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DEPARTMENT OF DEFENSE

TEST METHOD STANDARD

TEST PROCEDURES FOR PACKAGING MATERIALS



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FOREWORD

1. This standard is approved for use by all Departments and Agencies of the Department of Defense (DoD).

2. This standard was developed to document uniform test methods for the evaluation of materials used in military packaging applications. Reference to the test methods included herein ensures standardization of testing procedures and also eliminates unnecessary repetition of detailed test instructions within each individual packaging material specification.

3. The test methods included herein were previously documented in FED-STD-101. FED-STD-101A contained 250 test methods. This number was reduced to 172 in FED-STD-101B, then to 31 in FED-STD-101C, by deleting test methods no longer deemed relevant or as a result of supersession by industry standards. The remaining 31 standard test methods have recently been reviewed to verify current need and extent of usage. This review indicated that only 15 unique test method standards are required to support the testing of materials used in military packaging applications. These 15 test methods have been carried over to this Military Standard. A complete chronology of the above-mentioned history of packaging material standard test methods is included herein as Appendix A.

4. Comments, suggestions, or questions on this document should be addressed to: Commander, Naval Air Warfare Center Aircraft Division, Highway 547, Code 491000B120-3, Lakehurst, NJ 08733-5100 or emailed to thomas.omara@navy.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <http://assist.daps.dla.mil>.

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CHRONOLOGY OF PACKAGING MATERIALS TEST METHODS

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1. SCOPE

1.1 Scope. This document provides a centralized listing of detailed and uniform test methods that have been developed to evaluate relevant properties of materials used in military packaging applications. These standardized test methods may be referenced by number in specific packaging material specifications, as applicable, thus eliminating the need to repetitively detail the standard test method in each material specification.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, or 5 of this standard. This section does not include documents cited in other sections of this standard or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in section 3, 4, or 5 of this standard, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications and standards. The following specifications and standards form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

FEDERAL SPECIFICATIONS

QQ-S-698 - Steel, Sheet and Strip, Low Carbon.

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-PRF-131 - Barrier Materials, Watervaporproof, Greaseproof, Flexible, Heat-Sealable.

(Copies of these documents are available online at <http://assist.daps.dla.mil/quicksearch/> or <http://assist.daps.dla.mil> or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

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2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM) INTERNATIONAL

- ASTM-D471 - Rubber Property – Effect of Liquids, Standard Test Method for. (DoD adopted)
- ASTM-D996 - Standard Terminology of Packaging and Distribution Environments. (DoD adopted)

(Copies of these documents are available online at www.astm.org or from the American Society for Testing and Materials International (ASTM), 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.4 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. DEFINITIONS

3.1 General. Definitions of terms unique to this standard are listed below. Definitions of other terms commonly used in the packaging community may be found in ASTM-D996.

3.1.1 Blocking. Cohesion or adhesion between contiguous layers of similar or dissimilar materials in roll or sheet form which interferes with the satisfactory and efficient use of the material.

3.1.2 Curl. Tendency of an unrestrained flat strip of material to roll-up on itself.

3.1.3 Leak. Any opening in a container, contrary to design intent, that either allows the contents to escape or permits substances to enter.

3.1.4 Stain. Color changes formed only on a surface without any evidence of rust, pitting, etching, or deterioration of the surface.

3.2 Water vapor transmission rate (WVTR). The mass of water vapor transmitted through a given area of test material in a given time when the test material is maintained at a constant temperature, and when one surface is exposed to very low relative humidity and the other surface to a high relative humidity.

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4. GENERAL REQUIREMENTS

4.1 Test method format. The standard format used to describe packaging material test methods herein is as follows:

- a. Scope
- b. Apparatus
- c. Test specimens
- d. Test procedure
- e. Notes

4.2 Test documentation format. Documentation shall contain, as a minimum, the following information:

- a. A statement that the test was conducted in compliance with the procedure(s) detailed herein, or a description of any deviations from same.
- b. Identification of each specimen/material tested.
- c. Results of the test.
- d. An indication of compliance or non-compliance with specification requirements.

4.3 Test methods. Each standard test method is described in detail in section 5. The test method numbers have been retained from their originally assigned designations (see Appendix A).

4.4 Test room conditions. Unless otherwise specified, all testing specified herein shall be conducted on test specimens that have been conditioned for at least 24 hours in a test room maintained at 73 ± 3.5 °F and 50 ± 5 percent relative humidity.

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5. DETAILED REQUIREMENTS

5.1 Dimensional properties test methods.5.1.1 Test Method 1003 – Thickness.

5.1.1.1 Scope. This test procedure details a method for determining the thickness of flexible packaging materials. Method A is intended for use as rapid thickness test. Method B shall be used in laboratory thickness measurements.

5.1.1.2 Apparatus.

5.1.1.2.1 Method A. A handheld digital caliper reading in thousandths of an inch and calibrated accurate to within 0.001 inch.

5.1.1.2.2 Method B. A dial micrometer reading in thousandths of an inch and calibrated to be accurate within 0.5 percent or 0.001 inch, whichever is greater. The micrometer shall be securely mounted on a rigid test stand so that it measures the travel of a movable platen from a fixed base plate. The face of the movable platen shall be not less than 0.03 in², shall be not greater than 0.15 in², and shall travel perpendicular to the base plate. Contact pressure shall be not less than 1.5 lbs/in² and shall be not greater than 2 lbs/in².

5.1.1.3 Test specimens. Five specimens, each measuring 2 by 2 inches, shall be selected at random from representative locations of the material being evaluated.

5.1.1.4 Test procedures.

5.1.1.4.1 Method A. The digital caliper shall be zeroed by closing it manually and activating the zeroing mechanism. To make a measurement, the digital caliper shall be closed manually on a sample so that the sample is between all mating faces of the caliper. Within 5 seconds the caliper shall be read to the nearest 0.001 inch.

5.1.1.4.2 Method B. The dial of the micrometer shall be zeroed with the movable platen flush against the base plate at the above contact pressure. The test specimen shall be placed on the base plate so as to be centered beneath the movable platen. The movable platen shall then be lowered onto the specimen so as not to impact load the surface. Within 5 seconds, the micrometer shall be read to the nearest 0.001 inch.

NOTE

When 0.0001 inch accuracy is required, the thickness of 10 stacked specimens shall be determined and divided by 10.

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5.2 Strength and elastic properties test methods.5.2.1 Test Method 2015 – Curl.

5.2.1.1 Scope. This test procedure details a method to determine the curling tendencies of barrier materials when exposed to room temperature.

5.2.1.2 Test specimens. Specimens shall be selected at random to adequately represent any variation of the material being evaluated. Each specimen shall measure $12 \pm \frac{1}{16}$ inches by $36 \pm \frac{1}{16}$ inches. Six specimens shall be tested, three taken from each principal direction of the material.

5.2.1.3 Test procedure. The specimens shall be placed on a horizontal surface with the heat sealable (or inside) surface face up. After 30 minutes, the apparent horizontal length shall be measured to the nearest $\frac{1}{16}$ inch to determine the curling tendencies of the material. If any curling is observed in the opposing direction, specimens shall also be turned upside down for an additional 30 minutes and the apparent horizontal length measured. Percent curl shall be calculated as follows:

$$\text{Curl (\%)} = \frac{(36 - \text{Curled length}) \times 100}{36}$$

If the specimen curls back on itself so that contact is made, this shall be noted.

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5.2.2 Test Method 2017 – Flexing Procedure for Barrier Materials.

5.2.2.1 Scope. This test procedure details standard methods for repeatedly flexing barrier materials to simulate their use and handling.

5.2.2.2 Apparatus.

5.2.2.2.1 A Gelbo Flex-Tester or equivalent, as approved by the qualifying activity, shall be used to provide a standard flexing motion to materials being tested. The testing apparatus shall consist essentially of a 3 ½-inch diameter stationary head and a 3 ½-inch diameter movable head spaced at a distance of 7 inches from face-to-face at the starting position of the flexing stroke. The specimen supporting shoulders on each head are ½ inch wide. The motion of the movable head is controlled by a grooved shaft to which it is attached. For the full stroke operation the groove is so designed as to give a twisting motion of 440 degrees in the first 3 ½ inches of the stroke of the movable head followed by a straight horizontal motion of 2 ½ inches. The motions of the movable head are uniform except for that portion where the rotary motion is changing to straight translational motion. The motion of the machine is reciprocal, a full cycle consisting of the forward and return strokes. For an alternate short stroke operation, the movable head travels only 3 ¼ inches in each direction in such a manner that a twisting motion of only 400 degrees is imparted to the material. The flexing speed for all materials shall be 40 cycles per minute.

5.2.2.2.2 A heat sealer equipped with controls for temperature, dwell time, and pressure.

5.2.2.3 Test specimens. Four 8- by 12-inch specimens shall be cut from the barrier material, two in each principal direction. Four additional 9- by 13-inch specimens shall be cut out, two in each principal direction, and shall be aged by exposing the specimens in an atmosphere of 80 to 85 percent relative humidity at 160 ± 2 °F for 72 consecutive hours. At the completion of the aging exposure, the test specimens shall be returned to test room conditions for 4 hours and then trimmed to produce 4 aged specimens each 8 by 12 inches. Each test specimen, unaged and aged, shall be prepared for flexing by applying a ½-inch heat-seal (or joining by another means) to the two shorter edges of the sheet, thus producing an approximate 3 ½-inch diameter cylinder or sleeve 8 inches long.

5.2.2.4 Test procedure. The flexing procedure shall be performed in a test room maintained at 73 ± 3.5 °F and 50 ± 5 percent relative humidity. The sleeve, in cylindrical form, shall be positioned and clamped on the circular heads of the flexing apparatus. The drive shaft of the Flex machine shall be at dead center; i.e., perfectly horizontal, before flexing is initiated. Each sample shall be flexed for 20 cycles using the full stroke or short stroke as specified in the specification referencing this procedure. If not specified, the full stroke shall be used. The water vapor transmission rate after flexing shall then be determined by forming the specimen into a pouch and testing according to Method 3030 (see 5.3.6) of this specification.

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5.2.3 Test Method 2024 – Heat-sealed Seam Strength.

5.2.3.1 Scope. This test procedure details a method to assess the adequacy of heat-seals made on sheet materials (5.2.3.3) and those made in the fabrication of bags or pouches (5.2.3.4).

5.2.3.2 Apparatus.

5.2.3.2.1 Appropriate heat-sealing equipment with accurate controls of temperature, pressure, dwell time, or other sealing criteria required to fuse heat-seals of reproducible quality.

5.2.3.2.2 One-inch-wide clamps from which test weights can be suspended. The test weights shall be as shown in the table (see 5.2.3.5).

5.2.3.2.3 Test frame. A test frame to allow the weighted load to act freely on the test specimen.

5.2.3.3 Sheet materials. Figure 1 shall be used to cut eighteen 6- by 12-inch representative samples of the material being evaluated. Each of the samples shall be designated either Key 1 (as-received), Key 2 (heat-sealed and then aged), or Key 3 (aged and then heat-sealed).

5.2.3.3.1 Test specimen preparation and conditioning.

5.2.3.3.1.1 As-received (Key 1). Each of the 6 samples shall be folded in half with the crease perpendicular to the long axis and the 6-inch ends heat-sealed together. The fold shall then be cut off. Then three adjacent 1-inch-wide specimens shall be cut from the center of the sample and perpendicular to the heat-seal. One of the specimens from each sample shall be tested at room temperature (Test A), one from each sample at 100 °F (Test B), and the remaining one from each sample at 160 °F (Test C).

5.2.3.3.1.2 Sealed and then aged (Key 2). Each of the 6 samples shall be folded in half with the crease perpendicular to the long axis and the 6-inch ends heat-sealed together. These sealed samples shall then be aged at 160 °F for 12 days. Specimens shall then be cut and designated for test as in 5.2.3.3.1.1.

5.2.3.3.1.3 Aged and then sealed (Key 3). Each of the 6 samples shall be aged at 160 °F for 12 days. After cooling to room temperature, each of the 6 samples shall be folded in half with the crease perpendicular to the long axis and the 6-inch ends heat-sealed together. Specimens shall be then be cut and designated for test as in 5.2.3.3.1.1.

5.2.3.4 Fabricated bags and pouches. Three 1-inch-wide heat-seal specimens shall be cut from representative locations of the bag or pouch. Areas within 1 inch of the bag or pouch opening or corner shall be avoided when obtaining test specimens. Specimens shall be tested in an as-received condition at room temperature (Test A).

