CHAPTER 4

EXAMINATION AND EVALUATION PROCEDURES

There are a number of unique container integrity tests that can be performed on retort pouches. Appropriate methods should be obtained from the manufacturers of the containers or materials. Unfilled containers must be tested for bond strength. Filled containers, after thermal processing, must also be tested on a regular basis and results recorded.

Fusion is necessary for a good seal and exists when the opposing seal surfaces form a total weld. Such a weld is characterised by the inability to visually distinguish either opposing seal surface at the inner seal junction or after seal tensioning beyond the point of failure. On tensile failure (which can be produced by manual pulling), fusion exists when fracture of one inner ply at the seal junction occurs and there is delamination of one ply. If the seal peels away so that the inner seal surfaces are identifiable, fusion does not exist and the seals should be rejected.

Seals examined at the time of production may meet tensile and burst test criteria even though the seals do not have proper fusion. After a short (24-hour-plus) storage period, such seals may fail when subjected to handling tests such as vibration and drop cycles. Any processor producing flexible retort pouches should research the topic thoroughly to ensure that the production equipment and procedures will result in a fusion seal which will meet the seal specifications of the pouch material supplier. Destructive and non-destructive tests for assessing seals for fusion are described in the following sections.

4.1 CONTAINER EXAMINATION AND TESTS

The examination of retort pouches consists of a number of activities that will provide both quantitative and qualitative information:

1. visual inspection and external seal measurements to provide an initial assessment of seal integrity (this includes manufacturer's seals and tear notch area);

2. burst test to assess seal strength;

3. tensile test to verify seal weld (the laminate structure should fail before the welded seal fails).

4.1.1 Visual Examination

A careful external visual examination of containers and their seals is the primary means of detecting container defects. This involves:

1. removing the label from the container;

2. carefully examining the edges of each seal for any evidence of product in the seal area. No product (oil, etc.) should be visible;

3. measuring the width of the seals (manufacturer's and processor's) at a number of locations along the seal to ensure that they meet sealing machine specifications and the required minimum width of 3 mm; and

4. examining the seals by grasping the unsealed area of the laminate and exerting a steady pressure. Observe the package and pouch seals for signs of seal creep or delamination.

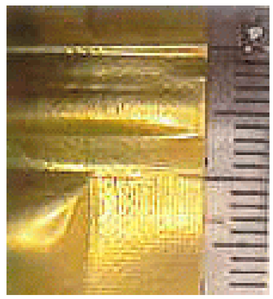
Visual examinations should be done at start up and every 30 minutes with one unit from each sealing head being tested and the results recorded[21,9]. A visual exam should also be done on the pouches slated for burst testing. Visual defects of concern include misaligned seals, flex cracking, product contamination of the seal, non-bonding, seal creep, delamination, and scratches.

Visual examinations should also be done after retorting since container damage could occur during the retorting, unloading, storage and packaging stages due to shingling of the pouches, rust in the retort and rough handling. Culls should be documented for defect type in order to assess the sealing operation.

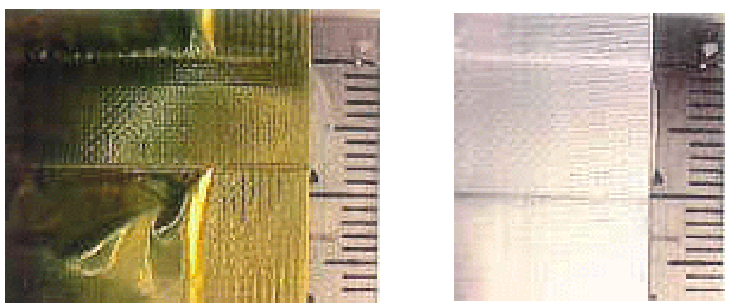
Figure 4.1 illustrates a normal fusion seal formed by a hot bar sealer and an impulse sealer. The heat seals can be either flat or profiled. Regardless of the physical shape of the sealing jaw or bar, the quality of the fused seal depends upon:

1. the temperature of the sealing surface materials;
2. the sealing jaw pressure;
3. the sealing dwell time;

4. the compatibility of the sealing materials;
5. the absence of seal area contaminants; and
6. the physical condition of the sealing surfaces.



Hot-Bar - normal seal



Thermal Impulse - normal

Figure 4.1 Closure Seal - Visual Inspection Criteria

4.1.2 Static Load Burst Test

A static load burst test (also called a compression test) may be used to determine the burst strength of a pouch as an indication of correct heat sealing conditions. A filled and sealed pouch of product, water or other non-compressible fluid is placed horizontally between two horizontal parallel plates connected to a load cell and indicating dial gauge. A standard weight is placed on the top plate for a set period of time. Pouches must withstand a force of 7.5 kg for 15 mm of internal seal length applied for 15 seconds[13].

Figure 4.2 illustrates a static load being applied across the faces of the pouch. The operator records either the force at which the pouch seal fails or, if a preset maximum force is applied, the time for which the pouch is held at that force.

