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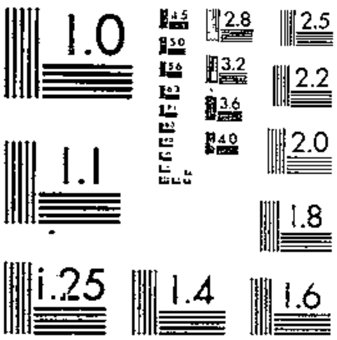

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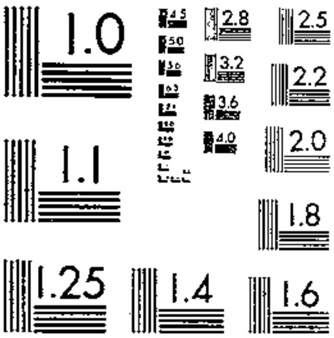

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Variables Affecting Film Permeability Requirements for Modified-Atmosphere Storage of Apples

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Variables Affecting Film Permeability Requirements for Modified-Atmosphere Storage of Apples

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SUMMARY

In 1962, a method was published for estimating film-liner permeability requirements for the storage of apples (*119*) (Literature Cited, p. 70). This method necessarily was based on data from many sources that probably contained unassessed variables. The purpose of the current study was to identify such variables, to define their importance, and to discover means of limiting their effects on film specifications.

Three lots of three varieties of apples from three sources were packed in five film types and stored under simulated commercial conditions for several months. Permeabilities of these films were mechanically measured at storage temperatures and results were compared with those computed by the RSAV△ formula. More than 50 experiments to reduce variance in permeability results were conducted. Only the more important of these are reported in this bulletin. These principally concerned method applications.

Summaries of these experiments presented no evidence for major changes in the basic concepts of the RSAV△ formula, but some changes in constants are suggested. Application and film-liner design formulas are presented and illustrated.

INTRODUCTION

This study was conducted to determine some of the variables that may be responsible for unpredicted modified atmospheres sometimes produced in sealed plastic box liners containing apples.

Opinions are divided what these variables are, what their relative importance is, and whether they can be sufficiently controlled to increase storage life of apples by specified beneficial atmospheres within sealed packages (*42, 48, 75, 122*).[1] It is the writer's view that many of these variables relate to mechanical faults of packaging films, that some involve film permeabilities, some concern liner seals, and some are based on doubts that films can be made to meet specified atmospheric requirements. Still other variables exist in the very techniques and methods intended to

[1] Italic figures in parentheses refer to Literature Cited, p. 70.

absolve variability. When these variables are fully identified, their complete reduction should ultimately follow.

It is probable, for instance, that by crystallization, cross-linking, and rigidity combinations (*76, 80, 81*), by monomer vaporization techniques (*14*), by laminates (*41, 85*), by changes in lamellar anisotropy and chain mobilities in amorphous regions (*10*), or by pore variations (*1, 2, 4, 20, 39, 84*), polymers and copolymers can be satisfactorily made to fit any need. If packers and designers can know what is wanted, it is probable that film specifications can be met.

Many attempts have been made to define film specifications (*25, 59, 60, 92, 98, 119, 123, 126*); these studies have contributed much basic information. In commercial applications, however, the problem has many variables that interact, and no single experiment is apt to yield the final answer (*17*). Many reports of experiments amenable to statistical evaluations have contributed both experience and information invaluable to the basic and applied research of this problem (*30, 34, 36, 42, 45, 49, 104, 108, 111, 119*).

This bulletin identifies the existence and incidence of certain objective and subjective variables believed to be of importance in the storing of produce in sealed films. It identifies those that probably should be assessed more closely. It offers considerable detail and emphasis on methods, in the hope that the cited variables ultimately may be satisfactorily controlled for the benefit of the produce industry.

GENERAL MATERIALS AND METHODS

Liners

One method for the specification of film liners for the storage of apples requires only the multiplication of five selected factors (*119*). This formula was applied, and film manufacturers and fabricators were solicited for any film type that approximately would meet one of four gas permeability specifications. The other requirements were that the films possess, at 32° F., adequate flexibility, have chemical inertness to foods, be reasonably odor free, and have sufficient strength for use as bushel-box liners. Film types, thickness, densities, and liner designs, other than lengths and widths, were left to the suppliers' choice.

Five liner designs were selected. Each experimental liner had to be fabricated before test fruits were purchased. This meant using liners that fit less well than desired for best results. Dimensions and other characteristics are given in table 1.

Test fruits

The 148 bushels of apples used in these experiments were selected at random from the warehouse stocks of commercial packers located in Virginia and West Virginia. All apples commercially graded size 113, fancy or extra fancy, and were tray-packed in telescope-type fiberboard boxes. Delicious and Stayman

TABLE 1.—*Mean dimensions and characteristics of film liners used in experiments*

[Sample size: 27 liners]

Item	Polypropylene	Rubber hydrochloride	Polyvinylchloride	Polyethylene	Polyethylene
Pigmented	No	No	No	Yes	No
Stock	Tube	Flat, folded once.	Flat, ends folded to middle.	Tube	Tube
Heat-seal	Across bottom.	Along two sides.	Middle seam and one side.	Across bottom.	Across bottom.
Gussets	None	None	4½ in. deep, 16½ in. on either side vertical seam.	Two 5 in. deep.	Two 7 in. deep.
Size[1] inches	33×31.75	33×33.5	32.25×32.25	33×34	33.38×32.5
Gross area[2] m.²	1.3519	1.4264	1.3420	1.4477	1.3996
Permeability area[3] m.²	1.2089	1.2090	1.1826	1.2090	1.2220
Kind of tie[4]	Single	Double	Single	Double	Double
Tie area[5] percent	10.5	15.2	11.8	16.4	12.6
Thickness[6] inches	0.00100	0.00075	0.00075	0.00150	0.00150
Density[7]	0.89–.91	1.097	ND	0.917	ND

[1] Width×length, unclosed, double sheet, within seams.

[2] Coefficient of variation among liners was 3.3 percent.

[3] Calculated by formula 12, p. 66, for each container requirement, then averaged for liner types. Variability within types 0.2–0.3 percent and among liner types 1.1 percent.

[4] The single closures were made by tying 3 tight loops of 5/16-inch cloth tape about the top of the liner, after twisting the top folds together firmly just above the produce. The double closure was a single closure with the top bent over and the double neck of folds again looped tightly 3 times and tied.

[5] Percentage of gross area; calculated by formula 15, p. 67, for each container type, then averaged for liner types.

[6] Manufacturers' estimates. See p. 53.

[7] Manufacturers' estimates; ND, no data given. See p. 54.

Winesap[2] were each supplied by three packers and Golden Delicious by two of these and a fourth packer; each packer supplied an equal quantity of the variety.

The apples were trucked to the Plant Industry Station, U.S. Department of Agriculture, at Beltsville, Md., and placed immediately in 31° to 32° F. storage. Thirteen boxes of apples were selected at random and set aside for later experiments. The remaining apples were tare weighed, coded, and repacked in trays, but with no further grading or sizing, into the five kinds of liners: three boxes of each of the three varieties from each of three growers in each liner type. No box pads were placed over the liners. The boxes were stacked five high on floor racks and

[2] The Stayman Winesap variety hereafter will be referred to as "Stayman."

spaced 2 inches between stacks and from the storage walls. Each stack contained apples of one variety and grower in five liner types. The order of the boxes in the stacks and of the stacks in the storage rooms was shifted with each test of the box atmospheres. A few experiments required slight variations in this storage procedure. These are discussed in the sections on the respective experiments.

The oxygen and carbon dioxide content in each of the 135 boxes was determined 12 times during the 6 months' storage period. The Orsat method was used, with the solutions modified to permit their use at 30° F. without precipitation or freezing.

At the conclusion of the storage tests, color photographs were made of each box of apples to show any apparent volume change in the unopened liner and to show the condition of the fruit in top and bottom trays. Tare weights again were determined for each box of fruit. About 20 fruits from each box were selected at random and inspected for internal breakdown. Damaged fruits were discarded. The others, after examinations were made for shrivel, scald, color, decay, and pressure tests, were set aside for additional experiments.

MODIFIED ATMOSPHERES IN LINERS

Atmospheres produced

None of the film samples received exactly met any of the alternative specifications requested. The resulting atmospheres in the packages thus were modified from those initially desired, but the films supplied served our purposes equally well to discern sources of variables.

Materials and methods

About 3,240 liner-atmosphere analyses were made by the Orsat method, under rigid controls, for the work here reported.

Results and discussion

On the basis of the analyses (summarized in table 2), it is believed the subjective errors of the Orsat method may be the source of much variation in the reported results in many papers. A more objective method of analysis would lessen variability in film permeability specifications. Some standardized form of gas chromatography may offer distinct advantages, but other forms of objective analysis also could be adapted.

The variations within the statistical blocks indicated a highly probable influence of interactions (table 2). These are discussed in following sections.

The atmospheres in double-tie liners appeared to be more favorably modified than those in single-tie liners. These data formed the basis for further studies reported in later sections of this bulletin.

RSAV△ formulations, (see lower half of table 17, p. 61)

TABLE 2.—*Measured atmospheres in film liners after 180 days at 31° to 32° F.*

Film type [1]	Apple variety	Gas present [2]					
		Final mean		Range [3]			
				CO_2		O_2	
		CO_2	O_2	Minimum	Maximum	Minimum	Maximum
		Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Polypropylene	Delicious	6.4	12.7	5.4	8.5	10.3	14.2
	Golden Delicious	6.9	12.2	6.9	8.7	9.2	12.2
	Stayman	6.7	11.6	5.7	8.8	9.4	14.1
Polyethylene (pigmented)	Delicious	4.4	6.0	4.1	7.3	1.6	6.7
	Golden Delicious	4.2	7.4	4.0	7.4	3.0	7.4
	Stayman	4.1	7.5	4.0	6.8	1.9	7.5
Polyvinylchloride	Delicious	4.7	14.8	4.2	7.0	11.5	15.2
	Golden Delicious	4.1	16.4	3.8	6.0	12.8	16.6
	Stayman	5.4	12.2	5.1	7.8	8.1	12.8
Rubber hydrochloride	Delicious	2.2	7.8	2.1	3.2	4.3	7.8
	Golden Delicious	2.0	10.2	1.8	3.1	5.0	10.2
	Stayman	1.9	9.7	1.8	2.7	4.8	9.7
Polyethylene (clear)	Delicious	4.7	9.2	4.6	7.7	3.9	9.6
	Golden Delicious	4.5	8.4	4.1	6.6	4.7	9.8
	Stayman	4.1	10.1	4.1	5.9	3.1	10.1

[1] See table 1, p. 3, for characteristics of film liners.
[2] Atmospheres at end of storage period.
[3] Values occurred among the totals for the 3 varieties and not within varieties. 9 boxes of 113 apples of each variety were packed in each type of film liner.

based on specifications of the films received and with known variables accounted for, would have forecast these atmospheres. However, film manufacturers and fabricators to date have little horticultural data on which to base formulations. Most of those who were asked for opinions when initial specifications were submitted to them replied that at least a 10-percent tolerance should be permitted. This tolerance does not seem to be unreasonable, based on the results of this study. It would seem to require but a reasonable application of the adjusted RSAV△ formula here presented plus a knowledge of what liner atmospheres are desirable and available. The results indicate some film types may show more promise than others. The satisfactory resolution of the involved variables is highly important.

It has been a matter of some concern how much weight should be given, in calculations of sealed-liner specifications, to atmospheres that have not reached equilibrium. These early storage periods have been reported to vary from 3 to 30 days (*65, 119, 123*). The basic data for table 2 showed extremes were limited to no one film type or apple variety. Three circumstances have initial bearing on such variabilities. First, only small quantities of carbon dioxide were found during this period. Second, the airspaces in an apple may total about 25 percent of its firmripe volume (*35*); hence, they would contain enough oxygen to support apple respirations for about a day.[3] Third, the initial oxygen inside the liner is enough at 32° F. for another 6 days (*119*). The remaining variability apparently is due to the interactions of storage incidents and time with selective film permeabilities. Curves of these data, plus those of results of others, indicate the proposal in the following section may adequately provide for preequilibrium periods.

Proposed definition of liner atmosphere

When a desired liner atmosphere is specified for fruit-storage purposes, it is important to the package designer that the specified value has a standardized meaning. Presently, it has none. It is proposed that this meaning be the effective atmosphere that the liner is expected to develop about the enclosed produce.

This physiologically effective atmosphere for fruit storage should be a mean, rather than an initial, equilibrium, or terminal value. Among 10 types of means considered in a series of exploratory calculations,[4] arithmetic means of measured atmospheres gave the highest variable results and time-weighted means gave the least. However, the calculation of time-weighted means was cumbersome, and the results were not always satisfactory.

More consistent and acceptable results may be obtained through use of the median atmosphere of each measured period. A mean of this type gave the lowest coefficients of variation for calculated permeabilities, gave almost equal coefficients for both carbon dioxide and oxygen, and can be rapidly calculated as:

$$G = \left[\frac{2(a_2 + \ldots + a_{n-1}) + a_1 + a_n}{2N-2}\right] \quad [1]$$

where: G = effective percentage of carbon dioxide (c) or of oxygen (o) in the liners during the storage.

a = measured percentage of the gas within the liners at successive inspections. If more than one measurement is made per inspection, the arithmetic average of them should be used.

a_1 = first inspection.

a_n = last inspection.

N = number of inspections.

It is important that the initial inspection should be made on the

[3] An apple weighing 187 g., with a volume of 236 cc., has about 14 percent intercellular oxygen that may be available at about 1.8 cc./kg./hr. (*69*).

[4] Based on results with three varieties of apples in one type of polyethylene film in 27 cases, for 180 days' storage.

day storage begins. This generally will be equivalent to the packing- or storage-room atmosphere. The efficiency of this formula is highest when inspections are made at equal intervals of time and the storage period is considered to be terminated at the last inspection.

Although only one film type was used to obtain the bases for the above suggestions, there appears to be no logical reasons for concern that the conclusions might be significantly different for other film types.

Effects of boxes on permeability of liners

It had been suggested that the permeability of liners may be inhibited by the boxes enclosing them and that this variable may need recognition in the final specification of liners. Two exploratory experiments were initiated to obtain information on this possibility.

Materials and methods

In the first experiment, apples in film liners placed in wire-mesh boxes were to have been compared with apples in similar packages placed in fiberboard cartons. Because of possible punctures of the liners by the mesh boxes, this experiment was abandoned after 2 months.

In the second experiment, atmospheres and apple conditions in liners alone were compared with those in liners put in boxes. Twelve boxes of Golden Delicious apples were sorted from among 45 boxes of fruits that had been in continuous storage at 31° to 32° F. for approximately 6½ months. The fruits were chosen for uniformity in ground color, for weight between 160 and 180 g., and for equal soundness and apparent maturity. The general condition of each apple was excellent. All apples were tray-packed, 113 apples per case, into similar polyethylene liners selected for the absence of visible pinholes, tears, or weak seams. All were closed with single ties, uniformly made as previously described. Half of the number of liners and enclosed apples were placed in boxes in such a way that no liner was subjected to packing stress and all liners were of equal apparent volume. The other half were not placed in boxes.

The boxes with enclosed liners and the liners without boxes were coded, randomized, protected by guard rows, and supported on angle irons. The arrangement was four boxes or liners per row, three rows high, plus the guard boxes. Storage was at 32° F. Liner atmospheres were sampled for carbon dioxide and oxygen over a period of 30 days, about 144 analyses.

Results

A summary of the results of the second experiment is given in table 3. The condition of the apples and the atmosphere compositions were not significantly different regardless whether or not the liners were enclosed in boxes.

TABLE 3.—*Mean effects of boxes on apples in liners* [1] [2]

Factor	Apples in liners not in boxes	Apples in liners in boxes
Firmness [3] ...... pounds..	9.1	9.2
Shrivel [4]	0	0
Scald [4] ...... percent..	1.2	1.0
Decay [4]	0	([5])
Splits [4]	0	0
Ground-color rating [4] [6]	3.3	3.0
Mean CO_2 [7] ...... percent..	3.7	3.7
Mean O_2 [7] ...... do....	12.8	12.4
Final CO_2 [7] ...... do....	3.4	3.2
Final O_2 [8] ...... do....	11.6	12.2

[1] Net weight of apples in each liner approximately 42.4 pounds.
[2] No significant differences between the pairs of means at the 5-percent level.
[3] 60 apples from each treatment were tested by the Magness-Taylor pressure tester.
[4] 678 apples from each treatment were inspected.
[5] Only 1 apple was decayed.
[6] Rating from U.S.D.A. standard ground-color chart for apples.
[7] Mean of 6 determinations at each of 6 time intervals on each treatment.
[8] Mean of 6 determinations on each treatment.

Ethylene in liner atmospheres

Reasons have been reported why liners for apple storage probably should be highly permeable to ethylene and other apple volatiles (*18*, *119*). The effects of volatiles on apples have been debated; but studies have indicated that respiration may be excessively stimulated by 1,000 p.p.m. of ethylene. At least 800 p.p.m. have been found in normal storages. Tomkins (*123*) noted that apples in trays covered with 150-gage polyethylene accumulated 755 p.p.m. of ethylene in 208 hours at 15° C. There is little information on the maximum amounts of ethylene that may accumulate in apple film liners during storage. It was thought such information might have a bearing on the present study.

There is practically no information on the permeability of liners to ethylene. This information is needed; but, because of local hazards, no attempts were made to obtain ethylene permeabilities.

Materials and methods

Ninety determinations were made by gas chromatography on the ethylene levels about Stayman and Golden Delicious apples stored in each of the five liner types. The boxes of Stayman and Golden Delicious apples for which the ethylene contents were determined had been stored for 9½ and 10½ months, respectively, a period somewhat longer than normal.

Results

The ethylene content was higher, on the average, in the two polyethylene and the rubber hydrochloride liners than in the other liners (table 4).

TABLE 4.—*Ethylene content in film liners containing apples stored at 31° to 32° F.*[1]

Variety and film type[2]	Liners examined	Ethylene content	
		Mean	Range
	Number	*P.p.m.*	*P.p.m.*
Stayman:[3]			
Polypropylene	18	377	21–666
Polyethylene (pigmented)	14	1,213	298–3,372
Polyvinylchloride	14	1,107	201–2,458
Rubber hydrochloride	9	2,008	525–4,900
Polyethylene (clear)	17	1,815	550–5,131
Golden Delicious:[4]			
Polypropylene	6	18	12–31
Polyethylene (pigmented)	1	9	--------
Polyvinylchloride	8	55	39–67
Rubber hydrochloride	3	220	213–363
Polyethylene (clear)	0	----	--------

[1] Determinations made by gas chromatographic techniques.
[2] See table 1, p. 3, for descriptions of liners.
[3] Stored continuously for 10½ months, with liners unopened to atmosphere.
[4] Stored continuously for 9½ months, but all liners, except the rubber hydrochloride liners, opened (to retie them more securely) about 20 minutes to the atmosphere approximately 30 days before tests were made.

