPTIS specifies the maximum quantity and associated rating for each material code.

Analytical instrumentation was capable of the identification and quantification of all offgassed products at or below their SMAC concentrations when tested at a test-material-mass-to-specimen-container-volume ratio of 5.0 ± 0.25 g/L. If the instrumentation cannot achieve this sensitivity, the minimum reportable concentration (reporting limit) for those offgassed products (except for formaldehyde) shall be reported. For formaldehyde, the analytical technique shall be capable of detecting a concentration of 0.1 ppm or current SMAC.

The recommended analytical instruments included a GC primarily using an FID, a GC-MS, and an IR, but also specified that special methods may be required to identify and quantify difficult to detect compounds. For example, the determination of formaldehyde may be performed using the proposed method of trapping on 2,4-dinitrophenylhydrazine cartridges for derivation and subsequent analysis by high performance liquid chromatography.

In some cases, the reporting limit for a compound is greater than the SMAC for that compound, but the ability to meet the SMAC requirement is known to be crucial. To determine if the compound offgasses at the SMAC, it is necessary to test more than the standard test-material-mass-to-specimen-container-volume ratio of 5.0 ± 0.25 g/L. The quantity of test material should be increased proportionally from the test-material-mass-to-specimen-container-volume ratio of 5.0 ± 0.25 g/L to a quantity that will allow the analysis to meet the SMAC requirement.

Test 7 target compounds used by NASA are found in ISO 14624-3⁴ Annex B. These include formaldehyde, methanol, benzene, vinyl chloride, acetonitrile, acetaldehyde, dichloromethane, ethylene oxide, 3-butene-2-one, 2-methyl-2-propenal, trichloroethene, 1,3-butadiene, propenal, chloropropene (as 3-chloropropene), 1,2-dichloroethane, acrylonitrile, furan, heptanal, 2-butenal, methylfuran (as 2-methylfuran), 2,5-dimethylfuran, CO, and ammonia.

NASA-STD-(I)-6001A,¹⁹ NASA-STD-(I)-6001B,²⁰ NASA-STD-6001B,² and NASA-STD-6001B w/Change 2²⁵ note an exception relevant to the gas standards described in ISO 14624-3⁴: The analysis of furfural in Mix B (as defined in ISO 14624-3:2005) was not required; however, this compound was useful as a diagnostic tool because it presented a meaningful challenge to the analytical system.

An addition added reportable quantities: This amount shall be determined by each analytical laboratory and based on analyzed concentrations of the specific compound. Explanatory material was offered: Compounds that have been identified, but for which the specific compound is unavailable as a standard, may have reportable quantities based on analyzed concentrations of a representative compound.

ISO 14624-3 Annex B provides a target compound list; no exceptions or additions are provided by NASA-STD-(I)-6001A,¹⁹ NASA-STD-(I)-6001B,²⁰ NASA-STD-6001B,² or NASA-STD-6001B with Administrative Change 2²⁴.

Non-mandatory guidance in both revisions stated the recommended analytical instruments include a GC, primarily using an FID, a GC-MS, and an IR spectrophotometer. Some analytical compounds may be more difficult to determine; therefore, special methods may be required to identify and quantify these compounds. For example, the determination of formaldehyde may be performed using the proposed method of trapping on 2,4-dinitrophenylhydrazine cartridges for derivation and subsequent analysis by high performance liquid chromatography.

Both NASA-STD-(I)-6001A¹⁹ and NASA-STD-(I)-6001B²⁰ stated in paragraph 7.7.4 Test Method p.: Addition (section 10.2 (to ISO 14624-3⁴)): If any strong odors are detected during the Test 7 procedures, the test lead shall note this and shall recommend that Test 6 (*Odor Assessment*) be performed.

NASA-STD-6001B² (August 26, 2011), *Flammability, Offgassing, and Compatibility Requirements and Test Procedures*, identifies *Determination of Offgassed Products* (Test 7). The procedure is identical to NASA-STD-(I)-6001A¹⁹ and NASA-STD-(I)-6001B.²⁰

The next update to Test 7 after the interim NASA-STD-(I)-6001A¹⁹ and NASA-STD-(I)-6001B²⁰ was NASA-STD-6001B.² Issued by the Office of the Chief Engineer, only minor changes/clarifications were made as NASA-STD-(I)-6001A and NASA-STD-(I)-6001B progressed to NASA-STD-6001B. For example, paragraph 7.7.2e (Test Summary) in NASA-STD-(I)-6001A was modified from “The overall rating of each material or assembled article shall determine the quantity of each material or the number of assembled articles that can be flown” to “The overall rating of each material or assembled article shall determine the quantity of each material or the number of assembled articles that can be flown concurrently,” with “concurrently” as the only addition.

