

# **BAM: Examination of Flexible and Semirigid Food Containers for Integrity**

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## **Bacteriological Analytical Manual. Chapter 22C Examination of Containers for Integrity**

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Flexible and semirigid food packages are composed mainly or in part of plastic materials. Closure is achieved by heat sealing or double seaming. The 4 main groups of packages that cause similar integrity concerns and that are examined by common methods are paperboard packages, flexible pouches, plastic cups and trays with flexible lids, and plastic cans with double-seamed metal ends.

The purpose of a hermetic closure is to provide a barrier to microorganisms and to prevent oxygen from degrading the food. Closure integrity is significant because sealing surfaces may contain food particles and moisture that contribute to heat-seal and double-seam defects. Critical control must be exercised in this operation. Visual examination will reveal most defects. For many flexible packages, seal strength may be ascertained by squeezing.

### **A. Package examination**

Note condition of package (exterior and interior) and quality of seals or seams; observe and feel for gross abnormalities, mechanical defects, perforations, malformations, crushing, flex cracks, delamination, and swelling. Measure dimensions as recommended by manufacturer of closing equipment or packaging material. Perform teardown procedure as described. Note condition of package and closure. If there is evidence that a package may lose or has lost its hermetic seal, or that microbial growth has occurred in the package contents, further investigation is required.

#### **1. Visual examination**

Use hand as well as eye. A magnifying glass with proper illumination is helpful. Rub thumb and forefinger around seal area, feeling for folds and ridges. Rub fingers over flat surfaces to feel for delamination, roughness, or unevenness. By sight and touch, determine presence of defects. Mark location of defects with indelible ink. See Fig. 30 for visual inspection criteria for closure seal.



Figure 30. Visual inspection criteria for closure seal.  
(Courtesy of Brik Pak, Inc.)

2. Examination of packages (see Tables 1 and 2)

Table 1. Test methods for plastic packages containing food (5)				
Test methods	Package type			
	Paper board	Flexible pouch	Plastic, heat-sealed lid	Plastic, double-seam metal end
Air leak testing	O	O	O	O
Biotesting	O	O	O	O
Burst testing	O	R	R	O
Chemical etching	O	O	O	NA
Compression, squeeze testing	R	O	O	O
Distribution (abuse) test	O	O	O	O
Dye penetration	R	O	R	O
Electester	O	NA	NA	NA
Electrolytic	R	O	R	NA
Gas leak detection	O	O	O	O
Incubation	R	R	R	R
Light	NA	O	O	O
Machine vision	O	O	O	O
Proximity tester	O	O	O	R
Seam scope projection	NA	NA	NA	R
Sound	R	NA	R	R
Tensile (peel) testing	NA	R	R	NA
Vacuum testing	NA	O	R	O
Visual inspection	R	R	R	R

Abbreviations: R, test method is recommended by NFPA Bulletin 41-L, Flexible Package Integrity Bulletin; O, other commercially accepted test method applications; NA, test method is inappropriate for this style package.

Table 2. List of visible package defects provided by National Food Processors Association (5)

Defect	Package type <sup>(a)</sup>			
	Paper board	Flexible pouch	Plastic, heat-sealed lid	Plastic, double-seam metal end
<b>Abrasion</b>	+	+	+	+
<b>Blister</b>	-	+	-	-
<b>Burnt seal</b>	-	-	+	-
<b>Channel leak(er)</b>	-	+	+	-
<b>Clouded seal</b>	-	+	-	-
<b>Compressed seal</b>	-	+	-	-
<b>Contaminated seal</b>	-	+	+	-
<b>Convolution</b>	-	+	-	-
<b>Corner dent</b>	+	-	-	-
<b>Corner leaker</b>	+	-	-	-
<b>Crooked seal</b>	-	+	-	-
<b>Crushed</b>	+	-	+	+
<b>Defective seal</b>	-	-	+	-
<b>Deformed</b>	+	-	-	-
<b>Deformed seal</b>	+	-	-	-
<b>Delamination</b>	+	+	+	+
<b>Embossing</b>	-	+	-	-
<b>Flexcracks</b>	-	+	+	+
<b>Foreign matter (inclusion)</b>	-	-	+	+
<b>Fracture</b>	-	+	+	+
<b>Gels</b>	-	-	+	+
<b>Hotfold</b>	-	+	-	-
<b>Incomplete seal</b>	-	-	+	-
<b>Label foldover</b>	-	-	+	-
<b>Leaker</b>	-	+	-	-
<b>Loose flaps</b>	+	-	-	-
<b>Malformed</b>	-	-	+	+
<b>Misaligned seal</b>	-	-	+	+
<b>Nonbonding</b>	-	+	-	-
<b>Notch leaker</b>	-	+	-	-
<b>Puncture</b>	+	+	+	+
<b>Seal creep</b>	-	+	-	-
<b>Seal leaker</b>	+	-	-	-

<b>Seal width variation</b>	-	-	+	-
<b>Shrinkage wrinkle</b>	-	-	+	-
<b>Stringy seal</b>	-	+	-	-
<b>Swell (swollen package)</b>	+	+	+	+
<b>Uneven impression</b>	-	-	+	-
<b>Uneven seal junction</b>	-	+	-	-
<b>Waffling</b>	-	+	-	-
<b>Weak seal</b>	+	-	-	-
<b>Wrinkle</b>	-	+	+	-

<sup>a</sup> +, Definition is applicable to that package type; -, definition is not applicable.

## II.

### i. Paperboard packages (20)

**Teardown procedures.** Unfold all flaps (except gable top packages); check integrity and tightness of transverse (top and bottom) and side (vertical or longitudinal) seals by firmly squeezing package. If package has longitudinal sealing (LS) strip, pull off overlapping paper layer at side (longitudinal) seal. Check air gap of longitudinal sealing strip application (about 1 mm). Squeeze package and check that there are no leaks or holes in the LS strip.

Next, on side opposite side seal, puncture container with sharp scissors and empty contents. Saving side seal portion, cut near fold at each end of package and down length of package to remove a large rectangular body portion. Observe this large rectangular body portion for holes, scratches, or tears anywhere on the surface. Pay close attention to corners of package, particularly directly under end seals and near the straw hole or pull tab, if present. Now cut remaining package in half through the center of the side seam. Wash both halves of remaining package and dry them with a paper towel. Mark to identify the package.