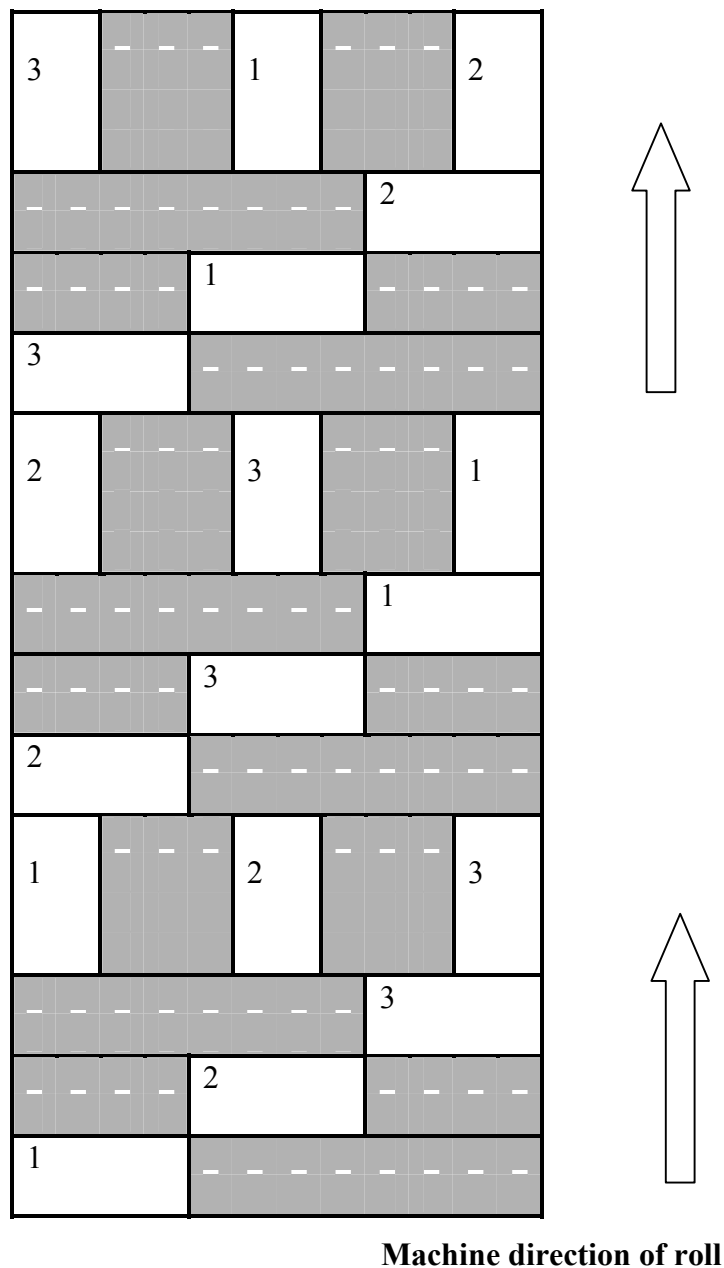
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5.2.3.5 Test procedure. Prior to testing, conditioned specimens shall have been exposed for a minimum of 1 hour as specified in 4.4. Each sealed specimen shall then be laid on a horizontal surface. A beveled (45 ± 5 degrees) plastic straight edge shall be slid between the unsealed ends of the specimen and held against the seal. On the outside of the specimen, the position of the straight edge against the seal shall be marked using a fine ball point pen. Specimens shall be tested using the temperature, weight, and time shown below.

Test	Temperature (°F)	Weight (ounces)	Time (minutes)
A	73 ± 3.5	56 ± 0.5	5
B	100 ± 2	32 ± 0.5	60
C	160 ± 2	10 ± 0.5	60

One end of each specimen shall be clamped so that the other end of the specimen hangs freely, at the test temperature. The test weight shall then be carefully attached to the free end of the specimen so as not to impact load the seal. The weight shall be allowed to act for the indicated time at the indicated temperature, whereupon the weight shall be removed and the specimen examined for separation of the heat-sealed faces. The beveled plastic straight edge shall be slid to the seal as before and the position of the straight edge marked. The distance between the two pen lines shall be measured to the nearest $\frac{1}{32}$ of an inch and shall indicate any seam separation.

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**KEY**

- 1 - 6- by 12-inch sample for "As Received" testing
- 2 - 6- by 12-inch sample for "Sealed before Aging" testing
- 3 - 6- by 12-inch sample for "Sealed after Aging" testing

FIGURE 1. Sampling method for seam strength test.

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5.2.4 Test Method 2065 – Puncture Resistance.

5.2.4.1 Scope. This procedure details a test method for determining the puncture resistance of flexible barrier materials used in military packaging.

5.2.4.2 Apparatus.

5.2.4.2.1 A test specimen holding fixture as shown on figure 2.

5.2.4.2.2 Compression test equipment for measuring the load required to puncture a specimen held by the above fixture.

5.2.4.3 Test specimens. Ten specimens, each measuring 2 by 2 inches, shall be selected at random from a representative area of the material being evaluated that is free of obvious flaws or defects.

5.2.4.4 Test procedure.

5.2.4.4.1 Calibrate the compression test equipment.

5.2.4.4.2 Attach the test specimen holding fixture to the compression test equipment so that the fixture plates are horizontal and the probe is perpendicular to the plates and centered on the specimen hole. The fixture shall be installed as shown on figure 2 or inverted.

5.2.4.4.3 Adjust the initial position of the compression test equipment so that the probe does not interfere with the installation of specimens. Zero the load.

5.2.4.4.4 Set the speed of the probe relative to the specimen holder at 20 inches per minute.

5.2.4.4.5 Install a specimen in the holding fixture by removing the hold-down plate and placing the specimen between the carborundum papers. Tighten the hold-down plate so that the specimen edges do not move during the test.

CAUTION

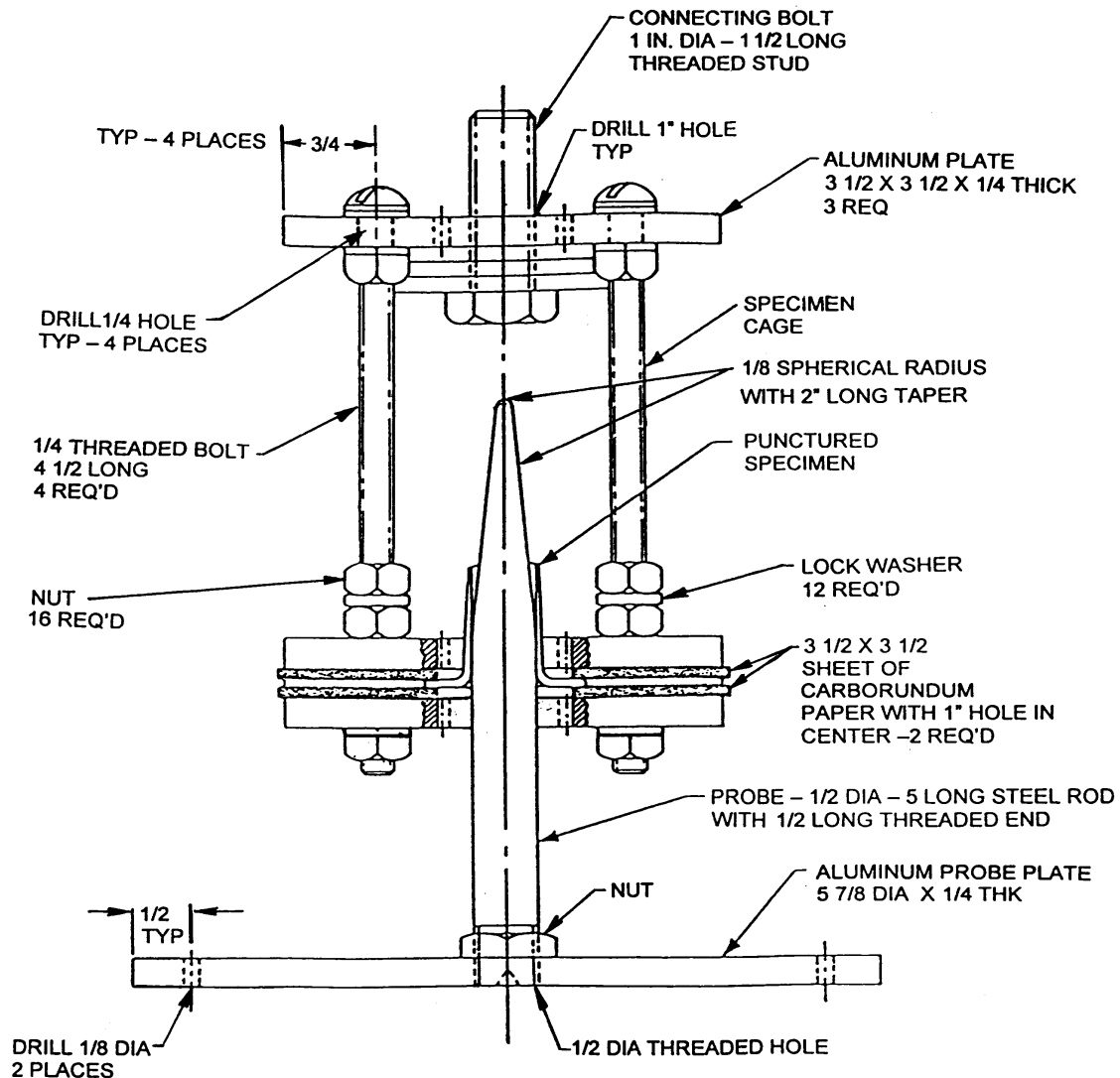
Ensure that the maximum compression load is not exceeded and that the probe does not make direct contact with the specimen holding fixture.

5.2.4.4.6 Start the test and record the maximum load (to the nearest 0.1 pound) measured by the compression tester. Repeat the test until 5 specimens have been tested in the sealable side up

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orientation, then average the results. Repeat the test until 5 specimens have been tested in the sealable side down orientation, and again average the results.

5.2.4.4.7 Note the lowest result.



NOTES:

1. ALL DIMENSIONS ARE IN INCHES.
2. ROUGH SURFACES OF CARBORUNDUM PAPER SHALL FACE EACH OTHER.

FIGURE 2. Test specimen holding fixture.

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5.3 Resistance properties.5.3.1 Test Method 3003 – Blocking Resistance.

5.3.1.1 Scope. This test procedure details test methods for determining the resistance of packaging materials adhering to similar or dissimilar packaging materials with which they might come in contact during their storage and service life.

5.3.1.2 Apparatus.

5.3.1.2.1 A circulating air oven that can be maintained at temperatures up to 160 ± 2 °F.

5.3.1.2.2 Two resilient pads, each measuring 2 by 2 by $\frac{1}{8}$ inches.

5.3.1.2.3 Two glass or smooth flat plates, each measuring 4 by 4 inches.

5.3.1.3 Test specimens. Eight specimens, each 3 by 3 inches, shall be selected at random from representative locations of the sheet material being tested.

5.3.1.4 Test procedure. The test specimens shall be stacked under room conditions in the following sequence:

Bottom smooth flat plate
Resilient pad
One test specimen, face up
One test specimen, face down
One test specimen, face down
One test specimen, face up
A neutral interleaving material (paper or foil)
One test specimen, face up
One test specimen, face down
One test specimen, face down
One test specimen, face up
Top smooth flat plate

All items shall be centered over the member beneath them. A weight which produces a 3.0 psi load on the specimens shall be placed on the top plate. The entire assembly shall then be placed in a circulating air oven maintained at 160 ± 2 °F for 24 hours. After removal of the stack from the oven, the weight shall be removed and the test stack shall be allowed to cool at test room conditions for 30 minutes. The specimens shall be disassembled and examined in sequence for any adhesion or cohesion between adjacent surfaces. If two surfaces appear to be blocked, the free end of one surface shall be vertically clamped so that the other surface hangs down freely. A

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200-gram weight shall then be gently attached to the corresponding free end of the second surface. If the two surfaces are not completely separated after 2 minutes, the specimens are considered blocked. Delamination or rupture of any test surface during separation is also considered blocking.

5.3.1.5 Notes. Procedure letters previously specified in packaging material specifications referencing this test method shall be disregarded. The method described herein is intended to replace both Procedure A and Procedure D with one standard test to determine resistance to blocking.

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5.3.2 Test Method 3005 – Contact Corrosivity.

5.3.2.1 Scope. This test method details a procedure to determine the corrosive tendencies of packaging materials when in intimate contact with a test surface.

5.3.2.2 Apparatus.

5.3.2.2.1 A circulating air oven that can be maintained at 150 ± 5 °F.

5.3.2.2.2 A desiccator chamber with drying agent.

5.3.2.2.3 A chamber that can be maintained at 120 ± 2 °F and 65 ± 3 percent relative humidity.

5.3.2.2.4 Rectangular steel weights, each measuring 1 by 1 by 3 inches, weighing 0.85 ± 0.05 pounds.

5.3.2.3 Test specimens. Three specimens representative of the material being tested shall be selected at random. When testing flexible sheet material, each test specimen shall measure 2 by 3 inches. When testing cushioning or blocking materials, each specimen size shall be not less than 1 by 3 inches. When testing granular material, each test specimen shall be approximately 20 grams of material ground to a size that passes a U.S. No. 40 sieve, but is retained by a U.S. No. 80 sieve.