Volume shrinkage in liners

It has been asked whether the volume adjustment factor, V, in the RSAV△ formula (*119*) is needed. The initial reasons for this factor were necessarily hypothesized, but also the writer had made numerous observations of boxes of apples without top pads over the liners as well as those packed with pads. For additional data whether this shrinkage factor might indeed be a false assumption traceable to depressions caused by the top pad, these pads were omitted from the boxes packaged for this study. Photographs of cartons containing less than a total of 21 percent carbon dioxide and oxygen indicate that the sides, as well as the top of the liners, do draw inward to that point where the atmospheric pressure in the liners is probably equal that outside (fig. 1). These photographs, characteristic of many observations, tend to sustain the hypothesis in (*119*) that the V factor probably is valid.

General condition of apples stored in test liners

Materials and methods

Although the primary purpose of this study was not to test the effect of various film liners on fruit-keeping quality during storage, the apples were examined after 9½ to 10 months' storage in the liners. The mean storage period at 31° to 33° F. for Delicious apples averaged 284 days, for Staymans, 294 days, days, and for Golden Delicious, 311 days. The relative humidity was uncontrolled. Certain subjective and objective tests were made on many individual fruits after this long holding period.

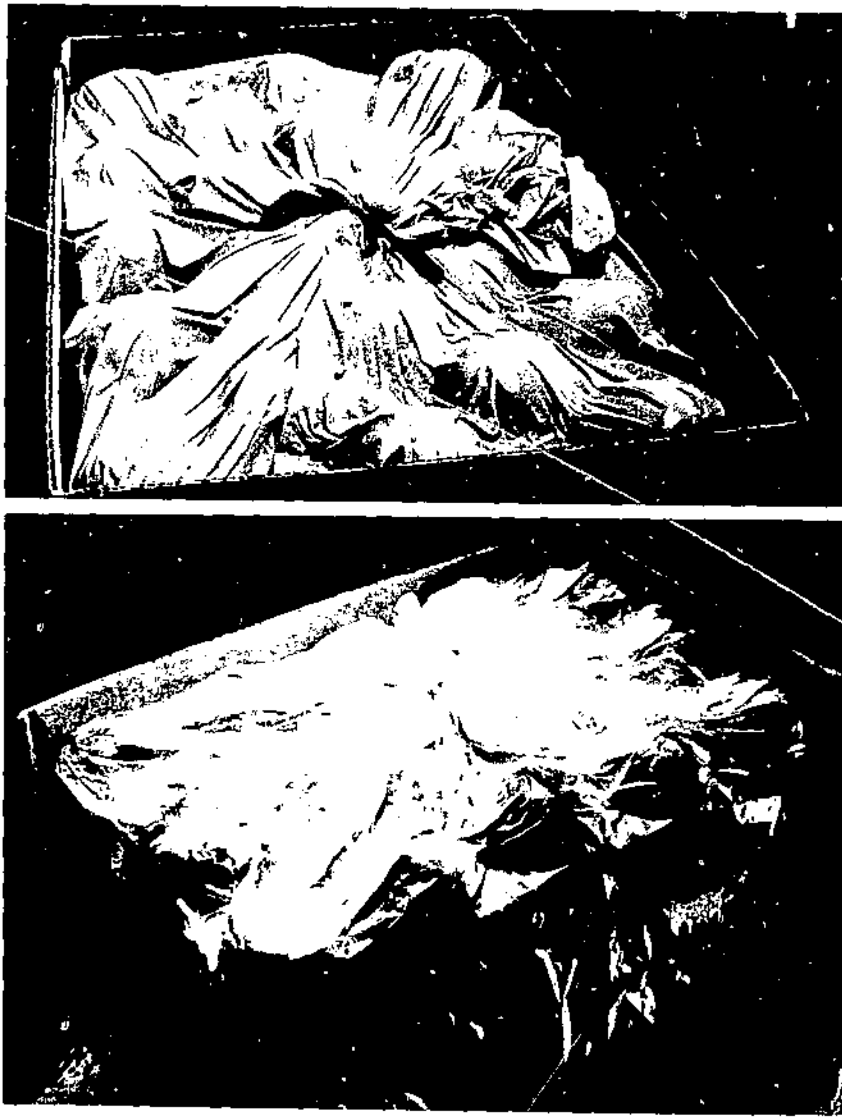

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FIGURE 1.—Volume shrinkage in liners containing less than 21 percent carbon dioxide and oxygen: *Top*, Polyethylene liner without box cover; *bottom*, polyvinylchloride liner with box cut away to show side depressions. No vacuum was present in either box.

A total of 4,575 apples were tested for firmness; 3,725 were inspected for internal browning; and 13,716 were examined for superficial scald, shriveling, decay, and splitting.

Results and discussion

As expected, differences in fruit firmness among sources and varieties of apples were statistically significant (table 5). (See also (*51*).) The loss in firmness was progressive with storage times. Differences in firmness due to the liners were not significant.

Color photographs of the apples after 180 days storage showed no difference in appearance of the apples stored in various liners. Likewise, at the end of the storage period (284 to 311 days) the condition of the fruits were similar in the various liners.[5] Highly contributory factors to these results include: all experimental fruits were commercially graded either fancy or extra fancy; all initially were without visible decay or defects; all were handled with care; all were stored almost immediately after harvest at a uniform low storage temperature; and all were stored in sealed liners moderately suitable for development of a desirable atmosphere.

As might be expected without the use of scald inhibitors, many of the Delicious and Stayman apples developed severe scald. Otherwise, all three varieties kept remarkably well and maintained a tree-fresh appearance.

The upper photograph in figure 2 shows the top and third tray from a box of Delicious apples after 9 months' storage. The lower photograph shows similar trays from a box of Golden Delicious.[6]

[5] Any of several modified atmospheres may theoretically give practically identical storage results (*119, table 5*).

[6] For possible interest in long-term storage, one such box of apples was further stored at 31° F. It was acceptably edible at 21 months.

TABLE 5.—*Condition of apples at termination of storage at 31° to 33° F.*

Variety	Firmness[1] Mean	Firmness[1] Range	Scald	Shrivel	Split	Rot	Internal browning[2]	Good
	Pounds	*Pounds*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Delicious[3] ...	9.6	9.0–10.3	75.0	0.1	1.1	1.4	8.3	14.1
Stayman[4] ...	11.1	10.8–11.5	40.6	0	.4	3.3	5.4	50.3
Golden Delicious[5]	9.5	8.7–10.6	3.8	1.5	1.4	.6	3.6	89.1

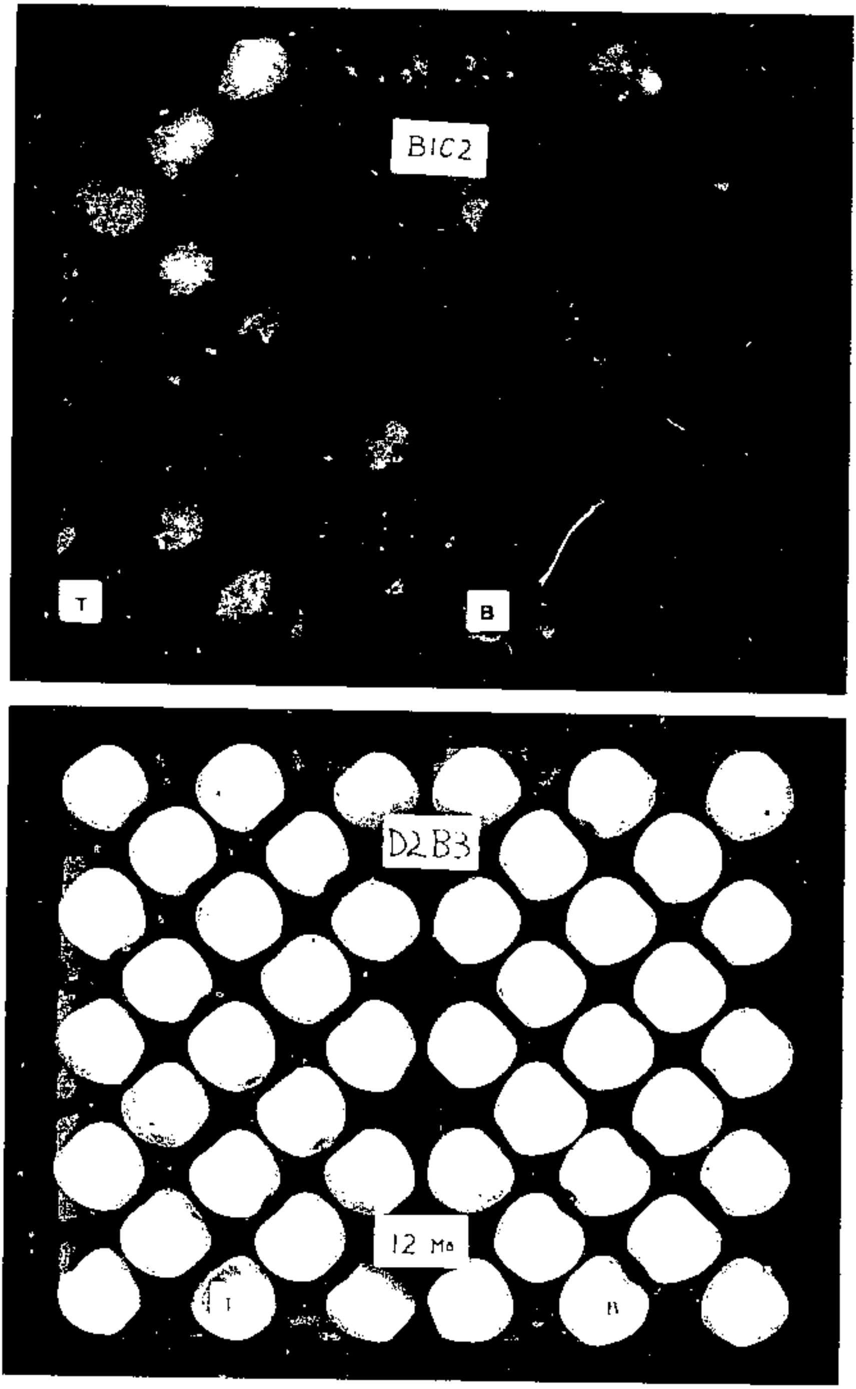
[1] Measured with Magness-Taylor pressure tester (*70*).

[2] From any cause. Measured by U.S. Department of Agriculture Optical Density Difference Meter (*12, 87*).

[3] 1,764 apples tested for firmness and 5,085 inspected for condition.

[4] 1,598 apples tested for firmness and 5,085 inspected for condition.

[5] 1,212 apples tested for firmness and 3,546 inspected for condition.



[illegible] Trays from boxes of apples stored in plastic liners: *Top,* Delicious [illegible] after [illegible] months at about [illegible] F.; *bottom,* Golden Delicious after [illegible] 12 months at about 32° F.

It must not be inferred that all boxes of apples packed in these or other types of film liners were of equal quality to those shown in figure 2. The photographs do show, however, that good-quality apples may reasonably be expected to retain quality for reasonable storage periods, provided they are properly packaged and refrigerated.

STORAGE CONDITIONS

The specification of box liners intended to produce modified atmospheres must necessarily include the desired storage temperature. This implies that the chosen temperature will be uniform throughout the storage room and within the liners.

Temperature in storage rooms

Tests were made to determine the variability in the temperatures in the experimental storage facilities. These facilities are believed to be not greatly different in age or efficiency from those used for most experimental storage investigations. They probably do not differ from most commercial installations of equal volume.

Materials and methods

The airflow patterns of each room were studied before the boxes of fruit were stored. This was done by evenly spaced ribbons. Replicate anemometer readings then were taken at each point where the pattern visibly differed. After the fruits were stored, 20 thermocouples in ethylene glycol were distributed among the boxes. Hygrothermographs, calibrated to the means of the thermocouple temperatures, provided spot checks on the room temperatures during the final months of the test storages.

Results and discussion

The airflow patterns within the storage rooms used for these experiments were highly constant at each test point where reliable measurements could be made; but air velocities and directions varied considerably from one test point to another. Some measurements could not reliably be made because of unavoidable baffling of the air currents by the anemometer supports or the observer. These measurements were not included in the means of any flow pattern. However, since storage rooms may be cooled by any of 11 or more ways and each with a different flow pattern (*103*), only sample data are given in table 6. These data are means observed at the ends of the rooms farthest from the refrigeration blowers.

These data also illustrate that cooling of storage rooms by blowers may occur either by convection plus conduction or by conduction alone. These data probably are not unusual. Ryall (*100*) noted that higher air velocities produced materially faster cooling than lower rates of air movement, although the air was 6° F. warmer at the higher velocity. Grierson-Jackson (*46*) found

TABLE 6.—*A sample of variability in cooling air velocities of storage rooms*

[Mean linear ft./min., 14 ft. from blower]

Distance from floor, feet	Variability of velocities at test point [1]—				
	A	B	C	D	E
6	201	263	103	1.5	0
4	4	10	15	0	1.5
2	0	65	0	0	0

[1] Test points were evenly spaced, left to right, across the room between unfilled boxes. Guard rows of boxes were placed about the test positions. Storage rooms were approximately 9 ft. high, 9 ft. wide, 15 ft. long, with a cooling unit at the end of the room and 8 ft. overhead. Airflow in positions D and E was toward the blower. Highest recorded air velocity in either storage room was 1,565 linear feet per minute.

wide ranges in cooling efficiencies in a study of 27 storages. These results probably explain why some freezing of fruits occurred only in the top trays of the bottom boxes of three stacks in one of our experiments and why some ice crystals formed on other fruits but the fruits did not show freezing injury.

Fortunately, there are known remedies for most airflow difficulties (*57*). For these experiments, a 16-inch oscillating fan centered 5 feet above the floor along a sidewall was used. The lowest speed was more effective than higher ones. Any combination of baffles was less effective than the fan in equalizing the airflow patterns, but no method was available to eliminate velocity gradients from the blowers to all other points. Any opening and closing of the storage doors disturbed normal air currents for several minutes. It is possible that adequate air curtains above and at the sides of such doors could do much toward lessening this variable.

The data derived from the experiments indicate that cooling within storage rooms can be positional within even small areas. This source of variability can affect experimental results; it should be given due consideration in the design and operation of experimental (and commercial) storages.

The flow patterns gave basis for the periodic shifting of box positions to obtain more equal test temperatures during the experiments. During the period that respiration atmospheres in film liners were being measured, the mean storage temperature in the room with Delicious apples ranged between 32.1° and 33.2° F., at a coefficient of variation of 1.8 percent. The range of storage temperature in the room with Golden Delicious and the Staymans was 31.4° to 32.4°, at the coefficient of variation of 1.4 percent. Thermocouple measurements in boxes indicated the mean fruit temperatures had smaller standard deviations than the rooms.

After the respiration data were collected, approximately 6 months after the tests began, the room temperatures were lowered to retain fruit conditions for later tests. The means of these lowered temperatures were 30.4° ± 0.4° F. for the Delicious and 29.9° ± 0.5° for the Golden Delicious and Staymans. This lowering of storage temperatures may have been responsible for the extended life of the film-packed apples.

Slight temperature fluctuations in commercial storages often are difficult to control. Probably they vary far more than the variations here reported. This point deserves attention, and any available remedies should be applied. Careful temperature control is important in the production of specified modified atmospheres in film liners containing produce.

Work by Denny (*29*) indicated that the permeability of certain plant tissues may vary as much as 8.3 percent per 1° F. variation. This observation was based on permeability to water, but gases were thought by Magness (*69*) to have about the same value. Gore (*45*) noted that, at a variability coefficient of only 1 percent, the respiration of fruits he tested was increased an average of 2.38 times per 10° C. (18° F.) temperature rise. In terms of film-liner permeability, such an increase in respiration would require a 13.2 percent increase in film permeability per 1° F. rise in the storage temperature. (See footnote to table 9, p. 22.) A variation of this magnitude suggests further emphasis on adequate control instrumentation and the use of a balanced refrigeration system plus any needed air curtains.

Temperature in boxes of apples

It has been presumed that temperatures in plastic-lined boxes may be somewhat higher than in unlined boxes of apples. If this were true it could mean that: (a) liner specifications probably should take this fact into account, and (b) it might be possible to keep apples in liners at a lower storage temperature and increase their storage life.

Materials and methods

Golden Delicious apples that had been stored in the pigmented-type polyethylene liner for approximately 7 months at 31° to 32° F. were used. The apples were sorted to a uniform size 113 and randomly divided into 14 boxes, nine with pigmented polyethylene liners and five without liners. Three boxes with liners and one without were evenly spaced among other boxes of apples along each of two opposite walls of the storage room. Six boxes, three with liners and three without, were placed in the center of the room, with guard boxes on either side of all boxes.

A thermocouple was fastened to the surface of each of five test apples—one in the center of each box, one at the center of each side, and one at either end of the bottom trays. A switchbox allowed each set of thermocouples in turn to be connected to a Honeywell-Brown potentiometer recorder. Readings were taken three times daily at the five points in each box. At the end of each third day the box positions were shifted until each box had been shifted five times. The 1,638 recordings were then analyzed for interactions, and the mean temperatures were determined.

Results and discussion

In 1957, Sainsbury (*102*) noted that apples in unvented cartons often took three times longer to cool than those packed in wooden boxes and that the fruit remained on the average 2° F. warmer than in wooden boxes. Only cartons with large vents and perforated trays cooled in about the same time as fruit in bulge-packed wooden boxes.[7] In 1960, Fisher (*38*) found no significant difference in the cooling rates of fruit in cartons with vents and the cooling rates of fruit in wooden boxes. These findings seem to warrant the inference that the temperatures of apples in liners may have a gradient between inside and outside the liners. Schomer and coworkers (*108*) reported that, after temperatures were stabilized in storage, temperatures in polyethylene-lined boxes averaged one-third degree higher than those in unlined boxes.

The experiments here reported were on apples identically packed. The 1,638 measurements among the replications indicated, at odds better than 99 to 1, that the position of the test packages in the stacks and the position of the stacks themselves influenced the temperature effects. The effects of these variations were determined and removed in the statistical analyses.

The data showed that room temperatures about the boxes used in these experiments averaged 29.1°±0.1° F., at odds better than 99 to 1. At the same odds, temperatures in the unlined boxes averaged 29.4°±0.1° and those in the lined boxes averaged 29.8°±0.1°. The variation coefficients respectively were: 1.1, 0.4, and 0.9 percent. The temperatures in the film-lined boxes averaged 0.35° (1.2 percent) higher than in unlined boxes, and 0.63° (2.2 percent) higher than the storage air. Although the differences between the means were statistically significant,[8] the temperature ranges were lower than those at which most commercial storage controls will operate. The results obtained agree very well with those of Schomer and coworkers (*108*).