NASA-STD-6001B w/Change 2²⁵: Administrative/Editorial Changes (April 21, 2016), *Flammability, Offgassing, and Compatibility Requirements and Test Procedures, Determination of Offgassed Products* (Test 7) was issued by the Office of the Chief Engineer. No changes were made to Test 7 from the previous version published in NASA-STD-6001B² (August 26, 2011), *Flammability, Offgassing, and Compatibility Requirements and Test Procedures, Determination of Offgassed Products* (Test 7).

A. Analytical Instrumentation

Major technical advancements of key instrumentation have been made since 1966 when Test 7 was first implemented. These include GC, GC-MS, and Fourier transform infrared (FTIR) spectroscopy, high performance liquid chromatography, hyphenated techniques such as GC with FTIR detection (GC-FTIR), data acquisition, and instrument software, the development and implementation of capillary columns instead of previously used packed columns, cryotrapping and cryofocussing techniques, improved interfaces, and ever-increasing sensitivity of instrumentation and purity of gases used in the test. The development and use of mass spectral libraries combined with improved separation and lower detection limits have greatly advanced the quality of data. The ability to detect and identify compounds not previously detectable has seen great advancements over the years due to advances in analytical instrumentation and techniques. Supporting systems such as programs that calculate toxicity values for offgassed items have become increasingly significant and helpful. These improvements have greatly enhanced the field of space toxicology and the ability of Test 7 to provide data of increasing value for the protection of the life, health, and safety of the astronauts.

B. Simulated Use Testing

Test 7 may be performed in a simulated use condition. Specifics are defined by test requests, but in general, a material or assembled article is placed in a test chamber, which may be a glove box or bell jar equipped with feed through and plumbed to sample the atmosphere or interface it with the required instrumentation. The material or assembled article is then activated in a manner and duration specified by the requestor before the test atmosphere is analyzed. Examples of simulated use tests include operation of a printer and a copy machine, cleaning and painting operations, application of ink from drawing devices, mixing epoxies, opening a bag containing an emergency escape mask, and operation of a three-dimensional (3D) printer.

C. Comments on Related Tests: Test 6, *Odor Assessment*, Test 16, *Determination of Offgassed Products from Assembled Articles*, and Test 12, *Total Spacecraft Cabin Offgassing*

These tests have or had relevance to Test 7 and are briefly discussed.

1. Test 6 (Odor)

Test 6 is an odor evaluation test established in ASPO-RQTD-D67-5A²¹ (May 3, 1967), *Apollo Spacecraft Program Office Nonmetallic Materials Selection Guidelines*, as “Odor and Carbon Monoxide.” If the Test 6 passed CO, the test material progressed to Test 7 (Total Organic). Test 6 was revised in MSC-A-D-66-3 Revision A⁹ (1967), MSC-A-D-66-3⁵ could not be found, to *Odor Test*, while CO was integrated into Test 7, *Determination of Organic Offgassing Products and Carbon Monoxide*. MSC-PA-D-67-13⁶, *Apollo Spacecraft Nonmetallic Materials Requirements* (February 9, 1968), Test No. 6 *Odor Test* stated the purpose of the test was to identify materials that are undesirable to the olfactory senses. A material that failed the test shall not be used in the habitable areas of the spacecraft. All materials subject to the odor test shall have previously been tested for CO content and total organics before being subjected to the odor test. If a material yielded over 25 micrograms of CO/gram of material, or over 200 micrograms of total organics/gram of material, no odor test was allowed and the material was rejected for odor (*sic* – carbon monoxide) and total organics. Carbon monoxide and total organics testing was performed by Test No. 7, *Determination of Carbon Monoxide and Total Organics*. MSC-PA-D-67-13 Addendum No. 1^{7,22} required a change to the test specified in MSC-PA-D-67-13,⁶ in which “All materials shall have been-tested for carbon monoxide (CO) and total organics (TO) contents before being subjected to the odor test. If the CO or TO concentrations of the gas sample exceed the maximum personnel safety limits of the test agency, the sample shall be diluted with pure oxygen until the maximum safe level is attained. This diluted sample will then be tested according to section 7.0. The results shall specify the dilution required to attain the safety limit and shall be reported as a special test.”