5.3.2.4 Test procedure.

5.3.2.4.1 Test panel preparation. The test surface for this evaluation shall be panels, each measuring 2 by 4 by $\frac{1}{8}$ inches. Panel material shall be low carbon steel conforming to QQ-S-698, condition 5. Panels shall be ground to remove surface scale, pits, and other irregularities from all surfaces. One of the large flat surfaces of the panel shall then be hand-finished with 240 grit aluminum oxide or silicon carbide abrasive sheet to a surface roughness of 6 to 12 microinches root mean square (RMS). The finished test panel shall then be wiped with surgical gauze and immersed in and then scrubbed in boiling absolute methanol. This shall be followed by two successive immersions in boiling absolute methanol each for 1 minute. Panels shall then be allowed to dry in clean air and used immediately.

5.3.2.4.2 Procedure.

5.3.2.4.2.1 Flexible sheet materials. Each 2- by 3-inch flexible sheet specimen shall be placed across the central portion of the test panel surface, as shown on figure 3. A glass slide 1 by 3 by $\frac{1}{16}$ inches with a weight superimposed and coinciding shall be centered on top of the specimen with the longitudinal centerline of the slide and weight coinciding with that of the test specimen as shown on figure 4.

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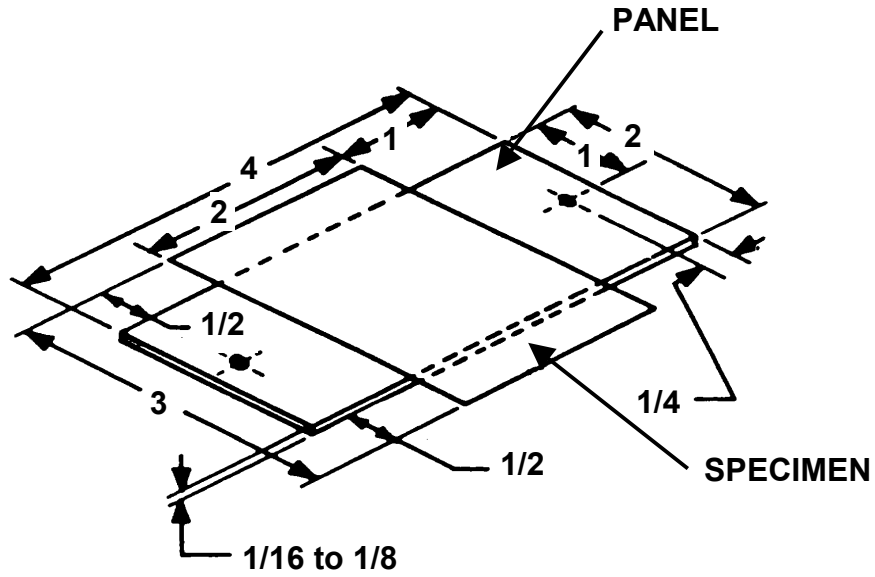
5.3.2.4.2.2 Cushioning or blocking materials. Cushioning or blocking material specimens shall be placed across the central portion of the test surface so that the specimen extends over the edges of the panel. Large specimens shall be placed off center so an area of the test surface not less than 2 by 2 inches shall remain uncovered.

5.3.2.4.2.3 Granular materials. If granular specimens are being tested, they shall be placed and leveled between parallel lines 1 inch apart across the central portion of the test surface of a panel. The specimen shall be carefully covered with a glass slide, and the steel weight shall then be placed upon the glass slide.

5.3.2.4.2.4 Exposure. In all cases the specimen and test surface so arranged shall be exposed for ½ hour in air maintained at a temperature of 150 ± 5 °F followed immediately by exposure in air at 120 ± 2 °F and 65 ± 3 percent relative humidity for 20 hours. At the end of the exposure period, the specimens shall be separated from the test surface that shall be immediately examined for evidence of corrosion. Note for each area – the one covered by the specimen, and the other not covered – whether or not corrosion occurred and a description including the severity and distribution of any corrosion.

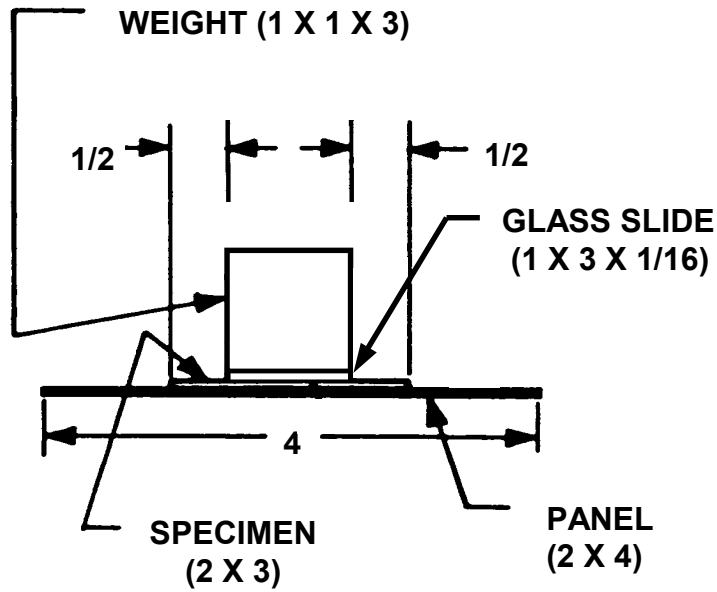
5.3.2.5 Notes. The given exposure environment and duration are such that corrosion is not visible on unprotected clean steel, but corrosion is visible on the test surface if the material under test has a tendency to induce corrosion. When test surfaces are of other metals, the exposure should be specified. To establish whether or not a specimen induces corrosion of a test surface other than steel, the severity of the test environment and duration of exposure thereto must be sufficient to closely approach incipient corrosive attack of the bare (control) specimen surface. This requires a preliminary test to establish the appropriate duration of exposure. When using aluminum test panels, exposure to the humid environment shall be 72 hours.

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DIMENSIONS IN INCHES

FIGURE 3. Flexible sheet material specimen placement.



DIMENSIONS IN INCHES

FIGURE 4. Contact corrosivity test assembly.

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5.3.3 Test Method 3015 – Delamination Resistance of Heat-sealable Laminated Materials.

5.3.3.1 Scope. This test procedure details a method to determine the delamination resistance of a laminated heat-sealable material in the presence of oil.

5.3.3.2 Apparatus.

5.3.3.2.1 An oven capable of maintaining a temperature of 160 ± 2 °F.

5.3.3.2.2 A heat-sealer equipped with controls for temperature, dwell time, and pressure.

5.3.3.2.3 Two smooth flat metal plates measuring 3 by 6 by $\frac{1}{4}$ inches and two 18-pound weights.

5.3.3.2.4 A sufficient quantity of the following test oils to conduct the prescribed tests:

a. IRM 903 as specified in ASTM-D471.

b. Di-2-ethylhexyl sebacate synthetic oil.

5.3.3.3 Test specimens. Five specimens, each 3 by 6 inches, shall be taken at random from material being evaluated for each test oil.

5.3.3.4 Test procedure. Each test specimen shall be folded in half, heat-sealable face to heat-sealable face, to produce a 3- by 3-inch specimen. A sharp crease shall be made in the specimen by placing the folded specimen between the two smooth flat metal plates and applying one of the 18-pound weights on top of the fold for 30 seconds. The specimen shall be unfolded and recreated in a similar manner (heat-sealable face to heat-sealable face) at right angles to the first crease by placing the specimen between the two smooth flat plates again and applying both 18-pound weights side by side on top of the fold for 30 seconds. A 3- by 3-inch pouch shall be made by folding the sample in half along the first crease and sealing along the two sides using the manufacturer's recommended sealing conditions. Heat-seals shall be $\frac{1}{2}$ inch wide.

Approximately 5 ml of oil shall be poured into each pouch, carefully keeping the sealing area free from oil. The open end of the pouch shall then be sealed keeping the enclosed air to a minimum. The pouches shall be promptly exposed in an oven maintained at 160 ± 2 °F, for 24 hours by hanging each pouch from the center of its sealed end. Pouches shall be removed from the oven, and after returning to room temperature, examined for any oil leakage. If a pouch shows oil leakage, the test shall be repeated with a fresh pouch and those results noted. The three sealed edges shall be cut off and all remaining oil shall be removed. The remaining sheet shall be pulled taut and examined for evidence of swelling, delamination, embrittlement, or other visible defects. A test for delamination of the face film shall be conducted at mid-length of the test specimen by placing the specimen between the thumbs and forefingers so that the thumbs rest on the heat-sealable face. The thumbs shall then be thrust forward and outward in a finger-snapping motion in such a manner that the heat-sealable face will delaminate if it is loosely bonded.

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5.3.4 Test Method 3027 – Water Resistance of Markings.

5.3.4.1 Scope. This test procedure details a method to determine the retention of markings on packaging materials after exposure to water.

5.3.4.2 Apparatus.

5.3.4.2.1 A 3-liter stainless steel beaker.

5.3.4.2.2 A magnetic stirrer and a stirring bar 2 inches long and $\frac{3}{8}$ -inch diameter.

5.3.4.3 Test specimens. Two or more specimens selected at random to adequately represent any variation in the material being evaluated shall each contain one complete set of dry markings.

5.3.4.4 Test procedure. Two specimens shall be taped to the inside of the beaker with pressure sensitive tape which remains adherent throughout test so that the markings are uncovered and facing the interior of the beaker. The beaker shall be filled with distilled water to within 2 inches of its top and covering the specimen completely. The stirring bar shall be added to the beaker and spun at 200 ± 40 revolutions per minute (rpm) for 2 hours at room temperature. After removal from the vessel, excess water shall be blotted off the test specimens. They shall then be examined for clarity, legibility, and shall not smear when lightly rubbed with one finger. The examination shall be repeated after the specimens have dried out at room temperature.

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5.3.5 Test Method 3028 – Water Resistance of Packaging Materials.

5.3.5.1 Scope. This test procedure details a method to determine the resistance of packaging material to delamination (ply separation) as a result of exposure to water.

5.3.5.2 Apparatus.

5.3.5.2.1 A vessel capable of accommodating and securing test specimens in a manner to allow unrestricted circulation of water around the specimens.

5.3.5.2.2 An oven capable of maintaining a temperature of 160 ± 2 °F.

5.3.5.3 Test specimens. Four specimens, taken at random such that the specimens represent any variation in the material being evaluated, shall be tested. Specimen size shall be 6 by 8 inches.

5.3.5.4 Test procedure. Test specimens shall be immersed in distilled water maintained between 68 to 86 °F. The specimens shall be supported in a manner allowing unrestricted circulation of water around the test material. The immersion period shall be 24 hours for fiberboard materials and 48 hours for flexible barrier materials. After removal from the water, each specimen shall be examined for evidence of swelling, delamination, embrittlement, or other visible defects. A test for ply separation of the face film shall be conducted at mid-length of the test specimen by placing the specimen between the thumbs and forefingers so that the thumbs rest on the heat-sealable face. The thumbs shall then be thrust forward and outward in a finger-snapping motion in such a manner that the heat-sealable face will delaminate if it is loosely bonded. Flexible barrier materials shall be further exposed in a circulating air oven maintained at 160 ± 2 °F for 24 hours. The specimens shall be re-examined for ply separation after being allowed to cool to room temperature.

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5.3.6 Test Method 3030 – Water Vapor Transmission Rate of Barrier Materials.

5.3.6.1 Scope. This test procedure details a method to determine the water vapor transmission rate (WVTR) of packaging barrier materials. The test is applicable to material either as received, after flexing, or after accelerated aging.

5.3.6.2 Apparatus.

5.3.6.2.1 A test chamber capable of maintaining a test environment of 100 ± 2 °F and 90 ± 2 percent relative humidity.

5.3.6.2.2 A heat-sealer with controls for temperature, dwell time, and pressure.

5.3.6.2.3 Porous heat-sealable tea bag stock.

5.3.6.2.4 An analytical balance.

5.3.6.3 Test specimens. Four 8- by 12-inch specimens for each test condition shall be selected at random from the sheet material being tested. Two samples shall be taken from each principal direction of the material.

5.3.6.4 Test procedure. The test specimens shall be folded in half (to 6 by 8 inches) and formed into a pouch by making a continuous seal along the 8-inch side (if it has not been sealed already; e.g. for flexing) and another seal along one 6-inch side, using sealing conditions as recommended by the manufacturer for the material. Seal widths shall be approximately ½-inch wide. A bag to hold the desiccant shall be formed by cutting a 3 ½-inch wide by 10 ½-inch long piece from the web of tea bag stock, folding in half to 3 ½ by 5 ¼ inches, and making a seal not more than ¼-inch wide along the two sides and the folded edge with the heat-sealer. The desiccant bag shall be filled with not less than 25 grams of anhydrous calcium chloride (4-20 mesh) then sealed. The desiccant bag shall then be immediately inserted into the test specimen pouch and the pouch sealed. The sealed pouch shall then be exposed in a test chamber maintained at 100 ± 2 °F and 90 ± 2 percent relative humidity for a stabilization period of 16 hours. After the stabilization period, the pouch shall be removed from the test chamber and a ½-inch wide strip cut from one end. The desiccant bag shall be immediately removed and replaced with a fresh preweighed desiccant bag. The pouch shall then be sealed and exposed in the test chamber for a period of 72 hours. After exposure, the pouch shall be removed and cooled for 5 minutes, then cut to remove the closure seal. The desiccant bag shall be immediately removed and reweighed. The weight gain of the desiccant bag (W) during the exposure shall be used to calculate the WVTR for the material being tested.