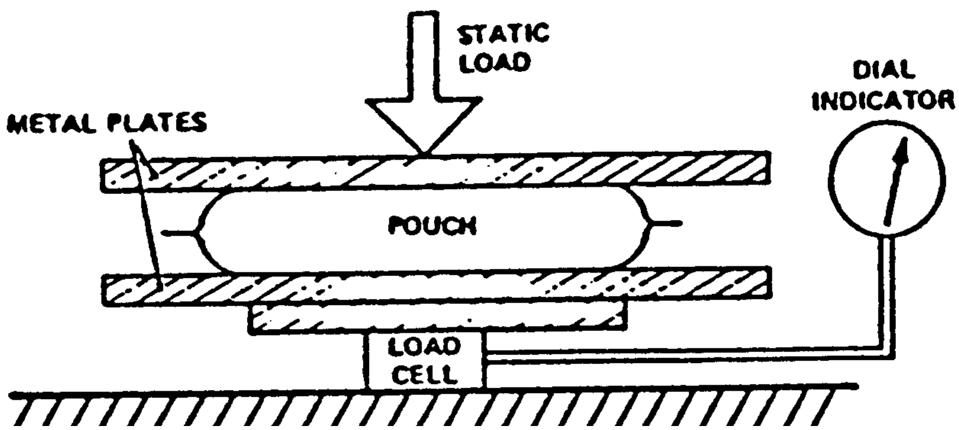


Figure 4.2 Static-Load Burst Test
(From Lampi, 1976, "Performance and Integrity of Retort Pouch Seals")[13]

When conducting a static load burst test the following points should be noted[8]:

1. The fail force of any pouch specification is related to the thickness of the filled pouch between the plates at the time of bursting. Since this thickness is determined by pouch internal dimensions and filled volume, the placement of the closing seal and product placement should be carefully controlled.

2. Product temperature can have a significant effect on the results of the test. Owing to heat transfer between the pouch contents and the pouch seals, a warm product or warm water can have a weakening effect on the seal. The degree of weakening depends on the pouch specification. As an example, a change in seal temperature from 30 °C to 40 °C may reduce the static load test result by as much as 35 %.

3. The seal damage caused by the static load test is generally less than that caused by the internal burst test because the force applied to the seals is from hydraulic pressure and it is relaxed as

soon as the pouch bursts. Thus the static load burst test is useful in locating and diagnosing seal faults. Also, the results of the static load burst test are generally more variable than those of the internal burst test, probably owing to the factors mentioned in points 1 and 2 above.

4. If the test is conducted with actual product in the pouch, the product must be capable of transmitting a hydraulic pressure to the seals; thus solid packs cannot be used for filled pouch burst testing.

5. After any burst test, intact test pouches of product must never be returned to production.

4.1.3 Internal Burst Test

The internal burst test is used as a good overall test for an hermetic seal including an indication of correct heat sealing conditions and a measure of the ability of a package to withstand transportation and handling. Internal pressure, applied by inflation with air, is used to stress the seals and the container's response is recorded.

Internal burst testing procedures involve the application of air pressure at a steady rate of 10 kPa/second (1 psig/second). Three internal burst testing procedures are as follows[1]:

1. The dynamic burst test, where inflation continues until the pouch bursts. The internal pressure at bursting is recorded. This test is used on fusion type seals.

2. The static burst test, in which inflation is stopped at a specified pressure and is held at that pressure for 30 seconds. Pass or fail is recorded. This test is used on fusion type seals.

3. The indexed burst test, in which inflation is stopped at a specified pressure such as 5 psig and held for 30 seconds, then inflated an additional .5 psig and held for 30 seconds, with inflation and holding periods continuing until the pouch bursts. The record includes the internal pressure at bursting and observations of seal separation. This test is used for peelable seals.

The internal burst test is to be completed before the tensile strength test.[9] It is generally recommended that internal burst tests be done before and after thermal processing as retorting and storage will reduce seal strength. Lampi[13] found that after sealing, pouches passed 240 kPa (35 psig) for 30 seconds, while after retorting and storage, the pouches passed 140 kPa (20 psig) for 30 seconds.

The internal burst testing standard, as outlined in *CAN/CGSB-32.302-M87 Use of Flexible Laminated Pouches for Thermally Processed Foods*[7] is 105 kPa (15 psig), held for 30 seconds with no evidence of seal rupture. Final seal width should be 3 mm or greater.[8] Note that pouches after retorting must meet these criteria, therefore, pouches prior to retorting will need to meet a higher criteria such as 140 kPa (20 psig) or greater.

There are two design types of internal burst testers.

1. Four-sided tester: a needle is inserted into a filled pouch and air is applied through the needle, as illustrated in figure 4.3;

2. Three-side-seal burst tester: an empty or emptied pouch is placed over an air source, the jaws are clamped to seal the pouch around the air source as illustrated in figure 4.4

In each case the pouch should be restrained. Restraint limits the angle of the seal which would otherwise increase with inflation. By limiting the angle of the seal, packages with strong seals fail at a higher pressure than when restraint is not used. This results in a noticeable difference in pressure at bursting between packages with strong seals versus weak seals.[1]

For pouches where the maximum thickness is less than 13 mm (½ in.), a heavy metal plate restrains the pouch thickness so the confining space will be no greater than 13 mm (½ in.). For all other pouches the confining space will be 10 % greater than the thickness of the container.[14]

