*Journal of Food Protection, Vol. 52, No. 6, Pages 412-415 (June 1989)*


# Leak Detection in Flexible Retort Pouches

**JAMES E. GILCHRIST, DHIRENDRA B. SHAH, DAVE C. RADLE[1], and ROGER W. DICKERSON, JR.***

*Divisions of Microbiology and Food Chemistry and Technology, Food and Drug Administration, 1090 Tusculum Avenue, Cincinnati, Ohio 45226*

(Received for publication November 21, 1988)

ABSTRACT

Retortable flexible pouches now contain sterilized foods, and a method is needed to identify holes in these pouches. The helium leak test and the fluorescence dye test for metal food containers were modified to find micron-sized holes in flexible retort pouches. The helium test and the dye test were about equal in ability to identify holes. The helium test properly identified 32 of 32 pouches with holes. The dye test identified 31 of the same 32 pouches with holes and was able to indicate the point of leakage. Neither method produced a false positive, but the helium test was more sensitive than the dye test or the original biotest.

Retortable flexible pouches are now used for packaging commercially sterile food products. The original testing of retort pouches used a biotest *(4)* to demonstrate that these laminated pouches adequately sealed processed food from contamination, and under proper conditions, trilaminated pouches provided an adequate barrier to prevent entry of microorganisms. Because of the time and manpower associated with the biotest, it is not considered practical for demonstrating the presence of leaks in flexible pouches, and a faster method is needed for routine compliance activities.

Several tests have been proposed *(1,3)* to measure pouch reliability against possible defects of material or seal strengths. However, these methods are limited to measurement of seal strength, burst pressure, and strength of the bond between laminations in the pouch materials. They do not permit testing for holes in the pouch.

In one procedure, a fluorescent dye method has been used to identify holes in pouches (unpublished private communication, W.R. Cole, Food and Drug Administration). The pouch is cut in half and the inside of both halves is washed with fluorescein dye. A hole is indicated if dye is observed on the outside of the package. Although the dye method has been used to locate leaks in flexible pouches, no fluorescent dye methods have been reported in the literature.

Previously, we developed and tested the Helium Leak Test *(2)* method for determining the presence of a hole in rigid metal cans. The method consists of applying an overpressure of helium to the outside of the can; the helium will leak into the can through any holes that might be present. This can is then pierced and headspace gas is tested for helium. the approach could not be used on flexible pouches because any pressure applied to the outside of a pouch would cause the sides of the pouch to flex until the internal pressure in the pouch equaled the applied pressure. Thus, no pressure difference would occur to force helium through the hole.

The objective of this paper is to describe modifications of both the helium leak test and the fluorescein dye test that provide identification of micron-sized holes in retortable flexible pouches. Results from the two methods are compared with results from the biotest.

## MATERIALS AND METHODS

The pouches used in this study were a trilaminate of polyethylene, aluminum, and polypropylene, having a thickness of 0.005 inch. Pouch sizes were 6 1/2" x 8 1/2" and 12" x 14" (Reynolds Metals Co., Richmond, VA)[a].

*Preparing holes in pouches*

Micron-sized holes were made in pouches by two different methods. In some pouches, holes were melted by a laser beam, producing holes with diameters from 17 to 81 microns (Precision Aperture, P.O. Box 10363, 314 E. Wallace Street, Ft. Wayne, Indiana 46854). The laser holes were approximately round with no tears or flaps.

In other pouches, holes were punched by forcing a stainless steel wire (0.004-inch diameter) through the laminate. This procedure produced apertures with tears and flaps; they were equivalent to round holes with diameters from 22 to 175 microns.

The pouch holes were measured by holding the laminate between two pieces of rubber that were open in the center. Helium (10 psig) was applied to one side of the laminate. Any helium passing through laminate displaced water from an inverted graduated cylinder. This event was timed. The rate of helium flow was proportional to the square of the hole diameter at that pressure. The hole size was calculated as previously reported *(2)*. This procedure was checked by estimating the hole size by using a microscope with an internal grid.