Evaluation procedures for seal quality differ between package designs, constructions, and sealing methods. Obtain specific procedures for a given package from the manufacturer. For example, seal evaluation may consist of starting at one end of the seal, and very slowly and carefully pulling the seal apart. In some packages the seal is good if the polymer stretches the entire length of the seal (that is, stretching of polymer film continues to a point beyond which paper and laminates have separated). In other packages, fiber tear can be seen the entire length of the seal (that is, raw paperboard is visible on both sides of the separated seal areas). This is known as 100% fiber tear and indicates a good seal. Test all 3 seals of each package half. Problems to look for are absence of (or narrow) fiber tear, lack of polymer stretch, "cold spots" (no polymer bond in seal area), and "tacking" (polymer melt but no stretch or fiber tear). For longitudinal sealing strip-type packages, additional

tests (such as centering examination, heat mark examination, and appearance of aluminum foil examination when stripped) should be made according to manufacturer's directions.

**Electrolytic and dye testing.** These tests differ according to each system manufacturer's filed procedure. Contact the individual manufacturer, obtain recommendations, and follow them.

ii. Flexible pouches (20)

1. **Teardown procedures.** Check tightness of both head and side seals by squeezing each package from each fill tube or sealing lane. Important points are corners and crossing of head and side seals. This is a rapid determination of obvious defects. Each seal must be accurately torn apart and evaluated for correct integrity. Carefully inspect edges of each head and side seal for evidence of product in seal areas. No product should be visible.

Observe width of each seal area. Width must comply with machine-type specifications: for example, 1/16 inch minimum on all head and side seals for fill tube or sealing lane machines. Look for presence of smooth seal junction along inside edge of seal. Open each package to check side seals and head seals. Visually inspect for such defects as misaligned seal, flex cracking, nonbonding, and seal creep. If applicable, tear the seals by doing a seal tensile strength test or a burst test. Then observe appearance of tear at each seal. Seals should tear evenly so that foil and part of laminated layer from one side of package tears off, adhering to seal on other side of package. The seal should appear rough and marbled. The seal is also adequate if the foil is laid bare across entire length of seal. Retain records of test results as required.

2. Other test procedures

**Squeeze test.** Apply manual kneading action that forces product against interior seal surface. The sealing surface must be smooth, parallel, and free of wrinkles. Examine all seal areas for evidence of product leakage or delamination. Packages that exhibit delamination of the outer ply on seal area but not at product edge should be tested further by manually flexing the suspect area 10 times and examining all seal areas for leakage or reduction in the width of the seal area to less than 1/16 inch.

**Seal tensile strength.** Results to be expressed in pounds per linear inch, average of sample (that is, 3 adjacent specimens cut from that seal) should not be less than specified for the material and application.

**Burst strength test.** With internal pressure resistance as the measurement to check all seals, apply uniform pressure, under designated test conditions, to a level of not less than specified for a material or application, for 30 s. Then evaluate seals to ensure that proper closure seal is still in effect.

iii. Plastic package with heat-sealed lid (20)

**Container integrity testing.** Peel test procedures of form fill and seal containers. Squeeze container side walls of entire set from a mold. Squeeze each cup to cause 1/8 inch bulge of lid area. Lid should not separate from package when package is squeezed. Observe sealing area for fold-over wrinkles in sealant layer of lidstock. From a first set of containers, visually observe embossed ring in sealed area for completeness. (Embossed ring should be at least 90% complete if present.) Remove a second set of containers (1 cup per mold) and gently peel back each lid at approximately a 45 angle. Observe the peeled area for a generally frosty appearance on both the lid and cup sealed surfaces. Observe entire package for holes, scratches, even flange widths, smooth inside surfaces, and any deformities caused by dirty mold or sealing die.

**Leak test procedures (optional).** These tests differ according to each system manufacturer's filed procedure. Contact the individual manufacturer, obtain recommendations, and follow them.

**Electrolytic test.** Plastic packages generally do not conduct a flow of low-voltage electricity unless a hole is present. Use a volt meter or amp meter to determine the presence of a closed circuit. If a voltage flow can be measured, use a dye solution to identify the presence of a hole.

**Dye penetration test.** Use a dye to locate leaks in packages or to demonstrate that no leaks exist.

**Air pressure or vacuum test.** Apply pressure or vacuum to a closed package to test for holes and to observe any loss of pressure or vacuum. Underwater vacuum testing may reveal a steady stream of small bubbles emitting from a hole in a package.

iv. Plastic cans with double-seamed metal ends (20)

Procedures for examining metal cans with double seams are described in Chapter 21 and in 21 CFR, Part 113. Use these methods to examine plastic cans with double-seamed metal ends. Make the following changes to 21 CFR 113.60 (a,1,i,a and b).

### III. Micrometer measurement system

**Metal cans.** Required: cover hook, body hook, width (length, height), tightness (observation for wrinkle), and thickness. Optional: overlap (by calculation) and countersink.

**Plastic cans with double-seamed metal ends.** Required in addition to seam scope examination: thickness and tightness. Compare seam thickness to that calculated from individual thicknesses of plastic flange and neck and metal end, excluding compound. Optional: cover hook, countersink, and width (length, height).

Seam scope of projector

**Metal cans.** Required: body hook, overlap, tightness (observation for wrinkle), and thickness by micrometer. Optional: width (length, height), cover hook, and countersink.

**Plastic cans with double-seamed metal ends.** Required: overlap, body hook, countersink, width (length, height). Optional: cover hook.

**Visual examination for plastic cans with double-seamed metal ends.** Required: tightness. Note compression of pressure ridge or flange during overlap measurement. Remove entire cover and examine pressure ridge for continuity. Under 21 CFR 113.60 (a,1,i,c) add the following: pressure ridge for plastic cans with double-seamed ends; impression around complete inside periphery of can body in double seam area.

### IV. Microleak detection

Microleak testing methods are not listed in order of sensitivity, nor is it necessary to use them all. Each test has advantages and disadvantages, depending on the package, equipment, and set of conditions. Optional methods are appropriate when additional information will clarify the nature of various package defects. Some test methods are not appropriate for some package materials, closures, or package styles. Refer to the manufacturer of the package or closure system for recommended test methods or see Table 1. Common methods are presented to provide the analyst with procedures and options. Visible defects of the 4 flexible package groups are summarized in Fig. 30.