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NOTE

To avoid the possibility of moisture change during weighings, the desiccant bag shall be transferred to a tared, closed, water vapor-impervious container for every weighing, while minimizing the time it is exposed to the open air.

5.3.6.5 Alternate procedure. When testing non-hygroscopic pouch materials which remain constant in weight during weighing, the following alternate simplified procedure may be used: After the stabilization period, the sealed pouch shall be cooled for 5 minutes, weighed and replaced in the test chamber. At the end of the period of exposure, the sealed pouch shall again be cooled for 5 minutes, weighed and the WVTR calculated, using the total weight gain (W) instead of that for the desiccant bag.

5.3.6.6 Determine the WVTR as follows:

$$\text{WVTR (grams/100 in}^2\text{/24 hrs)} = \frac{2400 \times W \text{ (grams)}}{\text{Interior pouch area (in}^2\text{) x Exposure time (hrs)}}$$

where the interior pouch area is determined by multiplying the seal to seal distances, length times width, to get the area of one side and then multiplying by 2 to get the total interior pouch area.

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5.4 General physical properties.5.4.1 Test Method 4031 – Vapor Inhibiting Ability (VIA) of Volatile Corrosion Inhibitor (VCI) Materials.

5.4.1.1 Scope. This test method details procedures to determine the corrosion inhibiting effectiveness of VCI materials in crystalline or liquid form, or VCI as a coating or treatment on substrate materials in the as received and after exhaustion (aged) conditions.

5.4.1.2 Apparatus.

5.4.1.2.1 Atomizer (see figure 5)

5.4.1.2.2 Assembled VIA test jar (see figure 6)

5.4.1.2.3 VIA test jar lids (see figure 7)

5.4.1.2.4 Exhaustion apparatus for crystalline and liquid materials (see figure 8)

5.4.1.2.5 Exhaustion apparatus for coated materials (see figure 9)

5.4.1.2.6 Steel test plugs as specified in figure 6.

5.4.1.2.7 Aluminum oxide or silicon carbide abrasive sheets (240 and 400 grit)

5.4.1.2.8 Stainless steel beakers, covers and hot plates for solvent cleaning

5.4.1.2.9 An air pump and flowmeter with regulator.

5.4.1.2.10 A forced draft oven with temperature controls.

5.4.1.3 Test specimens. All VIA tests shall be run in triplicate. In addition, a control test shall also be run using a VIA test jar but with no VCI test specimen. Specimen sizes for the different VCI materials covered by this test method are as follows:

- a. Crystalline VCI (as received) - 0.050 ± 0.005 gram (g) for each replicate
- b. Liquid VCI (as received) - 0.050 ± 0.005 g for each replicate
- c. VCI coated materials (as received) - Two strips of the material, each measuring 1 by 6 inches, for each replicate.

5.4.1.4 Test specimen conditioning. Specimens shall be tested (a) as received and (b) after exhaustion (by artificially aging the sample). Exhausted (aged) test specimens shall be tested for

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VIA immediately after the exhaustion procedure. Exhaustion is accomplished as follows:

NOTE

Outlets from the exhaustion apparatus shall be ducted out of the laboratory to avoid contamination of prepared test surfaces.

5.4.1.4.1 Crystalline VCI exhaustion procedure. The crystalline VCI material shall be introduced into the exhaustion conditioning assembly shown on figure 8 and as specified in 5.4.1.5.2.1, except that 0.10 ± 0.005 g of material shall be introduced. The holes in the lid shall be covered with tape. The assembly shall then be placed in a forced draft oven, with the inlet connected to the regulated air pump and the outlet connected to an exhaust duct. Air maintained at 50 ± 2 percent relative humidity shall be pumped into the jar containing the VCI at a rate of 100 cubic centimeters per minute while the oven is maintained at 100 ± 2 °F for 5 days. The jar containing the VCI shall then be removed from the oven and allowed to cool to room temperature prior to VIA testing.

5.4.1.4.2 Liquid VCI exhaustion procedure. The liquid VCI material shall be introduced into the exhaustion test assembly described on figure 8 and as detailed in 5.4.1.5.2.2 except that the large open end of the funnel shall be 2 inches from the bottom of the jar, and 25 cubic centimeters of material shall be introduced. The exposure shall be conducted as described in 5.4.1.4.1 except that the exposure conditions shall be at 75 ± 5 °F for 7 days.

5.4.1.4.3 Non-sealable VCI coated material exhaustion procedure. Samples of the material being exhaustion-tested shall be 2 by 8 inches. Plastic coated paper clips shall be placed at each end of the sample for ease of handling. A $\frac{1}{8}$ inch diameter hole shall be punched in the center of the sample approximately $\frac{1}{8}$ inch from the top edge. Each sample shall be placed in an assembly as shown on figure 9. The assembly shall then be placed in a forced draft oven, with the inlet connected to the regulated air pump and the outlet connected to an exhaust duct. Air maintained at 50 ± 2 percent relative humidity shall be pumped into the jar containing the sample at a rate of 100 cubic centimeters per minute while the oven is maintained at 140 ± 2 °F for 12 days. The test sample shall then be removed from the assembly and cut into two 1- by 6-inch specimens.

5.4.1.4.4 Sealable VCI coated material after exhaustion. This exhaustion test is the same as that for non-sealable VCI material except that the test sample shall be a 5- by 8-inch section of the material under test, folded to 2 $\frac{1}{2}$ by 8 inches and sealed using $\frac{1}{4}$ inch heat-seals on the open edges.

5.4.1.5 VIA Test.

5.4.1.5.1 Test plug preparation.

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NOTE

Test plugs treated with preservative may be purchased in the polished condition but shall be cleaned of preservative, then abraded and polished with both 240 and 400 grit abrasive sheets as specified below. The use of iron oxide abrasives and any abrasive paper that leaves residues not removable by the cleaning methods specified is prohibited. Some abrasive papers and cloths that are intended to be used dry or wet have been found to leave such residues.

The test plug shall be constructed of low carbon steel meeting the requirements of QQ-S-698, Condition 5. A cold finished round bar of this material shall be machined to the dimensions specified on figure 6. The undrilled end shall be hand-abraded to a uniform finish with 240 grit silicon carbide or aluminum oxide abrasive sheet. The abraded test surface shall then be hand-polished with 400 grit aluminum oxide abrasive sheet at 90 degrees to the previous abrasion marks and to a surface roughness of 4 to 6 microinches RMS for the test surface. Each polished plug shall be wiped clean with surgical gauze and examined at 10X magnification to ensure that all 240 grit marks have been removed and that the surface is uniform. Each plug shall then be placed in a container of reagent methanol until it can be further cleaned, but for no more than 4 hours. Further cleaning shall be accomplished by (a) immersion in a container of boiling reagent methanol and scrubbing the polished surface with surgical gauze, (b) immersion in a second container of boiling reagent methanol for 1 minute, and (c) immersion in a third container of boiling reagent methanol for 1 minute. During and after this cleaning process, the plugs shall be handled only with forceps and without touching the polished surface except for scrubbing with gauze. Upon removal from the third container of methanol, a plug shall be dried by waving it in the ambient air for 20 seconds then immediately installed in the VIA test jar fitted with the appropriate lid (see figure 7). Clean, low-lint laboratory tissue shall be used to force the plug into the stopper as shown on figure 6. The appropriate test procedure shall be initiated immediately.

5.4.1.5.2 VIA test setup. There are four different variations in the test setup depending on the nature of the material: crystalline VCI (see 5.4.1.5.2.1), liquid VCI (see 5.4.1.5.2.2), exhausted crystalline or liquid VCI (see 5.4.1.5.2.3), and VCI coated materials (see 5.4.1.5.2.4). VIA test lids are assembled for the different setups by fitting stoppers, aluminum tube, sleeve and test plug to the jar lids shown on figure 7. Following assembly of the test lids, all samples shall be tested in accordance with 5.4.1.5.3 without delay.

5.4.1.5.2.1 Crystalline VCI. An assembled VIA test lid shall be screwed onto a test jar. For material of this nature, 1 gram (g) of the material being tested shall be placed in a glass vial which shall be attached to the atomizer (see figure 5). The material shall then be sprayed into a test jar through one of the small holes in the lid until 0.05 ± 0.005 g, as determined by weight

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loss, is dispensed. As an alternate procedure, which shall be used only when specified in the material specification test method, 0.05 ± 0.005 g of the material being tested shall be placed in a 1 ½ by 2 inch pouch made from tea bag material and hung on a jar lid with coated wire. The tea bag shall not touch the jar and shall be centered vertically.

5.4.1.5.2.2 Liquid VCI. For material of this nature, 0.05 ± 0.005 g of the material being tested shall be weighed and placed in a standard 1 ounce wide-mouth jar. The vessel shall then be placed on the bottom of a test jar and an assembled VIA test lid set on the jar.

5.4.1.5.2.3 VCI in crystalline or liquid form after exhaustion. After conditioning, the exhaustion apparatus and specimen contained in it shall be used for VIA testing by removing the lid with the funnel attachment and setting an assembled VIA test lid on the jar.

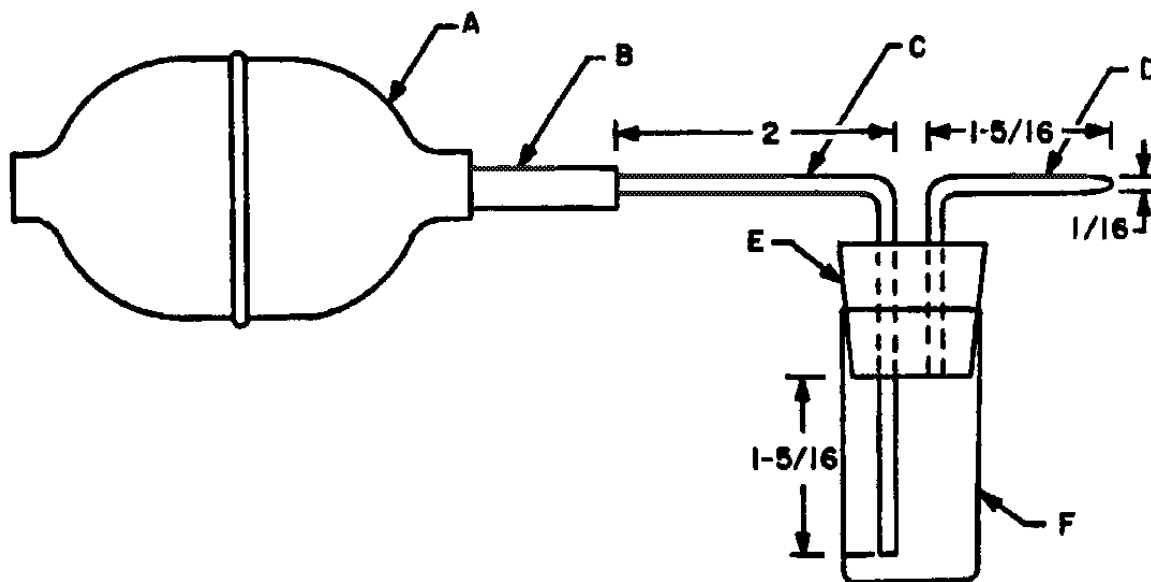
5.4.1.5.2.4 VCI in coated material form as received and after exhaustion. An assembled VIA test lid for coated materials shall be set on a test jar and two 1- by 6-inch specimens shall be inserted through its slots. The specimens shall be held in position by forming a ¼-inch tab at one end of the specimen. The tab is then taped to the outer surface of the lid. Alternatively, specimens shall be hung from coated paper clips attached to the lid. The treated surfaces of the test specimens shall face toward the center of the jar.

5.4.1.5.3 VIA test procedure. The lid shall be removed from each test jar and 10 cubic centimeters of a synthetic glycerin-water solution having a specific gravity of 1.076 at 75 ± 3 °F shall be introduced into the bottom of the test assembly shown on figure 6 (producing an atmosphere of 90 to 95 percent relative humidity). The lids shall be tightly screwed onto the jars and any holes in the lids and the jar-lid interface shall be sealed with tape. The test assembly shall be exposed to a temperature of 75 ± 5 °F for 20 hours. Each aluminum tube shall then be filled with cold water (32 to 35 °F). After 3 hours, the test lid shall be gently unscrewed and inverted to remove the water from the tubes. When dry, the polished surface of the test plug shall be examined for evidence of corrosion. If the polished surface of the control test plug shows no corrosion, the test shall be rerun.