A temperature difference of approximately 0.3° F. occurred between the storage air and inside the unlined boxes. This evidently was due to the insulation value of the fiberboard boxes. This difference would not have been due to unit thicknesses of box walls, and no single correction factor can apply to this variable, as discussed on page 18.

From the thermal conductivity values of plastics, one might expect polyethylene liners to lose heats of respiration of enclosed produce about 2.4 times as fast as polypropylene, about 2.1 times as fast as rubber hydrochloride, and 1.6 times as fast as polyvinylchloride. However, such comparisons can be misleading at the film thicknesses used in sealed packages of fresh produce. Thermal conductance values for the test liners are given in table 7. Data in the last column show that the rate of heat loss through one liner may be as much as 1.6 to 2.0 times the loss through an-

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[7] Golden Delicious apples frequently are packed in unsealed liners.

[8] By nonparametric sign test, based on 475 paired measurements, grouped into 25 equal cells, for each reported difference. See (*32*).

TABLE 7.—*Estimated heat conductance of test liners*

Film type [1]	Thickness [2]	Plastic-type conductance [3]	Test-liner conductance [4]
Polyethylene	3.81	8.0	7.3
Polypropylene	2.54	3.3	4.5
Polyvinylchloride	1.90	[5] 5.0	9.2
Rubber hydrochloride	1.90	3.8	7.0
Means	2.54	5.0	7.0

[1] See table 1, p. 3, for other characteristics of film.
[2] Mils × 10^{-3} × 2.54. Results are 10^{-3} cm.
[3] Heat conductivity in 10^{-4} cal./sec./cm.^{2}/cm./° C. difference. See chart insert "Plastic Properties" in *(40)*.
[4] (Col. 3 ÷ col. 2) × 0.35° C. Figures are 10^{-2} cal./cm.^{2}/sec./mean thickness/test temperature gradient of 0.35° C. (See text, p. 16.)
[5] Estimated.

other liner, but the ratios are not the same for the different liner comparisons. No significant differences in temperatures attributable to differences in insulation values of the liner types were found.

If one assumes that the heat of respiration of Golden Delicious at the test temperature was 730 B.t.u./ton/24 hr., the net weight per box was 43.03 pounds, and the permeable part of the liner was the thermal conductance medium, then the probable mean heat evolution of the fruits would be 3.80×10^{-6} cal./cm^2/sec. The mean heat conductance rate, from table 7, in similar units and at the 0.35° C. temperature gradient, shows the liners theoretically might dissipate this heat of respiration at the rate of 7.0×10^{-2} cal./cm.^{2}/sec., or about 1.84×10^{4} times the rate of heat production. Actually, this rate of heat loss may vary from this estimate. The calculations necessarily do not include that other insulation was present in the boxes, that refrigeration may not occur equally about each kilogram of fruit, and that heat is lost by convection and radiation.

Tissue-wrapped apples probably would lose heat less rapidly than these estimated rates. No literature was found on this subject. Oiled tissue wraps have been used for controlling scald, but other more effective means of control are available that do not require wraps (*50*). The primary interest of these experiments was identification of variations that affect liner permeabilities. For this reason the test apples were not wrapped or otherwise protected from scald. (See also p. 8.) Thus, no correction factor in the permeability formulas can be made for the use or omission of apple wraps and their effects on respirations and heat losses.

No analyses were made to obtain the times required for the test packages to reach temperature equilibriums. However, Schomer and coworkers (*108*) reported that three-fourths of the heat was removed in 58 hours from unlined boxes and in 76 hours from boxes with film liners. Fisher (*38*) found cooling time varied with the types of packs and with the distances between the rows and the stacks. In his experiments, the range in equilibrium times was 9.3 to 11.1 days to reduce the apple temperatures from 66° to 32° F. In the experiments here reported, the test apples had been in storage for 6 to 7 months and equilibriums were established.

Not all experimental cooling times and consequent storage life of produce will directly correlate with those found in commercial storages. Experimental packs are often stored in relatively small chambers where each package is readily accessible for inspections and measurements. Usually this means that the packages or boxes have one to five surfaces exposed to refrigeration air currents. And, from some surfaces the heat evolved from the respiration of enclosed produce may have to pass through only one or two thicknesses of fiberboard and possibly one pulpwood tray, but from other surfaces it may have to pass through many box thicknesses to reach the refrigerated air. In commercial storages, some boxes may have no surfaces exposed to moving refrigerated air, and those boxes on the outer sides of storage blocks may have only one or two surfaces exposed.

Sainsbury (*103*) noted that apples packed in wooden boxes and stacked in individual rows half-cooled in 27 to 50 hours and similarly packed apples stacked on pallets took 45 to 66 hours to half-cool. He noted that half-cooling times where convection is negligible varies almost with the square of the distance from the center of the packages to their refrigerated surface. In experimental chambers this distance may be but half the width of a box for a large part of the experimental lot. In commercial storages the mean distance would be much greater under almost any circumstance. The use of pallet lots both commercially and experimentally would lessen these variations.

Marcellin (*74, 75*) and Goidanich and Pratella (*44*) have reported results of storage of Golden Delicious, Delicious, and Jonathan apples in sealed plastic bags, at temperatures from 45° to 59° F. The Golden Delicious apples were held satisfactorily at 59° for 6 months. These are much higher temperatures than recommended for long-time storage in this country. Their basic work is of direct interest in a study of variables affecting film permeabilities. (See footnote 6, p. 11.) Further studies would be needed to develop adjustment factors for the RSAVΔ formula (*119*) to care for these higher temperature variables.

Atmospheres in storage rooms

Plastic-liner permeability phenomena vary with the partial pressures of the gaseous constituents inside the fruits themselves, those inside the sealed liner, those outside the liner, and with the interactions among these three atmospheres. For liner permeability specification purposes, it has been generally assumed that atmospheres inside storages will average the same composition as normal outside air. It now appears that this may be true only in the larger commercial warehouses. It may not be true in smaller warehouses and in pilot or experimental chambers.

The storage atmospheres for the present experiments were checked at 21 spaced time intervals. The data obtained very closely agreed with those found by Sainsbury and Gerhardt (*104*) in 30 storages in 21 commercial plants in the Northwest. The mean values and standard deviations in our storages were: oxygen, 20.7±0.1 percent; carbon dioxide, 0.3±0.2 percent. The ethylene content was 2 to 3 p.p.m., but this has since been found to vary greatly from this level.[9]

Twenty-one or more apple volatiles, and methods for their estimation, have been studied (*43, 77, 93, 95, 96, 129, 131*). The permeabilities of some plastic films to some volatiles have been estimated (*63*). There is information on the probable effects of volatiles in the functional diseases of fruits and vegetables (*93*). There seems to be no inference in these reports that an accumulation of apple volatiles[10] in liners is desirable and some indication that volatiles may be harmful. These data, and those reported on page 8, give further evidence that apple volatiles are present in greater concentrations within apple packages than outside them. The data tend to reaffirm that liners for apples should be as fully permeable as practical to such volatiles. There remains the problem for manufacturers that such films may need to be permeable to apple volatiles and yet nonpermeable to volatiles not found in apples.

If the 0.3 percent atmospheric carbon dioxide may be expected as a mean in commercial apple storages, as these few sample data may indicate, the 0.228 constant in formula 2 divisor (*119*) would need to be increased to 2.28. For package atmospheres to contain a low carbon dioxide percentage, this could mean a sizable increase in the carbon dioxide permeability requirements for the enclosing liner. At a barometric pressure of 760 mm., 1 percent oxygen and 2 percent carbon dioxide, this new constant would increase the film permeability requirement 20.3 percent over that estimated by the original formula. At 5 percent carbon dioxide, this increase would be 6.9 percent; and at 10 percent carbon dioxide, the increase would be 3.1 percent.

If the sample data are to be accepted as basis for a more valid estimate of the true carbon dioxide partial pressure constant, then the oxygen constant also should be changed in formula 3 (*119*) from 159.52 to 157.32. With this new constant, and with the percentage compositions of the package oxygen and carbon dioxide reversed in the examples above given, the corresponding increases in permeability required for oxygen could be 1.5, 1.8, and 2.4 percent. These are theoretical calculations. The differences in actual practice would be expected to be slightly less.

These lower increases for oxygen permeability, compared with

[9] These values are volume percentage at the test site and conditions. "Normal" atmosphere from sea level to 6.8 miles high is usually considered to contain 20.99 percent oxygen and 0.03 percent carbon dioxide, plus other gases of importance to this study only for their contributions to the total pressure of the atmosphere.

[10] For this report, "apple volatiles" means volatiles that have been found within apples. Some of these may occur also in other produce.

those for carbon dioxide, would indicate that they are less important. The reverse of this may be true. The effect of oxygen on the aerobic respiration of produce may reasonably be expected to be considerably greater than the effect of carbon dioxide. The amount of carbon dioxide produced depends largely on the amount of oxygen present. This variability was given numerical estimates for each gas in (*119, table 3*); and a comparison of the ranges and order of later estimates seems clearly to forecast that these relationships probably are relatively valid. This forecast of the greater effect of oxygen than of carbon dioxide also was well demonstrated by Anderson's studies (*9*) on the effects of various atmospheres on Delicious apples.

The relative humidity of the Delicious apple storage was about 82 percent. That for the Stayman and Golden Delicious was about 84 percent. Since none of the tested liners was hydrophilic, no efforts were made to have equal relative humidity in the rooms.

The average barometric pressure during the storage tests, at the mean test elevation of 57.71 m., was 759 mm., or 1012 mb. (millibars), with a coefficient of variation of the monthly mean about 0.05 percent. No records were kept of the daily variations. During the permeability checks of the test films, the monthly mean was 755.5±0.6 mm., with a coefficient of variation of 0.08 percent. All values were adjusted for variables due to temperature, capillarity, scale errors, and acceleration of gravity at the Plant Industry Station location.[11]

Permeability calculations usually are reduced to a standard barometric pressure of 760 mm. of mercury; this custom was followed in the various formulas of reference (*119*). This application has been misunderstood in a few instances to mean that storage atmospheres probably have a mean pressure of this value. The value of 760 mm.[12] was derived from the standard atmosphere weight at mean sea level.

APPLE RESPIRATION RATES

Methods of measurement

Respiration rates of 20 principal varieties of apples have been compiled for usual storage temperatures, and formulas have been presented for estimations at other temperatures (*119*). These rates were averages derived from reports of many workers, who used several experimental methods and whose results often approached the probable limits of ranges found by others. An additional range of data on three of these varieties was possible during the interim of the modified-atmosphere tests. These data were obtained at the storage temperature of 30° to 32° F.

The tests were by varieties; equal numbers of apples from

[11] Lat. 39°0′30″ N. (long. 76°53–7′ W.).

[12] Equivalent to 1013.25 mb. of standard mercury. International Committee on Weights and Measures, Paris, 1954. Conversions: 1 mb.=0.7500635 mm., or 1 mm.=1.333224 mb.

each of four sources were selected at random from 1,350 fruits chosen from the experimental lot of 135 bushels (p. 3). The apples from each source were further randomized into 12 equal lots, 12 apples each, for each variety and for each of two replications. The apples were sized by the packers; thus the test apples were of a random range of sizes sold commercially as size 113. The apples in each lot were tare weighed to the nearest gram.

Tests were run in duplicate by each of two methods simultaneously to obtain the respiration rates. The first of these methods was similar to that used by Haller and Rose (*47*), with only minor modifications. The second method was suggested by R. E. Anderson.[13]

This latter method used unmetered oxygen diffused into the desiccators from the storage atmosphere through a 10-inch length of 0.013-inch bore glass capillary tubing. All desiccators with their enclosed fruits were randomly arranged along one wall of a 30° to 32° F. storage room. Both equipment and apples were put in the test room, at least 1 week before any test. (At no time after initial storage began had the apples been subjected to any higher temperature.) The carbon dioxide released by the apples was absorbed in potassium hydroxide solutions that were collected and titrated at the end of each 48 hours. The indicators were phenolphthalein and methyl orange. The rinse water for the collections contained 500 ml. of glycerol per 3 gallons of distilled water. This amount of glycerol produced no difference in the subsequent titrations.

The free-air space in each desiccator was computed as the gross inside volume with all equipment and produce in place, minus the individual solid volumes of the removable pieces. The solid volumes were computed from equivalent water displacements. Each desiccator and its equipment was coded and used as a unit throughout the experiments. Fruit volumes were obtained as given on page 29. Equilibrium times were adjusted to these measurements.

An analysis of variance of the Delicious apple data showed there was no difference between the two methods of determining respiration rates. These data showed that an equal difference between means obtained by the methods could occur by chance alone in more than 50 percent of any similar sampling. Results by the two methods used were thus considered to be duplicate determinations and were combined for the analyses of variances for the three varieties of apples. Based on these data, Anderson's simplified method produced comparable results in the measurement of respired carbon dioxide, with less maintenance and setup time than that required by the Haller and Rose (*47*) method. The omission of measurement of oxygen consumption by the Anderson method was not a handicap for the purposes of this bulletin.

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[13] Research horticulturist, Market Quality Research Division, U.S. Department of Agriculture, Beltsville, Md.

Respiration rates

The mean rates of respiration for the three varieties are given in table 8. The coefficients of variation in respiration rates were 8.4 percent for Stayman, 7.8 percent for Delicious, and 4.0 percent for the Golden Delicious. The rates of both Delicious and Stayman tended to decrease with replications and time. This trend was present but less pronounced in the Golden Delicious. It is well established that respiration rates vary among varieties. The rates for each of these varieties also varied among the sources of the apples. The mean respiration rates for the Stayman and Delicious fell within probable ranges previously reported (*119*), although the confidence level was less for the Stayman data.

TABLE 8.—*Mean respiration rates of 3 varieties of apples from 4 sources at 30.7° ±0.03 F.*[1]

Source	Tests[2]	Rate of CO_2 produced per kilogram hour: Delicious	Stayman	Golden Delicious
	Number	*Mg.*	*Mg.*	*Mg.*
A	96	3.24	3.47	3.66
B	96	3.19	3.37	3.91
C	64	3.57	3.87	---
D	32	---	--	3.61
All sources[3]	288	3.33±0.26	3.57±0.30	3.73±0.15

[1] Standard deviation of temperature, 140 observations.
[2] Sample size: 864 apples from 135 cases of 113 apples each. (See p. 21.)
[3] Means and standard deviations were calculated from experiment data, not from mean rates given here. Extrapolation of data by formula $C_t = C_o \times 10^k$ would give for these varieties the respective 32° F. means of: 3.55±0.28, 3.80±0.32, and 3.97±0.16 mg./kg./hr.

Mean respiration rates for Golden Delicious have been reported for various storage temperatures, based on the weighted average results of different workers (*119*). Slightly more than half of these were measured at temperatures well above that normally used for the storage of Golden Delicious. The lowest probable limit of the mean rate at the reported temperature is about 4.7 percent higher than previous estimates, but it falls

TABLE 9.—*Revised estimated respiration rates of Golden Delicious apples stored unenclosed at 30° to 40° F.*

Temperature (° F.)	Rate of CO_2 production (Mg. of CO_2/kg./hr.)[1]
30	3.45 ±0.41
32	3.80 ±0.45
36	4.61 ±0.55
38	5.07 ±0.60
40	5.58 ±0.66

[1] Estimated by the formula $C_t = C_o \times 10^k$. See (*119*). A temperature variation of 1° F. will cause a geometric variation in these rates approximating 4.9 percent.

well within the probable lower confidence limits of those data reported by others at 32° F. It thus is proposed that the constants shown in table 7 of (*119*) should be raised to a mean between these two estimated ranges. On this basis, newly estimated probable rates and standard deviations for Golden Delicious for several storage temperatures are shown in table 9.

Effects of weight and size

The respective mean weights of the apples used in the respiration tests were: Delicious 179.1 g., Golden Delicious 174.3 g., and Stayman 187.4 g. These individual sample weights averaged greater than the mean individual weights in the total lot of 45 boxes in each variety, although the apples were sampled at random. This variation for Delicious, Golden Delicious, and Stayman, respectively, was 2.4, 0.9, and 0.9 percent greater. Respiration rates of apples usually are on a kilogram-weight basis, without regard to the size or number of apples used; this practice was followed for the results given in table 8. There is, however, the serious question whether the respiration rate of a few apples without adequate replication can be considered to represent a population mean, no matter how well randomized the sample. Much data have been produced on this basis. Analyses of those available have shown wide variabilities.

No published data were found that correlates apple sizes or surfaces to the respiration rates for the three varieties tested. Smock and Gross (*112*) concluded that their results with Wealthy, Grimes Golden, and Duchess apples did not emphasize that fruits of the same size should be selected for a given respiration study. However, much of their data showed a trend of increased respiration rate with increased apple surface area. The present data also showed a correlation trend of increased respiration rates to increased weight of the individual fruits, but the data are too few for acceptance for a reliable confidence interval.

The apples used in these experiments were purchased as size 113. Table 10 gives the proportion of the apples with mean

TABLE 10.—*Percentage of apples in respiration experiments that measured size 113 (2.83- to 2.94-inch diameter)* [1]

Source	Sample size [2]	Apples that measured size 113		
		Delicious	Golden Delicious	Stayman
	Number	*Percent*	*Percent*	*Percent*
A	192	77.1	...	35.4
B	288	70.8	64.6	37.5
C	288	45.8	56.2	12.5
D	96	. .	64.6	.
All sources	864	64.6	61.8	28.5

[1] Most others measured larger.
[2] Randomly chosen from 1,350 fruits that represented 135 boxes of 113 apples each.

equatorial diameters of 2.83 to 2.94 inches (size 113), as measured with a Cranston [14] apple thinning gage. Whether the total surface of these apples would have equaled the surface of an equal number of size-113 apples was not determined. Actual surface proportionality to respiration rates was not an auxiliary objective in these experiments. Further exploration of this possible variable may be required to determine any need in film-liner specifications to balance any effect of apple size or surface area. (See also section "Apple Weights, Sizes, and Volumes.")

Effects of equilibrium rates

There seems to be little or no doubt that the respiration rates of apples enclosed in film liners do not reach equilibrium for several days after the liners are sealed. To a much lesser extent it is probable that apples within any respiration chamber also require an equilibrium period. It is questioned whether the introduction of oxygen at atmospheric pressure within a test chamber thus may not produce a different metabolic rate of respiration than occurs within a sealed liner, where diffusion of the oxygen would be much slower in reaching the apple atmosphere. No reports were found to give further evaluation of this possibility. If such difference exists, it may be necessary to establish R values (*119*) only on accepted equilibrium respiration rates.

