Designation: D4991 – 07 (Reapproved 2015)

Standard Test Method for Leakage Testing of Empty Rigid Containers by Vacuum Method[1]

This standard is issued under the fixed designation D4991; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the testing of empty containers for resistance to leakage under differential pressure conditions such as those which can occur during air transport. It is suitable for testing rigid containers intended for the transportation of some hazardous liquids in accordance with the United Nations Recommendations On The Transport Of Dangerous Goods (UN TDG) and the International Civil Aviation Organization Technical Instructions For The Safe Transport Of Dangerous Goods By Air (ICAO TIs).

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D996 Terminology of Packaging and Distribution Environments

D3078 Test Method for Determination of Leaks in Flexible Packaging by Bubble Emission

2.2 *United Nations Document:*

ST/SG/AC.10/1 Recommendations On The Transport Of Dangerous Goods[3]

2.3 *International Civil Aviation Organization:*

DOC 9284-AN/905 Technical Instructions For The Safe Transport Of Dangerous Goods By Air[4]

3. Terminology

3.1 *Definitions:* Terms and definitions used in this test method may be found in Terminology D996.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *rigid container*—a container sufficiently rigid that under a positive pressure of 100-kPa gage the volumetric expansion does not exceed 0.5 %, such as a glass bottle.

4. Summary of Test Method

4.1 An empty rigid container to be tested is pressurized by immersion in an ethylene glycol-water solution in a transparent test chamber and subjected to a gradually increasing partial vacuum. The container is observed for signs of leakage, as evidenced by escaping air bubbles during depressurization or fluid in the container after re-pressurization.

5. Significance and Use

5.1 Containers may be pressurized in accordance with this test method without modification to the closure or to the body of the container. This test method may be used for testing rigid containers intended for the transportation of some liquids by air in accordance with the ICAO TIs or in accordance with the UN TDG.

5.2 This test method establishes the point at which leakage commences, with a limit of approximately 95-kPa (13.8-psi) differential. See Test Method D3078 for flexible packages.

5.3 This test method may not be suitable for some packages, such as packages with paper cap seals, where the test fluid may rapidly deteriorate the packaging.

6. Apparatus

6.1 *Transparent Vessel,* large enough to permit the test specimen(s) to be immersed in the test fluid with a minimum headspace of 20 %, capable of withstanding at least 1½-atm pressure differential, fitted with a flat vacuum-tight cover is required.

6.2 *Vacuum Gage, Inlet Tube from a Source of Vacuum, and Outlet Tube to the Atmosphere,* shall be sealed into the cover. The inlet and outlet tubes shall be equipped with handoperated valves. The vacuum gage shall be laboratory quality with a full-scale range from 0 to 100 kPa (0 to 14.5 psi) with

[1] This test method is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.21 on Shipping Containers and Systems - Application of Performance Test Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from United Nations Sales Section, United Nations, New York, NY 10017.

[4] Available from Intereg Group, Inc., 5724 N. Pulaski Rd., Chicago, IL 60646.

minimum graduations no greater than 2 kPa (0.3 psi) and accuracy to within 1 %. A suitable moisture trap, to prevent back-flow of liquid, should be fitted between the vacuum gage and the source of vacuum.

6.3 *Solution of Ethylene Glycol in Water, 50 % by Volume* for use as a test fluid, at ambient temperature unless otherwise specified, is required. Where a test temperature other than ambient is specified, the solution shall be at the specified test temperature. Other solutions may be required for high and low temperatures.

NOTE 1—If ethylene glycol antifreeze solution is used, it should be clarified by filtering through activated charcoal.

6.4 A suitable means to hold the test specimen(s) with the closure(s) not more than 25 mm (1 in.) under the surface of the test fluid is required. The method of restraint should not affect the results of the test.

7. Test Specimens

7.1 Test specimens shall consist of containers, including their closures. Test specimens of containers which are in production shall be representative samples, taken at random.

7.2 Containers shall be empty containers which previously contained any substance and were cleaned of the contents prior to testing. New containers and closures, where available, should be used.

7.3 Each container shall be closed for testing in the same manner as it would be closed for actual shipment. All closures shall be installed using the techniques or torques specified by the closure manufacturer, container manufacturer, or shipper. Close containers at ambient pressure and temperature.

7.4 Where a test temperature other than ambient is specified, the sample containers shall be conditioned at the test temperature for at least 24 h, and until they reach equilibrium of temperature with the conditioning atmosphere.