*Helium test procedure for microleaks in laminated flexible pouches*

The helium test consists of cutting open one end of a pouch, emptying its contents, adding dry sand, and resealing the pouch. Subsequent steps are the same as those used for rigid cans *(2)*. The

[1]*Food and Drug Administration, Cincinnati District Office, 1141 Central Parkway, Cincinnati, OH 45202.*

[a]*Mention of company or product names implies neither endorsement nor criticism by the Food and Drug Administration.*

dry sand provides a structure inside the pouch that prevents the pouch from collapsing and provides interstices for helium to enter the pouch during pressurization.

*Test procedure.* One end of a filled pouch was cut open, emptied, and all residue was rinsed from it. The inside lips of the pouch opening were blotted or wiped dry. Sand was added to the pouch to about half the normal pouch volume; e.g., about 250 ml of sand for a 6 1/2 x 8 1/2 pouch. The inside lips of the pouch opening were wiped free of sand particles and coated with high vacuum silicone grease (Dow Corning Corporation, Midland, Michigan). The vacuum grease was spread about 1/2" to 3/4" from the edge of the pouch opening. The sides of the pouch were pushed together to remove excess air and the lips of the pouch opening were pressed together to ensure that sand did not enter the vacuum grease. The open end of the pouch was rolled around a 7/32"-outside diameter tube (K and S Engineering, Chicago, Illinois) and sealed with a 1/4" paper binder (Fig. 1) (Slide-N-Grip Backbone Paper Binder, 11" x 1/4", C-Line Products, Inc., Number 34447, Des Plaines, Illinois). A 1/4"-outside diameter tube (K and S Engineering, Chicago, Illinois) was placed in the paper binder to provide proper positioning of the clamp around the opened end of the pouch (Fig. 1).

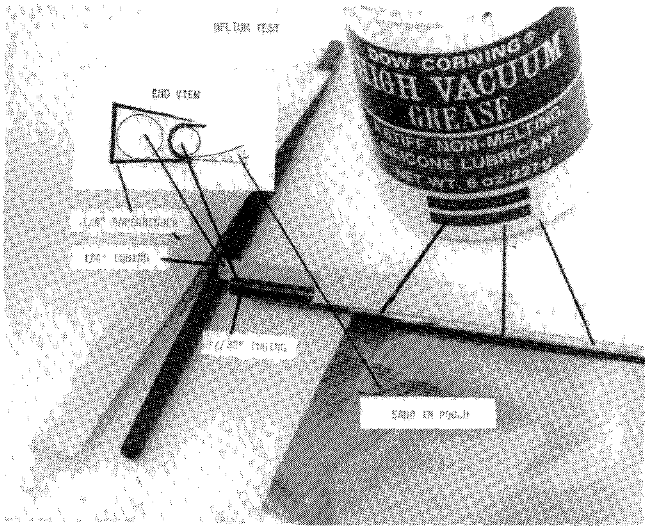


Figure 1. *Materials needed for the helium test.*

Sealed pouches were placed on their side in the helium pressurization tank. The pressurization tank was a 10-gal paint tank, as described previously *(2)*. The pouches were placed in the tank so that the sand in each pouch was distributed evenly. The tank was pressurized to 30 psig with helium and held at this pressure for 30 min. The pressurized helium was released from the tank and the pouches removed. The rate of pressurization and exhaust was 3-5 min for the 10-gal tank. Each pouch was pierced with a syringe and a sample of pouch gas (approximately 500 μl) was injected into the calibrated gas chromatograph. Helium was measured by the gas chromatograph (Fisher Model 29) with dual thermal conductivity cells and dual in-line columns that operated under ambient temperature. The columns, gases, and conditions were as described previously *(2)*. If helium content was 1% or greater, the pouch was recorded as a leaker.

*Dye test procedure for microleaks in laminated flexible pouches.*

The dye test consists of cutting open one end of a pouch, emptying its contents, adding fluorescein dye solution, resealing the open end, and placing weights on the pouch to force dye through any holes in the pouch.

*Preparation of fluorescein dye solution* The fluorescein dye solution consisted of 100 ml triethylene glycol, 300 ml water, 15 g glycerine, 3 g wetting agent (such as Triton X-100), and 3 g sodium fluorescein, technical grade *(1)*.