Effects of barometric pressures

There is some evidence that respiration measurements may give different values if made at different mean barometric pressures, inasmuch as film-liner permeabilities depend on the difference in partial pressures of the respiratory gases inside and outside the liner. By experiments covering 10 or more years, Brown (*16*) showed unmistakably that respiration rates of potatoes correlate with variances in barometric pressures of the earth atmosphere. Scanning experiments were made by the writer during the winter of 1962–63 to compare the variability of barometric pressures within apple liners with those outside. One of these experiments is here reported. (*See* Market. Res. Rpt. 842.)

A compensated anaeroid barometer was sealed inside the film liner so that it could be read through a porthole in the carton. Pressures were compared with those of a Fortin-type mercurial barometer located adjacently at the same height. Observations of the comparable pressures were made four times daily for about 3 weeks. The measurements were made in a room controlled for constant temperature and humidity. Two boxes and two different liners were used in this experiment. The two boxes were of the same fiberboard construction. They were used consecutively and not concurrently. Each was placed alone on a shelf. Each box contained 109 size-113 apples. No pressure measurements were made in boxes in a stack.

In summary, differences between the two pressures on a daily

[14] Cranston Machinery Company, Oak Grove, Oreg.

basis were slight; with inside pressures lagging behind the atmospheric pressures, except approximately at midpoint between wide swings.[15] This relative variation of the inside-package pressure with the outside pressure was expected; but the presence of the time lag was not, and this lends weight to the possible interaction of additional variables in film permeabilities.

The interactions that now appear more reasonable include: (a) The partial pressures of respiratory gases in sealed packages can be varied by protracted changes in weather outside the storage; (b) the barometric level at the time packages are sealed and the initial tightness of the film about the produce may be among causes of variation in package atmospheres that have not reached equilibrium; (c) the permeability of package films may be a variable whose specification perhaps should be based on at least one storage season at stated storage conditions. It appears logical that in the future barometric pressure may come to have some importance, along with temperature and relative humidity, in the preservation of packaged fruits and vegetables. Barometric pressure has not heretofore been considered as a possible storage factor, and no published records were found of the range of barometric pressures in apple storages. This simple experiment does not indicate any need at this time to adjust either of the permeability formulas for internal package pressures other than as already provided.

APPLE WEIGHTS, SIZES, AND VOLUMES

Weight and sizes of individual apples

Materials and methods

Each fruit was weighed to the nearest gram. The sample was 1,481 apples, from a lot of 148 boxes of 113 apples each. All were sold as size-113 apples. Mean equatorial diameters of the fruits were measured to the nearest 0.01 inch. No size allowances were made for the shape of the apples. The size divisions chosen are given in table 11.

TABLE 11.—*Identification of apple sizes used in this bulletin* [1]

[Circumferences converted to circle diameters]

Diameter limits	Diameters of apples in size [1]—						
	80	88	100	113	125	138	150
	Inches	*Inches*	*Inches*	*Inches*	*Inches*	*Inches*	*Inches*
Upper	3.28	3.18	3.06	2.94	2.83	2.73	2.65
Lower	3.19	3.07	2.95	2.84	2.74	2.66	2.59
Mean	3.24	3.12	3.00	2.89	2.78	2.70	2.62

[1] Measurements were made at the widest diameter and without allowance for varietal form of the apples.

[15] Irregular daily oscillations are usual in the temperate zones. In the tropics, diurnal oscillations occur with great regularity according to the hour of the day.

Results and discussion

Most commercially boxed apples (not prepackaged) are sold as "size 113," "size 100," etc., based only on the number of apples in the container.[18] But there seems to be no standard method by which apples are sized.

The percentages of apples by diameter sizes in the 148 boxes used in these experiments are given in table 12. This table shows that while 83.6 percent of the apples probably were size 113 or larger, only 49.7 percent were size 113 if measured in accord with the median ranges given in table 11.

Much sizing of apples is done on the basis of individual apple weights. The mean net weights of the 5,085 apples of each of the three varieties used in these experiments, and sold as size 113, averaged: Delicious, 174.9 g.; Golden Delicious, 172.7 g.; and Stayman, 185.8 g. From table 12 it is clear these weights really are the means of the several sizes included in each box and were not the mean weight of size 113 only.

The mean weights of the apples that measured size 113 were: Delicious, 167.4 g.; Golden Delicious, 166.8 g.; and Stayman, 164.4 g. A comparison of these mean weights with those for the apples sold as 113's indicates the mean differences were, respectively, 4.5, 3.5, and 13.0 percent.

By Duncan's multiple range test, the combined data for all varieties show the mean weight of apples from source C was significantly larger at the 5-percent level than the weights of apples from any other source. There was no significant difference at the 5-percent level between the mean weights of the apples from source A and B. An early study (*109*) showed that apple sizes also may vary from year to year, from tree to tree, and

[18] These usually have greater volumes but less weight than a standard bushel. As separately defined by container Acts of 1916, 1922, and 1928, a standard bushel contains 2,150.42 cubic inches, and 50 pounds. The sizes of containers most used for apples are given in (*119*).

TABLE 12.—*Percentage of apples by sizes in 13 boxes packed as fancy or extra-fancy size 113* [1]

[148 box lot]

Variety	Percentage of apples in commercial size [2]—								
	72	80	88	100	113	125	138	150	163
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Delicious	0	0	1.5	23.7	65.6	7.2	1.8	0	0.3
Stayman	0.3	0.6	10.5	46.5	39.1	3.1	0	0	0
Golden Delicious	0	0	1.6	17.0	44.4	29.3	6.8	0.8	0
Total	0.1	0.2	4.5	29.1	49.7	13.2	2.8	0.3	0.1

[1] Samples were selected at evenly spaced intervals within variety lots: 3 boxes each of Delicious and Golden Delicious and 7 boxes of Stayman.

[2] Sizes were based on mean equatorial diameters of apples.

among parts of the same tree, with respective coefficients of 6.8 to 9.6, 7.0 to 9.7, and 6.3 to 8.8 percent.

These data indicate that it probably is not practical to base permeability formulations for film liners on specific sizes of apples or too strictly on a range of sizes about a mean size per box. There is some basis for judgment in the data that commercial practice may sustain a designated mean size within something less than one standard deviation. The data indicate that net box weights, rather than individual fruit weights, may be more reliably used in determining permeability requirements.

Net weight of apples in boxes

Materials and methods

Gross and tare weights to the nearest 0.1 pound were obtained for each of 135 boxes of apples. (See p. 3.) A carefully leveled and calibrated dial scale was used for weighing. Since all work was to be done in the 30° to 32° F. rooms where the apples were stored, the scale was moved to these rooms.

Results and discussion

The net weight of boxed apples is a necessary factor in calculating specifications of plastic liners intended to produce modified atmospheres in the package. Few data, however, are available on either the net weight of a box lot or on the sizes of the individual fruits. Spurling and Bain (*114*) considered a firm tray pack for export purposes to contain 40 to 42 pounds of apples. Measurements on a limited commercial sampling of apples offered for sale in the Washington, D.C., area indicated that apples of any one actual size weigh about the same, but this weight differs slightly with varieties and with sources. These differences are caused in part by the use of various commercial methods to sort these sizes. Some packers sort by individual fruit weights; others use one of several methods based on a diameter of the apple; and some grant preferential tolerances to some varieties. It is probable that there is also some shifting of tolerances within each variety according to seasonal volumes. On most boxes, only the quality grade and number of apples they contain are given. The net weight of contents usually is not given.

A preliminary estimate of a mean net weight per box of 41.15 ± 0.57 pounds has been used for illustration purposes (*119*). This estimate necessarily presumed two alternative practices: (a) that only apples of a stated size would be packed in any container; or (b) that the boxes would contain a mean weight with an even distribution of weight sizes and numbers on either side of the mean. A purpose of this study was to gain additional information on the variance of these presumptions in commercial practice.

None of the three varieties of test apples was packed in a single size per container. This eliminated presumption (a), on

TABLE 13.—*Mean net weights of apples in film-liner boxes* [1]
[45 boxes of size-113 apples of each variety]

Film type [2]	Net weight of—		
	Delicious	Stayman	Golden Delicious
	Pounds	*Pounds*	*Pounds*
Polypropylene	43.5	46.4	43.1
Rubber hydrochloride	43.5	46.4	43.2
Polyvinylchloride	43.8	46.5	43.5
Polyethylene (pigmented)	43.3	46.3	42.8
Polyethylene (clear)	43.7	45.7	42.5
Mean [3]	43.56±1.86	46.28±0.63	43.03±1.81

[1] Each weight is the mean of 3 boxes from each of 3 sources.
[2] See table 1, p. 3, for characteristics of film liners.
[3] The standard deviation for each mean includes the variation in 15 averages of 3 boxes each per variety.

the basis of this sampling, but does so only inasmuch as there are no defined tolerances for any size.[17]

Weights of the boxes of test apples are shown for each liner in table 13. The means of the three sources were significantly different at the 1-percent level, according to the analysis of variance and Duncan's multiple range test. However, on the average, the weight of apples of any one variety packed in any one liner was not significantly different (at the 5-percent level) from the weight of the same variety packed in any other liner. At the 1-percent level, there was a significant difference between the mean box weights of the different varieties. The coefficients of variation of the mean weights within varieties were 4.3 percent for Delicious, 1.4 percent for the Stayman, and 4.2 percent for Golden Delicious.

If net weights per box were computed on individual apple weights and the percentages of each size found per average box, the box weights would have been 43.41, 45.91, and 41.51 pounds, respectively, for Delicious, Stayman, and Golden Delicious. Compared with the true weights among the 135-box sample, these computed weights would be within the standard deviation of the mean true weights of the varieties.

Based on these data the calculated percentage distribution in the various sizes may have approximated the actual distribution. It also appears that the originally estimated box weight of 41.15±0.57 pounds may be too low for a true mean of a general population. This observation would be predicated on the supposition that packers will continue to pack an assortment of sizes clustered about a mean size rather than pack only the size labeled on the box plus or minus one standard deviation from this true size.

[17] Considerable data were collected on this subject. These may be presented separately at a later time.

Based on this assumption and that the test varieties of this sample may logically be weighted by their relative importance in crop volumes, 43.86±1.68 pounds becomes an additional estimate of an all-variety mean, based on the present sample of eastern apples. Since the original estimate was based on western apples, it would seem that the true general mean probably would fall between these values. This would give the proposed mean net weight of 42.51±1.12 pounds. This value, based on present sampling, could serve as an all-purpose mean, at least for the more popular sizes of 100 and 113. As illustration, 113 apples of size-113 Delicious, Golden Delicious, or Stayman would weigh within the error limits.

Apple volumes

Materials and methods

Apple volumes were obtained from measurements of the water they displaced when submerged. The water temperature was controlled ±0.5° C. The density of the water was determined by pycnometer for a range of temperatures above and below that of the test room. The apples had attached stems. The sample size was 1,317 apples, unsorted as to source except there were 1,194 eastern-grown apples and 123 western-grown apples.

Results and discussion

The free-air volume[16] in a box of apples affects the time required for liner atmospheres to come to equilibrium. This is a source of variation in the estimation of liner performance in producing modified atmospheres. Whether a range of apple sizes or one size only is packed in liners affects this volume without doubt; but whether this change is a significant one has not been determined. There also has been some consideration that liner atmospheres at the time of sealing perhaps could be preconditioned, and thereby reduce the preequilibrium period. This period has been observed to extend 6 to 30 days; its elimination may considerably lengthen the shelf life of produce. Some data have been obtained with western apples (*119*). The present data cover eastern apples (table 14).

On the basis of data in tables 12 and 14 and assuming that size distributions of any pack probably shift with the predominant size, the total fruit volume per box for labeled sizes 80 through 125 should approximate 23,000±850 cc. This mean reasonably agrees with the previous estimate of 22,400 cc. that was based on a sample smaller than this one (table 14). This difference does not materially disturb the basis for the original calculations (*119*).

[16] This is the inside package volume not occupied by fruit, liners, wrappers, trays, or cell dividers.

TABLE 14.—*Volumes of individual apples of 3 varieties*

Variety and factor[1]	Volume of apples in size[2]—						
	80	88	100	113	125	138	150
Delicious (187):							
Volume ....cc..	280	264	231	209	186	174	155
Variation[3] percent .	6.9	7.9	9.1	10.1	4.6	4.7	6.2
Stayman (565):							
Volume ....cc..	252	231	210	195	179	0	0
Variation[3] percent .	4.0	1.1	0.9	0.7	1.4	0	0
Golden Delicious (565):							
Volume ....cc..	0	246	230	208	190	176	0
Variation[3] . percent .	0	5.7	1.8	1.5	0.8	1.7	0

[1] Numbers in parentheses indicate the number of apples tested.
[2] Based on mean equatorial diameters of apples.
[3] All volumes are individual means. Samples were randomly chosen by case lots from 7 sources.

Apple-tray volumes

Materials and methods

The volumes of pulpboard apple trays were obtained by water displacement in a manner similar to that used in obtaining apple volumes. A wire cradle kept the trays submerged with the help of two 200-g. weights.

Results and discussion

Trays used in apple boxes vary in design. The corners of some are notched, some diagonally cut, and some rounded. Some have holes in the bottoms of the cups; some do not. The islands between cups have different heights; and some islands have holes, and others do not. Occasionally, some trays in an apple box appear to be older than the other trays. It has been questioned whether these differences may be disregarded in determining free-air volumes and in considering uniform atmospheres.

It was found that these differences in total could cause as high as a 44 percent coefficient of variation in the volumes of these trays; but this variation would amount to only 1.4 percent of the free-air volume of the box and thus may be considered negligible in importance compared with other measurable variables.

For the development of uniform modified atmospheres in sealed packages, apple trays with rounded corners, islands with holes, and fruit pockets with holes would seem to have advantages over some other design variations.

LINER FAULTS

Film faults are recognized as a source of variation in producing modified atmospheres in packaged produce; but few data have been presented showing the incidence and kind of imperfections observed. Schomer and coworkers (*108*), in 1954, reported that of 280 sealed liners examined in commercial apple-packing plants in Washington State 5 to 25 percent were not airtight. In 1963, Hartman and coworkers (*53*) noted that 25 percent of their 0.7 mil rubber hydrochloride film samples and 20 percent of their 1.0 mil polyethylene samples contained pinholes, but they gave no sizes or numbers. Sargeant (*107*) reported that of 46 carefully sealed packets only 28 had perfect seals. Tomkins (*121*) mentioned that one pinhole per bag is not unusual, but he reported mostly on small bags and containers.

Other workers have studied how to detect the presence of leaks in plastic containers but not especially how to classify them or trace their sources. Tomkins (*121*) offered a method for estimating when pinholes are present; but his method would seem to include other leaks as well. Other workers have proposed tests for unidentified leaks in pouches (*26, 88, 125*), in bags (*54, 56, 73, 110, 130*), and in films (*21, 22, 53, 58*). Most of these methods are limited to small packages, to laboratory tests, and special uses. The tests often preclude further use of the samples or packages. A few of the methods are elaborate and expensive.

Initial tests

Materials and methods

At the conclusion of the storage periods summarized in table 2 (p. 5), some measures of the existence and extent of liner faults possibly present in the 135 test liners was determined by the Tomkins method. Tomkins (*121*) reported that, when the decline in the oxygen (O_2) percentage in a package does not equal at least 1.5 times the rise in carbon dioxide (CO_2) percentage, his experiments invariably disclosed imperfections in the bags or their seals. Our final atmospheres, means of three boxes each of three apple varieties, from three sources, packed in five liner types, were tested for such indications by the formula:

$$\left[\frac{21-(\%O_2)}{\Delta\ \%\ CO_2}\right] = > 1.5 \qquad [2]$$

where: 21 is the approximate percentage of oxygen in a normal atmosphere; ($\%\ O_2$) is the percentage of oxygen at the final inspection; and "$\Delta\ \%\ CO_2$" is the difference between the final and initial percentage of carbon dioxide present in the package. Both gases are given in percentages to one decimal place.

After initial photographs were taken of each packed box of apples, the liners were removed, the top of each liner was severed immediately below the liner tie, and each top was tagged and set aside for the two studies of liner seals reported on later (p. 34 and p. 38). The liners were then tested by inflation with compressed air at about 2-pounds-per-square-inch pressure to disclose obvious splits, tears, and punctures detectable from the escaping air. A few liners were tested by immersion in a vat of water. The liners were then cut into 8- by 11-inch rectangles for development of other techniques of flaw detections.

Results and discussion

Among the 45 ratios obtained, the means for the liner types were: polypropropylene, 1.31; tinted polyethylene, 3.30; polyvinylchloride, 1.38; rubber hydrochloride, 5.81; and clear polyethylene, 2.64. The respective ranges of each were: 1.1–1.5, 3.0–3.7, 1.0–1.7, 5.0–6.7, and 2.0–2.9. The general mean ratio for the five types was 2.41. At odds of 19 to 1, there was no ratio difference due either to varieties or sources of apples. At odds of 99 to 1, there was a difference among the ratios due to liner type or to faults associated with the liner type.

In defense of the ratio test, more than half the liner faults were found among three liner types that gave the lower ratios; and the faults were about evenly divided among the three. It is a little less easy to account for the differences between the tinted and clear polyethylenes and between either of the polyethylenes and the rubber hydrochloride. There seemed to be no correlation of the ratios with the thickness of the films. On the bases of these data, it would seem that minimums of acceptability for the ratios might well be correlated with liner compositions; hence, perhaps a minimum ratio should be established for each type of liner.

Also, in partial support of the general mean ratio of 2.41, it was observed during the storage tests that 23 percent of the liners were imperfectly sealed regardless of the extreme care used in forming, packing, and handling the liners. The division of these observed faults were classified as: liner splits and tears, 8.1 percent; pinholes or punctures, 0.7 percent; faulty seams or heat seals, 8.9 percent; failure to include all folds beneath the twist ties, 2.2 percent; thin or stretched areas, several; possible stress cracks due to deterioration of films, 3.0 percent. Most of these faults are believed to be of minor significance; and in every case possible the liners either were repaired immediately when a fault was found or the liner was replaced. Most of these faults were observed before any equilibrium of the liner atmospheres. A few were suspected and found after equilibrium through use of the Tomkins test.

Attempts to detect imperfections in liners before use were limited to close observation of the seams and inflation of the liners with air. These attempts were not very satisfactory. The tests depended upon the quietness of the room plus subjective acuity of vision and hearing that varied with the inspector. Immersion of the liners in water after inflation was not practical. Not only did this present the problem of drying the liners but the pressure necessary to submerge the liners often damaged them.

It is probable commercial practice to discard film liners with grossly obvious faults; but neither routine nor sequential sampling tests are used by packers to insure that their liners are without faults. They rely upon manufacturers to perform this service.