Measure packages before testing for microleaks. Mark visually detected defects to aid location during or after microleak testing (non-water soluble markers are recommended). Record all results, methods used, and environmental conditions (temperature, relative humidity) and retain these records. Conduct all tests in the standard laboratory atmosphere of  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity. When this is not possible, report temperature and relative humidity along with test results (14).

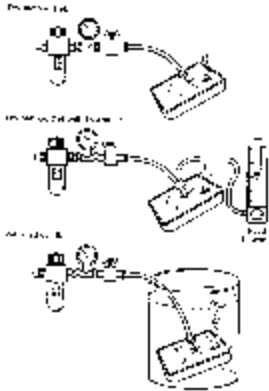


Figure 31. Air leak testing of packages.

1. Airleak testing (5) (Fig. 31)

i. Dry method

1. Materials

Compressed air with regulator  
Needle, valve, hoses  
Pressure gauge or flow meter

2. Procedure

Puncture container wall with needle. Inject air while increasing at 1 psi/s until a standard pressure is reached. Standard pressure used for testing should be less than the normal unrestrained burst pressure for the package. Observe pressure gauge for loss of internal pressure over a 60 s period. If a flow meter is used, observe for airflow, which indicates presence of openings in the test package. Dye testing may be used to locate air leaks that are not visible with the dry method. Inject air to create internal pressure within the package without causing it to burst. Observe all surfaces and seals for air leaks. Observe flow meter for indication of air loss from the package.

ii. Wet method

1. Materials

Compressed air with regulator  
Needle, valve, hoses  
Water  
Transparent container to observe bubbles

2. Procedure



Inject air to create internal pressure within the package without causing it to burst. Immerse package in water and inspect visually for a stream of bubbles emitting from a common source.

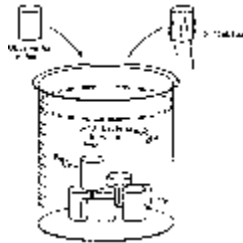
iii. Results

**Positive.** - A steady stream of bubbles comes from the package at one or more locations.

**Negative.** - No bubbles are emitted from the package.

**False positive.** - Bubbles are emitted from point at which needle entered package; or bubbles cling to surface of the package after package is submerged in water.

**False negative.** - Food particles block holes through which air might escape from defective package; or air pressure used is insufficient to force air through minute holes in package.



- 2.
3. Figure 32. Biotesting of packages.
4. Biotesting (5,21) (Fig. 32)

The objective of biotesting is to detect the presence of holes in hermetic packages by placing them in an agitated solution of fermentation bacteria in water for an extended period of time.

Obtain representative packages and submerged them in an agitated solution of active bacteria. The bacterial concentration should be  $>10^7/\text{cm}^3$ . The temperature of the solution that surrounds the packages should be maintained at a temperature that permits rapid growth of the bacteria within any packages they may enter. However, growth of the bacteria in the liquid surrounding the submerged packages is not desirable. The bacteria must cause fermentation of the product within the package if they penetrate and must not be pathogenic. Packages should be flexed during immersion to expose cracks and holes to incursion. The solution that surrounds the packages should be maintained at a temperature that permits rapid growth of bacteria within defective packages. After biotesting, packages are incubated for 3 weeks at 95-100F. This test should be used only to evaluate new package designs or to validate packaging systems. It should not be used as routine quality control procedure. Other methods are cheaper, simpler, and just as reliable.

a. Materials

Water bath with temperature control and agitation solution of

*Enterobacter aerogenes* for foods, pH >5.0. Solution of

*Lactobacillus cellobiosus* for foods, pH <5.0

Sample packages

Apparatus to flex packages

Incubator

b. Procedure

Obtain representative samples. Mix active bacteria in water at about  $1.0 \times 10^7$ /ml. Immerse samples in mixture. Agitate water bath and flex sample for 30 min. Remove packages and rinse with chlorinated water. Incubate samples for 2 weeks at 95-100F. Observe packages for swelling for 3 weeks. Open each package by cutting in half across the middle, leaving a hinge and observe contents for spoilage. Thoroughly wash insides of both halves from each spoiled package. Subject each half to a dye test to locate leaks.

c. Results. Report location of leaks.

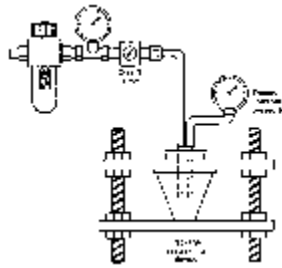


Figure 33. Burst tester.



Figure 34. Pouch air burst tester.

5. Burst testing (5) (Figs. 33 and 34)

The objective of burst testing is to provide a means for determining the ability of a hermetically sealed package to withstand internal pressure (psig). The entire package is subjected to uniform stress and failure generally reveals the weakest

point. Both restrained and unrestrained burst testing may be used. Restraint limits expansion by minimizing the angle of the package seal, which becomes greater as a package is inflated. With restraint, packages with strong seals fail at greater internal pressure than do packages with weak seals. Thus, use of a restraining device during burst testing permits noticeable separation between packages having strong or weak seals.

Fused seals are stronger than the walls of a flexible package. Burst failure generally occurs adjacent to fused seals. Peelable seals are weaker than the walls of a flexible package, and less pressure is needed to induce pressure failure. Lower pressure and a longer time increment are required to burst test peelable seals.

Dynamic burst testing involves a steady increase of internal pressure until failure occurs. Static burst testing involves a steady increase in internal pressure to a pressure less than failure, followed by a 30-s hold. Both methods are used for packages with fused seals. Peelable seals are burst-tested by inflating at a steady rate to a point less than failure pressure and held for 30 s, followed by a 0.5 psig pressure increase and another 30-s hold. Pressure and time indexing is continued with observation of the seal area for seal separation (peeling) until failure occurs.

- . Materials Compressed air or water  
Regulation valve  
Needle with gasket and pressure tubing  
Solenoid with timer(s)  
Pressure indicator(s), digital or gauge with sweep hand  
Restraining device (optional)
- i. Procedure

Use empty sealed package, or cut and remove contents of a filled package. Place package in restraining fixture (if used). Pierce package with gasketed needle(s) and inject air or water. Inflate at 1 psig/s.