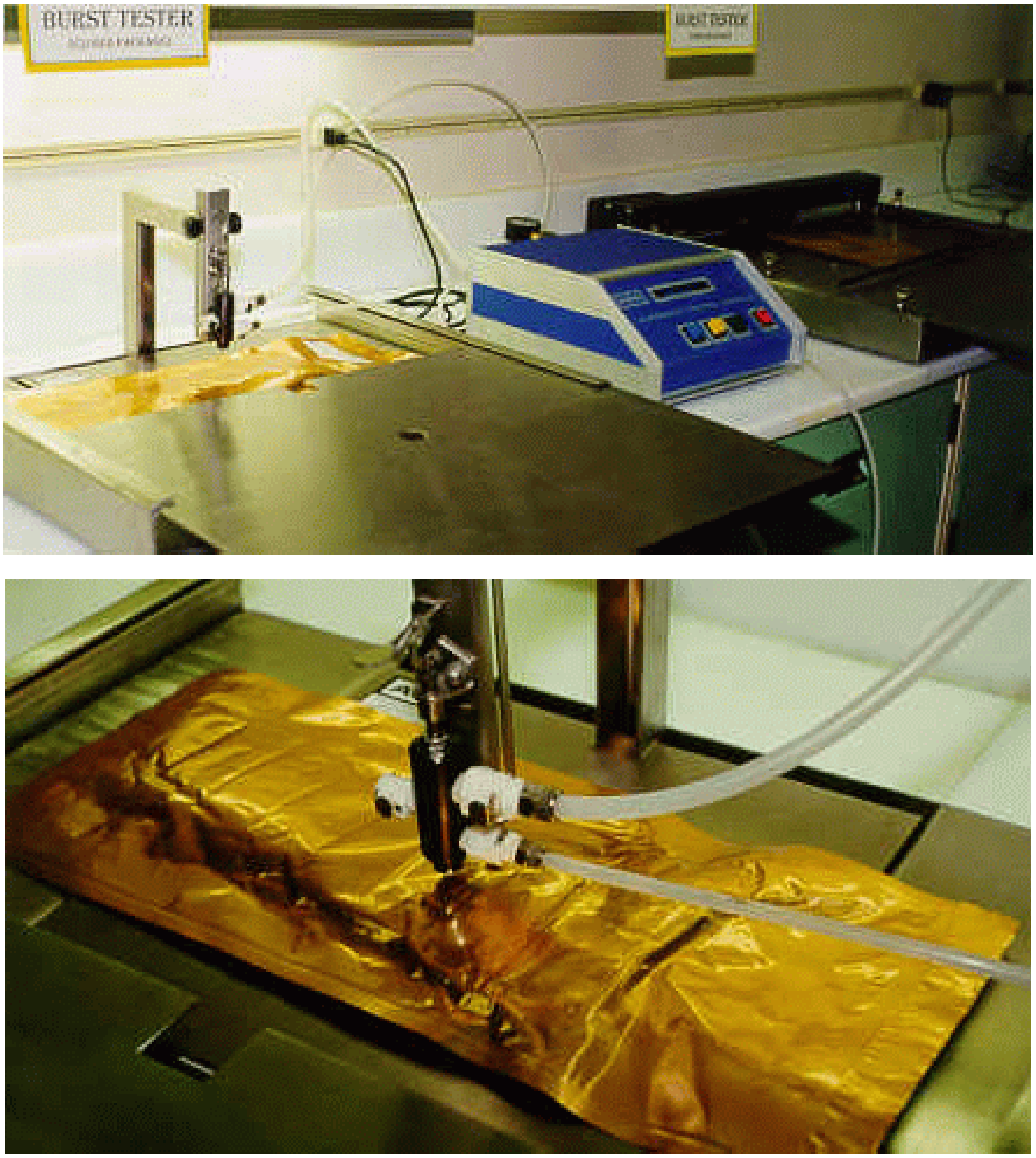
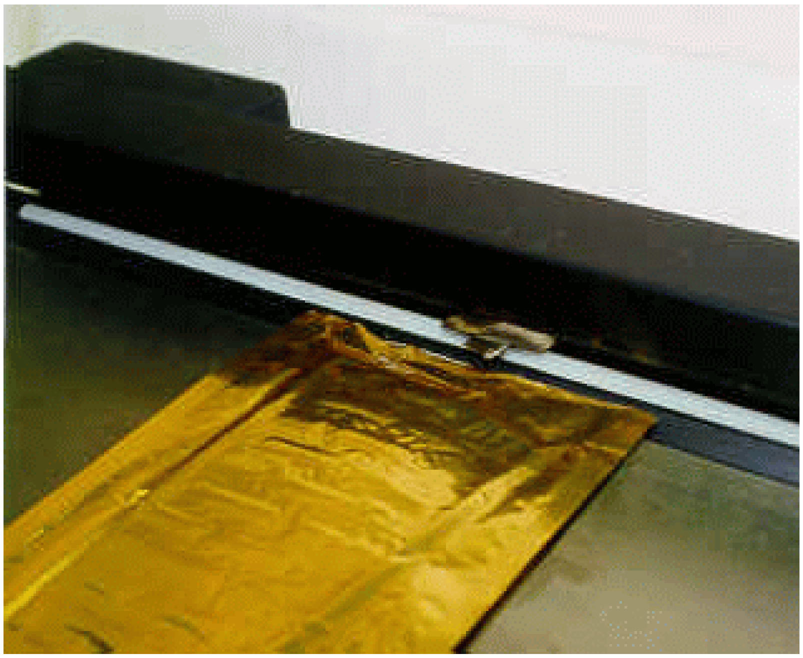
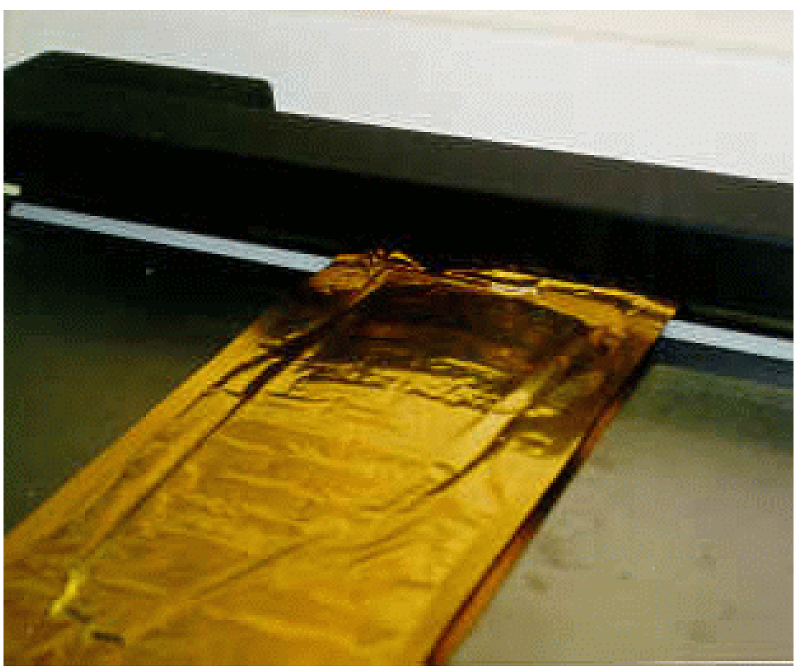