The receipt of liners without faults should be paralleled with due precautions that liners are not damaged during packing of fruit. The splits or tears in 8.1 percent of the test liners occurred most often at the seams; these were due principally to sharp corners on the packing trays. As previously noted, the sample trays were variously cut. The rounded corners gave far less trouble than other types of cuts. Some of the tears doubtlessly were due also to the use of liners not correctly proportioned to the boxes and their contents. (See section "Film-Liner Design, Dimensions, and Tolerances.")

Pinhole leaks

Pinholes in plastic films have been mentioned in reports on packaging more often than any other flaw. Means have been presented to detect them in films (*21, 22, 53*); to forecast their existence (*25*); to estimate whether they are present during storage periods (*121*); and, perhaps more importantly, how to prevent them (*4, 41*).

Materials and methods

Several methods for detection of pinholes were attempted, but only two proved to be of even minor worth compared with the general applicability of the Tomkins method previously mentioned.

For the first method, ten 1-inch squares were cut from each of five liners of each of the five plastic materials. Twelve microscopic fields of each of these 250 samples were examined at ×28 magnification for pinholes larger than 0.012 millimeters. Some additional inspections were made of microtomed cross sections of the films placed between sheets of cellophane and imbedded in Fisher Tissuemat.

The second method was similar in principle to that reported by Carson (*22*) and later by Hartman, Powers, and Pratte (*53*). A hole 1 cc. deep was machined into a brass block used also to cut uniform film samples. A rubber ring gasket, 5 millimeters thick, was tightly fitted into this well. For each test, a mixture of powdered sucrose and 1 percent finely granulated crystal violet dye was placed in the ring in sufficient amount to be level with the shoulder of the rubber gasket. On this was placed the cut film sample. A heavy copper ring held the film taut and in contact with the sugar-dye mixture. Approximately 5 ml. of distilled water was put on top of the film. With the film held in place tightly, it was reasoned that the water would seep through pinholes present and dissolve the dye by diffusion through the sugar. The dye spots would approximately locate the pinholes.

Results and discussion

Among the 3,000 or so microscopic fields in the 250 random samples of the five liners, only 28 fields contained one or more pinholes greater in diameter than 0.012 mm. The microscopic cross sections of the films revealed nothing of usable importance to the present study.

The Carson-Hartman method was adequate to detect pinholes in the film samples if the holes were about 1 mm. or more in diameter; but the results were not always positive when smaller holes were present. Since the results from this method were erratic, the data were discarded and the method abandoned as unsatisfactory. As most packaging films are not easily wetted over an entire surface, possibly the addition of some wetting agent to the water placed on the film samples would cause the water more readily to pass through any pinholes present. By the method, as used, few pinholes were found. It would appear from these few data that pinholing perhaps has been overrated as a film-liner fault.

Not all pinholes can be presumed to be small. On one occasion a roll of packaging film was received from the manufacturer with the free edge stapled at either end and at two places near the middle. This evidently had been done in the shipping department to keep the roll from unwinding. It is hoped this was an isolated instance; yet the fact that it occurred suggests that not all personnel are fully aware that packaging films for many purposes should be free of holes.

As to possible tolerances for pinholing, there are few data on which to base a judgment for variability. Freeman and coworkers (*41*) reported that leakage through holes 0.1 mm. and less, at low temperatures, were in the range of the true permeabilities of plastic films. Presumably this implies that the permeabilities of such films are not adversely affected by holes this small; i.e., that gases may pass through such holes no more readily than through the other parts of the film surface. It is suggested that the numbers of holes and sizes could make a difference in their importance. It is conceivable that the presence of the smallest of holes in sufficient number may greatly alter the calculated permeability of a plastic film. It is suggested that any calculated probability of the presence of such holes or their importance can be no more reliable than the degree to which the test samples truly represent the population of all liners from all sources of variation. The far more reliable answer is their elimination.

Seam leaks

The method of sealing liners is one of the important variables in the satisfactory production of modified atmospheres in packages. The seam has invariably been found to be the weakest part (*73, 88*). This circumstance may have had a large part in the erratic results obtained by Ryall and Uota (*101*), Smock and Blanpied (*111*), and Hardenburg and Anderson (*48, 49*). Smock and Blanpied reported that half of the liners from one source were found to be leaky and had to be discarded. This is a circumstance especially suspected wherever gusset liners have been used. Dale (*26*), Schomer and coworkers (*108*), Sherman and Mannheim (*110*), Tomkins (*120, 123*), and others have reported unidentified seam and seal leaks; but particular trouble spots have not been reported in the literature. This may have been due to the lack of any identifying test. Attempts were made to develop such a test.

Materials and methods

Various dye solutions and liquids with low surface tensions were applied to many liner seams in the hope of finding something that would move visibly through any imperfections in the seals. None of the liquids performed better than a few drops of diluted[30] india ink applied directly where the seams joined. Wherever a leak was present, the ink was drawn into the imperfection by capillary action and was visible on the opposite side of the imperfection in a few seconds.

Poststorage tests by the india ink method were made on 90 liners—27 gusset-type liners from each of two commercial fabricators and 36 liners without gussets.

Results and discussion

All the gusset-type liners from one commercial fabricator had an air leak at one or more junctions of the double and single seams. Gusset-type liners from the other source showed no such leaks. In these tests, 27 percent of the 90 liners had one or more seam leaks. About half of these occurred at the bottom of the liner, at the junctures of double and single seams. The other half occurred along single seams.

The ink migration paths through the seams were rarely straight, and they often were of greater length than the seam widths. This could account for the variability of gas transmissions of liners from the same lot when sealed with the same time-heat periods. It further suggests the improbability that gas transmissions through such paths can reliably be subject to mathematical prediction.

Points C, figure 3, show the appearance of two major seam faults of different magnitudes. Points A show the almost invariable presence of tiny leaks at the juncture of the double and single seams of gusset liners. Figure 4 shows a seam leak at the right-hand gusset; none of significance occurred at the left-hand gusset. The bag was commercially made and of good quality. Leaks through box liners have a similar appearance; and, thus, can be unmistakably located by the ink test to facilitate corrections in subsequent fabrications.

Much research has been done on the sealing of plastics. Technical data are now available on the heat sealing of polyethylene (*31*, *99*), polypropylene (*117*), and other polymers (*99*). For difficult bonding, work has been reported on sealing by high frequencies (*23*), by ultrasonics (*27*), and by infrared (*106*). The thermodynamic relations of heat-sealing times, pressures, web

[30] The dilution was 4 drops of 5-percent reagent ammonium hydroxide per ounce of ink.

tensions, diffusivities, viscosity limits, fusion points, and quenching are involved, but these basic papers are offered for further investigations.

All sealing methods presume positive identification of the film to be sealed. This usually is not a problem for either manufacturers or fabricators, but conceivably it could be for others in industrial laboratories or research workers who do not regularly work with plastics. Simple qualitative tests for the more common plastics have been given by Dressel (*33*) and in reference (*91*). (For more precise tests, see Film-Liner Identities, p. 40.) The sealing of film bags and liners no longer should be a problem; but the complete solution requires development of a commercial method of checking that all seams are unquestionably hermetically secure.

Sargeant (*107*) developed a highly reliable apparatus for line or check testing of closed packages anytime during their life use. It is not clear that this apparatus could effectively test liners inclosing produce in rigid or fiberboard shipping containers. It is probable that the equipment could be suitably adapted to do so.

At the least, it would seem that the simple india ink test could be applied at commercial speeds to provide fabricators with a quality control method to assure that liner seams are without leaks. The visible presence of the ink could do much to assure buyers that such tests had been made in their behalf. The costs would be far less than the costs of quality losses through unsatisfactory storage.

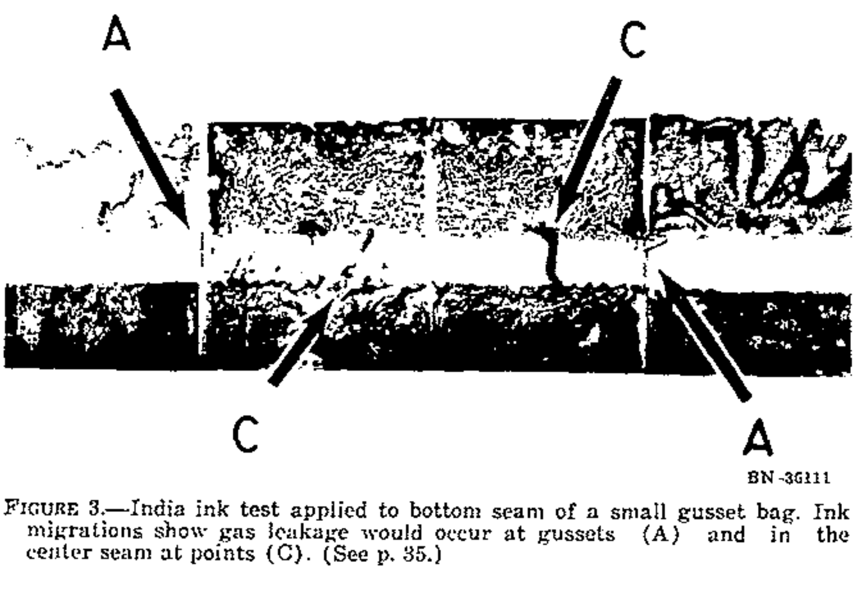

FIGURE 3.—India ink test applied to bottom seam of a small gusset bag. Ink migrations show gas leakage would occur at gussets (A) and in the center seam at points (C). (See p. 35.)

FIGURE 4.—India ink test applied to bottom seam and to the twist seal of a small gusset bag. (See p. 38 for discussion.)

Twist-seal leaks

The twist-seal tie as a method of closing liners, supposedly hermetically, has been used for many years in apple-packing plants and apple-packaging experiments. The india ink test used for testing completeness of seam seals was considered as a possible test for twist-seals.

Materials and methods

Diluted india ink [20] was applied to the center of the deepest folds. If the tie was leaky, the india ink could be observed below the tie in a time commensurate with the fault. The time periods varied from 5 to 40 minutes or more. This suggested not only the need for a better tie or closure but also the need for a faster test. Some comparisons were made in the interest of each of these goals.

About eighty 4-inch strips were cut widthwise from undamaged 1½ mil polyethylene liners. Ties were cut from 5/16 inch cotton cloth tape,[21] carpenters' chalkline, and 8-ply cotton twine. Three inches from an open end of a liner strip, one of the three types of ties was wrapped twice about the gathered piece and tightly tied with a hard knot. Twenty-five ties were made with each type of binding.

Air at 1-inch gage pressure was maintained in a manifold fitted with a quick-acting ball valve and a ½-inch rubber hose outlet. Flush into the end of this hose was fitted a short length of copper tubing. Twenty drops of the india ink solution were placed in the center of the liner folds. The folds were quickly tied to the tube-supported hose, and air pressure was released to the liner top at the instant a stopwatch was started. The time for the ink to appear beneath the ties quickly indicated the effectiveness of the three types of seals.

Results and discussion

The india ink test proved to be a valuable tool in testing the efficiency of various twist-seals, as well as of seams. The time required for the ink to be forced through the twist-seals tied with carpenters' chalkline averaged 1.30 minutes; those tied with the cloth tape, 1.25 minutes; and those tied with twine, 1.43 minutes. The coefficients of variation, respectively, were 33.8, 13.6, and 14.0 percent. Although the tape ties were not so secure as either the cord or twine ties, they produced a less variable tie and did not damage the liners. Both the cord and twine did damage the neck of some of the samples.

From this study of 75 twist-seals it was found that, no matter how tightly the ties seemed to be made, the india ink visibly passed through 88 percent of the ties if they were made in the usual way. However, when the folds of test liners were tied once

[20] See footnote 19, p. 35.
[21] Fed. Spec. DDD-T-101c. Not gummed.

then folded over and tied again, a "double-twist" seal, only a little less than 23 percent of the liners showed any ink migrations. On the basis of these experiments, it seems reasonable that gas molecules probably move through any twist-seal or tie; but this movement is, at least, much slower through a double tie than through a single one. (Provisions have been included in calculations for liner specifications for the use of double ties, p. 65.)

It is probable that leaks at twist-seals may in the past have been more frequent and important than other leaks and that the twist-seal variable is badly in need of correction. A far better way to close liners might be to seal them instead of tying them. It is believed that a commercial heat sealer could be developed to produce a top seal of the same type as the bottom one and that this could be done without too great difficulty.

Thin places in liners

Thin places, strains (*133*), stresses (*19*), and creases (*92*) have been listed as causes of variability in the permeability of packaging films. The effects of such flaws have been measured, and attempts have been made to predict their occurrence. There seem to be no objective tests to indicate their presence, and they cannot always be readily observed subjectively. A method was sought to detect them with greater sureness. The best method was based on light diffraction principles reported by Zimm (*134*) and Debye and Bueche (*28*).

Materials and methods

Rectangles 2- by 4-inches were cut from a random selection of the 8- by 11-inch samples of the different liners. Each of these in turn was placed between two plates of single-strength window glass, and the assembly was held at about a 30° angle to a directed beam of light. When the film samples then were observed from a 90° angle to the incident light, many of the stressed areas and creases became clearly visible.

The thin places in liners were occasionally visible as puckered areas. These, too, became more visible when the liners were tested by the diffracted light method. Some microscopic observations were made of thin places mounted as cross sections between sheets of cellophane imbedded in Tissuemat.

Results and discussion

As mentioned on page 32, several thin places were observed in the 135 test liners. These were not recorded by frequency of their occurrence because it was believed this would grant an undue bias to their importance. First, no instance of "fish-eye" shape faults that have been mentioned in the literature were found in the liners. Instead of a double-convex shape, each microscopic cross section of the thin places showed them to be double-concave. Secondly, the permeability of these thin places may differ from that of the rest of the film as the ratio of their mean thicknesses.

The mean thickness of any fault of regular shape would be equal to a normal volume of the fault minus the volume loss due to the fault, divided by the fault area. However, among the test liners no fault of any regular shape was found. For these reasons, plus the enormous task of adequate sampling by microscopic inspections, any evaluation of thin places by numbers or percentage of occurrence is without practical significance.

It appeared probable that in this sample the thin places were caused by stretching of the films subsequent to their manufacture. Stretching in some instances may have been due to pulling the twist-seal too tightly. It often was due to sharp corners on the packing trays. In a few instances thin places developed at points where the apple stems caught on the liner or pressed against it as the liner was pulled taut for tying.

Although it has been stated in the literature that the effects of such faults have been measured, it is difficult to believe that such measurements have effectively discounted the possible inclusion of faults other than thin places. For such measurements to be meaningful the permeability measurements would have to be confined to the areas of the thin places alone. Attempts to do

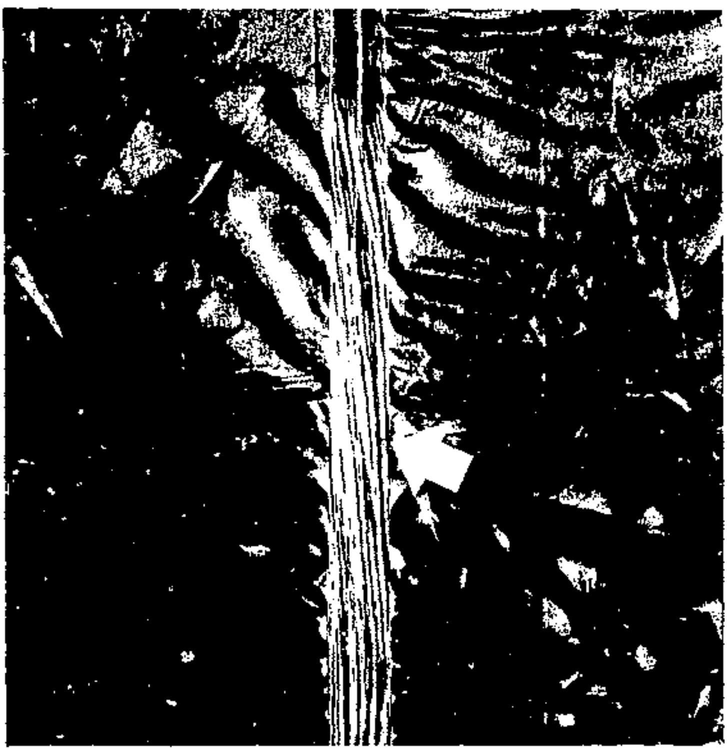

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FIGURE 5.—Diffracted light photograph (X 3) of faults in a film liner. (See p. 39.)

this, and then to compare the results with those of an adjacent film sample, were not satisfactory with the permeability equipment available for these tests.

The appearance of thin places, creases, folds, and tensile stress in liners is illustrated in figure 5. Diffracted light shows clearly the vertical thin place at the middle of this photograph is a series of light peaks and valleys of varying thickness, number, and orientation. The stress exhibited was produced by deformation of the film; it is noticeably different in appearance from stress caused by deterioration. The creases can be identified by their irregularly oriented straight lines. Rarely were these accompanied by apparent stress, but conceivably they could be so accompanied if the crease is very severe and maintained. The folds seemed to show no stress, yet clearly stress must have existed to cause the deformations shown. Based on these observations, the elimination of such faults should be far more practical than any calculations of their probable existence.

Film-liner identities

The identification of packaging films is important for proper sealing, in tracing the nature of liner faults, and in checking film orders. During the handling of the several hundred pieces of film during these investigations there were occasional doubts whether a film sample was from one coded folder or from another. There was a question whether a film shipment was of the type specified. There also was the question whether certain chemical changes might have taken place in some films. These questions were beyond the original purposes of this study, but the answers were important to the total results. Thus, a method was sought to identify each test film quickly, qualitatively, and nondestructively.

Materials and methods

Adaptation experiments in 1961 suggested use of a ratio recording infrared spectrophotometer.[22] A reference file of 86 such analyses of plastic films was compiled for any needed comparison. (See also (*37*).)

The five types of film liners used in these studies were identified by their infrared transmission curves (fig. 6). One hundred and seventeen analyses were made.

Results and discussion

A study of the analyses showed this method to be rapid, perfectly repetitive, and fully satisfactory to identify packaging films, to compare lots, and to detect changes in any lots caused by aging or chemical variations.

The method proved useless to detect or evaluate the presence of pinholes in a film sample. The reproduction of major and minor transmission curves was excellent when the same un-

[22] Perkin-Elmer, Model 13, NaCl prism, 1.0μ–15.5μ.