5.4.1.6 Test surface evaluation. Each test surface shall be examined under 10X magnification. A rating shall be attributed to each surface based on the number of corrosion spots found: 0 = no spots, 1 = 1 spot, 2 = 2 spots, 3 = 3 spots, 4 = 4 or more spots. In addition, it shall be noted whether any of the spots exceed 250 micrometers in diameter (determined using 40X magnification and a corresponding standard of comparison).

5.4.1.7 Notes. The exposure environment specified herein is intended to evaluate the corrosion preventive effectiveness of the VCI material being tested with respect to steel surfaces. For other metals, the exposure conditions and test surface would have to be adjusted to evaluate the effectiveness of VCI material.

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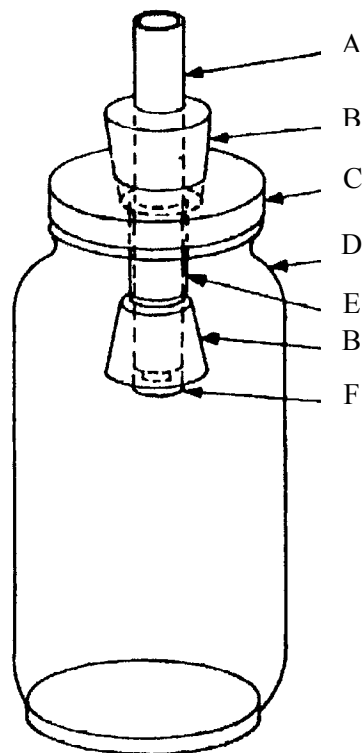


DIMENSIONS IN INCHES

- A - Rubber bulb
- B - Rubber tubing - 1-inch long
- C - Pyrex glass tubing - $\frac{1}{8}$ -inch ID, length to suit
- D - Pyrex glass tubing - $\frac{1}{8}$ -inch ID, length to suit, with $\frac{1}{16}$ -inch nozzle
- E - Rubber stopper - No. 4 with holes to accommodate glass tubing
- F - Glass vial - 2 inches in height by $\frac{7}{8}$ inches ID

FIGURE 5. Atomizer (dispenser for introducing VCI into test assembly)

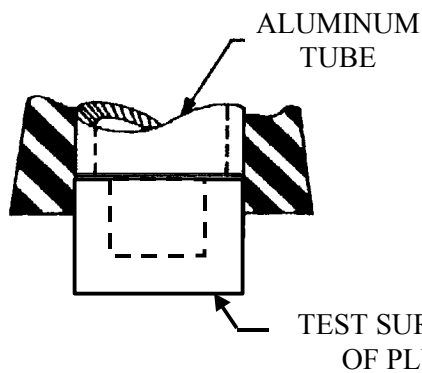
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- A - Aluminum tube – 4 ½ in length, 5/8 OD and ½ ID
- B - Rubber stopper – #6-½ rubber stopper with ½ hole drilled through center (2 required)
- C - Jar lid – See figure 7 for details
- D - Jar – Quart size, 6 ¾ inches in height, 3 ½ ID
- E - Insulating sleeve – ½ ID thin rubber tubing, length 1 ½
- F - Test plug – 5/8 OD, ½ long with 3/8 deep, 3/8 ID flat bottom hole drilled in center

NOTES

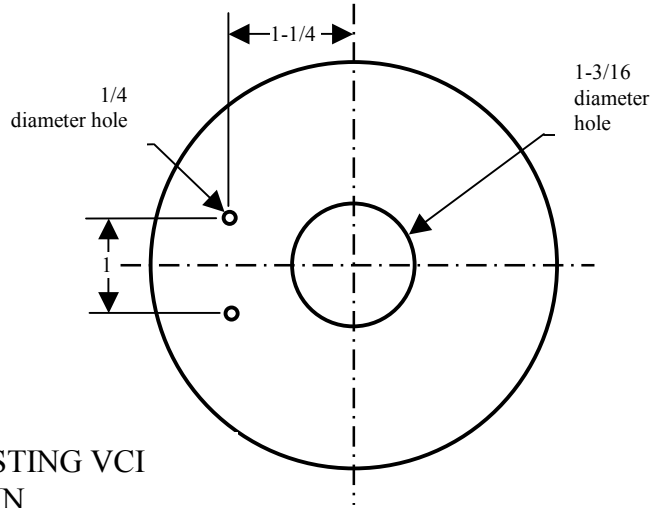
1. Dimensions in inches
2. All parts of the assembled test lid shall be in contact with adjacent part



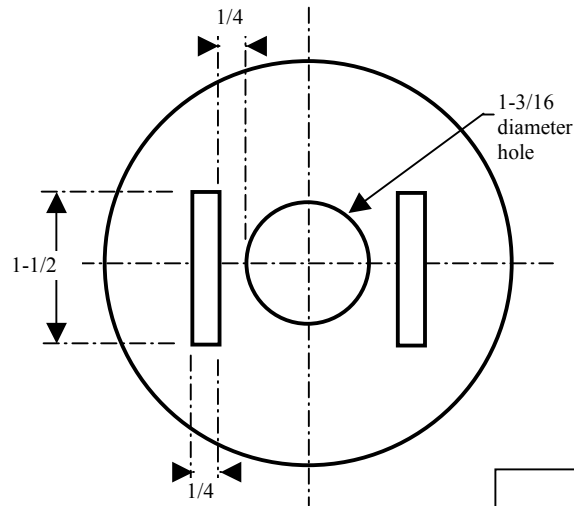
ENLARGED VIEW OF
BOTTOM OF SPECIMEN
HOLDER ASSEMBLY

FIGURE 6. Test assembly.

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LID FOR TESTING VCI
MATERIAL IN
CRYSTALLINE OR LIQUID
FORM



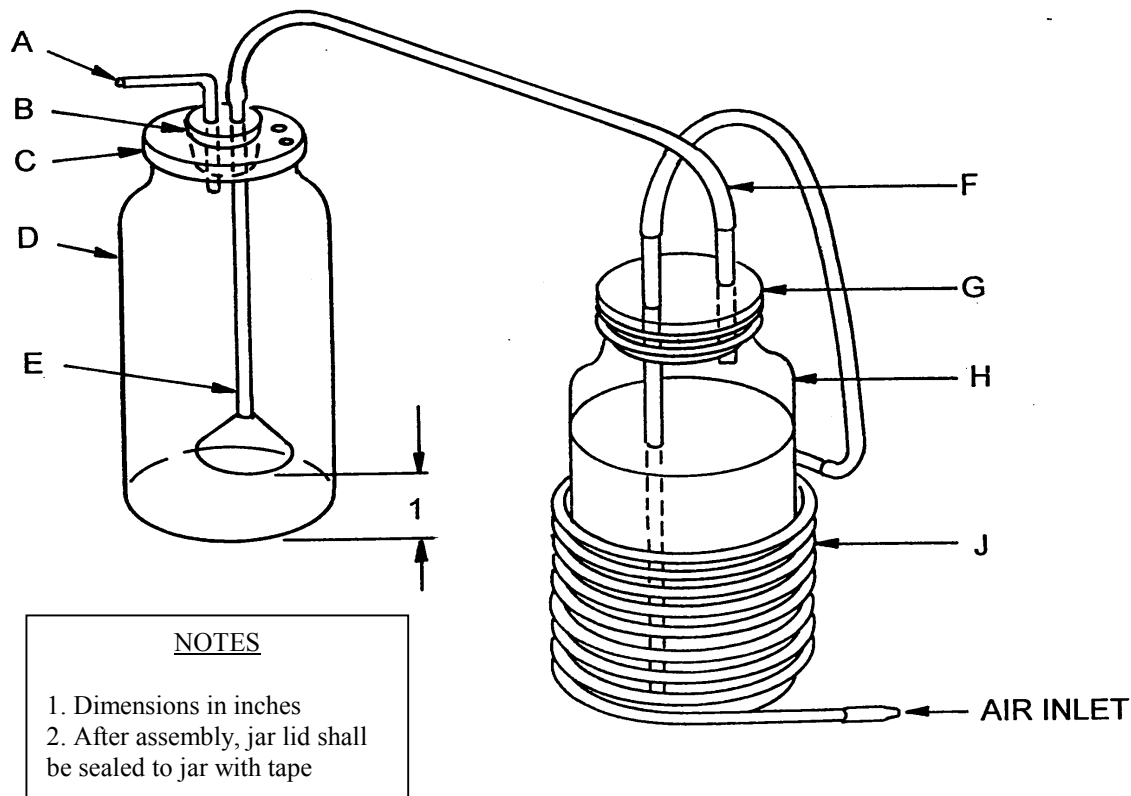
LID FOR
TESTING VCI
COATED
MATERIALS

NOTES

1. All dimensions in inches
2. Both lids to be plastic screw type to fit quart size jar.

FIGURE 7. Jar lids.

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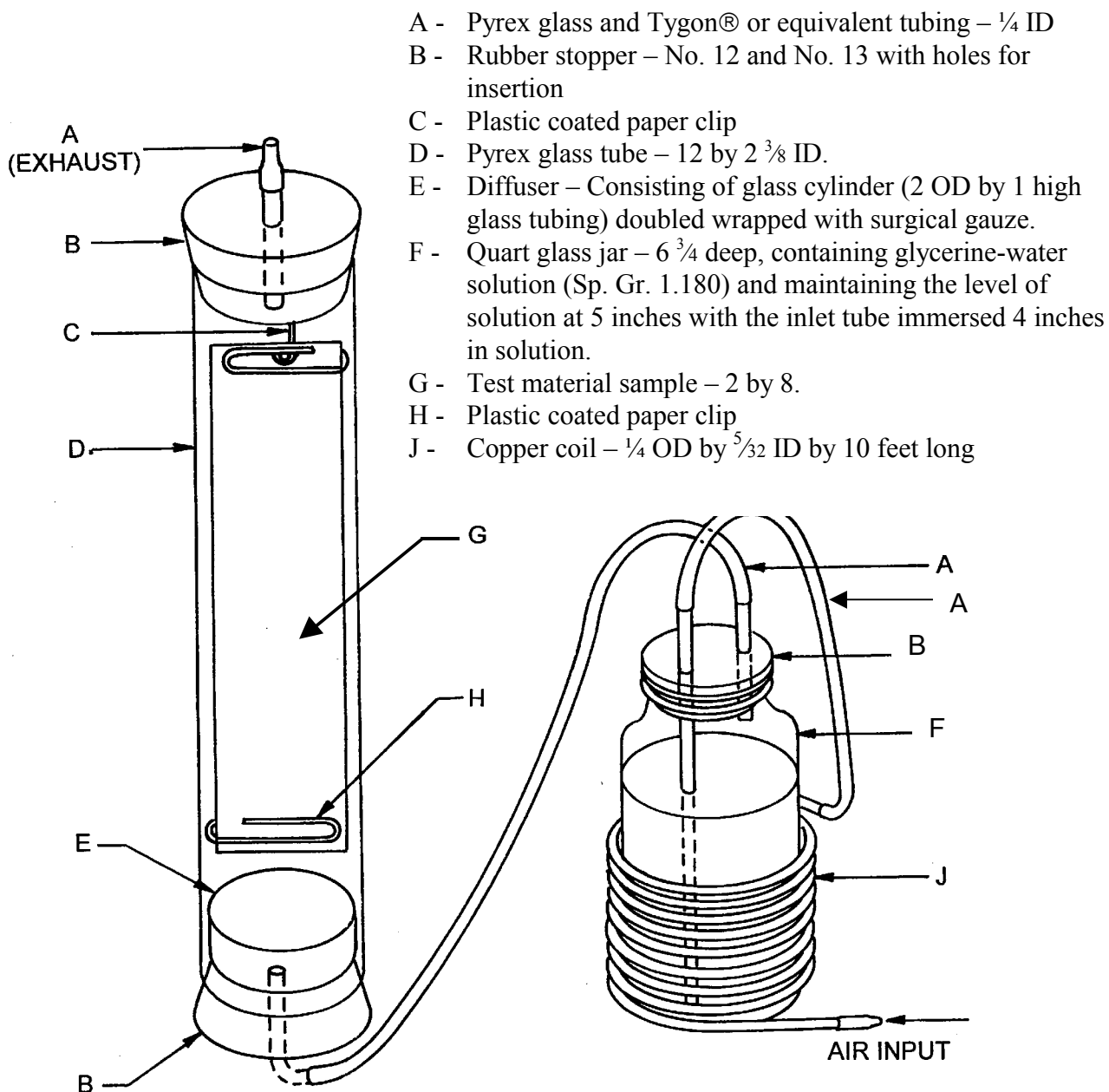


- A - Pyrex glass tubing – $\frac{1}{8}$ ID, length to suit
- B - Rubber stopper – With two holes to suit insertions
- C - Jar lid – See figure 7
- D - Jar – Quart size, $6\frac{3}{4}$ inches in height, $3\frac{1}{2}$ ID – See figure 6, D
- E - Pyrex glass funnel – Approximately 2 ID at mouth, 8 inches long
- F - Pyrex glass and Tygon® (or equivalent) tubing – $\frac{1}{4}$ ID
- G - Rubber stopper – With two holes to suit insertions
- H - Quart glass jar – $6\frac{3}{4}$ inches deep, containing glycerine-water solution (Sp. Gr. 1.180) and maintaining the level of solution at 5 inches with the inlet tube immersed 4 inches in solution
- J - Copper coil (OD $\frac{1}{4}$, ID $\frac{5}{32}$, length 10 feet)

FIGURE 8. Apparatus for exhaustion of VCI material in crystalline form.

