Designation: D3078 – 02 (Reapproved 2021)ε1

Standard Test Method for Determination of Leaks in Flexible Packaging by Bubble Emission[1]

This standard is issued under the fixed designation D3078; 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.

ε[1] NOTE—Keywords were added editorially in January 2021.

1. Scope

1.1 This test method covers the determination of gross leaks in flexible packaging containing a headspace gas. Test sensitivity is limited to 1×10^{-5} atm cm^3/s (1×10^{-6} Pa m^3/s) or even less sensitive as indicated in a recent interlaboratory test (reported in Section 12).

1.2 Small leaks may not be detected by this procedure. Viscoelastic effects on the products, or entrapped air, become significant and prevent passage through small openings. Positive pressure inside the pouch after the vacuum is drawn may force the product to plug small leaks. The size of the leak that can be detected is dependent upon the products contained, the nature of the packaging material, and the test parameters selected.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E425 Definitions of Terms Relating to Leak Testing (Withdrawn 1991)[3]

E515 Practice for Leaks Using Bubble Emission Techniques

F98 Recommended Practice for Determining Hermeticity of Electron Devices by a Bubble Test (Withdrawn 1990)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *leak*—any opening in a flexible package that, contrary to intention, either allows the contents to escape or substances to enter.

4. Apparatus

4.1 *Vacuum Chamber*—Any transparent container capable of withstanding approximately one atmosphere pressure differential, fitted with a vacuum-tight cover. A vacuum gauge, an inlet tube from a source of vacuum, and an outlet tube to the atmosphere shall be connected to the chamber cover. The inlet and outlet tubes shall be equipped with hand valves. Attached to the underside of the cover shall be a transparent plate that will closely approximate the inside dimensions of the container and be such a distance from the top of the container that when it is two-thirds filled with fluid, the attached plate will be positioned 1 in. (25 mm) under the fluid.

5. Materials

5.1 *Immersion Fluids*—Use an immersion fluid which does not degrade the package being tested. Fluids with a low surface

[1] This test method is under the jurisdiction of ASTM Committee F02 on Primary Barrier Packaging and is the direct responsibility of Subcommittee F02.40 on Package Integrity.

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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] The last approved version of this historical standard is referenced on www.astm.org.

tension are generally more sensitive. Examples include water, water treated with a wetting agent, denatured alcohol, and mineral oil. Other possible fluids are listed in Practice E515 and Recommended Practice F98.

6. Sampling

6.1 The number of specimens used in the test sample may be varied according to the nature of the product, its cost, its size, and whether the specimens are taken from a production line in a normal packaging operation, or are few in number, or are to be used only for purposes of comparative evaluation of procedures or materials.

7. Test Specimen

7.1 *Flexible Package,* with or without its intended contents.

8. Conditioning

8.1 The test sample and test fluid shall be at equilibrium with normal room temperature.

9. Procedure

9.1 Submerge the specimen in fluid contained in the vessel within the vacuum chamber. The uppermost surface of the specimen shall be covered by not less than 1 in. (25 mm) of fluid.

NOTE 1—Two or more small packages may be tested at the same time, provided that they are placed in such a manner that all parts of every package under test can be observed for leakage during the test.

9.2 Set the cover on the vacuum chamber, close the outlet valve, and turn on the vacuum so that the gauge rises slowly (approximately 1 in. Hg/s) to a selected vacuum level. The vacuum level chosen should be as large as possible in order to ensure optimal sensitivity of the test. Limiting factors will include package fragility, the degree of package expansion, and the test-fluid vapor pressure.

9.3 During the rise in vacuum, observe the submerged specimen for leakage in the form of a steady progression of bubbles from the flexible container. Isolated bubbles caused by entrapped air are not considered as leaks. Also note the approximate increase in package volume. The pressure differential of the test is inversely related to the volume increase of the sample; therefore, large volume increases significantly detract from the severity of the test. Flexible packaging with little or no headspace cannot be reliably evaluated with this test method.

9.4 Hold the vacuum for a specified time period; 30 s is recommended, but this may be set at the tester's discretion.

9.5 Release the vacuum, remove the lid, and examine the specimen for the presence of test fluid inside the specimen.

10. Interpretation of Results

10.1 If there are bubbles definitely attributable to leaks in a specimen during the rise of vacuum, or when held at full vacuum, the specimen fails the test.

10.2 If test fluid attributable to a leak is inside a specimen, the specimen fails the test.

10.3 If there are no bubbles observed attributable to leaks, and if no test fluid attributable to a leak is inside a specimen, the specimen passes the test.