*Test procedure* One end of a filled pouch was cut open, emptied, and all residue rinsed from the open pouch. Unless the pouch was unused and empty, it was soaked (inside and outside) in warm detergent solution for a minimum of 10 min and then rinsed with water.

Fluorescein dye solution (about 1/10 of normal pouch volume) was added by pumping or pipetting half of the solution over each interior side. Air from the pouch was removed by hanging liquid in the pouch over the edge of a bench top and smoothing the pouch sides together with a paper towel blotter to adsorb surplus dye. The open end of the pouch was rolled around a 7/32"-outside diameter tube and sealed with a 1/4" paper binder (Fig. 2) in the same manner as for the helium test. The sealed pouch was placed between clear plates and weights (10 to 20 lb) were placed on the top plate (Fig. 2). Some leaks were noted with the unaided eye within 10-15 s. The pouch with weights was allowed to stand for a maximum time of 15 min. The pouch was removed from the plates and a U.V. light source (Mineralight shortwave U.V. lamp, Ultraviolet Products, Inc., San Gabriel, California) was used to see any leaking fluorescent dye. The presence of dye on the outer surface of the pouch indicated a hole.

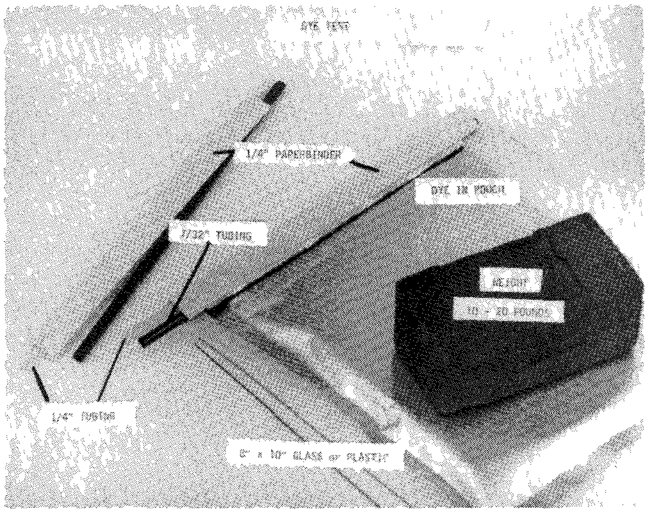


Figure 2. *Materials needed for the dye test.*

*Biotest*

Laminated pouches were filled with Tryptic Soy Broth and sealed with a commercial pouch sealer (Swiss Vac, Model CJ100, Gunther Kruse Packaging, Inc., Downers Grove, Illinois). The pouches were commercially sterilized (20 min @ 121°C) in a still retort (Model RDT1-3, Dixie Canner Equipment Co., Athens, Georgia) with air overpressure during cooling.

Gas producing *E. coli* 0A8112 were grown overnight at 37°C in Tryptic Soy Broth, reaching a concentration of $10^8$ *E. coli*/ml. The sterile laminated pouches were placed in the *E. coli* broth for 2 h and agitated each 15 min. Subsequently, the pouches were rinsed with tap water and incubated at 37°C for 20-24 h. A pouch was considered to be contaminated by bacteria and recorded as a leaker if it was: (a) swollen, (b) the headspace contained a large percentage of carbon dioxide and hydrogen, and (c) the broth was turbid.

## RESULTS AND DISCUSSION

Both the helium leak test and the fluorescent dye leak test identified very small holes. A total of fifty retort pouches, new and unused for food storage, were tested by both methods. Thirty-two of the pouches were deliberately made defective by a laser hole or a punched hole.

A comparison of the helium and dye test identification of the same holes indicated that both tests were very sensitive (Table 1). The helium test identified 32 of 32 holes and the dye test 31 of 32. There were no false positives among the eighteen control pouches. The control and experimentally pierced pouches were randomly mixed during testing.