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ALL DIMENSIONS ARE IN INCHES

FIGURE 9. Apparatus for exhausting VCI coated material.

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5.4.2 Test Method 4034 – Transparency.

5.4.2.1 Scope. This test procedure details a method for determining the degree of transparency of packaging materials.

5.4.2.2 Apparatus.

5.4.2.2.1 A laser printed 600 dots per inch legibility standard, using Times New Roman font size 16 (body size = 0.167 ± 0.005 inch) in black lower case letters and at least ten digits on ultrawhite paper (96 lumens minimum).

5.4.2.3 Test specimens. At least three specimens, each 3 by 5 inches, selected at random from the material being tested.

5.4.2.4 Test procedure. The characters on the legibility standard shall be examined when viewed through the material being tested when the test material is held 3 inches away from the legibility standard.

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5.4.3 Test Method 4046 – Electrostatic Properties.

5.4.3.1 Scope. This test procedure details a method for determining the electrostatic properties of packaging materials. These properties are determined by measuring the dissipation rate for an electrostatic charge induced on the material being evaluated.

5.4.3.2 Apparatus.

5.4.3.2.1 A 1/8-inch thick aluminum calibration panel measuring 3 by 5 inches.

5.4.3.2.2 A source capable of producing zero to 15 kilovolts, positive and negative.

5.4.3.2.3 An electrometer with a full scale reading of 0.01, 0.1, 1.0, 10 and 100, or a recording oscilloscope with a response of 1 microsecond per division, or equivalent.

5.4.3.2.4 An electrostatic test unit, fabricated as specified on figure 10.

5.4.3.2.5 A test chamber (see figure 11) capable of maintaining the test environment specified in 5.4.3.4.2. The chamber shall be provided with hand holes for mounting and removal of test specimens.

5.4.3.2.6 A single channel pen type recorder with speeds of 0.5, 1.0, 2.0, 4.0, and 8.0 inches per minute and per second.

5.4.3.2.7 Four RG 114/U cables for use as follows:

- a. 5-inch cable for the connection between the electrostatic detector and the output connector on the electrostatic test chamber.
- b. 34-inch cable for connection between the electrostatic test chamber and the electrometer.
- c. Two 31½-inch cables for connections between the electrometer and the recorder.

5.4.3.2.8 High voltage wire as needed to convey the charge from the high voltage source to the electrostatic test unit and to ground as required.

5.4.3.2.9 Three-position control switch for connecting the test specimen to the high voltage source or to ground or to a neutral potential.

5.4.3.2.10 The equipment shall be assembled as specified on figure 12.

5.4.3.3 Test specimens. Three specimens for each pre-test conditioning shall be selected at random to represent any variation of the material. Each specimen shall be 3 by 5 inches and shall

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be free of defects such as holes, cracks, and tears. If the specimen is coated, the coating shall be continuous.

5.4.3.4 Test procedure.

5.4.3.4.1 Pre-conditioning. Prior to testing, expose 3 specimens for 12 days in a circulating air oven uniformly maintained at 160 ± 5 °F; 3 additional specimens in a horizontal position for 24 hours under a continuous water shower; and 3 more specimens in an atmosphere uniformly maintained at 73 ± 5 °F and 50 ± 5 percent relative humidity.

5.4.3.4.2 Test environment. All specimens shall be placed in the electrostatic test chamber for a minimum of 24 hours immediately before testing. Perform tests in an atmosphere uniformly maintained at 73 ± 5 °F and 12 ± 3 percent relative humidity. This relative humidity can be obtained by inserting sufficient anhydrous calcium chloride into the electrostatic test chamber. The anhydrous calcium chloride shall be replaced as required to maintain required relative humidity.

5.4.3.4.3 Calibration. The test equipment shall be calibrated as follows:

- a. Turn on all apparatus and allow to warm up, as noted in the operations manual for the particular apparatus.
- b. On the electrometer, set "multiplier" switch to provide a half-scale reading when the test voltage is applied, the "operate" switch at "zero check," and the meter to read positive charge.
- c. Adjust the high voltage for 5 kilovolts (KV) positive output.
- d. Mount the 3- by 5- by $\frac{1}{8}$ -inch aluminum calibration panel between the electrodes in the electrostatic test unit so that the detector head is directly in the center of the panel. Tighten the four wing nuts to secure the panel.
- e. Set speed of the recorder chart to 1 inch/minute. Turn on recorder.
- f. Set "operate" switch on the electrometer to "operate."
- g. Turn three-position control switch to high voltage.
- h. Observe that the reading on the recorder is identical with the measurement on the meter. Adjust the recorder if necessary.
- i. Turn three-position control switch to ground to remove the charge from the test panel.
- j. When the electrometer meter reaches zero, stop the recorder and turn the "operate" switch on the electrometer to "zero check."
- k. Repeat the calibration procedure for 5 KV negative. Set the appropriate controls on the apparatus for negative charge.

5.4.3.4.4 Testing. Each conditioned specimen, when tested, shall be mounted vertically between the electrodes and the wing nuts tightened to ensure intimate contact between specimen and electrodes. The test shall then be conducted as follows:

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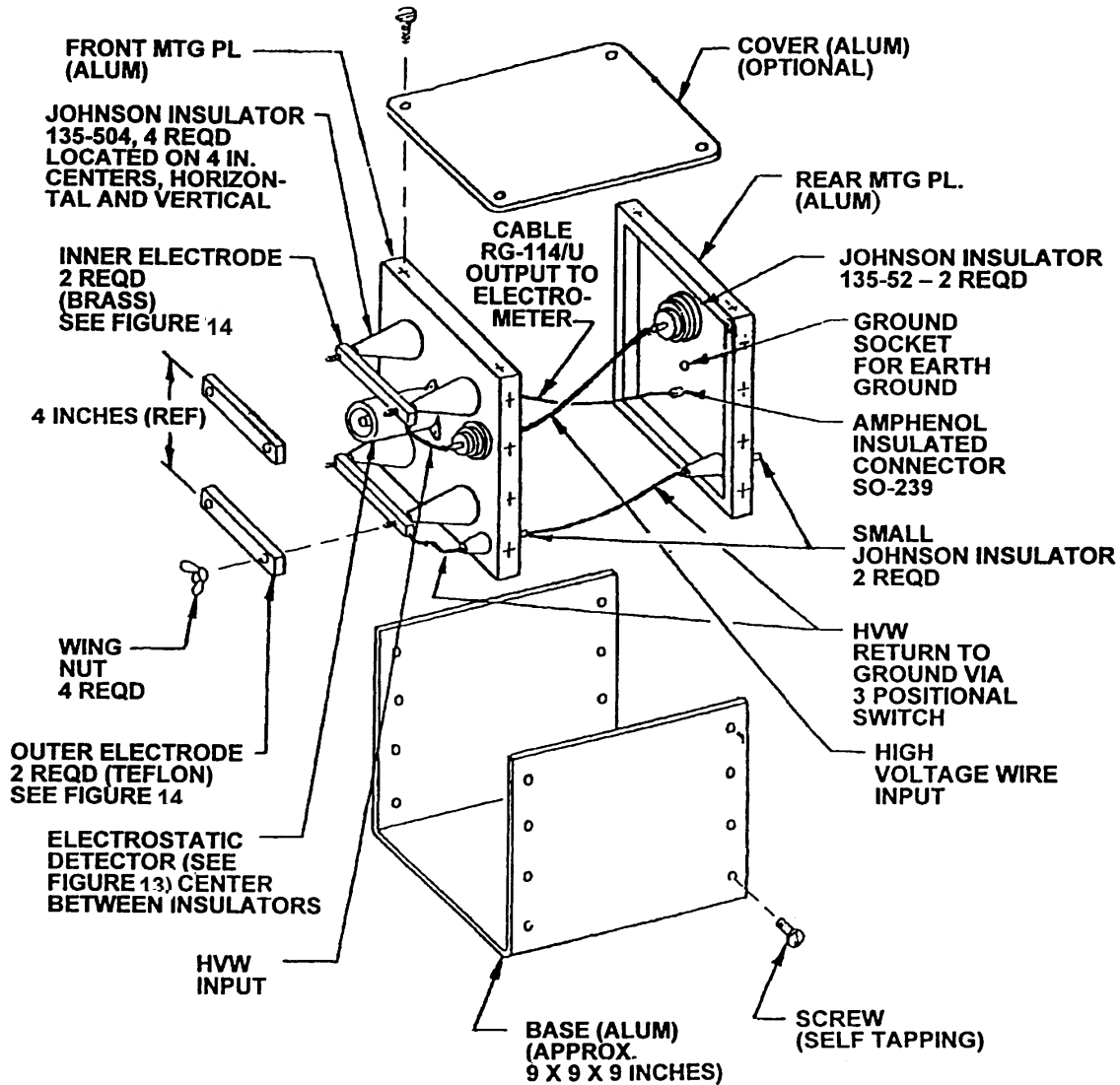
- a. Set recorder chart speed to 0.5 inch/second and turn on recorder.
- b. Set electrometer meter switch to indicate positive or negative charge, depending on the high voltage to be applied.
- c. Adjust the high voltage for the desired 5 KV potential.
- d. Set "operate" switch on electrometer to "operate."
- e. Turn the three-position control switch to high voltage.
- f. When the meter reading stops increasing, indicating the specimen has received its maximum charge, turn the three-position switch to ground position.
- g. When the meter needle reaches zero or after 10 seconds, whichever comes first, stop recorder and move "operate" switch to "zero check." Each specimen shall be charged 3 times for both positive and negative charges, allowing specimen to remain grounded for 10 minutes after each charging cycle to remove any residual charge on the specimen. For non-homogeneous materials, both surfaces shall be charged by reversing the face of the material in contact with the inner electrodes. At the conclusion of each charging test, measure the horizontal distance on the recorder chart from the point where the specimen was grounded to the point where the needle reached zero. With the speed of the chart known, the decay time for each specimen in seconds can be calculated.

5.4.3.5 Notes.

5.4.3.5.1 The purpose of this procedure is to evaluate the electrostatic buildup and dissipation properties of packaging materials used to protect electronic parts that are susceptible to damage by electrostatic discharge.

5.4.3.5.2 A Keithley 621 Electrometer may be used. Other settings may apply if another electrometer of different design is used.

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NOTE: AFTER ASSEMBLY, THE BRASS DISC OF THE ELECTROSTATIC DETECTOR SHALL BE ADJUSTED SO THAT THE DISTANCE BETWEEN THE DISC AND A MOUNTED SPECIMEN IS 1 INCH.

FIGURE 10. Electrostatic test unit.