**Dynamic method.** Continue inflation at 1 psig/s until failure occurs. Record internal pressure at failure.

**Static method.** Inflate at 1 psig/s to specified internal pressure, and hold at specified pressure for 30 s. Record as pass or fail.

**Indexed method.** Inflate to 5 psig and hold for 30 s, inflate additional 0.5 psig and hold for 30 s. Continue increase and hold sequence until failure occurs. Observe peelable seal separation. Report internal pressure at failure.

ii. Results

**Positive.** Pressure failure occurs below specified level of performance, indicating a hole in the package.

**Negative.** No pressure failure occurs below specified level of performance.

**False positive.** A leak is present at point where air or water is injected into package and pressure cannot be maintained.

**False negative.** A small leak occurs, but is not sufficient to reduce pressure noticeably.

6. Chemical etching (5)

Multilaminate and composite packaging materials may be etched to remove overlying layers, revealing the hermetic seal of packages that have polyolefin heat seals. This allows comparison of visually detected package defects on the external surface before etching and within the seal area after all external layers have been removed.

**Composite paperboard packages.** The outer layers of a package are removed by tearing, abrasion, and chemical action to expose the sealant layer intact. By photographing or photocopying the package before etching, the etched seal can be compared with the photograph to determine the significance of visually discernible defects.

. Materials

Water bath and heater with thermostat  
Three 1-L Pyrex glass beakers  
Running tap water  
Graduated cylinder  
Automatic stirring device (heated is preferred)  
Drying oven equilibrated to 65°C (150F)  
Paper towels  
Rubber gloves, protective goggles, apron, tongs  
Fume hood with chemical-resistant surface

Chemicals for etching of paperboard aseptic packages:

hydrochloric acid (HCl) solution, 3.7 N  
acidified solution of copper chloride (CuCl<sub>2</sub>)  
saturated solution of bisodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in water

- Preparation of solutions. **CAUTION:** Always pour acid into water; never pour water into acid.

Pour 0.5 L of concentrated HCl into 1 L of cold distilled water. Pour slowly, as heat will be produced when acid and water mix. Stir until mixed completely. Cover to prevent evaporation. Solution will be 3.7 N HCl.

Pour 0.5 L of concentrated HCl into 1.5 L of cold distilled water. Add 10 g of  $\text{CuCl}_2$ . Stir until completely mixed. Cover beaker and let warm to room temperature before using.

Pour enough  $\text{Na}_2\text{CO}_3$  into a container to make a saturated solution at room temperature. Some undissolved  $\text{Na}_2\text{CO}_3$  should remain on bottom of beaker after stirring.

- Procedure

Cut transversal seal from package approximately 1 inch from end. Identify multiple samples by notching cut edge with scissors. Manually strip paper from sample to be etched. Place sample in hot HCl solution ( $65^\circ\text{C}$ ) for 5 min. Remove sample with tongs and immerse it in  $\text{Na}_2\text{CO}_3$  solution to neutralize the acid. Remove sample from the  $\text{Na}_2\text{CO}_3$  solution with tongs and rinse it in running tap water. Pull off polyethylene layer that lies between paperboard layer and aluminum foil.

Using a glass stirring rod to manipulate the sample, drop it into the  $\text{CuCl}_2$  solution so that it is completely immersed. Observe closely while stirring to ensure that the heat of the reaction does not damage the polyethylene sealant layer as the foil is dissolved. Remove from solution.

Dip sample in  $\text{Na}_2\text{CO}_3$  solution to neutralize it, and then rinse it with water. Press sample gently between soft absorbent paper towels and place in oven at  $65^\circ\text{C}$  ( $150^\circ\text{F}$ ) until dry. Apply alcohol-based dye solution to inner and outer seal edges. (See fluorescein dye solution formula, described above).

Observe pattern of ink dispersion and check for leaks and channels within fused seal area. Use overhead projector to enlarge seal samples and provide a more accurate visual inspection.

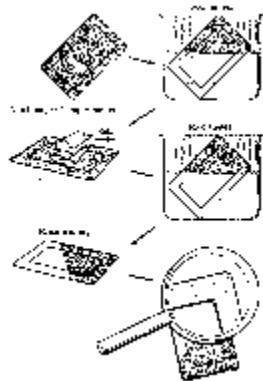


Figure 35. Chemical etching of package seal.

### Retortable pouches (Fig. 35)

#### 1. Materials

Two 1-L Pyrex beakers  
Running tap water  
Paper towels  
Rubber gloves, apron  
Protective goggles, tongs  
Fume hood with chemical-resistant surface  
Chemicals for etching retortable pouches  
6 N HCl solution, commercial grade  
Tetrahydrofuran (THF), commercial grade, stabilized

#### 2. Procedure

Cut off end of pouch and remove contents. Wash inside of pouch. Dry the pouch. Cut all but suspected area away from area of interest, leaving about 1 inch adjacent to seal. Soak sample in tetrahydrofuran (THF) to remove outer polyester layer by softening adhesive and/or inks. Do this in a fume hood; wear protective gloves resistant to THF. (If separation cannot be obtained, proceed to next step.) Remove most of the ink and adhesive from aluminum foil with THF and paper towels. Soak remaining structure in 6 N HCl in a fume hood to remove aluminum foil by etching. Rinse sealant layers with water and dry with paper towels.

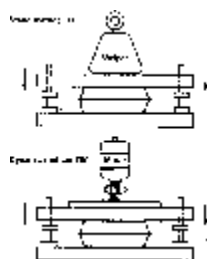


Figure 36. Compression testing of packages.

#### 7. Compression test (5) (Fig. 36)

Place a filled and sealed food package on flat surface and apply pressure while observing for leaks.

- #### 0. Materials
- Flat surface or conveyor belt
  - Sealed package

Heavy flat object or mechanical press

Timer

1. Procedure

**Static method.** Place sealed package on flat surface and lay a flat-surfaced weight on it. Observe effect of weight on integrity of package seals over time. A similar test may be performed by applying a constant weight to a package moving on a conveyor belt. The speed of the moving belt determines the time of compression.

**Dynamic method.** Use a press to continually increase the force applied to a package at a constant rate. Observe the maximum force required to cause failure of the package.