Figure 4.3 Internal Burst Test Equipment - 4-sided tester

Pouches are restrained between two plates, shown in the open position (top picture)



3-sided seal tester showing the open end of the pouch being connected to the air source. The top plate is shown open.



Pouch open end in position over the air source. Open end is sealed between seal-bars, which clamp together around the air source during the test.

Figure 4.4 Internal Burst Test Equipment - 3-sided tester

4.1.4 Tensile Testing

Tensile tests are used as a quality assurance tool for assessing the inherent sealing qualities of flexible packaging films and are employed as an optional test. Regardless of the specifics of the technique (sample widths, equipment, and variable crosshead or jaw separation speeds), the tensile test can best be used for surveillance of the sealability of materials and as a spot check on sealing conditions and equipment operation.[13]

Prior to carrying out the tensile testing, it is recommended that the heat sealed specimens be conditioned using 23 ± 2 ^{0}C (73.4 ± 3.6 ^{0}F) and 50 ± 5 % relative humidity as the standard conditioning atmosphere[3]. A minimum of 40 hours conditioning is recommended, although some materials may require longer conditioning times. Judgment should be exercised in the selection of conditioning times and procedures, which may be necessary to meet specific test objectives.

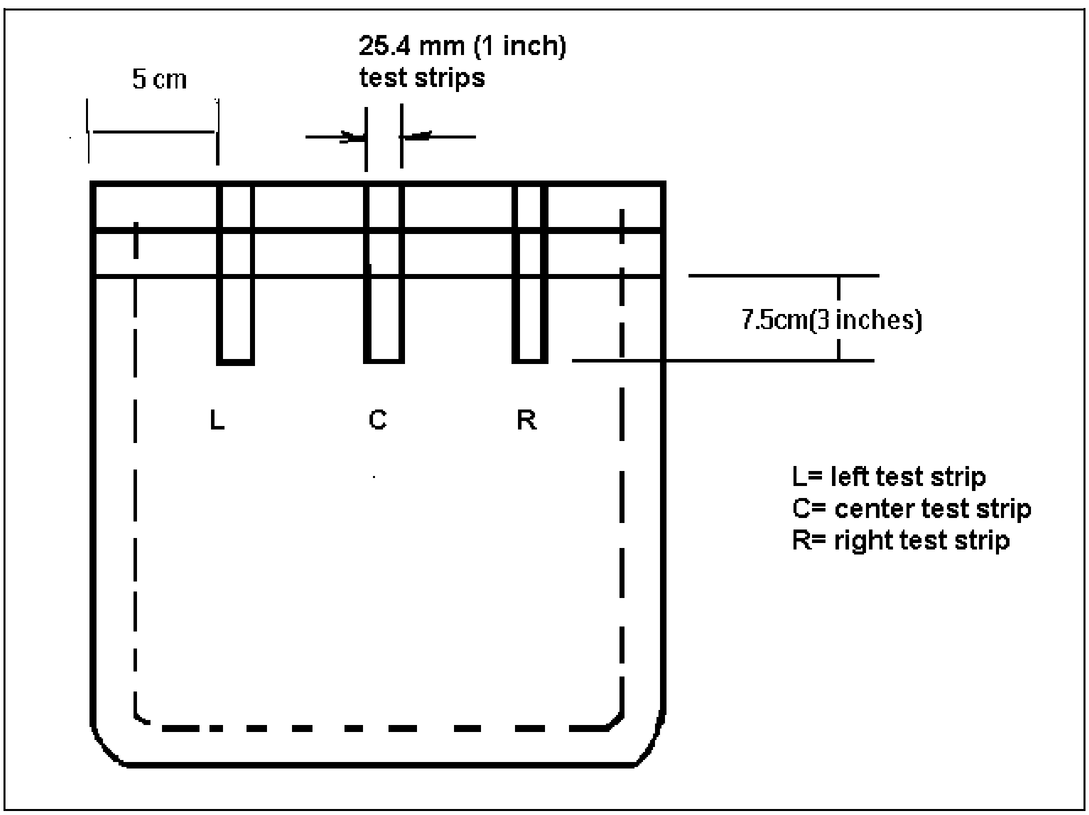


Figure 4.5 Measurement Locations for Seal Strength Test

Test strips which are 25.4 mm (1 in.) wide and at least 75 mm (3 in.) long are removed from the pouch seal as shown in figure 4.5. The edges must be clean-cut and perpendicular to the direction of the pouch seal.[3]

Each leg of the test strip is then clamped in the tensile testing device (as shown in figure 4.6). The sealed area of the test strip should be equidistant between the clamps and the recommended distance between the clamps is 25 to 50 mm (1 to 2 in.). The test strip must be aligned in the clamps so that the pouch seal line is perpendicular to the direction of pull. The seal must not be stressed prior to the initiation of the tensile test procedure.[3]

The seam is slowly pulled apart. The rate of loading should be between 250 and 300 mm/min (10 and 12 in/min).[3] The force required to pull the seal apart is recorded in newtons/metre of width (pounds-force per linear inch). At least three adjacent samples should be taken from each seal being tested and average of the sample is compared to manufacturer's specifications.[21]

Sample Test Strip