Test 6 progressed as a required test with changes including criteria for performance based on Test 7. NHB 8060.1A¹⁶ noted that, “The results of Test No. 7 shall be reviewed before conducting this test so that members of the odor team are not inadvertently exposed to toxic offgassing products.” Other changes included testing procedure, criteria for odor panel members, test equipment (olfactometer/mask terminology), human research policy, allowable time from offgas to odor test, odor panel standards formulations, and others until NASA-STD-(I)-6001A,¹⁹ where Test 6 was no longer required. Test 6 was not deleted in NASA-STD-(I)-6001A but became a supplemental test (with a name change to “Odor Assessment” that remained through the NASA-STD-6001B w/Change 2²⁵ to Habitable Flight Compartments and to Breathing Gases environments. As discussed in NASA-STD-(I)-6001A, supplemental tests may be conducted to support the system hazard evaluation for materials that do not meet the criteria of the required tests; data based on other appropriate and applicable test methods also may be used to support the evaluation. Test 6 remains a supplemental test in NASA-STD-6001B.² Another relationship between Test 6 and Test 7 was specified in NASA-STD-(I)-6001A, where it was stated in an addition to the adopted and tailored ISO 14636-3 that, “If any strong odors are detected during the Test 7 procedures, the test lead shall note this and shall recommend that Test 6 (*Odor Assessment*) be performed.” This addition persisted through NASA-STD-6001B w/Change 2.

2. Test 12 (Spacecraft Offgassing)

NASA-TM-79493¹³ (December 1970) introduced Test No. 12, *Guidelines for Total Spacecraft Offgassing Test*. This test was designed to measure the total quantity and individual components of the products offgassed by a spacecraft during manned and unmanned chamber tests and, thereby, determine the toxicological safety of the spacecraft during manned flight. Test 12 was similar to a Test 7 except that a spacecraft, unmanned and manned, was both the test chamber and the test article.

A chronology of Test 12 appearances in the NASA standard test documentation reviewed is as follows:

- NHB 8060.1, Test No. 12, *Guidelines for Total Spacecraft Offgassing Test*
- NHB 8060.1A, Test No. 12, *Guidelines for Total Spacecraft Offgassing Test*
- NHB 8060.1B, Test 12, *Total Spacecraft Cabin Offgassing Test*
- NHB 8060.1C, Test 12, *Total Spacecraft Offgassing*
- NASA-STD-6001, Test 12, *Total Spacecraft Offgassing*
- NASA-STD-(I)-6001A, Test 12, Total Spacecraft Offgassing. Deleted.
- NASA-STD-(I)-6001B, Test 12, Total Spacecraft Offgassing. Deleted.

In the NHB 8060.1 description of Test 12, testing of the spacecraft interior areas of interest was performed in two configurations: unmanned and manned. Spacecraft conditions such as power on requirements were not specified, but the test pressure and gas mixture conditions to be used during the test were designated in accordance with the requirements of each program and shall not be less, quantitatively, than those used in the manned flight vehicle. Gas sampling lines from the spacecraft to the analytical equipment provided the capability for continuous on-line, real-time

monitoring of carbon dioxide, nitrogen, oxygen, and other spacecraft atmospheric contaminant concentration. This capability necessitated use of a carbon dioxide analyzer, oxygen analyzer, nitrogen analyzer, mass spectrometer, and any necessary instruments selected from Test No. 7 (GC-MS, GC with a minimum of two detectors, thermal conductivity and hydrogen flame ionization units with appropriate recorders, and an electron capture detector as a recommended supplementary detector), and infrared spectrometer. A cold (40 °F) water trap was located upstream of the on-line real-time instrumentation and a cryogenic trapping system was required. Cryogenically trapped samples were collected from the cabin and analyzed during all phases of the test. Posttest, the cabin gas sampling line was purged with high purity nitrogen as soon as possible after completion of the exposure period to remove and cryogenically trap any condensables (a maximum of 18 hr was allowed). The line was heated to 170 ± 5 °F during purging and continued for one hour after purging. The cryogenic traps were collected and analyzed. The water trap, upstream of the on-line real-time instrumentation and the cryogenic trapping system, was dismantled after the nitrogen purge and analyzed.