**Squeeze test.** Apply a manual kneading action that forces product against the interior seal surface area. Examine all seal areas for evidence of product leakage or delamination. Packages that exhibit delamination of the outer ply on the seal area but not at product edge should be tested further by again manually flexing the suspect area 10 times and examining all seal areas for leakage or short-width.

2. Results

**Positive.** Holes form in package or its seals or seams, with measurable movement of top plate or deflection on a force gauge.

**Negative.** No loss of hermetic integrity, and no measurable movement of top plate or deflection on a force gauge.

**False positive.** Underfilled or weak packages deflect in a manner that simulates failure without loss of hermetic integrity.

**False negative.** Holes form in package but food product closes off the holes, permitting pressure to increase within package.

8. Distribution (abuse) test (5)

Packages are subjected to vibration, compression, and impact at levels typical of the distribution system for which they are designed. After the test, which is a conditioning regimen, the packages are examined. Defects are quantified and described in relation to package failures observed in normal distribution. Fragility is eliminated by design changes in the package system. Whenever possible all samples should be incubated for 2 weeks at 100F before abuse-testing (Fig. 37).

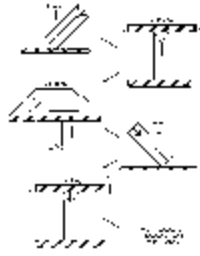


Figure 37. Distribution abuse testing.

- . Materials Packages to be tested
  - Drop tester
  - Vibration table
  - Compression tester
  - Standard laboratory conditions  $23 + 2^{\circ}\text{C}$ ,  $50 + 5\%$  relative humidity
  - Incubator at 100F to contain all test packages
- i. Procedure. **See** ASTM D-4169 Standard Practice for Performance Testing of Shipping Containers and Systems (15).

Select distribution cycle 6 for flexible packages in shipping cases transported by motor freight. Before testing, incubate all packages for 14 days at 100F and inspect visually for defects.

Perform the following 10 steps (**see** Section 9 of ASTM D-4169) (15).

0. Define shipping unit - Shipping unit to be tested is a typical pallet load.
1. Establish assurance level - Assurance level II will be used, based on value and volume of shipment.
2. Determine acceptance criteria at assurance level II: **Criterion 1** - no product damage; **criterion 2** - all packages in good condition.
3. Select distribution cycle (DC) - DC-6 will be used for pallet shipments.
4. Write test plan (values for X must be determined before conducting the test). Select representative samples for test. Condition samples to  $23 + 1^{\circ}\text{C}$ ,  $50 + 2\%$  relative humidity, in accordance with Practice D 4332 (14).
5. Perform tests in accordance with test plan in step 5, as directed in the referenced ASTM standards and in the special instructions for each shipment.
6. Evaluate results - Examine products and packages to determine if acceptance criteria have been met.
7. Document test results (16) - Write a report to cover all steps in detail.
8. Report fully all the steps taken. At a minimum, the report should include all the criteria in step 10.



9. Description of product and shipping unit DC and test plan
  - Assurance levels and rationale
  - Number of samples tested
  - Conditioning used
  - Acceptance criteria
  - Variation from recommended procedures
  - Condition of specimens after test

After testing, examine all failed (positive) packages to determine location and cause of damage. Incubate all containers that do not fail (negative) during testing for 14 days at 100F and inspect visually for defects before destructive testing by other methods listed in this chapter.

ii. Results

**Positive.** A package loses hermetic integrity during any one phase of the testing protocol or during the incubation period that follows.

Type test	ASTM Method	Level
Handling (12)	D-1083	One impact on 2 opposite base edges from X inches
Stacking (10)	D-642	Compression to X lb (individual container) * Search 3-100 Hz at 0.5 g peak. Dwell 10 min at 0.5 g peak
Vibration (13)	D-999 Method C	
Handling (11)	D-959	One impact on 2 opposite base edges from X inches
	(12) D-1083	
	(17) D-997	
	(18) D-775	
Stacking (10)	D-642	Compression to X lb (individual container) *

\*Alternative full pallet load compression test, X lb per bottom tier container.

7. **Negative.** A package retains hermetic integrity through the test, and contents do not show evidence of microbial growth after incubation.

**False positive.** A package appears to be defective, yet confirmational testing by incubation or dye penetration reveals that no loss of the hermetic barrier occurred during the abuse test.

**False negative.** A package appears to pass testing but later exhibits failure when incubated.

8. Dye testing (5)

Dye or ink is applied to inside surface of a cleaned package at the seal or suspected location of failure and observed to determine whether it can pass through to the outside (Figs. 24, 38, 39).



Figure 24. Can piercer and gas collection apparatus.

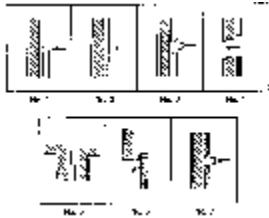


Figure 38. Dye testing.

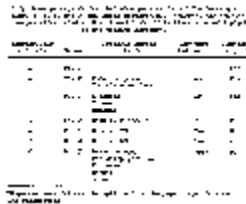


Figure 39. Dye testing results.  
(Courtesy of Brik Pak, Inc.)

- a. Materials Disposable plastic gloves  
Dye solution: 1 L of isopropanol (solvent) and 5 g of rhodamine (powder) mixed (or other appropriate dye solution)  
Sink  
Scissors or knife  
Oven to dry sample packages  
Paper towels  
Magnifying glass or low-power microscope
- b. Procedure

Open and empty a package; wash, and dry by wiping or by oven drying (180F, 15 min). Apply low surface-tension solution containing dye along the closure

or on side of package at suspected location of hole. The solution moves by capillary action through the hole and appears on opposite side of package wall. After dye is completely dry, cut package with scissors and examine the hole closely.

Cut open cans, tubs, or bowls through bottom (leaving seal areas or double seams untouched) and remove product. Cut pouches and paperboard containers along equator, leaving a hinge (so that both ends can be tested), and remove product. Wash package with water containing mild detergent, rinse thoroughly with tap water, and wipe dry. Holding package upside down and at slight angle, place 1 drop of dye solution at inside edge of seal surface. Rotate to allow dye to wet entire inside seal circumference.

**CAUTION:** A number of dyes are known or suspected to cause cancer. Rhodamine B is a possible carcinogen. Wear disposable plastic gloves and avoid skin contact with dyes.