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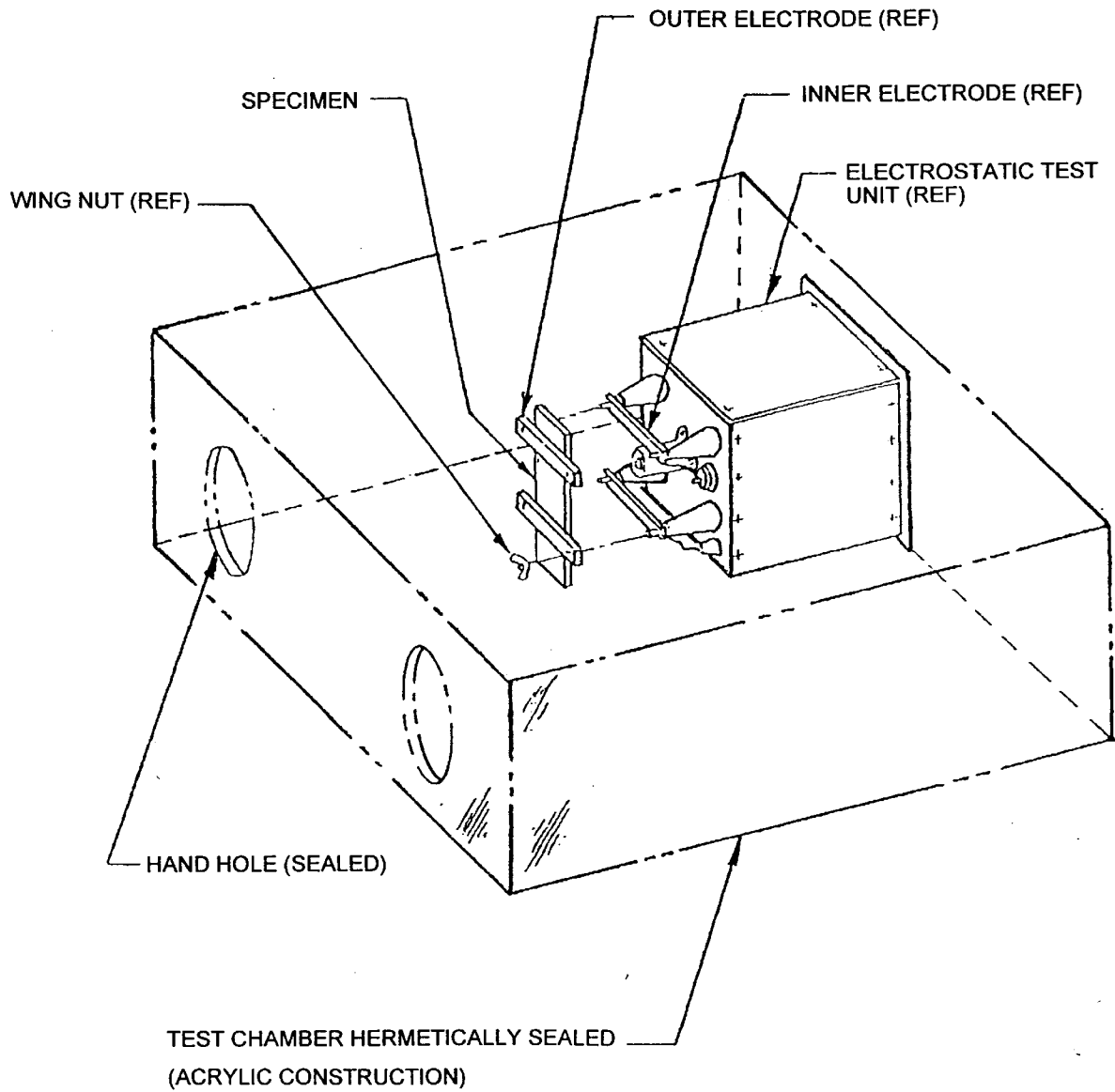


FIGURE 11. Electrostatic test chamber.

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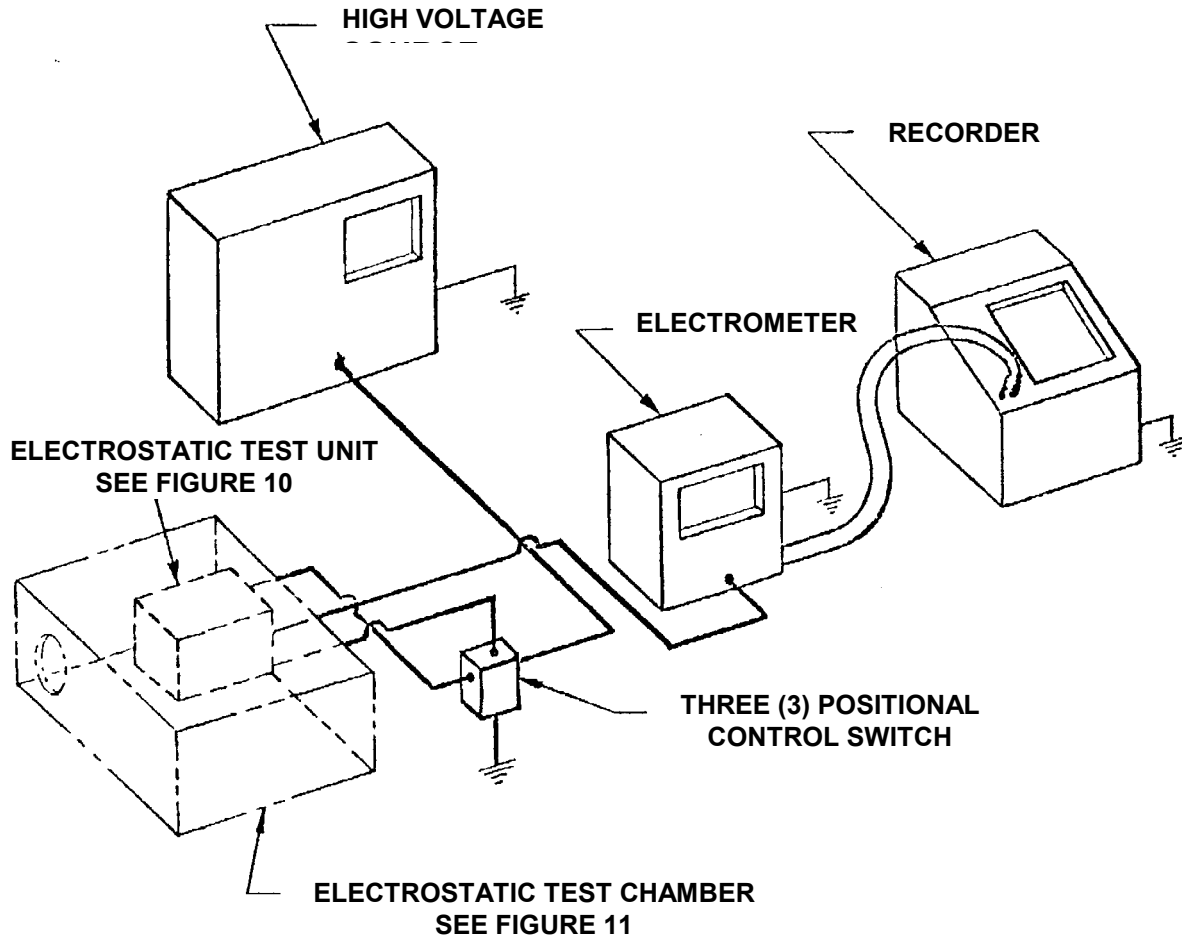
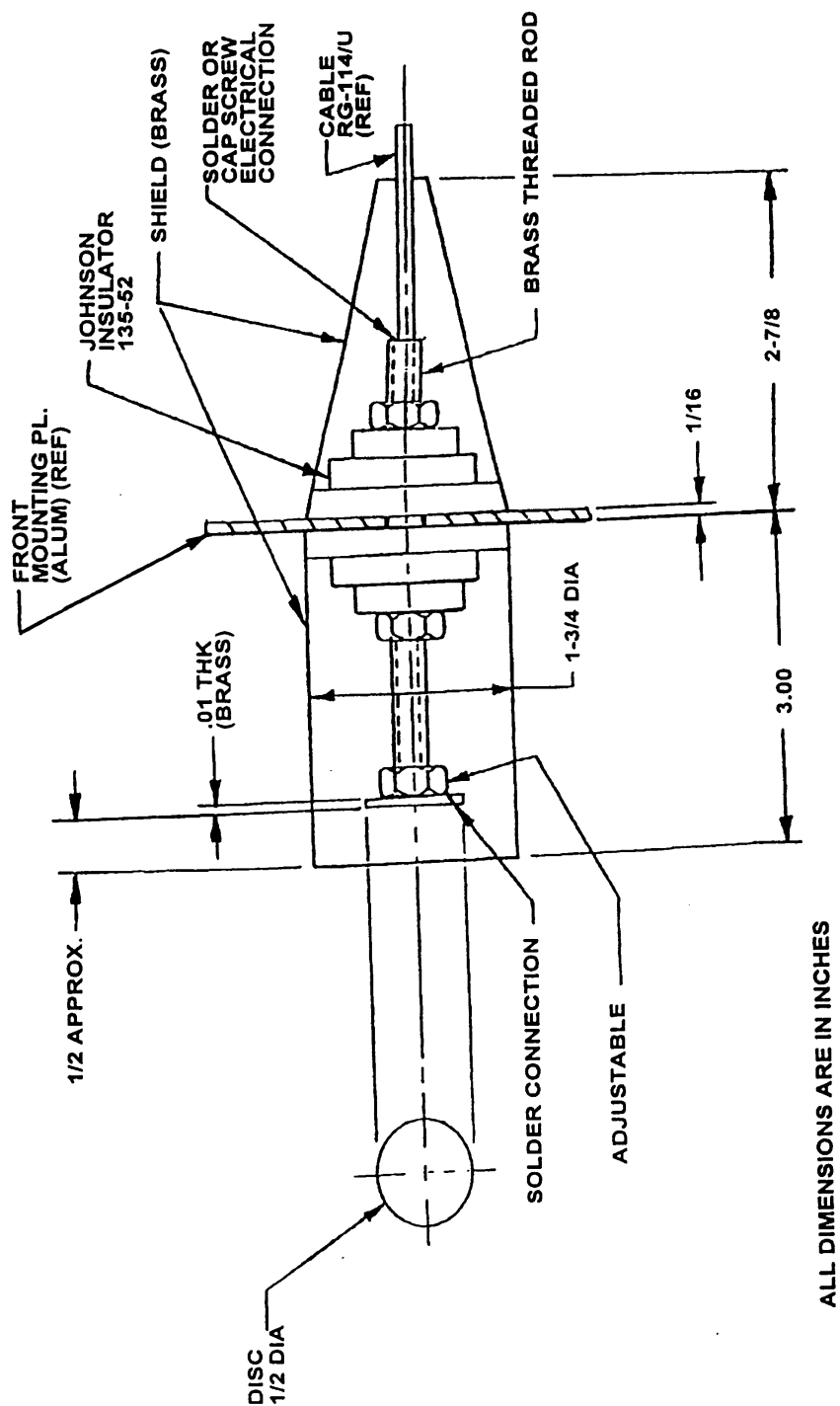


FIGURE 12. Electrostatic test arrangement.

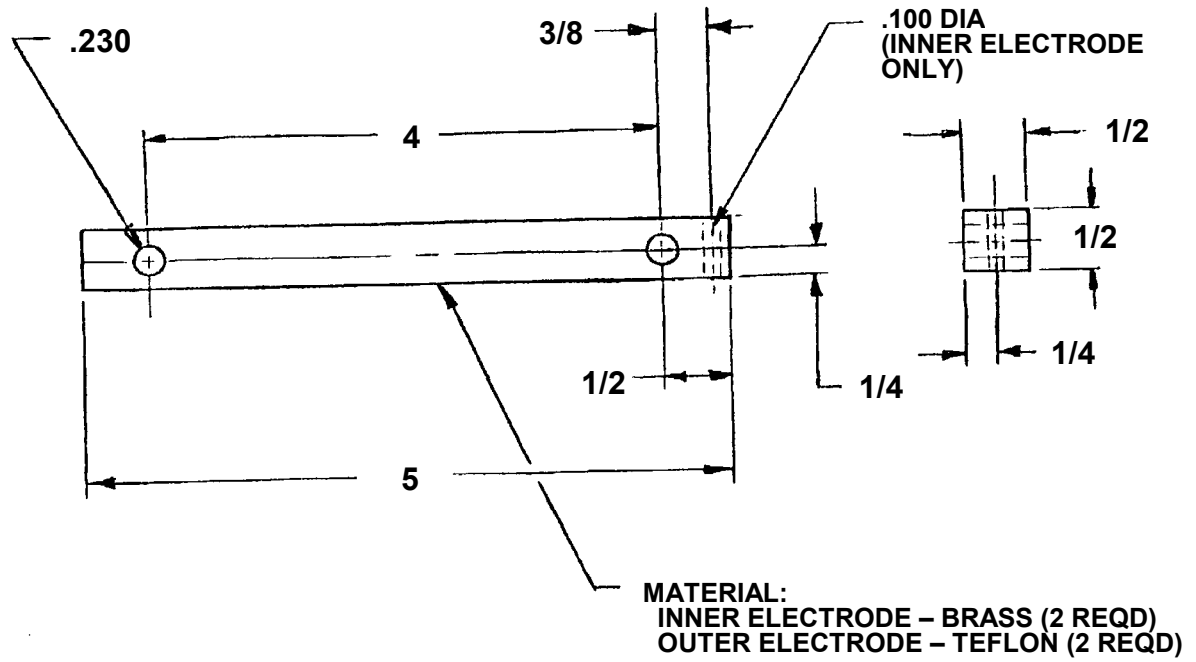
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ALL DIMENSIONS ARE IN INCHES

FIGURE 13. Electrostatic detector.

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ALL DIMENSIONS ARE IN INCHES

| FIGURE 14. Electrode.

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5.5 Properties of containers.5.5.1 Test Method 5009 – Leaks in Containers.

5.5.1.1 Scope. This test procedure details methods to determine leakage of air or liquids from sealed containers. Five test methods are specified; choice of method is determined by the physical characteristics of the container being evaluated (see 5.5.1.5).

5.5.1.2 Apparatus.

5.5.1.2.1 Vessels of sufficient size to allow complete submergence of the container being tested.

5.5.1.2.2 A vacuum pump and pressure gage if the vacuum retention test is being conducted.

5.5.1.2.3 A pressure gage and a supply of compressed air if the pneumatic pressure test is being conducted.

5.5.1.3 Test specimens. A specimen shall be one container and contents (actual or simulated), packed and sealed as for shipment.