The NHB 8060.1A¹⁶ Test 12 procedure was similar to that in NHB 8060.1¹⁵. The NHB 8060.1B²³ Test 12 procedure changed modestly from NHB 8060.1A with samples taken from the spacecraft cabin atmosphere in sets of three gas cylinders rather than continuous monitoring, and no cryogenic traps or water traps were included. “All possible flight hardware” were required onboard the spacecraft at the time of the test, and all flight hardware not available must be subjected to Test 16. NHB 8060.1C¹⁷/NASA-STD-6001¹⁸ brought further modifications to Test 12 with the implementation of specific good laboratory practices and a requirement that grab sample sets be obtained at intervals sufficient to establish a plot of the offgassing trend, which allowed the JSC Toxicology Group to predict toxicity of the vehicle air at first ingress following docking with a space station. At least five samples of the atmosphere were collected. In the next iteration, NASA-STD-(I)-6001A¹⁹ deleted Test 12 and it remained deleted through NASA-STD-6001B.²

In perhaps another embodiment of Test 12 or a similar test, Musgrave, Larsen, and Sgobba²⁶ (2009) discussed spacecraft module testing. When a large module, such as Spacelab, Spacehab, or an International Space Station segment is planned to fly for the first time, offgas testing of the whole module is performed by a “special procedure.” Ideally, the module is to be outfitted fully with all cargo onboard before the test. Musgrave, Larsen, and Sgobba discuss that this is rarely feasible and a correction has to be made based on the weight of nonmetallic materials that are not present. The module is sealed up, and samples of the atmosphere are taken at multiple locations over a period of several days. The specifics of the test are customized for the application.

3. Test 16 (Determination of Offgassed Products from Assembled Articles)

Test 16 made a single appearance in NHB 8060.1B²⁴ after which it was combined with Test 7. Test 16 established the criteria for a screening test to determine the suitability of assembled articles for use in space vehicle crew compartment environments. This test established was a screening test to determine the suitability of assembled articles for use in space vehicle crew compartment environments. The criteria were established with respect to production of potentially toxic volatile offgassed products. An assembled article was defined as any component “black box” or assembly of components that represents the article to be used in a spacecraft. The quantity of each offgassed product of an assembled article shall not have resulted in a projected spacecraft concentration in excess of the MAC value for that product. The test conditions for the pertinent materials category were designated by the cognizant NASA Center program office, and these test variables represented the most hazardous condition anticipated in the spacecraft. The test was fundamentally similar to Test 7, and results included the identity of organic and inorganic offgassed products and their quantities in μg (per article), rather than $\mu\text{g/g}$ for a material.

4. Test 7 (Condensates)

Several of the early Test 7 procedures discuss condensates/condensables and their analysis and reporting. Offgassed and condensed products are different (gas versus liquid phase). In more recent times, the determination of volatile condensable products is performed independent of Test 7 in accordance with NASA SP-R-0022,²⁷ *Vacuum Stability Requirements of Polymeric Material for Spacecraft Application* or ASTM E595,²⁸ *Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment*. The incorporation of condensed phase offgassed products as analytes in Test 7 and attention to them in Test 7 appeared to reach a maximum in NHB 8060.1A,¹⁶ where it was clearly stated that, “Condensables (organic and inorganic) shall be determined at room temperature. The condensed products shall be flushed from the test cell by solvent rinse or an equivalent method.” Analysis and characterization of condensed products were phased out of Test 7 in subsequent revisions of NHB 8060.1 and NASA-STD-6001.

ASTM E595²⁸ *Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment* promulgated in 1977 described a screening technique to determine the total volatile content of materials when exposed to a vacuum environment. Two parameters were measured: total mass loss (TML) and collected volatile condensable materials (CVCM). An additional parameter, the amount of water vapor regained (WVR), could also be obtained after completion of exposures and measurements required for TML and CVCM. Subsequent revisions of ASTM E595 were issued in 1984, 1990, 1993, and thereafter. SP-R-0022²⁶ (1969) and later SP-R-0022A²⁹ (1974), *Specification Vacuum Stability Requirements of Polymeric Material for Spacecraft Application*, were earlier NASA specifications. ASTM E595 eventually replaced the analysis and characterization of condensable materials and was largely ASTM’s version of the original NASA procedures. NASA-STD-(I)-6016³⁰ (2006) and NASA-STD-6016³¹ (2008) called out the use of ASTM-E595 for thermal vacuum stability testing of nonmetallic materials exposed to space vacuum and gave acceptance criteria of ≤ 0.1 percent collected volatile condensable materials (CVCM) and ≤ 1.0 percent total mass loss (TML).