TABLE 1. *New 6 1/2" x 8 1/2" laminated pouches tested for leaks by helium and dye tests.*

| Hole size[a] (microns) | Dye test | Helium test (% helium) |
|---|---|---|
| | *Pouches 1-25* | |
| *Laser holes* | | |
| 17 | + | 11 |
| 20 | + | 25 |
| 30 | + | 12 |
| 35 | + | 10 |
| 38 | + | 10 |
| 40 | + | 11 |
| 45 | + | 2 |
| 63 | + | 9 |
| 70 | + | 10 |
| 71 | + | 16 |
| 81 | + | 20 |
| *Punched holes* | | |
| 22 | + | 9 |
| 26 | + | 15 |
| 30 | - | 6 |
| 37 | + | 12 |
| 41 | + | 9 |
| 60 | + | 10 |
| 65 | + | 5 |
| 95 | + | 5 |
| 97 | + | 12 |
| 102 | + | 12 |
| 125 | + | 12 |
| 165 | + | 14 |
| 175 | + | 17 |
| | *Pouches 26-50* | |
| *8 Punched holes[b]* | + | 8-38 |
| *18 Controls* | | |
| (Pouch not pierced) | - | 0 |

[a]Equivalent diameter of a round hole that would pass the same amount of helium as the aperture under test.
[b]Hole size not measured.

In considering differences between the holes punched with a stainless steel wire or melted by a laser beam, the laser holes could be made smaller than the punched holes. The punched holes, however, had flaps of material that sometimes obstructed them during testing.

During the development of the retortable pouch, the biotest had been used to demonstrate integrity of flexible pouches *(4)*. Consequently, we compared results from the biotest, the helium test, and the dye test. Twenty-six pouches with laser holes were filled with Tryptic Soy Broth, closed with a commercial sealer, and processed to achieve commercial sterility. Each pouch was then tested by the biotest, the dye test, and the helium test, respectively.

Under the conditions of the still retort and bacterial exposure, the biotest found that bacteria passed through holes down to about 20 microns (Table 2). The helium test found more holes than either the biotest or the dye test. The smallest hole size measured by helium flow was 3 microns. The dye test was positive on most holes to about 20 microns, but was less consistent than the biotest. The remeasurement of hole size was performed to confirm that holes were still present, if the dye test failed to detect them. With rigid metal cans, the hole sizes tend to decrease with additional handling and manipulations *(2)*; this also occurred with pouches (Table 2).

The holes used in this study were between 3 and 175 microns. In comparison, the hole from a small straight pin is about 500 microns. The small holes (3 to 57 microns) demonstrated some reduction in size when remeasured (Table 2). As with rigid metal cans *(2)*, micron-sized holes in flexible pouches are easily partially obstructed or closed by the contained material. There were two observations in Table 2 where, although the first measurement indicated a hole and the helium leak test was positive, the remeasurement indicated the hole was closed.

In another pouch (Table 2) the first hole-measurement indicated absence of a hole, even though a laser hole had been drilled. The helium test was slightly positive for a leaker (1% helium), and remeasurement indicated the absence of a hole. This may have occurred because the helium leak test for pouches was conducted at 30 psig, while the laminated material was held in a jig and subjected to only 10 psig to determine the hole size. The hole size, as measured by flow rate, was determined at only 10 psig so as not to stretch and distend the laminate in the holding jig while the measurement was being made. The helium leak test could also be performed at 10 to 20 psig, but some sensitivity would be lost. Above 50 psig, some false positives might be obtained.

The dye and the helium tests were about equal in their ability to identify newly made holes in unused pouches (Table 1). In several instances, however, the dye test was negative, the helium test was positive, and the helium flow rate identified the size of the hole (Table 2). When this occurred, we thought that the helium pressure might have opened some partially clogged holes, then shown as positive by the dye test. for this reason, a second dye test was applied when the first dye test was negative and the helium test was positive. However, only in one case did the reapplication of

TABLE 2. *Biotest, dye test, and helium leak tests compared, using the same holes in laminated pouches.*