Let dye solution dry completely. Very slowly peel the seal completely and observe the frosty, white, sealed surfaces for evidence of dye. In some packages the innermost laminates must be carefully observed for stretching as the seal is peeled.

c. Results

**Positive.** Dye penetrates hole in package, indicating loss of hermetic barrier.

**Negative.** Dye does not pass through the package (wall or seal).

**False positive.** Solution dissolves packaging material, creating hole in package, or dye is accidentally splattered on outside of package, indicating hole or leakage where none exists.

**False negative (for paperboard only).** Solution penetrates holes in hermetic barrier layers but fails to reach outside of package where it would be visible.

9. Electester (5)

The objective is to determine changes in viscosity of liquid foods after incubation of filled packages (Fig. 40).

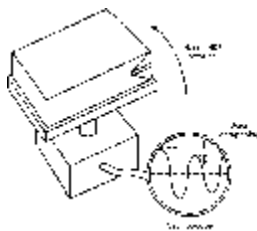


Figure 40. Electester

Microbial fermentation can cause changes in the viscosity of still liquids. If all factors are constant, shock waves will dampen at different rates in liquids with different viscosities. Incubation of shelf-stable liquid foods and nondestructive testing of each package may identify containers that have been subjected to microbial activity.

- a. Materials: Packages filled with still, liquid food, incubated  
Electesting device  
Fixture to restrain test packages
- b. Procedure

Remove representative samples from production line and incubate at 95°F for 4 days. Place packages containing still liquids in restraining device with largest flat surface of package facing downward. Rotate package 90 horizontally and back to its original position very rapidly; do this only one time. The motion creates a shock wave. Fixture holding the package is precisely balanced to minimize outside interference and minimize dampening as shock wave moves back and forth within package. Motion is sensed and displayed on an oscilloscope with alarms alerting operator to vibrations that dampen more quickly or more slowly than normal for a specific liquid food product. Examine contents with a microscope and determine pH to confirm spoilage if there is any doubt.

- c. Results

**Positive.** Wave dampens more quickly or slowly than normal, indicating change in product viscosity.

**Negative.** Rate of wave dampening is within range established by testing "normal" liquid product that did not display microbial spoilage during incubation.

**False positive.** Range of acceptance is too narrow, and normal product is incorrectly identified as spoiled.

**False negative.** Range of acceptance is too broad, and spoiled product is incorrectly identified as normal.

## 10. Electroconductivity (5)

The objective is to detect holes in hermetic packages by sensing the flow of electrical current. Plastics are generally poor conductors of electricity. Consequently, plastic food packages without holes will form an effective barrier to mild electrical current; therefore, this method may be used to detect minute breaks in plastic food packages.

A detectable flow of low-voltage electrical current generally indicates that the hermetic barrier has been lost.

- a. Materials: 1% NaCl in water (brine solution)

Scissors

Battery 9V, three 12-inch lengths of wire, 9V light bulb, or a conductivity meter (VOM). Remove insulation from each end of wires. One wire from positive pole goes to light bulb that has a wire as probe. The second wire from the negative pole is the other probe (Fig. 41).

Plastic bowl large enough to submerge package

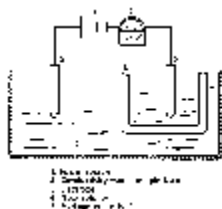


Figure 41. An electrolytic cell for leak examination.

- b. Procedure

Obtain sample food package and cut off one end with scissors. Aseptic paperboard packages and flexible pouches may be cut on all but one edge along package equator and folded 180 on uncut side to form 2 equal halves. Wash samples to remove all food contents and any dried plugs that may occlude holes. Oven drying at 180F is recommended but not required before immersion. Wipe the cut edges with a paper towel if necessary, as wet edges may result in false-positive test results. Place samples in bowl containing brine solution and partially fill sample with brine so that it stands upright and is almost completely submerged. Place conductivity meter or light bulb with one probe inside the package and the other outside the package. Submerge both probes into their respective brine solutions. Test the other half of package similarly for current flow.

- c. Results

**Positive.** Current flow indicates break in hermetic barrier.

**Negative.** No current flow indicates hermetic barrier exists.

**False positive.** Aluminum foil conducts electricity. A pinhole or partial break through inner layers of a package may expose the foil layer, resulting in false-positive test result. Dye testing will confirm presence or absence of holes. Moisture may form a bridge over cut edge of a package, creating a false positive.

**False negative.** Dried product may occlude minute holes in a package. If plugs do not rehydrate quickly, they will not conduct electricity when packages are immersed.

## 11. Gas detection (5)

The objective is to detect microleaks in hermetically sealed packages with sensors tuned to detect only gas leaking from within package. The package must be a barrier to the test gas so that the rate of gas permeation through the package wall will not raise the normal background concentration in atmosphere of testing area. Gas concentrations may be detected by impact to a sensor. The sensor may be a heated element in which electrical resistance varies in relation to gas molecules removing heat as they impact. Examples of test gases suitable for package include oxygen, nitrogen, hydrogen, carbon dioxide, and helium.

### a. Procedure for detecting helium leak

Gas obtained from storage tanks or air fractioning may be used to displace headspace gases within food packages before closure. Concentration of gas within package must be greater than the concentration of that gas in the atmosphere where packages are tested. There are three modes for detection: ASTM E493, inside-out tracer mode (6); ASTM E498, tracer probe testing mode (7); and ASTM E499, detector probe testing mode (8). Slight compression of a package may assist the movement of gas molecules through microleaks.

### b. Results

**Positive.** Detection of gas concentrations greater than the normal atmospheric concentration indicates break in hermetic barrier of sample package. Confirm with dye testing to locate hole in sample package.

**Negative.** No detection of test gas concentration greater than the normal atmospheric concentration indicates hermetically sealed container.

**False positive.** Detection of gas concentrations in excess of the normal background level may result from increase in test gas concentration in the testing area. Test background concentration before and after testing sample. Packages with high permeability may lose gas.

**False negative.** Internal gas concentration may be reduced through absorption by the product, reaction with a component inside the package, or permeability if over an extended storage period.