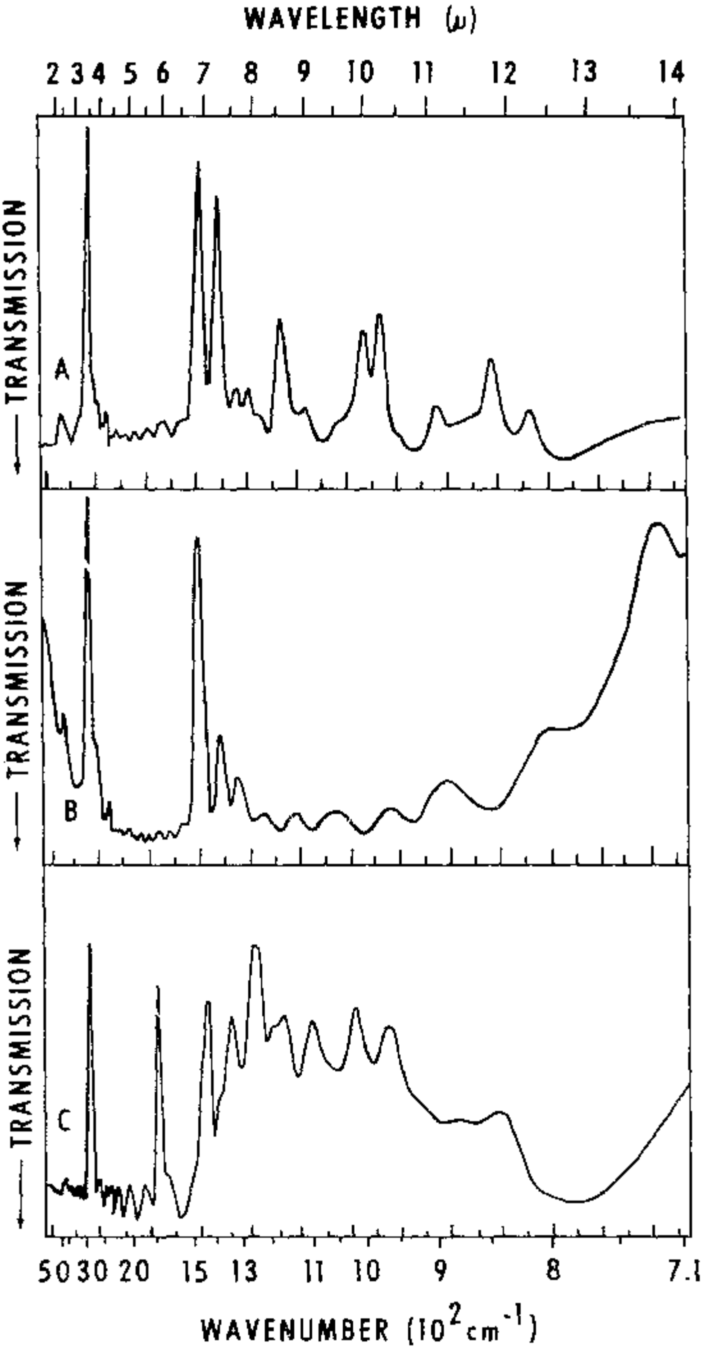

FIGURE 6.—Film transmission of infrared rays, NaCl prism: *A*, Polypropylene; *B*, tinted polyethylene; *C*, polyvinylchloride; *D*, rubber hydrochloride; *E*, clear polyethylene.

touched sample was used. However, the curves often differed slightly in minor peaks when test samples were from different liners of the same type, sometimes even when they were from different parts of the same stock-film roll, and in a few instances when they were from different parts of the same film liner. If film samples may vary in composition, as these infrared analyses indicate, some differences in permeabilities are more easily accounted for. Higher transmission gain was helpful in detecting

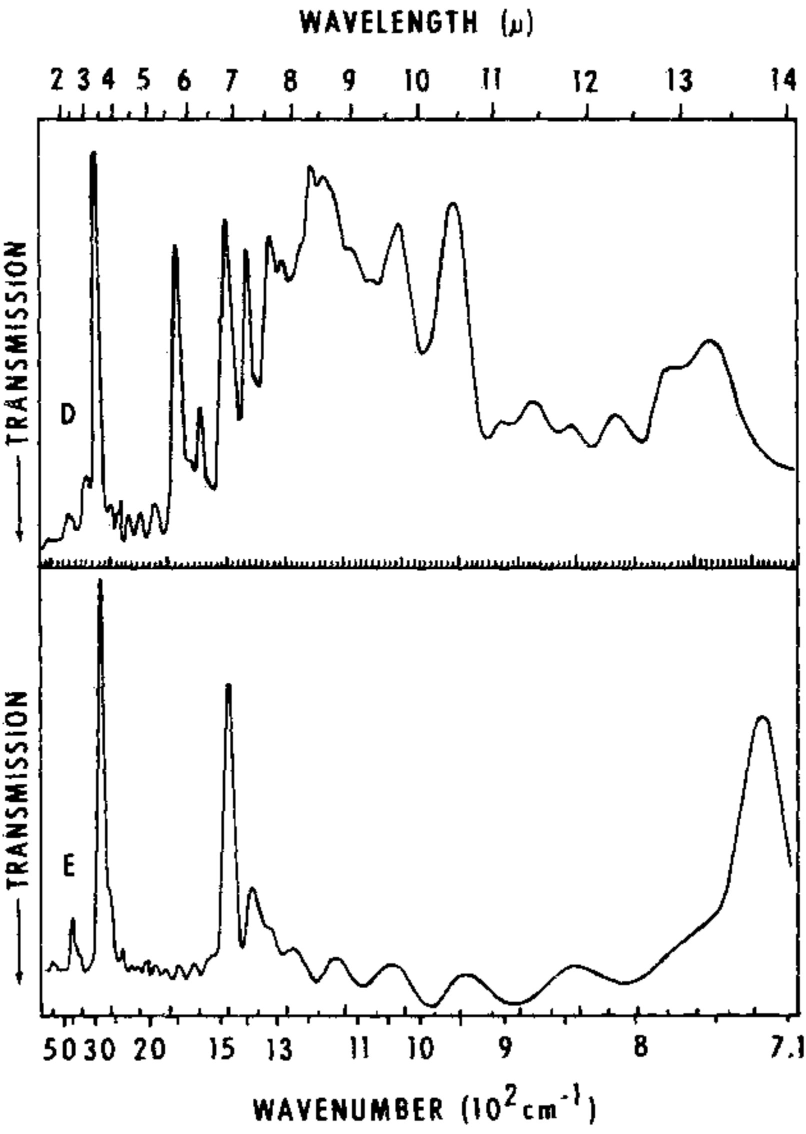

FIGURE 6.—Continued

small comparative differences among samples, but this also presented the problem of more electrical noise. Regardless of these few difficulties, the method proved to be a valuable identification tool. (See also (*13*).)

Film deterioration

At the conclusion of the storage tests brittleness was observed in some liners. (See also (*113*).) It was reasoned that this condition may have caused variability in film permeabilities. Accordingly, nine of the worst films in each apple-variety lot were set aside for rating and further study. Code numbers later showed these 27 liners were polypropylene.

Materials and methods

Infrared light transmissions of the films were measured in the frequency range of 1.8μ to 12.5μ. These transmissions then were compared with those of similar but unused film samples to discern whether chemical changes had occurred in the films. The 27 liners were subjectively graded (table 15).

Results and discussion

Nine of the worse liners contained either Delicious or Stayman apples. These varieties develop storage scald easily and did so in the absence of oiled wraps. It was found that 44 percent of the deteriorated liners contained apples from one grower who may have treated his apples with a chemical scald inhibitor. At the 11 wavelengths, there were changes between 3.0μ and 11.56μ. Similar comparisons of other film types showed slight spectral shifts in polyethylene at other wavelengths, but none of these showed any brittleness.

Worthington[23] earlier had noted some darkening of films con-

[23] Unpublished data. Courtesy of John T. Worthington, research horticulturist, Market Quality Res. Div., U.S. Dept. Agr.

TABLE 15.—*Subjective grading of brittle polypropylene film liners about 6 months after storage tests*

[Sample size: 27 liners]

Apple variety in liner	Liner damage (brittleness)[1]		
	Very slight	Slightly objectionable	Objectionable
	Percent	*Percent*	*Percent*
Delicious	14.8	0	18.5
Stayman	18.5	3.7	11.1
Golden Delicious	22.3	7.4	3.7
Total	55.6	11.1	33.3

[1] Very slight, some brittleness was present; slightly objectionable, film might be used for a short additional time; objectionable, films showed some evidence of cracking.

taining apples displayed under fluorescent lighting. He then stretched polypropylene and polyethylene film over breakers containing 2,000 p.p.m. of an antiscald suspension(ethoxyquin). At high humidity, the polyethylene darkened and was found to fluoresce under 3,660 Å ultraviolet light. The polypropylene was unaffected. At low humidity, neither film darkened and both fluoresced. The films not exposed to the antiscald suspension neither darkened nor fluoresced. Worthington's experiment demonstrated that the antiscald preparation was volatile and apparently was adsorbed by the films. Whether absorption also took place was not clearly established, although it was found that the darkening of the films could not be reduced by subsequent washing with the antiscald suspending medium. Neither of these test films became brittle.

Additional experiments by the writer confirmed Worthington's results, but brittleness was not developed in any sample by ethoxyquin. Brittleness was developed however when an aged but unused sample was suspended in air in direct sunlight during the daytime and under a dual 15-watt 3,500° fluorescent lamp at night. The brittleness was objectionably present at the end of 52 days. Brittleness also was later found in a stock roll of polypropylene that had been stored in the dark at 75° F. for about 2 years. These experiments suggest that the scald inhibitor did not cause brittleness.

The appearance of film aging in its early stages was accompanied by stress cracking, or crazing, visible by diffracted light (fig. 7). These observations were made by the writer's method for detecting thin places in liners (p. 39).

Polymer degradation can be attributed to several factors that depend on the polymer structure and conditions (*68*). Blake and Thompson (*13*) illustrated the aging of rubber hydrochloride. They noted that spectral absorption increases as the film ages. Wall (*127*) considered polymer degradations mainly to be reac-

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FIGURE 7.—Light diffraction photographs of film: *Left,* Normal film; *right,* crazing. (See p. 39.)

tions in the side chains rather than merely the result of weak links in the molecule. Wright (*132*) recorded that plastics may be aged by heat, light, chemicals, bacteria, or simply by long storage, with distinct losses in physical, mechanical, optical, and electrical properties.

Wright (*132*) cautioned that almost all plastics may deteriorate within 2 years. But he stated that the aging of polyvinylchloride and polyethylene (among others) can be delayed up to 3 years by stabilizers. Without stabilizers polyvinylchloride can be made to age within a few hours, and polyethylene will degrade rapidly under natural weathering at an equivalent of 100 to 900 hours in a fadeometer. According to Wallder and coworkers (*128*), the aging of polyethylene can be proportionately delayed as much as 20 years by the addition of as little as 2 percent carbon black. Several stabilizers exist for polyvinylchloride, such as metallic soaps, organic nitrogen bases, and organo-tin compounds. It seems reasonable to expect that aging of packaging films can be delayed sufficiently for any commercial storage period and thus eliminate the aging variable in the use of film liners for modified-atmosphere fruit storage.[24]

FILM-LINER PERMEABILITIES

Film-liner permeability measurements proved to be the greatest source of unassessed variables among those studied. Film permeability has been measured by many methods. Most of these have been used at a convenient room temperature, with the results extrapolated to other temperatures by one of several theoretical concepts. It is unfortunate that all permeability instruments do not expand and contract at the same rates, do not have equal sensitivities and precisions, and do not have equal usefulness at all temperatures. Apparently few, if any, film permeability measurements have been made at 30° to 31° F.—the usual storage temperatures of apples. Reliable values at these temperatures can be obtained by few methods. The methods here used were not wholly satisfactory, but they seemed to be the best available that might simulate commercial conditions.

Major and Kammermeyer (*72*) wrote, "We have made thousands of permeability measurements and we have reluctantly come to the conclusion that they are anything but simple." It is because of the interaction of variables, most of them associated with the equipment itself, that reported permeability measurements apparently vary so widely. The identification of some of these variables, a major objective of this study, is the obvious initial step toward their ultimate solution.

There is very little in the literature that can be of direct help in determining how one should partition his analyses of variances among equipment, operator, and materials. Suppliers of the plastic materials used in this study were asked what percent tolerance

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[24] Films used in the present study were experimental types. Commercial types may be more stable and without these faults.

in permeability should be considered reasonable. The consensus of the replies was that ±10 percent should be allowed, principally for variances in equipment control. Karel and coworkers (*61*) wrote that measurements of the same sample of film indicated the precision of their method was of the order of 5 percent. But they concluded there can be only permeability ranges for a given plastic material because variations exist between different batches of the same plastic and between samples. Taylor, Karel, and Proctor (*116*) noted in measurements of oxygen permeability a coefficient of variation, "due to inherent variability of samples," was of the order of 5 to 15 percent.

Materials and methods

Equipment

The permeability experiments were conducted in an insulated room with temperatures automatically maintained. The room was baffled to provide a reasonably even airflow to all parts of the 1,185 cubic-foot volume. Temperature checks were made at each equipment piece by either a standardized thermocouple or thermometer. Mean room temperatures for the different experiments were obtained from 20 thermocouples distributed over the room and recorded on a Honeywell-Brown potentiometer. Temperature fluctuations were small.

Clocks and stopwatches were calibrated for the temperatures at which the instruments were to be operated.

An anaeroid and a secondary standard barometer were used for most of the work. The latter instrument was standardized daily and was calibrated for expansion, elevation, capillarity, and gravity.

The absolute vacuum gage and all secondary gages were calibrated against master gages at the barometric pressure of the experimental periods, and correction curves were drawn for each. Gage pressures were converted to the absolute where this usage was appropriate.

The test gases for all experiments were purchased in size 1 or 1A tanks. All tanks, with regulators and gages attached, were kept at the storage temperature for at least 24 hours before use.

The gas tanks were connected through diaphragm regulators to a heavy steel manifold of about 1.3 liters capacity. This manifold led to a vacuum pump. The other end of the manifold was connected through a moisture filter to the inlet valve of the permeability apparatus. The equipment and fittings were checked daily for gas leaks both by vacuum-retention times and a low-tension detergent.

The permeability-measurement apparatus (fig. 8) was essentially that designed by Major (*71*), with necessary adaptations.

Since some of the permeability measurements were made at 0° C., water could not be used in the capillary tube to measure the gas permeated through the plastic samples. The indicator chosen for most of the work was a mol-fraction mixture of

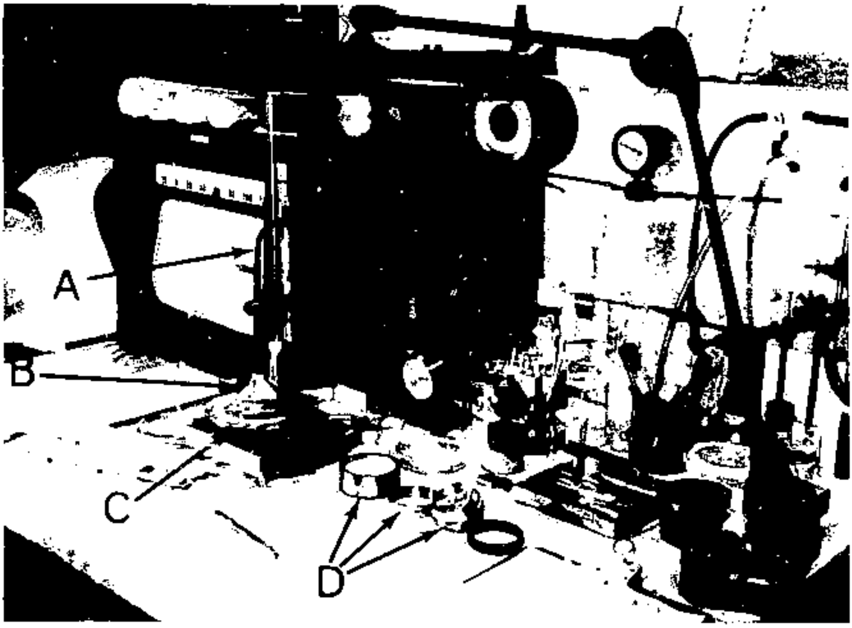

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FIGURE 8.—Assembled film permeability apparatus: A, Gas supply line; B, demountable gas-inlet cone, containing diffuser plate, ring gasket, distributor paper disk, film sample, and thermocouple; C, capillary measurement tube, leveled support, and plastic syringe; D, film sample cutter.

1,2,3-propanetriol with one-half percent concentrated copper sulfate solution, some Janus Green B, and Saffron-O. F-values for this indicator were substituted for those supplied by the manufacturer for distilled water.

The temperatures at which permeation through the plastic samples took place were obtained by placing a thermocouple inside the instrument cone just above the film sample. Shielded temperature sensors, placed adjacent to the capillary entrance, further checked the approximate gas temperature after permeation of the sample.

Whatman No. 1 filter paper was used in all permeability tests to provide equal diffusion of the gas over the surface of the film sample. These disks were cut with a circular die and block similar to that shown in figure 8. The thickness of the papers was obtained as a mean of 420 measurements on 140 disks. This mean, to nearest 0.0001 inch, converted to 0.0147±0.0005 cm. The diffusible area per disk was computed to be 13.48 cm.[2] These values were used in the needed calculations and test formulas.

Test samples

Circular disks, 50.04 mm. in diameter, were die punched from the 8- × 11-inch rectangles cut from each test liner. (See fig. 8.) The samples were kept at the storage temperature of the tests in desiccators. Ninety percent relative humidity in each desiccator was maintained by distilled water containing 33 percent commercial glycerine and 1 percent copper sulfate solution. The lids were sealed with vaseline.

Film thickness of the film types was measured to the nearest 0.0001 inch for calculations of the permeability values. These measurements did not always agree with those established by the manufacturers for commercial identification purposes.

Permeability measurements

Before each permeability test, all measurement instruments were compared with a checklist of standards for the test temperature and adjusted if necessary. The gas manifold was exhausted and then filled to the test pressure with the test gas. The rest of the system was purged with the gas, and thereafter the testing procedure was essentially that suggested by the permeability instrument manufacturer. The gas supply was maintained at a constant pressure. All tests were made by the same operator. At least duplicate measurements were made on each of the 135 film liners at 20°, 10°, 5°, and 0° C. Only 609 of these were used in calculations. The others were test runs.

The upstream gas pressures were varied, as test runs of the various types indicated would be needed, from 20 to 75 pounds' gage pressure. These pressures were converted into millimeters of mercury pressure at densities corresponding to the test temperatures. In practice, it was found unnecessary to adjust these gage values for local barometric pressures.

The formula used to convert the test observations into milliliters of gas permeated per square meter of diffusible film-liner area, in 24 hours at 760 mm. of mercury pressure differential, at the test temperature, was

$$\frac{2.89\times10^{8}\times C\times L\times P_{b}}{K\times t\times P_{g}} \qquad [3]$$

where:

2.89×10^{8} = a constant derived from the product of the standard temperature in ° K, the minutes in 24 hours, and the ratio of the diffusible area of the sample to a square meter of liner.

C = capillary tube calibration in standard milliliters of test gas per scale unit.