Positioned in Clamps

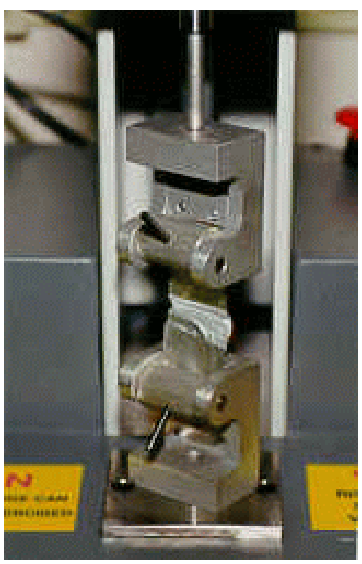
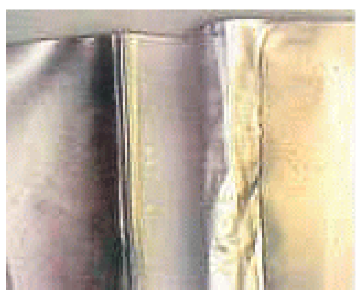


Figure 4.6 Tensile Test Equipment Showing Sample being Tested

Note that the tensile test measures the total force required to cause failure over the total width of each sample strip. The detection of channels or stress points and the effect of occluded particles or other small weak areas within the seal obscured by the adjacent high-strength areas.

Observe the appearance of the tear at the seal. In a properly formed seal, the inner ply from each side of the pouch are fused or welded completely such that when the seal area is pulled apart, the seal does not peel apart at the original surfaces. Instead, delamination should occur such that the foil and part of the laminated layer from one side of the pouch tears off, and adheres to the seal area on the other side of the pouch. The seal should tear evenly and will appear rough and marbleised[21,13] as shown in figure 4.7.

Typical Failure of Acceptable Seals (with Fusion)



Typical Failure of Unacceptable Seals (Lack of Fusion)

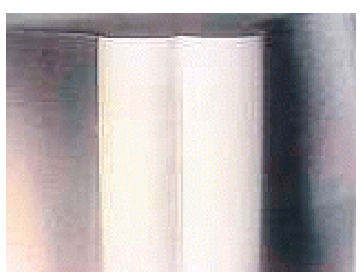


Figure 4.7 Tensile Test Closure Seal Visual Criteria

The tensile test report should include the following:

- identification of materials being tested;
- seal width being tested;
- test conditions (temperature and humidity);
- type of failure observed (delamination, peel or film break);
- maximum force required to cause seal failure for each test strip (expressed in N/m of test strip width, or lb/in of test strip width); and
- other pertinent information (e.g., statistical calculations, percent elongation before failure).

4.1.5 Residual Air Test - Destructive Test

The quantity of residual air can be measured during the tear down examination. The amount of allowable residual air is recorded as a part of the registered process (generally the maximum is 10 cc[26,12], but it may vary as long as this critical factor is specified in the thermal process). Too much residual air can exert excessive pressure on the seal during retorting or result in a product cold spot. Too little residual air can result in flex cracks forming around the edges of solid product as the pouch puckers upon cooling.

The test is performed by holding the pouch under water under a funnel attached to a graduated cylinder filled with water. A corner of the pouch is cut open under the funnel and the air is squeezed out. The amount of residual air in the pouch is measured as the water displacement in the cylinder (as shown in figure 4.8).