## 12. Incubation (5)

The objective is to determine whether a package has lost hermetic barrier by holding containers at an ideal temperature for sufficient time to ensure microbial growth. Hermetic integrity is the condition that bars entry of microorganisms into a package. Growth of microorganisms indicates either insufficient processing or loss of hermetic barrier. Growth may be observed as gas formation, change in pH, growth of viable organisms, or changes in the appearance of food.

- a. Materials Insulated box or room to serve as an incubator  
Heater with thermostat  
Storage racks  
Recording thermometer  
Temperature recording charts  
Knife, scissors  
pH meter  
Inoculating loop and flame  
Sterile culture dishes and tubes, and culture media
- b. Procedure

Obtain representative sample packages containing processed product. Inspect all samples visually for defects. Place packages in incubator for recommended period of time at recommended temperature.

### **Products stored in incubator at 95°F (35°C)**

- FDA products - 14 days
- USDA products - 10 days

### **Products stored in warehouse**

- 85-95°F (29-35°C) 30 days
- 70-85°F (21-29°C) 60 days
- 60-70F (16-21°C) 90 days

Visually inspect packages for evidence of spoilage. Open and inspect all (or some) packages for visible signs of microbial growth, aroma, and change in pH. Never taste incubated product if spoilage may have occurred. Aseptically obtain product samples to culture microbiologically and confirm cause of spoilage. Conduct appropriate integrity test on package to identify presence or absence of microleaks. Dispose of product safely. Autoclave any product or packages showing spoilage before disposal.

- c. Results

**Positive.** Spoilage has occurred and is evident as swelling, putrefactive odor, change in product pH from normal, or change in appearance.

**Negative.** Spoilage has not occurred.





During heat sealing of transparent and translucent plastic materials, energy is added, providing free movement of polymer chains. Close packing and increased hydrogen bonding occurs, resulting in alignment of carbon chains and increased crystalline structure. Differences between random, oriented, and crystalline configuration affect both light absorption and transmission in these materials. A seal sample placed between 2 polarizing filters is first illuminated by polarized light. To enhance color changes resulting from differences in crystalline structure, rotate the other filter to block most of the transmitted light. Inspect visually to determine degree of crystallinity within fused seals. Uniform crystallinity, seen as uniform color tone along the inner edge of the primary seal, is one indicator of fusion. Areas that are not fused appear as a different color. Colors differ with materials and thickness.

1. Materials: Light bulb, white, 40, 75, 100 W  
Polarized camera filters, 2 each  
Frame to hold filters and permit free rotation of both filters in line with light bulb and sample
2. Procedure

Obtain a clean transparent seal sample. Turn on light. Place seal sample between polarized filters. Rotate one filter to obtain maximum difference in color between fused seal and nonseal area. Examine fused seal area for uniformity.

- d. **Visible light.** The objective is to detect holes in packages by sensing transmitted and reflected visible light. Package is placed over low-wattage light bulb in darkened room to enhance visual inspection. Aluminum foil will block all light transmission except where holes and flexcracks in foil are present. Close inspection is required to determine whether other lamina overlay holes in foil layer. Dye testing is required to establish presence or absence of minute holes. Chemical etching may be used to remove materials external to polyolefin seals. Magnification of etched seals with backlighting aids inspection.

1. Materials: Light bulb  
Scissors  
Sink with running water  
Paper towels  
Darkened room  
Indelible marking pen  
Dye (optional)
2. Procedure

Remove contents, wash, and dry container. Inspect package for light leaks. Mark location of light leaks with a marking pen; draw a circle around the defect location. Closely examine defects for presence of holes through all layers. Use dye test to verify presence or absence of holes.

### 3. Results

**Positive.** A hole through all layers is detected in a package.

**Negative.** No light leaks are detected.

**False positive.** A hole in the foil layers permits light to pass, but no holes exist in overlying layers and hermetic barrier is maintained.

**False negative.** A hole through all layers is not aligned so that light can be transmitted.

### 14. Machine vision (5)

The objective is to detect holes in hermetic packages by computer evaluation of images with previously defined patterns of acceptance. This system is designed to eliminate visual inspection of packages. Packages are positioned before a camera to present a consistent pattern. The video image obtained is digitized. Both grayscale and color density may be evaluated. The computer compares coded patterns with acceptable patterns stored in memory. Some systems evaluate one image at a time. Others use parallel computers to evaluate different segments of the video image in less time. Patterns that do not match the acceptance criteria are rejected and the package is automatically rejected from the production line.

- a. Materials: Video imaging system  
Computer with stored images for acceptance criteria  
Strobe light (optional)  
Packages
- b. Results

**Positive.** Image does not match acceptance criteria.

**Negative.** Image matches acceptance criteria.

**False positive.** Image was not presented to camera correctly and does not match acceptance criteria.

**False negative.** Acceptance criteria include defects.

### 15. Proximity devices (5)

The objective is to detect holes by measuring changes in the shape of hermetically sealed packages as a function of time. The position of a package containing metal may be established by the strength of a magnetic field, detected with a galvanometer. By comparing 2 readings as a function of time, a determination can be made as to whether the shape of a package has changed.

- a. Materials: Proximity detection system  
Computer with stored acceptance criteria  
Packages
- b. Procedure

Compare multiple packages to a standard value. Fix limits of acceptance or alter automatically by computing a running average and standard deviation. Packages displaying stronger or weaker disturbances to a magnetic field sensed by a galvanometer may fall outside of the limits of acceptance. Mark these packages for removal from packaging line.

Read magnetic fields of single packages at one location and, after a period of time, make a second reading at a downstream location. If shape of container changes, mark package for removal from packaging line. Confirm with dye testing to locate holes in packages.

- c. Results

**Positive.** Disturbance of magnetic field exceeding limits of acceptance.

**Negative.** Disturbance of magnetic field within limits of acceptance.

**False positive.** External disturbance of magnetic field or imprecise positioning of package resulted in values that exceeded limits of acceptance.

**False negative.** Distortion of package sufficient to cause disturbance of magnetic field outside normal range of acceptance.

#### 16. Seam scope projection (5)

The objective is to measure critical dimensions in the closure profile of plastic packages. Packages are cut in cross-section to reveal all components in their proper thickness and relative position. The cut edge is magnified with a projector to aid measurement and visual inspection.