| Laser hole size (microns)[a] | Biotest[b] | Dye test | Helium test (% helium) | Remeasured hole size[a] (microns) | Dye test repeated[c] |
|---|---|---|---|---|---|
| *6 1/2" x 8 1/2" pouch* | | | | | |
| 0 | - | - | 1 | 0 | - |
| 3 | - | - | 3 | 4 | - |
| 6 | - | - | 3 | 6 | |
| 10 | - | - | 0 | 0 | |
| 13 | - | + | >30 | 10 | |
| 17 | - | - | 7 | 14 | - |
| 19 | - | - | NM[d] | 0 | |
| 21 | - | - | 2 | 3 | - |
| 22 | + | NM | NM | 5 | |
| 23 | - | + | 25 | 11 | |
| 23 | - | NM | 2 | 0 | |
| 24 | + | + | 30 | 12 | |
| 42 | + | + | 20 | 28 | |
| 43 | + | - | >30 | 18 | + |
| 47 | + | - | >30 | 27 | - |
| 51 | + | + | 20 | 16 | |
| 51 | + | + | 15 | 4 | |
| 51 | + | - | >30 | 30 | - |
| 51 | + | + | 5 | 14 | |
| 54 | + | + | >30 | 37 | |
| 54 | + | - | 22 | 30 | - |
| 56 | + | + | 27 | 14 | |
| 57 | + | + | 15 | 37 | |
| *12" x 14" pouch* | | | | | |
| 32 | - | NM | 5 | 7 | |
| 47 | + | NM | 5 | 0 | |
| 48 | + | NM | 2 | 6 | |
| *5 Controls* (6 1/2" x 8 1/2" pouch) (not pierced) | - | NM | 0 | NM | NM |

[a]From helium flow rate.
[b]After sterilization in still retort.
[c]Dye test repeated after the helium test and remeasurement of hole size had been completed.
[d]NM - not measured.

the dye test identify a hole. Thus, something kept the dye from flowing through the holes.

Both the dye and the helium tests require the pouches to be sealed. Since sealing of laminated pouches requires higher temperatures and pressures than pure plastic materials, we used a commercial sealer to seal the pouches for the biotests and some of the helium leak tests. For laboratories that do not have a commercial sealer, we developed an adequate seal that was made from a book binder and two pieces of tubing. The pieces that are used for the seal are shown in Fig. 1. When sealing a pouch for the helium leak test, a viscous paste (silicone, high vacuum grease) must be smeared on the inside lips of the pouch in order to insure an adequate seal.

We did not find any other satisfactory methods of resealing a retort pouch, except the commercial sealer for retort pouches and the paperbinder with silicone vacuum grease. An Audion 500 watt impulse sealer was not sufficient to seal the laminated pouches. None of 9 types of glue would hold the pouches closed. A lighter weight grease (Vaseline) used with the paper binder clamp allowed some leakage and was not tested further. Care must be taken with any method of sealing a pouch. Even with a commercial sealer, water, dirt, or wrinkles can result in a poor seal.

With the dye test, a "leaker" was considered as one with excessive dye present on the pouch surface. If the light-blocking aluminum foil was broken, but not the plastic layers, then dye could be seen through the clear plastic when no hole was present. It was also difficult to avoid small dye droplets that were caused from splattering. Similarly, if a high intensity lamp was placed inside a pouch in a darkened room, a break in the aluminum foil could be quickly found, but this was not always a hole.

The helium leak test was more consistent at identifying small leaks than the biotest or the dye test. The dye test will find the point of leakage whereas the helium test and biotest will not. When the dye test is used, the internal surface of the pouch must be washed with detergent. Earlier tests, without the detergent washing step, produced conflicting results.

**ACKNOWLEDGMENTS**

The authors thank Meredith Grahn, FDA, Cincinnati District Laboratory for permission to compare leak testing procedures in their laboratory. We also thank Diana Redmond for typing and assistance with the preparation of this manuscript.

**REFERENCES**

1. Anonymous. 1984. U.S. Food and Drug Administration, Bacteriological Analytical Manual, Chapter XXIV, 6th ed. Association of Official Analytical Chemists, Arlington, VA.
2. Gilchrist, J. E., U. S. Rhea, R. W. Dickerson, and J. E. Campbell. 1985. Helium leak test for micron-sized holes in canned foods. J. Food Prot. 48:856-860.
3. Lampi, R. A., G. L. Schulz, T. Ciavarini, and P. T. Burke. 1976. Performance and integrity of retort pouch seals. Food Technol. 30:38-48.
4. Maunder, D. T., J. F. Folinazzo, and J. J. Killoran. 1968. Bio-test method for determining integrity of flexible packages of shelf-stable foods. Food Technol. 22:615-618.