5.5.1.4 Test procedures.5.5.1.4.1 Vacuum retention technique.

5.5.1.4.1.1 Specimen preparation. Provisions shall be made for connection of a tube to evacuate air and installation of a gage to indicate any loss in vacuum pressure. Such provisions shall be a tube and gage sealed into openings at the corners of a seam of a flexible container or a drilled and tapped hole for a plug or a valve stem incorporated in a rigid container or other acceptable devices that can be either sealed or removed from the container without adverse effect.

5.5.1.4.1.2 Testing. Connect the vacuum pump to the specimen and evacuate the air until the desired vacuum is attained. The vacuum pressure shall be 9 ± 1 millimeters of mercury or $5 \pm \frac{1}{2}$ inches of water. The required vacuum may be drawn more than once to ensure that equilibrium within the specimen is reached. When the specimen is evacuated to a constant specified pressure, stop evacuating air and note the vacuum pressure gage reading. After 10 minutes, the gage shall be read again to determine if there is a loss in vacuum pressure, indicating a leak.

5.5.1.4.2 Pneumatic pressure technique.

5.5.1.4.2.1 Specimen preparation. Provisions shall be made for connecting a tube or clamp-in valve to the specimen. Either attach a pressure gage to the specimen or use a low-pressure hand type tire gage to sense any loss in pressure. A tube or valve shall be sealed into an opening at

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one end of a seam in a flexible container, a hole drilled and tapped with a plug or a "clamp-in valve" stem incorporated in a rigid container, or other methods that will permit removal and seal without adverse effects of the serviceability of the container.

5.5.1.4.2.2 Testing. Pressurize the specimen with air from compressed air supply. Gradually introduce air until either the prescribed pressure in the specimen is attained or leakage becomes apparent. The pressure (P) in psi shall be calculated as follows:

$$P = \frac{\pi S}{(d_1 + d_2)}$$

where S = the specified strength of the barrier seams
 d_1 and d_2 = the two smaller dimensions of the package.

(For example, to test a package 10 by 6 by 4 inches enclosed in MIL-PRF-131 barrier material, the pressure shall be the specified strength of MIL-PRF-131 barrier seam (3 ½ lb./in.) times pi (3.14) divided by the sum of the smaller dimensions (6 + 4 inches); that is, the pressure shall be 1.1 lb./sq. in. For other sizes other pressures shall be calculated in a similar manner or read from an appropriate curve.)

CAUTION

Pneumatic pressure may cause explosive failure of weak specimens. The applied pressure should be no greater than necessary to reveal leaks.

When the specimen is pressurized to a constant specified pressure, read and record this initial pressure. After 30 minutes, read and document the final gage pressure. If no change is noted between the initial and final gage pressure, the item is considered satisfactorily sealed.

5.5.1.4.3 Squeeze technique. Squeeze technique is applicable only to flexible specimens.

5.5.1.4.3.1 Specimen preparation. During final sealing of the specimen, entrap as much air as possible within the specimen.

5.5.1.4.3.2 Testing. Either submerge the specimen 1 to 2 inches under water and, while squeezing the specimen to force air to the area under observation, observe all seams and surface for leakage; or coat all seams, joints, or other areas likely to leak with a bubble-supporting film and observe each for leaks while squeezing the specimen to force air to the area under observation.

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5.5.1.4.4 Hot water technique. Submerge the specimen in water heated to a temperature at least 50 °F above the initial temperature of the specimen but not over 110 °F for wax-dipped specimens. While holding the specimen submerged with the uppermost surface covered by not more than 1 inch of water, observe for at least 15 seconds to detect leakage. The specimen shall be rotated and observed repeatedly until all of the specimen has been examined. Total time in hot water shall not exceed 8 minutes.

5.5.1.4.5 Submersion (or immersion) techniques. The specimen shall be submerged so that the uppermost surface is beneath the water surface not less than 1 inch or more than 2 inches for 1 hour or longer in water maintained at a temperature at least 40 °F below the temperature at which the specimen is sealed. After submersion and before opening the specimen, carefully dry the outside of the specimen where the opening will be made. Then open the specimen and inspect the inside for leakage.

5.5.1.5 Notes.

5.5.1.5.1 Selection of technique. The most appropriate technique depends principally upon the construction, size and weight of the unit pack, and the information needed. The hot water technique is appropriate for large unit packs. The squeeze technique is appropriate for small unit packs constructed of flexible materials such as plastic film. The vacuum retention technique does not specifically locate leaks and may not indicate the existence of tiny leaks in a large unit pack. The submersion (or immersion) technique for detecting water leakage is not as sensitive as the air leakage tests, but it is appropriate to reveal whether or not water might leak into the unit packs and, depending upon the duration of the test, gives some indication of the extent to which the materials used in the pack are waterproof. The pneumatic pressure technique is primarily appropriate for rigid containers. Neither the hot water nor the pneumatic pressure techniques are appropriate for rigid containers that are sealed with tapes; the submersion technique shall be used.

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6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. This standard is a listing of uniform test methods that have been developed to evaluate properties of materials used in military packaging applications. It is intended that the test methods detailed in this standard be referenced (by number) in packaging material specifications. This eliminates the need to repetitively detail any standard test method in each material specification.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number, and date of the standard.

6.3 Subject term (keyword) listing.

Blocking resistance	Leakage
Contact corrosivity	Packaging materials
Curl resistance	Seam strength
Delamination	Test methods
Electrostatic properties	Volatile corrosion Inhibitors
Flex testing	Water vapor transmission rate

6.4 Changes from previous issue. The margins of this standard are marked with vertical lines to indicate where changes from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

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APPENDIX A

CHRONOLOGY OF PACKAGING MATERIALS TEST METHODS

A.1 SCOPE.

A.1.1 This appendix provides a cross reference and tracks test methods of government packaging test methods. This appendix is not a mandatory part of this standard. The information contained herein is intended for guidance only.

A.2 APPLICABLE DOCUMENTS. This section is not applicable to this appendix.

A.3 HISTORY. This Military Standard supersedes FED-STD-101C. The latter standard originally contained approximately 250 test methods for evaluation of military packaging materials. This number has been steadily reduced as a result of supersession by non-Government documents and deletion of methods as a result of little or no usage thereof.

A.4 CHRONOLOGICAL SUMMARY. To assist users following up references to test methods specified in FED-STD-101, all test methods that were listed in FED-STD-101C and FED-STD-101B are listed in Table A.I. Disposition of each of these methods is identified as well as a cross reference to the 3-digit test method identifier that was used in FED-STD-101A. All test methods listed in Table A.I that are dispositioned as "deleted" have been removed from the system over the years with no superseding information. Any document specifying these deleted test methods should be updated to reflect currently available alternative military or industry test method standards. Test method chronological summaries have not been changed as a result of Revision A to MIL-STD-3010.

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TABLE A-I. Test methods chronological summary.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
1001	X			238
1002	X			316
1003		X	MIL-STD-3010	332
1004		X	ASTM-D3652	---
2001	X			160
2002	X			255
2003	X			244
2004	X			162
2005	X			230
2006	X			231
2007		X	*TAPPI T810 or *TAPPI T807	250
2008		X	ASTM-F36	292
2009	X			293
2010	X			294
2011	X			296
2012		X	ASTM-D695	297
2013		X	ASTM-D2221	300
2014	X			165
2015		X	MIL-STD-3010	183
2016	X			304
2017		X	MIL-STD-3010	308
2018		X	ASTM-D1098	309
2019		X	ASTM-D790	310
2020		X	ASTM-D3499 ASTM-D3500 ASTM-D3501 ASTM-D3502 ASTM-D3503	311

* = Document is not DOD adopted.

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TABLE A-I. Test methods chronological summary – Continued.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
2021		X	ASTM-D1037	312
2022	X	X	ASTM-D1238	170
2023	X			242
2024		X	MIL-STD-3010	259
2025	X			313
2027	X			110
2028	X			340
2029	X			155
2030		X	ASTM-D3499 ASTM-D3500 ASTM-D3501 ASTM-D3502 ASTM-D3503	265
2031	X			112
2032	X			321
2033		X	ASTM-D2808	348
2034	X			234
2035	X			326
2036		X	*TAPPI T414	327
2037	X			248
2038	X			246
2039	X			247
2040		X	ASTM-D638	245
2041	X			225
2042	X			228
2043	X			224
2044		X	ASTM-D3953	227
2045	X			226
2046		X	ASTM-D1004	328

* = Document is not DOD adopted.

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TABLE A-I. Test methods chronological summary – Continued.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
2047	X			218
2048	X			107
2049	X			158
2050		X	ASTM-D3330	---
2051		X	ASTM-D3889	---
2052	X			---
2053		X	ASTM-D3813	---
2054		X	ASTM-D3654	---
2055		X	ASTM-D3654	---
2056	X			---
2057	X			---
2058		X	ASTM-D3662	---
2059	X			---
2060	X			---
2061		X	ASTM-D3759	---
2062		X	ASTM-D3759	---
2063		X	ASTM-D3759	---
2064		X	ASTM-D3811	---
2065		X	MIL-STD-3010	---
2071	X			---
3001	X			254
3002	X			337
3003		X	MIL-STD-3010	223
3004	X			341
3005		X	MIL-STD-3010	338
3006	X			339
3007	X			299
3008	X			229
3009	X			192

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TABLE A-I. Test methods chronological summary – Continued.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
3010	X			305
3011	X			163
3013	X			167
3014	X			306
3015		X	MIL-STD-3010	260
3016	X			261
3017	X			262
3018	X			187
3019	X			266
3020	X			233
3021	X			125
3022		X	ASTM-D779	270
3023	X			269
3024	X			268
3025	X			274
3026	X			329
3027		X	MIL-STD-3010	285
3028		X	MIL-STD-3010	345
3029		X	ASTM-D724	106
3030		X	MIL-STD-3010	286
3031	X			333
3032		X	ASTM-D3816	
3033		X	ASTM-D3833	
4001		X	ASTM-D4157 ASTM-D4158	287
4002	X			219
4003	X			289
4004	X		MIL-STD-2073-1	290
4005		X	ASTM-D1894	291
4006	X			256
4007	X			298

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TABLE A-I. Test methods chronological summary – Continued.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
4008	X			301
4009		X	ASTM-D1531	303
4010	X			222
4011	X			257
4012		X	ASTM-D1310	307
4013	X			177
4014	X			235
4015	X			131
4016	X			315
4017	X			317
4018		X	ASTM-E462 ASTM-E619	220
4019	X			174
4020	X			263
4023		X	ASTM-C148	320
4024	X			173
4025	X			267
4026		X	ASTM-B117	322
4027	X			323
4029	X			123
4030	X			134
4031		X	MIL-STD-3010	344
4032		X	ASTM-D1545 ASTM-D562	280
4033	X			281
4034		X	MIL-STD-3010	282
4035	X			283
4036		X	ASTM-D570	284
4037	X			271
4038	X			272

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APPENDIX A

TABLE A-I. Test methods chronological summary – Continued.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
4039	X			325
4040	X			133
4041	X			273
4042	X			275
4043		X	ASTM-C177	185
4044	X			276
4045		X	ASTM-D1003	277
4046		X	MIL-STD-3010	347
4047		X	ASTM-D3611	
4048	X			
4049	X			
4050		X	ASTM-D3815	
5001	X			217
5002	X			335
5003		X	ASTM-D642	295
5004	X			168
5005		X	ASTM-D4169	214
5006	X			302
5007		X	ASTM-D4169	216
5008		X	ASTM-D4169	213
5009		X	MIL-STD-3010	241
5010	X			314
5011		X	ASTM-D4169	236
5012		X	ASTM-D4169	212
5013	X			243
5014		X	ASTM-D4169	240
5015	X			239
5016		X	ASTM-D4169	209
5017		X	ASTM-D4169	210
5018		X	ASTM-D4169	215
5019		X	ASTM-D4169	278

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TABLE A-I. Test methods chronological summary – Continued.

FED-STD-101B and/or FED-STD-101C <u>Test Method</u>	<u>Disposition</u>		Superseding <u>Document</u>	Cross-reference to FED-STD-101A <u>3-Digit Test Method</u>
	<u>Deleted</u>	<u>Superseded</u>		
5020		X	ASTM-D4169	279
5021		X	ASTM-D1008 ASTM-D895	252
5022		X	*TAPPI T410	204
5023		X	ASTM-D4169	211
5024		X	*TAPPI T400	205
5025	X			319
5026	X			264
5027	X			208
6001	X			253
6002	X			203
6003	X			129
6004	X			135
6005	X			206
6006	X			207
6007	X			126
6009	X			288
6010	X			144
6011	X			237
6012	X			157
6013		X	ASTM-D1030	249
6014	X			318
6017	X			221
6018	X			232
6019	X			251
6020		X	ASTM-D549	324
6021	X			330
6022	X			331
6023	X			349

* = Document is not DOD adopted.

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Custodians:

Army – SM
Navy – AS
Air Force – 11

Preparing activity:

Navy – AS
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Review activities:

Army – AR, AV, CR3, GL3, MI, MR
Navy – OS, SA, SH
Air Force – 99

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