The volumetric measurements of air may be corrected to atmospheric pressure by Boyle's law:

$$V_1 = \frac{(P_a - W_h)V_m}{P_a}$$

where:

V_1 = Volume of air at atmospheric pressure (mL)
P_a = Atmospheric pressure (inches of mercury)
W_h = Pressure of water level in graduated cylinder (inches of mercury)
V_m = Volume of measured air (mL)

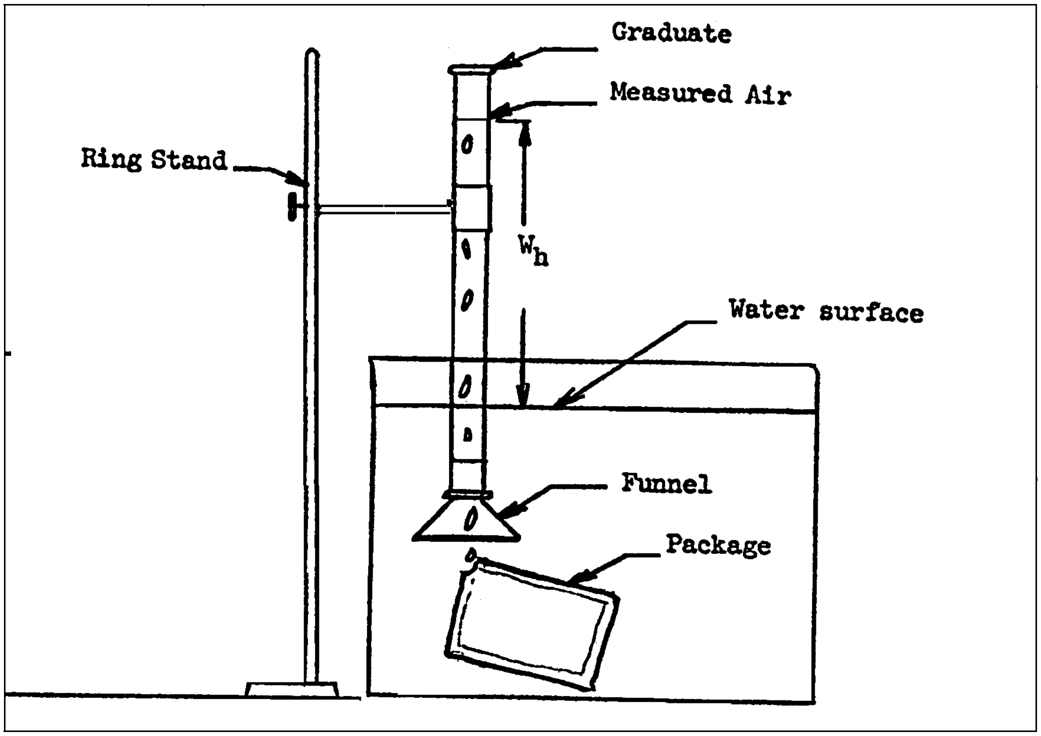


Figure 4.8 Residual Air Test Equipment

4.1.6 Residual Air Test - Non-Destructive Test

The non-destructive test involves the principle of natural buoyancy, where the volume of gas is determined by entering measured values into an equation derived from Archimedes' principle, Boyle's law, and the combined gas law. A non-destructive method to determine the volume of air in hermetically sealed flexible packages has been studied for its applicability as an acceptance test in specifications. The general principle of this method involves weighing the package while it is suspended in water and then reducing the environmental pressure until the gases in the flexible package expand sufficiently so that the package is in a state of neutral buoyancy (as shown in figure 4.9).

Processors routinely using the non-destructive testing procedure need to validate the results of the following non-destructive residual air test calculations, with destructive residual air test results.

The equation used to determine the volume of air in a flexible package is as follows:

$$V_1 = \frac{P_2\ (D)}{P_1 - P_2}$$

where:

V_1 = Volume of air (gas) in package at pressure P_1 (mL)
P_1 = Atmospheric pressure at the time of the test (in. of mercury)
P_2 = Pressure at time package is in a state of neutral buoyancy in water (in. of mercury)
D = Weight of package in water at pressure P_1 (g)

Note: the temperature is kept constant and the density of water is assumed to be 1 g/1 mL.