- a. Materials: Knife, saw, or scissors  
Microprojector  
Micrometer, calipers, ruler, or measurescope
- b. Procedure

Cut directly across seal or closure with knife, saw, or scissors and remove section containing adjacent material. Magnify cross section. Compare observed dimensions with criteria for acceptance or rejection provided by manufacturer of package or closure machine. Accept or reject sample.

- c. Results

**Positive.** Dimensions of sample exceed limits of criteria for acceptance.

**Negative.** Dimensions within limits of criteria for acceptance.

**False positive.** Magnification with incorrect scale or measuring error results in rejection of acceptable sample.

**False negative.** Measuring error results in acceptance of defective sample.

## 17. Sound (5)

**Ultrasonic.** The objective is to passively sense air moving through small orifices in packages possessing internal vacuum or pressure by monitoring the presence or absence of high-frequency sound waves.

- a. Materials: Microphone  
Audiofilters  
Oscilloscope with alarm system  
Packages
- b. Procedure

Place packages in a chamber to eliminate external disturbances and subject to changes in external pressure. Air movement through small holes in package wall generates ultrasonic sound waves. A microphone senses the vibration. Audiofilters eliminate all frequencies except those of interest.

- c. Results

**Positive.** Package exhibits ultrasonic whistling sound, indicating a leak is present, permitting air to enter or exit package.

**Negative.** No sound is emitted by package within range of frequencies monitored.

**False positive.** Background noise occurred within range monitored.

**False negative.** Hole does not emit a noise within the range monitored, or hole was occluded by moisture or food.

**Echo.** The objective is to actively sense the frequency of echoes in hermetically sealed containers. When a package possessing a vacuum is tapped, the tightness of the package creates a sound that is audibly different from that of the same package without a vacuum. Two changes can be monitored: frequency and amplitude. Changes in frequency (vibrations per second) are recognized as differences in tone (pitch). Changes in amplitude are recognized as 2 relative difference in volume. Loss of hermetic integrity will result in microbial growth within the contents of a food

package during incubation. Changes in sound accompany changes in viscosity. Consequently, this method may be used as a nondestructive test for a number of product/package combinations.

- d. Materials: Control sample  
Samples to be evaluated  
Tapping device (electronic device or unsharpened pencil to be used like a drumstick)  
Incubator
- e. Procedure

Obtain sample packages, either newly packed or incubated, and a control package (known to be properly sealed) containing the same product as sample packages. Tap the section of the package covering that is taut. Listen to the echo for differences between packages. Commercial devices are available that electronically monitor the echos, allowing for a less subjective determination.

- f. Results

**Positive.** Package displays audibly different sound, indicating loss of hermetic integrity.

**Negative.** Package resonates at same frequency as control package.

**False positive.** Differences in vacuum level or fill volume create different sounds in test packages.

**False negative.** Audible difference between control package and test package cannot be differentiated.

## 18. Tensile strength (5)

The objective is to measure the tensile strength required to cause separation of peelable or fused seals. A section of a seal is obtained by cutting a 1/2 or 1 inch strip perpendicular to the seal edge. The strip is then clamped by opposing grippers and pulled at constant speed and defined angle until failure is obtained. The peak force required to fully separate the 2 halves is recorded as the strength of the seal.

- a. Materials: Sample packages  
Sample cutting apparatus  
Scissors (sample dimensions are critical to precision)  
Tensile strength testing device
- b. Procedure. **See** ASTM D-882 - Standard test methods for tensile properties of thin plastic sheeting (9).

Remove representative sample from production line. Cut open sample and remove contents. Do not disturb seal to be tested. Cut a segment of the seal to produce a test strip. Test strip must be cut perpendicular to the seal to be tested. Secure both ends of test strip in separate clamps. With screwdriver, move one screw clamp away from the other, creating a 180° separation of the seal. Observe force required to fully separate seal. Fixtures are required to hold samples at angles different from 180°.

c. Results

**Positive.** Sample separates at peak tensile strength less than established standard.

**Negative.** Sample separates uniformly at peak tensile strength greater than or equal to established standard.

**False positive.** Sample separates at peak tensile strength less than established standard because of equipment miscalibration or greater separation speed of jaws.

**False negative.** Sample separates at tensile strength greater than or equal to established standard. However, a different portion of the same sample failed at a tensile strength less than the standard.

19. Vacuum testing (5)

The objective is to cause the movement of air out of a sealed container through leaks by using external vacuum within a testing chamber. Closed packages are placed inside a sealed testing chamber and vacuum is created to cause movement of air through leaks in the packages. Deflection of the package may be measured as a function of time to determine whether leakage has occurred. If vacuum chamber contains water, bubbles from holes in packages may be observed.

- a. Materials: Bell jar (glass or plastic) with tight-fitting lid  
Water to cover package within bell jar  
Weighted fixture to keep package below water level during test  
Vacuum pump  
Vacuum gauge  
Valve  
Grease for tight gasketing of lid on chamber
- b. Procedure

Obtain representative sample from production line. Place one sample inside vacuum chamber. Evacuate chamber. Observe package swelling and any movement of air (bubbles) or product through holes that may be present or may have developed. When vacuum is released, observe packages to

determine if original shape is retained or if atmospheric pressure causes sample to appear slightly crushed.

c. Results

**Positive.** Leak in test package causes air or product to escape through holes in container. Container ruptures or lid separates because of weak closure. When vacuum is released, package appears distorted or crushed by atmospheric pressure.

**Negative.** Package distorts under vacuum but no loss of product or air is observed. When vacuum is released, package assumes its original configuration.

**False positive.** Air clinging to surface of package or within paper laminates is mistaken for bubbles emitting from a defect.

**False negative.** Food particles prevent movement of air out of a hole in container while under vacuum.

20. Visual inspection (5)

The objective is to visually observe defects in food packages. Representative samples are obtained from production line. External surfaces are examined for holes, abrasions, delamination, and correct design. Critical dimensions are measured and observations recorded.

a. Materials: Strong light without glare, for visual inspection of packages  
Measuring devices, such as ruler, calipers, micrometer  
Scissors or knife

b. Procedure

Refer to examination procedures for paperboard packages, flexible pouches, plastic packages with heat-sealed lids, and plastic cans with double-seamed metal ends.

c. Results

**Positive.** Visually detected defect.

**Negative.** No visually detected defects.

**False positive.** Visual identification of defect not actually present.

**False negative.** Defect is present, but not visually detected.