7.5 Unless otherwise specified, test at least three specimens.

8. Preparation of Apparatus

8.1 Assemble the apparatus in accordance with Fig. 1.

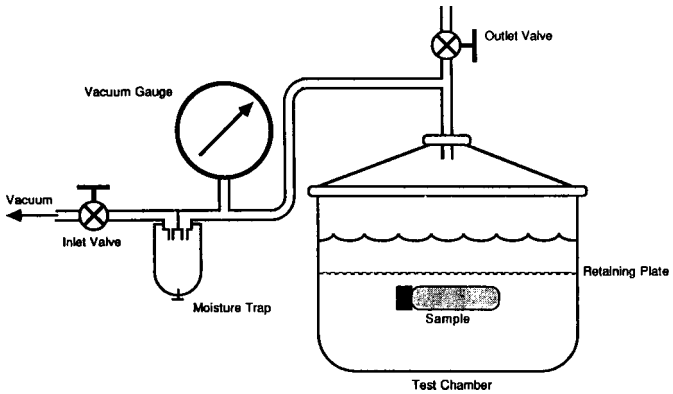

FIG. 1 Typical Test Setup for Leakage Testing of Containers by Vacuum Method

8.2 Prior to testing, draw a vacuum to at least 95-kPa (13.8-psi) differential and maintain this vacuum for 10 min in order to remove air dissolved in the test fluid.

9. Procedure

9.1 Immerse the test specimens in the ethylene glycol-water solution inside the transparent test chamber. Remove as many bubbles as possible from the outer surface of the test specimens by agitation or other means.

9.1.1 More than one specimen can be tested at one time provided that there is sufficient space in the test chamber to allow clear observation of each specimen. If this is done, affix the test specimens so closures are sufficiently separated to permit individual observation of each, to detect leakage.

9.2 Seal the lid, open the hand valve on the inlet tube, and close the hand valve on the outlet tube to the atmosphere. Turn on the vacuum so that the gage rises slowly (from 30 s to 1 min) to a pressure differential of 95 kPa (13.8 psi), or other specified pressure differential. The vacuum can be controlled by cracking open the hand valve on the outlet tube. During the rise in vacuum, observe the test specimen(s) for leakage as evidenced by a continuous stream or recurring succession of small bubbles. Isolated bubbles caused by trapped air are not considered evidence of leakage. Maintain the vacuum for a minimum of 10 min. For test specimens which are single packagings or combination packagings in whole or part of plastic material, maintain the vacuum for 30 min. Then turn the vacuum off and release it slowly by opening the hand valve on the outlet tube. Keep the test specimen(s) immersed for at least 30 s at ambient pressure. Remove the test specimen(s) and examine the inside for fluid.

9.3 Leakage is indicated by a continuous stream or recurring succession of bubbles. Fluid within the test specimen is also evidence of leakage.

10. Report

10.1 Report the following information:

10.1.1 Identification of the containers and closures, using manufacturer's part numbers and descriptions or using exploded view drawings or cross-sectional drawings, including detailed data on material construction,

10.1.2 Total number of specimens tested,

10.1.3 Number of specimens tested at one time,

10.1.4 Test pressure requirement, where applicable,

10.1.5 Whether test was performed at ambient temperature and if not, the test temperature. If a test solution other than 50 % ethylene glycol was used, identify the solution,

10.1.6 Whether test specimens passed or failed the test, where applicable,

10.1.7 Whether each test specimen leaked and at what pressure differential,

10.1.8 Whether test fluid was apparent in each test specimen after testing,

10.1.9 A statement to the effect that all tests were made in full compliance with the requirements of this test method, or noting any variations and detailing them, and

10.1.10 The name and address of the testing agency, the date, and the signature of a responsible representative of the testing agency.

11. Precision and Bias

11.1 No information is presented about either the precision or bias of this test method for measuring leaking of rigid plastic containers since the result is non-quantitative. It is recommended that test sensitivities be determined and a positive and negative control be used when validating the test method.

11.2 Once validated, the user does not have to apply the positive and negative controls before each test.

12. Keywords

12.1 containers; leakage testing; packaging; vacuum method

APPENDIX

(Nonmandatory Information)

X1. TABLE X1.1

TABLE X1.1 Pressure Conversion Table—Pressure Differential[A]

KiloPascals	Pounds per square inch (psi)	Inches of mercury in Hg (20°C)	Bar
10	1.45	2.96	0.10
20	2.90	5.93	0.20
30	4.35	8.89	0.30
40	5.80	11.85	0.40
50	7.25	14.82	0.50
60	8.70	17.78	0.60
70	10.15	20.75	0.70
75	10.88	22.23	0.75
80	11.60	23.71	0.80
85	12.33	25.19	0.85
90	13.05	26.67	0.90
95	13.78	28.16	0.95
100	14.50	29.64	1.00
101.325	14.70	30.03	1.01325

[A] psi × 6.894757 = kPa
in. Hg × 3.37411 = kPa
bar × 100 = kPa