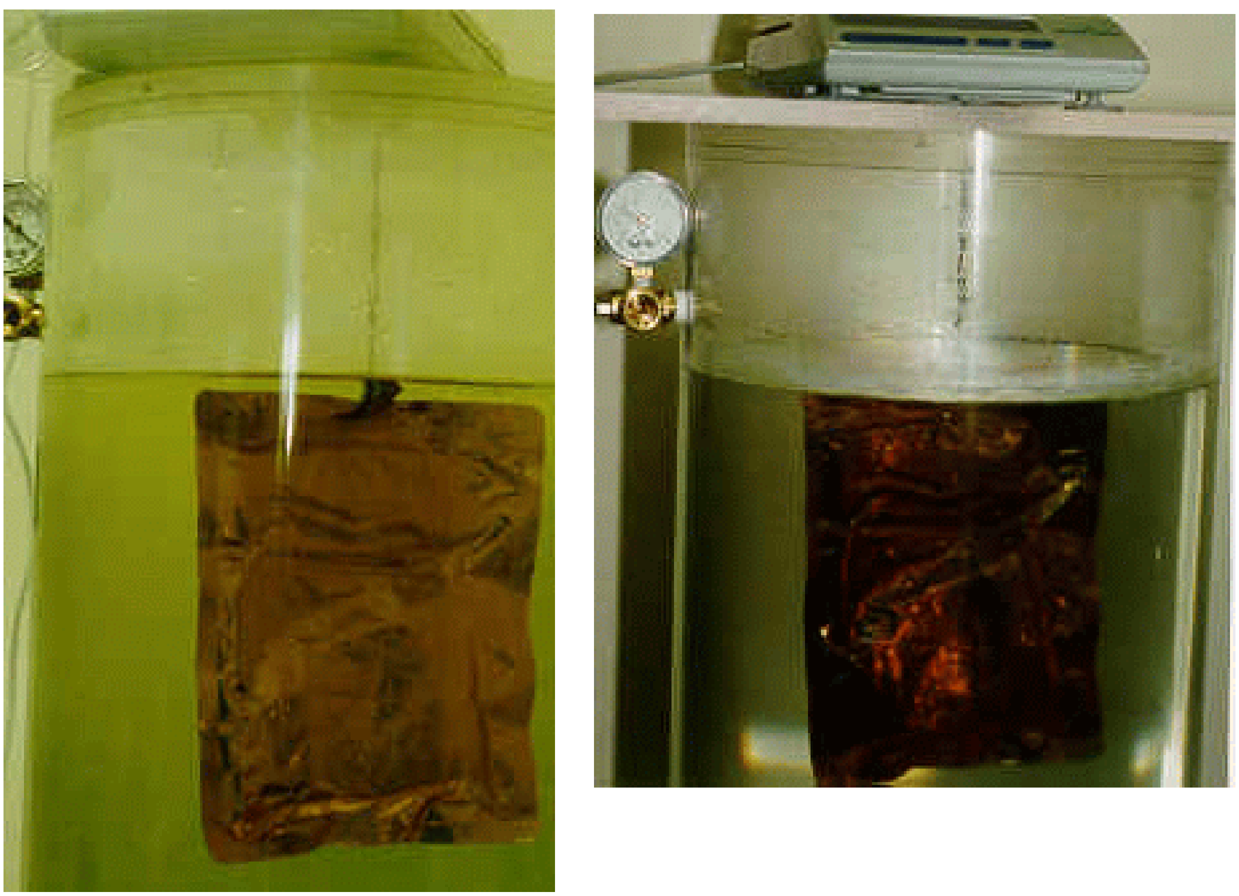


Figure 4.9 Weighing the Package in Water

To obtain the required data for the equation, the test package is placed into a transparent cylindrical vessel containing water. The water should contain a wetting agent to minimise the effect of any air bubbles clinging to the external surface of the package. The test package is initially weighted from its position suspended in the water, just below the water surface (this is weight D).

A vacuum is then applied to the cylindrical vessel containing water and the package. The package is observed for signs of leakage, such as a steady stream of escaping bubbles from the leakage location.

To obtain neutral buoyancy, the vacuum in the transparent cylindrical vessel is increased, gradually allowing the air in the package to expand, causing the package to rise to the surface (refer to figure 4.10). The pressure is further adjusted until the package is at the neutral buoyancy position just below the water line. The pressure reading inside the vessel is taken at this point by means of a vacuum gauge and is equal to P_2 in the equation.

Note that the values of P_1 and P_2 are in inches of mercury. A typical atmospheric reading for P_1 may be 30 inches of mercury. In this case, if the vacuum gauge reading is 0 at atmospheric pressure, then a reading of 10 inches of mercury while under vacuum will be equivalent to 20 inches of mercury for the value of P_2.