D. International Standardization

The European Space Agency (ESA) document ESA-PSS-01-729-ISSUE-1³²(1989), *The Determination of Offgassing Products from Materials and Assembled Articles to be used in a Manned Space Vehicle Crew Compartment*, was a specification defining a test procedure for the determination of the trace contaminants released by nonmetallic materials under a set of closely controlled conditions. The test procedure covered both individual materials and assembled articles. The test provided data for aid in the evaluation of the suitability of assembled articles and materials for use in a space vehicle crew compartment. The data obtained concerned the nature and quantity of organic and inorganic volatile contaminants evolved when the articles and materials were subjected to the crew compartment environment. Preparatory conditions, test performance, acceptance limits, and quality assurance were covered. NASA NHB 8060.1B²⁴ was referenced in this document. The basic equipment necessary for the performance of this test consisted of a sealed test chamber, a sampling capability, and the necessary analytical equipment (gas sampling system, GC with a minimum of an FID, MS preferably GC-MS, and IR). The test conditions were 50 °C in normal air (21 percent O₂), 1 atmosphere pressure (at 50 °C), and 72 hr. The sample analysis identified the quantity of CO offgassed in $\mu\text{g/g}$ of material tested, quantity of total organics (expressed as pentane equivalents) offgassed in $\mu\text{g/g}$ of material tested, and the identity and quantity of each contaminant offgassed in an amount in excess of 10 $\mu\text{g/g}$ of material tested. As a preliminary screening test, a material must offgas concentrations lower than 25 $\mu\text{g/g}$ CO, 100 $\mu\text{g/g}$ total organics, and any organic present in a concentration >10 $\mu\text{g/g}$ should be identified.

The European Space Agency document ECSS-Q-ST-70-29C³³ (2008), *Space Product Assurance – Determination of Offgassing Products from Materials and Assembled Articles to be used in a Manned Space Vehicle Crew Compartment*, is one of the series of the European Cooperation for Space Standardization (ECSS) standards intended to be applied together for the management, engineering, and product assurance in space projects and applications. ECSS-Q-ST-70-29C defines a test procedure for the determination of the trace contaminants release by nonmetallic materials under a set of closely controlled conditions. The test procedure covers both individual materials and assembled articles. Test conditions are 50 ° ± 2 °C in an atmosphere of at least 20.9 percent oxygen, with the balance nitrogen or argon, 1 atmosphere pressure (at 50 °C), and a duration of 72 ± 1 hr. The sample analysis shall identify the quantity of CO offgassed in $\mu\text{g/g}$ of material tested, the quantity of total organic in $\mu\text{g/g}$ of material tested (expressed as pentane equivalents), and identity and quantity of each contaminant offgassed in a concentration in excess of 10 $\mu\text{g/g}$ of material tested. This specification gave informative analytical instrumentation and parameter information. NASA-STD-6001¹⁸ was provided as a reference.

The International Organization for Standardization (ISO) promulgated ISO 14624-3⁴, *Space Systems—Safety and Compatibility of Materials—Part 3: Determination of Offgassed Products from Materials and Assembled Articles* in 2006 with defined exceptions, clarifications, and additions specified in each NASA-STD-6001.¹⁸ A product of Technical Committee 20, *Aircraft and Space Vehicle*, Subcommittee 14, *Space Systems and Operations*, and Working Group 1, *Design Engineering and Production*, ISO 14624-3 corresponded in part with NHB 8060.1C¹⁷/NASA-STD-6001¹⁸ Test 7 from which they were derived, but was unsuitable for NASA use as a stand-alone document due to the additions, exceptions, and clarifications established by NASA to meet NASA test requirements in NASA-STD-6001 revisions that reference ISO 14624-3.

The British Standards Institution (BSI) adapted ISO 14624-3 shortly after it was issued and published BSI BS ISO 14624-3,³⁴ *Space Systems—Safety and Compatibility of Materials—Part 3: Determination of Offgassed Products from Materials and Assembled Articles* (2006). This British Standard reproduces verbatim ISO 14624-3:2005 and implements it as the United Kingdom national standard. The BSI standard correspondingly provides less detail than the NASA documents from which they were derived and does not stand alone for NASA use due to the additions, exceptions, and clarifications established by NASA in NASA-STD-6001 revisions that reference ISO 14624-3. NASA does not refer to any BSI standards in any of its standard test documents.

V. Conclusion

NASA Test 7 standard test protocols appear to originate with the Apollo program in 1967 and progressed throughout Skylab, the Space Shuttle Program, and International Space Station to the present edition used in 2019. Test 7 is actively used to qualify materials and articles used for a variety of current NASA programs. The test evolved in a number of ways; Apollo-era tests were conducted in oxygen atmospheres and current tests are performed in atmospheres specified by a program. The type of analytical instrumentation called out in the test protocols did not change much because GC, MS, GC-MS, and IR were all available since 1967; however, advances and improvements to those instruments enabled greater sensitivity and ability to detect compounds. Based on history, the authors do not foresee any reinstatement of historical methods, but do foresee a continuation of standard test conditions and continuous improvement in analytical methods as the need to identify and quantitate (at increasingly low concentrations) as the list of toxicologically important compounds also grows.

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