L = the number of whole scale units traversed by the indicator bubble during the test time.

P_b = barometric pressure at the instrument level, corrected for instrument errors and the density of mercury to 0° C., minus any effective vapor pressure of the indicator, in millimeters.

K = test temperature of the film surface in ° K.

t = test duration to nearest 0.01 minute.

P_g = gage pressure of test gas in millimeters of mercury at 0° C. density.

In practice this formula was further simplified where several test conditions were identical.

Auxiliary equipment variables

During this study, it was noted that insufficient temperature control was the largest single source of variability since it directly or indirectly affected almost every other variable. Blowers caused cool spots unless properly baffled. Heavy steel cylinders used to store the gases resisted temperature changes necessary for subsequent tests, and time allowances were required so that the enclosed gases would be at the test temperatures desired. Vacuum equipment was less efficient at lower temperatures and took longer to produce vacuums readily obtained at higher temperatures. Gages calibrated at one temperature did not equally respond at a different temperature; and they required extra care to obtain true readings at the colder temperatures. Mechanical timing devices were found more variable at the colder test temperatures than were synchronous motor-driven types. Gas leaks were more frequent at the colder temperatures. Extra care was needed to remove moisture from the test gases that were used at 0° to 10° C. Questionable permeability readings frequently were traced to the presence of tiny ice crystals at the surface of the test film or along the capillary of the permeability instrument.

Barometry variables

The problems associated with the barometry of our studies were extensive. Fortunately, practically all were fully identified and excellently treated in two publications (*15, 52*). Among problems not so readily solved were those of frequent moisture particulates and frequent jarring caused by the opening and closing of doors of adjacent storage rooms.

Indicator variables

The 1,2,3-propanetriol solution was the best general gas-transmission indicator among the 52 compounds considered and tested. Both the base and water in this solution were chemically pure; thus, the mole fraction of the water content was presumed to be the source of most of the vapor pressure. The vapor pressure of the base was about 3.0×10^{-5} mm./0° C. When a mole fraction of 0.9582 was used, the derived vapor pressures for the indicator at 20°, 15°, 10°, 5°, and 0°, were, respectively, 16.80, 12.25, 8.824, 6.270, and 4.388. These values were sufficiently correct for use in the permeability formulas. Pure 1,2,3-propanetriol solidified at approximately 0°.

Since carbon dioxide readily dissolves in water, the indicator slugs in the capillary were kept short in length. Failure to recognize this was responsible for some variation in early results. These variations were reduced by vacuumizing the test solutions, followed by saturating with the test gas immediately before each use of the indicator. Each test temperature required this procedure.

The rapid closing of heavy doors in the test building frequently caused the slug movement to cease momentarily and then to jump forward. When these tests were repeated, the total slug movement for an equal time period was never the same.

Some early variations were caused by fragments of mycelium in the indicators used at 20° C. These inclusions were responsible for some irregularity in slug movements. The copper sulfate additive lessened occurrences.

Capillary-tube variables

A microscopic crack at the epoxy-cemented joint of the capillary caused variable results before it was discovered. A slight vertical warp along one part of this tube gave unit/time ratios at this part that were not equal to those above it. Short-length measurements were not proportional to those for the full lengh of the capillary. This suggested the use of a specific length of the capillary for all subsequent tests. Several substitute tubes also were found to have variable bores. A final selection required calibration for each increment of the scale rather than a division of the total usable volume into equal units. All tubes were recalibrated for each test temperature.

Variations in results were lessened when successive indicator slugs were released to the capillary before the previous slug reached the full length of the tube. Timing was started when the second slug rate became uniform. This usually required about two scale units. Treatment of the end of the capillary with Desicote [23] aided abrupt discharge of the slugs.

These methods reduced capillary-tube variations from a coefficient of 17.4 percent to 1.9 percent. The capillary tube constant should be as accurate as possible since the results are converted to a square-meter basis for apple-box liners.

Film-sample variables

It appears probable, from the summary of results of the permeability tests, that some of the values may not have been obtained at final solubility of the test gas in the film sample. Efforts were made to use only those values that could be reasonably repeated. However, a carefully obtained initial value sometimes would shift to other values if the sample film were continually subjected to the test gas. Variations in permeability in a few samples continued to occur for more than 10 hours with all test conditions held constant, yet no defects could be found in the samples or in the test equipment or conditions. Adequate preconditioning of the film samples with the test gas seemed to be the only remedy of any help on this problem.

[23] Fisher Scientific Company.

Most permeability instruments in some manner hold the test film sample rigidly between two chambers of the test cell. Chamber halves put together by twisting one into the other may wrinkle, stress, or damage film samples more easily than those held together by adequate, even clamping. (See also (*19, 118*).)

Sealing of the film sample to the diffusion chamber shoulder became more of a variable at lower test temperatures. Many of the greases tried either had vapor pressures of their own or became too viscous for a thoroughly reliable seal. Some caused gradual deterioration of the O-ring gaskets, a variable difficult to locate. Colorless fresh vaseline was the best grease tried.

Determination of the true diffusion surface of the film sample can be the source of a variable. It is usual to assume that gas transmission of the film sample occurs only through an area equal to one side of the filter-paper diffusion disk. It may occur also through the edge of the filter disk and through that area of the film sample that is beyond the filter-paper edge yet inside the compression point of the O-ring. The larger of these two areas could mean an error of 4.3 percent; the edge area could add another 1.4 percent. Large drawings scaled from microscopic measurements indicated the edge is involved but the other area probably is not if (1) the test gas is maintained at a gage pressure, (2) the filter disk permeability is considerably greater than that of the thinnest film of high permeability, and (3) the shoulder supporting the film is free of scratches or imperfections leading to the diffusion grid. A more favored construction would be that this shoulder area be reduced to a minimum that will effect a reliable seal.

The thickness of the film sample was not built into formula 3, page 49, because this variable is the easiest maneuvered by the manufacturer to approximate a specification. For testing whether a film sample meets a specification, however, it becomes necessary to know this measurement. Manufacturers usually will be able to state the mean thickness of a film, plus or minus 0.1 or 0.2 times the mean for a certain percent sample, based on so many hundreds of sample readings. Thickness usually is expressed to the nearest quarter mil. This measurement may not be precise enough for permeability purposes for fresh produce. Also the manufacturers and consumers or designers may measure film thickness differently and with different results.

The ASTM (*7*) suggests that film thickness measurements be made with a micrometer and reported as the average of five determinations, made uniformly throughout the specimen, with the results recorded to the nearest 0.0001 inch (1/10 mil). Major and Kammermeyer (*72*) suggested an improvement of the ASTM method in which nine thicknesses of a film are measured at one time and at a standardized point. For the current study, the least variation among methods of measurement was found when the disk film samples were folded diameterwise once and radialwise twice to produce eight equal sectors per disk. The mean of the measurements at three places over each folded sector divided by 8 gave the mean thickness of the sample. Such measurements were made on a total of 1,907 samples of five film types.

An analysis of variance was made to estimate whether there was a difference between measurements made at eight different points on disks without folding and disks folded to equal eight thicknesses. At a critical F-value of 4.54 for 19 to 1 odds, the calculated F of 0.84 indicated the agreement between the averages was acceptable; and thus, on the basis of equal samples of 16 means, the sector method probably gives equal accuracy and less variation than the single-thickness method of measurement.

The sector method of measurement was used for thickness determinations of the five film types (in table 16). Coefficients of variation between tests of different samples of the same film ranged from 1.8 to 26.2 percent. Four of the five film types averaged within 0.1 to 4.1 percent of the thickness stated by the manufacturers. About 75 percent of these slightly exceeded the stated thickness. The average thickness variation for the fifth film type was 15 percent. Thickness variations may in part be due to measurement methods, but they also may in part be due to manufacturing controls that heretofore have been believed to be adequate.

Among desirable film qualities submitted by a film committee of the Society of Plastics Industry, in 1957, were standards for gage tolerances for general-purpose film (*94*). These tolerances were ±25 percent for 1.0 to 1.5 mils, or ±20 percent for 1.5 to 6 mils if film is less than 36 inches wide. For film over 36 inches wide, the tolerances were ±25 percent for 1.5 to 2.0 mils. It is now probable these latter tolerances are unsatisfactory for films intended to produce specified atmospheres in produce packages.

There has been some question whether package atmospheres reach equilibrium faster in thicker films or in the thinner ones. Tomkins (*123*) found the thinner ones produced a faster equilibrium in trays of apples covered with 150-gage polyethylene. Graphs by Salunkhe and Dhaliwal (*105*) showed that fruits took almost twice as long to reach equilibrium in a 2-mil film as in a 1.5-mil film. This variable was not further evaluated in the present study. The formula proposed on page 6 can be adjusted for

TABLE 16.—*Measurements of film thicknesses by the sector method*

Film type[1]	Mean thickness		Number of measurements
	Mils[2]	*Cm.* (10^{-4})	
Polypropylene	1.15±0.30	2.92±0.76	442
Polyethylene (pigmented)	1.53± .03	3.88± .07	324
Rubber hydrochloride	.78± .02	1.98± .05	459
Polyvinylchloride	.75± .03	1.90± .07	324
Polyethylene (clear)	1.51± .05	3.84± .14	358

[1] See p. 3 for manufacturers' estimated thicknesses for the samples supplied.

[2] Thickness in mils is more realistic for thin films, but many data are quoted in the c.g.s. system of column 3.

any effect of this variable in correlations of package atmospheres to permeabilities of package films.

Film-density variables

The relationship of the density of packaging films to their gaseous permeability was basically delineated by the work of Barrer (*11*) and van Amerongen (*8*). However, a workable correlation suitable for all film types, at all temperatures, seems not to have been satisfactorily resolved. This may in part be due to interactions of the polymer melting points, the degree of crystallization and resulting amorphous regions of the resins, the branching and cross-linking of the molecules, and the diffusion temperatures. The practical effects of density on the permeability of films was pointed out by several researchers (*62, 83, 115*) in 1957–58. Hardenburg and Anderson (*48*), in 1959, in apples packed in films of various densities, found that the film-liner permeabilities apparently varied inversely with the densities. (See also *24, 79, 80.*)

A preliminary study of some of the films used by Hardenburg and Anderson (*48*) and of the limited data on a few other films indicated that the oxygen permeability of such films may have approximated the hyperbolic cosine of the density at 0° C., yet only the log of the permeability equaled the density at 23°. These formulas gave the respective correlation coefficients of 0.95 and 0.98 among the 16 regressions tested. On the other hand, the carbon dioxide permeability of these same films also was approximately equal to the hyperbolic cosine of the density at 0°, but at 23° the permeability was about equal the log of the density. A study of some similar films of another manufacturer indicated that the log of the carbon dioxide permeability of these films gave a linear regression with the log of the densities. These different results seem to indicate that still other variables may be involved in such attempted correlations of densities and permeabilities.

A possible source of some variability may be due to misinterpretation of density classifications and their limits. The polyethylene of the early 1940's had densities ranging from about 0.910 to 0.925. About 1955, a process made possible densities of 0.940 to 0.965; and the following year, an intermediate density of 0.935 was developed. The American Society for Testing Materials (*7*) thereupon proposed that films having densities of 0.910 to 0.925 should be designated as type I;[26] those of 0.926 to 0.940, type II; and those of 0.941 to 0.965, type III. See (*62*).

As a part of this study, graphs were drawn for the carbon dioxide and oxygen permeabilities of data in papers that gave also the densities of the films. These graphs, obtained principally for a study of the effects of temperature on film permeability, showed clearly a very wide range of permeabilities due to the

[26] Often referred to as "low density," the only density of principal usefulness for nonperforated packaging of fruits and vegetables.

effect of density within type I films. It is insufficient for the purposes of packaging fresh fruits and vegetables to assume that any type I film may be substituted for any other type I film to produce a specific modified atmosphere in a sealed package. These data clearly indicate that the density of a proposed film should be precisely known for reliable results.

Methods for the measurement of polymer densities have been available for several years.[27] However, O'Connell (*86*) has pointed out that the average density reading on a given resin is higher today than it was years ago, because then there was no universal method for density measurements. He also observed that a polyethylene sample prepared by slow cooling may show a density of 0.927 but that the sample may have a density of 0.923 without special conditioning. This is enough of a change to shift a film from a type II to a type I. The resin density may be further varied by the processing technique. ASTM (*7*) methods D792–60T and D1505–60T have done much to improve accuracy and uniformity in resin and film specifications; surely density measurements will be less variable in the future.

The density of packaging films is of concern in the mechanical measurement of permeabilities. Karel and coworkers (*61*) reported that as much as 50 hours were required to obtain steady-state gas flows through 2-mil Mylar film. He cautioned that shorter times may result in errors of measurement of as much as 50 percent. Denser films usually will require higher gas pressures on the upstream side to produce measurable permeabilities within economical test times. However, films gradually may develop stresses or small tears and punctures if the pressure is too high. Often these errors are difficult to detect without benefit of sequential analysis techniques. (See also (*67*).)

Diffusion-disk variables

The Whatman No. 1 filter paper used in the permeability apparatus allowed ready diffusion of the test gases and had a highly uniform texture. Any paper chosen should have a smooth hard surface, have a dry strength at least equal Whatman No. 1, be thin and highly porous, be of high uniformity, and have general availability. A thick or rough paper will change the diffusible area of the film. A paper too thin or weak will be pressed into the diffusion grid and thus reduce the diffusion area of the film. The paper should be highly porous relative to the most permeable film with which it is to be used; otherwise, the permeability of the paper and film is measured rather than that of the film alone. Even limited tests seemed to confirm these variables.

Successive tests of a film sample require at least one re-position of the filter-paper diffusion disk. This method gave less variability than either an original disk not re-positioned or a substitute disk. A re-position of the film sample did not account for

[27] Especially see (*7*, *82*, *124*) for methods and bibliographies of techniques.

the total variability; but a re-position of both filter disk and film did approximate it.

O-ring variables

The rubber ring that seals apart the upstream and downstream chambers of the permeability cell required replacement many times during the 0° to 5° C. tests. At these temperatures the rings were less elastic and leakage occurred. It was essential that these rings be without mold marks and entirely free from microscopic pits, or surface fissures. A tetrafluoroethylene ring of a suitable size might make a less variable gasket.

Considerable variability occurred when the cell halves holding the test film evidently had been screwed together tightly enough to distort the film and present a greater diffusion surface. Hence, the lightest tight-fit was found by trial, for each sample, each time the gas pressure or the test temperature was changed. A small amount of vaseline on the upper and lower crowns of the ring was needed to tighten the gasket sufficiently and yet not tear or wrinkle the thin film samples. A vapor error would have been small at the temperatures vaseline was used. With flawless O-rings and smoothly machined ring grooves, no variable traceable to compression of the ring was found, so long as some compression was present.

Purging variables

It remains a problem how much purging of equipment is necessary to insure that a diluent gas is not present and measured concurrently with the test gas. In this respect, measurements by a thermal conductivity cell would have advantages Best results were obtained when the equipment upstream from the cell was vacuumized before introduction of the test gas. The cell then was purged at low pressure during its assembly with the film in place. The upstream gas valve was closed momentarily for the final quarter turn of the cell assembly. This procedure lessened the variability due to dilutions.

The voids in the filter-paper disks were computed to be equal to 2.1 scale units. Some of the film samples doubtlessly contained a mixture of dissolved gases. Our equipment did not reliably precondition the film sample and filter disks. Acceptable variability occasionally required as many as eight complete 15-unit purges at permeation pressures. A considered solution was to vacuumize the entire test areas, place an indicator slug in the capillary, and then admit the test gas. Applications of the method would not be simple. A battery of permeability instruments held at permeation pressures for several hours would be a better method.

It was found important to cleanse the capillary at each closedown period and each time a different test gas was used. The capillary was flushed several times with freshly boiled distilled water, followed by three flushings with a mild detergent compatible with the tube material. This was followed by three flushings with distilled water and then drying with the test gas. Sample tubes were tested for variability by immersion in the detergent for 1 week, with calibrations before and after immersions.

Conversion variables

Since permeability standards have not been universally accepted, work has been reported in various systems of measurements. The conversion of these sometimes is involved. It has caused results to be variably interpreted. As the metric system may be universally adopted for its ease of conversions, the system suggested by ASTM *(7)* D 1434–58 has been followed in this bulletin. It is of advantage to the industry as a whole to use an identical standard in reporting results.

Tables of film permeabilities have been variably interpreted when tabular values have been headed by some factor such as "$\times 10^{9}$" or "10^{6}." When only thin films are concerned, such headings should be used only to mean that the tabular figures shown have been multiplied by the "power of 10" shown in the heading. For instance, the value "2.05" in a table headed by 10^{9} is "2.05×10^{9}." and would be so shown were sufficient tabular space available.

Test-temperature variables

For the mechanical measurement of the permeabilities of films, it is generally presumed that the ambient temperature is also the temperature of the gas permeating through the film. This is a possible variable. The test gas may not be at the ambient temperature of the instrument nor at the temperature inside the instrument. The true temperature of the gas may vary according to its pressure and whether the gas supply at the same pressure has been allowed to come to equilibrium with the temperature of the surfaces over or through which it passes. It was found in these tests that this difference could be between 0.7° and 2.3° F. This difference is small, but results in permeability are logarithmic to temperatures and temperature differences.

Recorded permeabilities often lack sufficient temperature specifications for the data to be of real value to prospective users. Common difficulties are: (1) the temperature at which the data were obtained is omitted; or (2) the data were obtained at a temperature not used in the commercial storage of produce, and no guidance is given for interpolations of the data to other temperatures. Permeability rates are meaningless without unmistakable temperature data, and rates preferably should be given for at least two temperature points.

When two rates are given, the user then may establish loci on a nomograph to estimate equivalent permeabilty rates at other desired temperatures *(90)*. Also, equivalent permeability rates (P) may be obtained by interpolation from a semilog chart of given rates plotted against reciprocals of corresponding absolute temperatures. Many workers have used this relation, varied as:

$$\text{Log } P = 1000/(273 + {}^\circ \text{ C.}) \qquad [4]$$

The results agree well with those obtained by plotting logarithms of permeability rates against centigrade temperatures equivalent to logs of the vapor pressures of ethyl alcohol. This method, too, is reliable, but it requires drafting special reference lines. (See also (*90*).)

If a permeability rate is available for only a single temperature, the data still may be extrapolated to other temperatures by an Arrhenius-type equation into ml./sec./cm.^{2}/mm./cm. Hg.-units.

$$\text{Log } P = (\log P_o) - [E_p(10^3)/4.58(273^\circ + {}^\circ\text{C.})] \qquad [5]$$

where P_o and E_p are constants specific for each type and density of film, for each permeated gas of interest (*6, 8, 11, 55, 66, 90, 97, 115*). These constants should be available from film manufacturers. They should be specific and not adapted from other data.