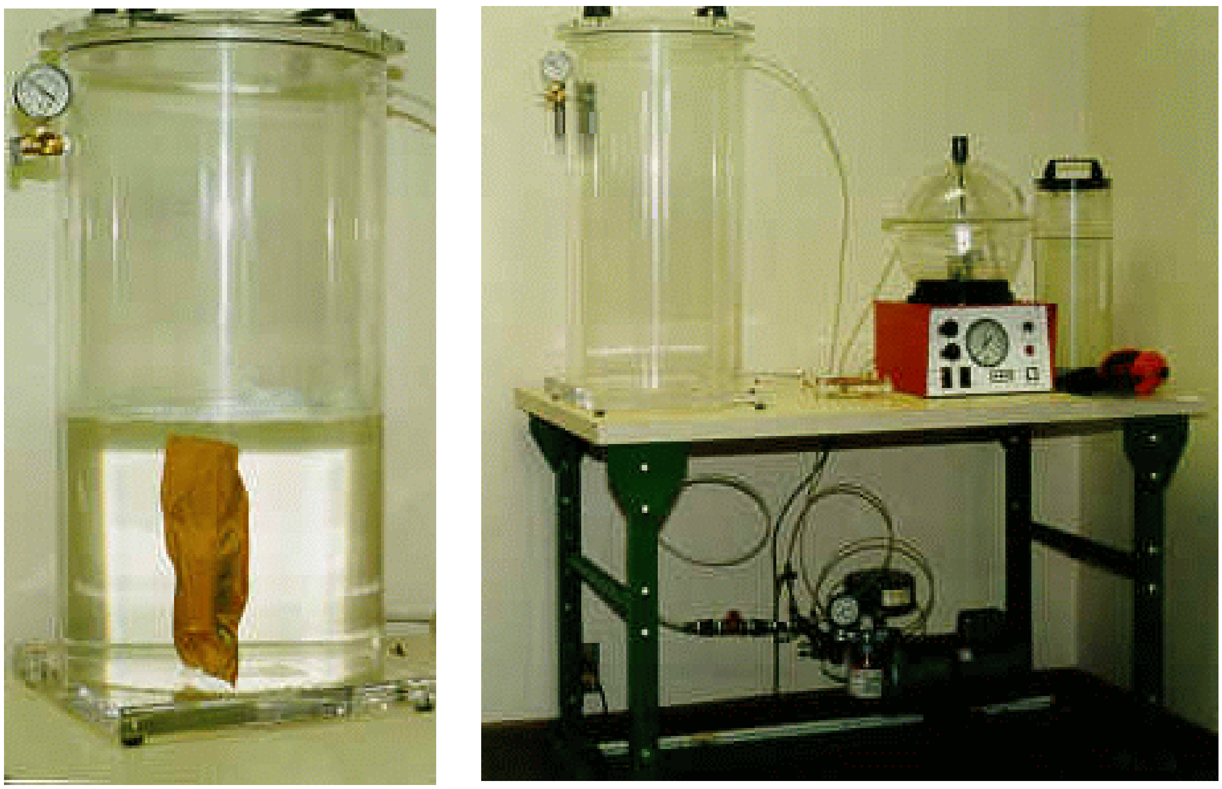


Figure 4.10 Test Apparatus for Determining Neutral Buoyancy Pressure

It may not be possible to achieve neutral buoyancy for some large size packages (850 g) which contain a very small amount of air (in the order of 1 mL or less). This condition should not detract from the value of the non-destructive test method for the following reasons:

1. if the air content were so low as to be inadequate to cause or sustain neutral buoyancy, the air volume in relation to the package size would in such instances meet the required specifications; and

2. it is unlikely that a precise measurement of a very low volume of air could be obtained for a package, containing actual foodstuffs, using the conventional destructive test method.

4.1.7 Dye Test

The dye test can be used to identify the location of very minute holes. The container is cut open, contents removed and the inside is cleaned to remove oil and water. The dye (containing isopropanol and rhodamine B) is squirted on the inside surface and along the inside seals with a syringe. After drying for 2 hours, the outside of the container is observed with UV light to detect dye that has penetrated through any microleaks [11].

One problem with the dye test is that the solvents used to carry the dye may attack the plastic, causing false-positive results. The dye test should be used as a diagnostic test which is used to pin-point the location of micro-sized holes that have been detected by other tests.

A number of studies have been conducted on the permeability of plain films and laminated materials to a variety of bacteria. The result of these studies demonstrates that retortable laminates do not allow bacterial penetration unless an actual fracture in the laminate exists. Where actual fractures exist, they can be readily detected by dye stain techniques that penetrate the defect. Consequently, aluminum foil flex cracks in the structure represent no immediate microbiological hazard unless the crack is accompanied by cracks in the plastic components of the laminate which would allow for dye stain penetration completely through the laminate [14].

4.1.8 Incubation Test

The retorted product is held at temperatures that would encourage the growth of "spoilage" organisms over a pre-determined period of time (i.e., 25 °C for 2 weeks). If growth is detected then the hermetic barrier has been compromised. Bacterial growth may be identified through standard microbiology tests and/or the presence of gas in the container.

It is difficult to establish a non-destructive microbiological test for heat-processed pouches unless statistically acceptable sample numbers have been taken. This sample size is unacceptably high and it is not feasible to complete microbiological testing on all such samples. The best compromise is to hold all production for 10-14 days and examine for swollen or blown spoilage prior to shipping.

A small sample should be incubated at relevant temperatures for microbiological testing to accumulate trend data over a period of time. **Microbiological testing of the sample pouches should be considered an inspection procedure and should not become a substitute for proper production line controls.**

4.1.9 Gas Leak Detection

Gas leak detection tests have been used successfully in the detection of micro-leaks. However, the procedure, equipment and time to perform the test all combine to make this test non-production oriented.

4.2 INSPECTION OF SEALED RETORT POUCHES

Retort pouch processors are encouraged to develop a testing protocol by working with the sealing machine manufacturer and the pouch material manufacturer. The evaluation methods for seal quality may differ between package designs, pouch construction and sealing methods. The frequency of pouch examinations shall be conducted with sufficient frequency to ensure adequate seals.

If the processor doesn't have specific testing procedures for the pouch from the pouch manufacturer, the following table 4.1 will be used for the minimum frequencies of tests and recommended sample sizes.

VISUAL SEAL EXAM - EXTERNAL SEAL APPEARANCE

Inspection Items	Frequency	Sample Size
Check for: - wrinkle across the seal - misaligned seal - inclusion in the seal - width of seal - flex cracks - delamination	At the sealing machine as often as possible. Minimum time between inspections is 30 minutes. Also after set-up and sealer adjustments.	1 pouch from each sealer position.

FILLED POUCH TESTING

Inspection Items	Frequency	Sample Size
Seal strength, and Width of seal	- Once per retort load (pre-cook), also - After shutdown exceeding 30 minutes, or - After settings to sealer are changed __________ - Once per retort load (post-cook)	- 1 pouch from each sealing position __________ - a minimum of 4 pouches
Residual gas test	- Once per retort load (post-cook)	- 1 pouch

EMPTY POUCH INSPECTION

Inspection Items	Frequency	Sample Size
- Pouch dimensions - Pouch shape - Tear notch - Delamination - Abrasions - Day code correct stock	Daily. Also, when changing pouch size or opening a new box from the manufacturer.	- Random sample of at least 20 units.

Table 4.1 Retort Pouch Inspection Schedule