11. Report

11.1 Report the following information:

11.1.1 A statement that the test was conducted in compliance with this test method or a description of the deviations from this test method.

11.1.2 Identification of the specimen and the specific material tested.

11.1.2.1 Identification of the test fluid and the maximum vacuum level employed;

11.1.2.2 A statement regarding the approximate average and range of sample expansions when at maximum vacuum; and

11.1.2.3 The time period held at maximum vacuum.

11.1.3 A statement whether or not leakage occurred, and if possible, a report of the location of each leak.

11.1.4 A statement of the number of specimens included in the test and the number of failures, if any.

11.1.5 When the test is performed to check compliance with requirements, a statement that the sample did or did not meet the requirement, and identification of the source for the requirement.

11.1.6 When the test is conducted to evaluate or compare products, materials, or methods, a statement of any observations that may lead to improvements.

12. Precision and Bias

12.1 An interlaboratory test[4] was conducted to determine each participating laboratory's ability to detect leaks of various sizes when tested at various vacuum levels in accordance with the test method. If a leak was detected, the participant was then asked to quantify the size of the leak by determining the time required to leak ½ mL of air.

12.1.1 Details of the test protocol, including a description of the apparatus, are in preparation and will be available as a research report.

12.1.2 Three laboratories participated with each providing three independent researchers. Each of these tests were replicated three times by each researcher. The same measured leaks and test apparatus were used at each location.

12.1.3 The three vacuum levels tested ("low", "medium," and "high" were relative terms used only to differentiate conditions), were:

Low vacuum	12.5 ± 0.5 in. Hg
Medium vacuum	18.5 ± 0.5 in. Hg
High vacuum	24.5 ± 0.5 in. Hg

12.1.4 The leaks used ("big", "medium," "small," and "very small" were relative terms used only to differentiate variables) were characterized by a helium leak detector as listed below. By most standards, all of these leaks were quite large.

Big	6 E-02 cc/s He
Medium	7 E-03 cc/s He
Small	3 E-03 cc/s He
Very small	1 E-04 cc/s He

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F02-1029. Contact ASTM Customer Service at service@astm.org.

12.1.5 Precision, characterized by repeatability, *Sr, r*, and reproducibility, *SR, R* has been determined for the materials as shown in the tables that follow.

12.1.6 Values reported below are in seconds to leak ½ mL of air except for the last material, "very small", which was either reported as bubble produced within 1 min (1) or no bubble produced (0).

12.1.7 Precision Statement for Test Condition: Low Vacuum (12 in. Hg)

Materials	Average	*Sr*	*SR*	*r*	*R*
Big 6E-02	64.778	7.772	14.186	21.762	39.721
Medium 7E-03	181.185	10.653	12.601	29.828	35.284
Small 3E-03	739.148	30.264	100.144	84.738	280.404
Very small 1E-04	0.000	0.000	0.000	0.000	0.000

Under this relatively weak vacuum (12 in. Hg), the "very small" leak at 1 E-04 cc/s Helium did not produce any bubbles within 1 min, which is taken as "non-detectable." All other leaks were easily detected.

12.1.8 Precision Statement for Test Condition: Medium Vacuum (18 in. Hg)

Materials	Average	*Sr*	*SR*	*r*	*R*
Big 6E-02	26.111	2.667	3.918	7.467	10.971
Medium 7E-03	82.111	4.073	6.196	11.406	17.350
Small 3E-03	365.000	18.963	32.549	53.096	91.138
Very small 1E-04	0.037	0.192	0.192	0.539	0.539

With this medium level of vacuum (18 in. Hg), the "very small" leak produced a bubble 1 time out of 27 trials. This is essentially "non-detectable." All other leaks were easily detected.

12.1.9 Precision Statement for Test Condition: High Vacuum (24 in. Hg)

Materials	Average	*Sr*	*SR*	*r*	*R*
Big 6E-02	10.481	1.000	2.187	2.800	6.122
Medium 7E-03	30.407	1.678	4.650	4.698	13.019
Small 3E-03	119.037	9.724	24.987	27.227	69.962
Very small 1E-04	0.778	0.000	0.441	0.000	1.235

With this highest vacuum level used (24 in. Hg), the "very small" leak produced a bubble in 78 % of the trials. This seems to indicate that a leak of the size of 1 E-04 is "detectable" most of the time but is close to the detection limit of the method and test conditions.

13. Keywords

13.1 bubble emission; determination; flexible packaging; leaks