There is little doubt from these data that both carbon dioxide and oxygen permeabilities of films probably form linear regression trends on temperature. However, plots of the permeabilities of polyethylene to carbon dioxide from these formulas and data from 15 sources in the literature, indicate that the regressions are not always parallel for the same densities, resin types, or brands of film. Reasons for this variability are unknown. More often, similar data for oxygen permeabilities did appear to be parallel. Similar data for other kinds of films are too few to warrant conclusions. In tentative summary, a safer procedure would be to use such formulas to predict variations of permeability by temperature only when the formulas have shown agreement with experimental data for the particular density, type, and kind of film for which interpolations are needed. Extrapolations, always questionable, should be given consideration only with due caution.

Landrock and Proctor (*64*) cautioned that permeability rate units should be corrected to their values at the standard temperature and pressure (STP) of 0° C. and 760 mm. pressure; but there should be no possible confusion to the reader that the permeability measurements were made at 0° C., if this was not the case. Any application to data by the formula

$$\text{cc's} \times \left[\frac{273}{(273 + {}^\circ\text{C.})} \times \frac{(\text{local barometric pressure})}{760} \right] \qquad [6]$$

only adjusts the gas-volume cubic centimeter to a standard size. Many present-day writers indicate that this adjustment of their data has been made by using as a subscript to their units of measurement the letters "STP." This practice supplemented at the end of the specification by the information "@ _ _ _ °C.," the test temperature, should eliminate confusion.

Unassessed variables

Although many variables that may affect film-liner permeability values have been reported on in this section, there were others that should be mentioned. Among these are the possible effects of mixed gases on the permeabilities of films and prolonged permeations.

On two occasions, single-film samples were noted to have apparently different permeability rates at night and during the following day. The permeability rate of one of these samples increased 65.4 percent overnight from the previous day rate that had held steady for 18 hours. The permeability rate of the other sample increased 24 percent over the previous-day steady state and then dropped to only a 3.4 percent greater rate during the following day. A very thorough check of the instruments and recorders showed no apparent change in any of the recorded conditions or controls. Barometric pressures were not recorded automatically nor continuously. There was no knowledge whether these film samples may have contained any absorbed volatiles.

There is a possibility that the permeability of films may be varied, especially in hydrophilic plastic laminates, when the plastic becomes wet by respiration water during storage. Oswin (*89*) noted a 5-percent linear expansion in wet regenerated cellulose film and recorded that Morton, in 1935, estimated that pore sizes of 5 Angstroms in dry film may become four times as large when the film becomes wet. The relative humidity in apple liners is high throughout the storage period.

ASTM (*7*) designation D 1434–58 for testing the gas transmission rates of plastic sheeting prescribes that the steady state of gas transmission is to be considered achieved when the loss of water or volatile additives from the specimen are negligible. It is reasonable that as gases are removed from the film their effects on measured rates also would be removed and net transmission rates would decrease. Although this may occur during the usual mechanical test of permeabilities, any such result usually is obscured by a net rate increase until the steady state is apparent. This leaves some doubt whether either moisture or volatiles are entirely removed from the specimen films during a test. There is reasonable doubt also whether such losses occur from films enclosing produce, where concentration gradients would be much less than under mechanical test conditions. Either combination of these events thus would appear to present an unassessed variable between mechanically measured permeabilities and those obtained by enclosed produce.

Meyer and coworkers (*78*) studied the variability of polyethylene permeability to oxygen in the presence of carbon dioxide, at test temperatures between 0° and 60° C. It thus seemed not urgent to study the effects of the presence of mixed gases on the permeability of the five test films. To have included such studies would have required equipment more elaborate than probably is used commercially.

Measured versus estimated permeabilities

It was not questioned at the start of this study that differences in permeabilities among various films would be present, but the magnitude of variable interactions became apparent only as the study progressed. It was fortunate that the possible presence of at least some of these had been postulated and provisions made for their study. A major objective of the study was that the mechanical measured permeabilities of the various films should be compared with those biologically obtained and translated, by the RSAV△ method (*119*), into forecasts, or estimates, for film specifications. A summary of these results is presented in table 17.

The RSAV△ method remained a basic practical tool for estimating the permeabilities that might be developed in the various films, under the varied packaging conditions. Actual values, rather than estimated values, were substituted into the formula where these could be determined. The *R* values were computed from actual case weights and from probable respiration rates at the storage temperatures inside the liners. The *S* values were based on the liner measurements computed by formulas presented in detail in the next section. The calculations of the *V* and △ factors were performed as originally proposed in (*119*). A major change (as explained in following paragraphs) in the *A*-value calculation permitted both halves of this factor to provide for oxygen and carbon dioxide interactions.

As stated (*119*), the package-atmosphere constants *A*, in the RSAV△ formula, were estimated from limited available data. The present study allowed a greatly expanded test of these constants at several temperatures, for three varieties of apples from three or more sources, and packaged in five different films. These data indicated at odds considerably greater than 99 to 1 that the atmospheres in these packages were probably modified by an interaction of the carbon dioxide and oxygen present in the packages. The effect of the carbon dioxide may have been less than that of the oxygen, at the atmospheres obtained in the test packages. It was found that a slight adjustment of the *A* factors for oxygen, plus use of the original values for *A* for carbon dioxide, would approximate an interaction factor for either gas when the two halves of the *A* factor were multiplied together. (See also (*5*).)

The oxygen constants (*119, table 3, p. 9*) thus may be revised to read consecutively, beginning with 3 percent, 0.411, then 0.453, 0.500, 0.552, 0.610, 0.673, 0.743, 0.820, 0.906, and at 21 percent, 1.000. The carbon dioxide constants remain as given. The regression formulas become:

(a) for carbon dioxide: $\text{Log}\ (10Y_c) = 0.99720 - 0.01193X_c$ [7]

and (b) for oxygen: $\text{Log}(10Y_o) = 0.54899 + 0.02147X_o$ [8]

In each of these regressions, Y equals the constant-half for that gas, as a decimal; and X equals the percentage of the gas, as a whole number. Data were not available on which to base any ex-

TABLE 17.—*Estimated and measured permeabilities of film liners to carbon dioxide and to oxygen*

Type of closure and film [1]	Apple variety [2]	Atmosphere [3]		Permeability [4]					
				Carbon dioxide			Oxygen		
		Carbon dioxide	Oxygen	Estimated [5]	Measured [6]	Mean difference [7]	Estimated [5]	Measured [6]	Mean difference [7]
Single-tie:		*Percent*	*Percent*			*Percent*			*Percent*
Polypropylene	(1)	6.6	12.4	7,170	2,180		5,200	850	
	(2)	6.8	12.2	7,050	2,100	227.1	5,060	830	494.4
	(3)	7.3	11.3	6,650	2,100		4,660	830	
Polyvinylchloride	(1)	4.8	14.5	7,630	3,480		5,280	360	
	(2)	5.0	14.1	7,470	3,400	115.6	5,000	340	1,312.5
	(3)	5.3	13.2	7,060	3,400		4,410	340	
Mean for total						158.3			734.1
Double-tie:									
Polyethylene (clear)	(1)	4.8	8.2	10,940	10,710		3,500	3,430	
	(2)	4.8	8.1	10,910	10,480	2.1	3,500	3,350	3.5
	(3)	5.2	7.5	10,490	10,480		3,480	3,350	
Polyethylene (pigmented)	(1)	4.5	5.4	10,470	10,240		2,520	2,460	
	(2)	4.6	5.2	10,400	10,020	2.3	2,540	2,380	6.2
	(3)	5.0	4.9	10,110	10,020		2,610	2,380	
Rubber hydrochloride	(1)	2.1	7.9	14,160	13,980		1,810	1,780	
	(2)	2.1	7.8	14,030	13,600	1.3	1,810	1,720	4.0
	(3)	2.3	7.2	13,540	13,600		1,810	1,720	
Mean for total						1.9			4.5

[1] See table 1, p. 3, for characteristics of film liners.
[2] (1), Delicious; (2), Golden Delicious; (3), Stayman.
[3] Time-weighted mean atmospheres in liners during tests, at 32°–33° F.
[4] $ml._{(STP)}$ (mil/24 hrs./m.^{2}/760 mm. Hg./33.4° F. for all Delicious and 32.6° for all Golden Delicious and Stayman.
[5] Estimated by adjusted RSVA△formulas. See text for details. Figures rounded to nearest 10.
[6] Mechanically measured under controlled conditions and interpolated to storage temperatures of test corps. Figures rounded to nearest 10.
[7] Differences computed as: $100\,\frac{(\text{estimated} - \text{measured})}{\text{measured}}$.

tension of the table below 3 percent oxygen. It appeared from the results in table 17, for the double-tie data, that these constants gave reasonable correlations in the test data of this study.

The design of the RSAV△ formula was intended to include adequate safety factors between film specifications and atmospheres actually produced within packages of produce. It was intended that a film having a permeability anywhere between rather wide limits might be used to produce a stated atmosphere within narrow limits. Experimental results have now indicated that this is basically probable. This practicality requires that measurements, conditions, and controls must be determined and maintained as precisely as equipment permits. Whenever dependent specifications are formulated, it therefore is suggested that means plus standard deviations should be determined for best results.

Ratios of gas permeabilities of film

Various workers have inferred that the permeability of a plastic film to a given gas bears a constant ratio to the permeability of that film to other gases and that this ratio is independent of temperature. The permeability measurements of this study were used to examine this theory further. More references have related to polyethylene ratios; thus the following results concern that film. Results with other film types were similar.

Based on 122 permeability measurements, ratios of carbon dioxide to oxygen permeabilities at 0°, 5°, 10°, 15°, and 20° C., respectively, were: 5.08, 4.63, 4.16, 3.79, and 3.45. The difference between the lowest and highest ratio was approximately 47 percent. To test whether such differences might be due to unusual sampling or methods, similar ratios were calculated from data for eight other film types given in private correspondence or published material. These data, with a single exception, showed the ratios increased as the temperatures decreased toward 0°. The increase varied—from as little as 31 percent to 112 percent—within the data and among techniques. These data suggest that ratios, constant for a specific film and a specific condition, may not forecast results at a different temperature, for different conditions, and for different film types.

These results also suggest that the permeability of one film type should be given as a ratio to the permeability of another film type only in accord with experimentally determined measurements over a temperature range. Technical brochures frequently have not been entirely clear on these points; thus, unintentional illusions have been given.

FILM-LINER DESIGN, DIMENSIONS, AND TOLERANCES

It was anticipated in the experimental design of these studies that film-liner dimensions might prove to be a major source of variation in film permeabilities. It would be pointless to specify a permeability rate for a packaging film if the fabricated liner should not fit the produce volume.

Therefore, liner variations were partitioned and formulas were sought to discount the effects of each through efficient liner design. It is believed the simple formulas presented in this section sufficiently correlate these important considerations. The effects of these design formulas are combined within the section on "Formula Adaptation."

Materials and methods

The five different liner designs used in these experiments were described in the first section. These were offered by different designers for a single size of container. An examination of these five designs showed considerable variation in the dimensions of material available for tie-seals, in seam widths, in the widths and lengths of the liners available as diffusional areas, in the presence or absence of gussets and their depths, and in the material (tubing or flat sheets of plastic) from which the liners were made. It seemed obvious that in some designs some material was needless and in others the liner could have been better proportioned to fit the needs of the produce to be enclosed.

For analysis of these differences, three or four liners of each type in turn were fitted tightly to a clothbound metal box made to fit precisely inside the fiberboard apple containers used in the storage tests. All creases and folds were outlined and the areas determined, coded, and sectioned so that they might be put together in different combinations on a second sheet of plastic that was to enclose the same metal container. From these data, formulas were devised to describe the areas believed to have a part in permeability considerations.

Most of these areas were mathematically simple, and the deviations of their means were very low. Considerably greater variance occurred in the liner areas of the tied-top closures. This difficulty was earlier anticipated; hence the "tie-off" [28] of the original liners had been removed (p. 31). These tops, used in the india-ink tests, were opened flat and their individual outlines were traced on superimposed sheets of thin paper. Comparisons of these tracings, a few at a time over a bank of shadowbox lights, afforded a trial-mean shape suggesting an hyperbola relation. A formula for this area was derived, simplified, and tested against a mean integrated tracing of the original tops. This hypothesized area proved to be statistically satisfactory.

The rest of the problem concerned derivation of general formulas to describe the principal dimensions of liners for any plane-surface container. The dimensions selected as having present or future importance were: length, width, one-side surface area of the liner, total permeable area, liner seams, area of liner above

[28] The "tie-off" is considered that area gathered and tied to close the liner.

produce, permeable part of the liner top, and the nonpermeable part of the top.

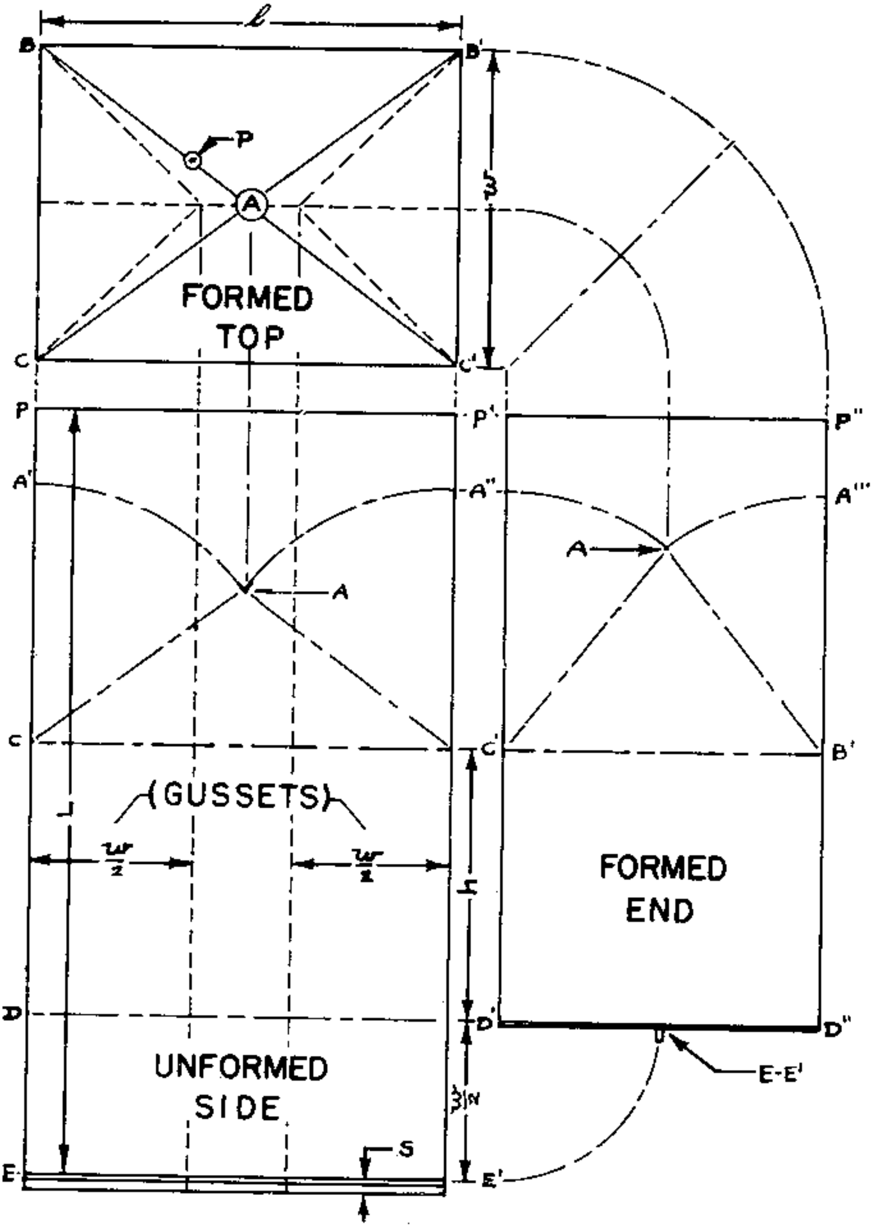

FIGURE 9.—Proportionate dimensions of gusset liners for boxes: AP, 3 inches (minimum); CP, (CB/2) + AP; all other dimensions depend on the height, width, and length of box required.

Liner-design formulas

The following developed formulas are believed applicable to liners for any plane-surface container but not to liners for basket containers or containers of other shapes. The factors have been simplified to those of box height (h), width (w), length (l), and a selected seam allowance (S). The formulas require dimensions to be expressed in inches to two decimal places. Where trade usage of a result requires or suggests any conversion to metric dimensions, this can easiest be done in the final answer. All symbols have the same meaning in each formula.

Whether the liners are made from tubing or flat stock and with or without gussets, affects only the location of the seams. These designs take approximately the same amount of material, and the same basic formulas can be used. Since gusset liners fit containers more neatly than liners without gussets, the gusset design is illustrated (fig. 9). For a neat fit, the gussets should be positioned as shown and each made as deep as one-half the width of the box. The formulas allow for a double-twist seal closure of 3 inches. (See "Seam Leaks" and "Twist-Seal Leaks.")

Length requirements

The double-thickness length of liners, except for seams, is given by

$$\mathrm{L} = \frac{(2h + w + \sqrt{w^2 + l^2} + 6)}{2} \qquad [9]$$

The total length of material required for fabrication of the liner can be calculated by formula [9] plus the seam allowance (S) explained in "Liner Seams," p. 66. The answer is in inches.

Width requirements

The width refers to the width of tube stock or to the width of double-thickness flat stock needed to form a liner either with or without gussets. It is given by

$$\mathrm{W} = w + l \qquad [10]$$

Care should be observed that dimension (W) is not confused with the italic letter (w) used for box widths in the various formulas. When side seams are used in the liner design, seam allowances must be added to formula [10]. Answer is in inches.

Surface area

The surface area for the use intended here includes both permeable and nonpermeable parts of the liner, but it does not include any seams. Formulas [9] and [10] are combined to give the surface area, thus:

$$\mathrm{A}_t = \frac{2\mathrm{WL}}{1{,}550} \qquad [11]$$

This formula was basic to the derivation of formula [12] and is useful to test the derived results in any applications. The usefulness of the formula in permeability calculations is increased by an end-result, as expressed, in square meters. In order to use formula [11] in figuring liner costs, the seam areas must be added to the numerator. The answer is in square meters.

Total permeable area of liner

The total permeable area is the important part of the liner in permeability specifications. It is calculated by the formula

$$A_{tp} = \frac{2WL - [(\sqrt{w^2+l^2}+6)(w+l)] + wl + 0.7854(w^2+l^2)}{1{,}550} \qquad [12]$$

This area often is considered to be the same as the surface area of the liner. But there is real and important difference between the two. Calculations based on 23 accepted tariff specifications for bushel-size [29] apple boxes showed this area to be 11.8 to 12.8 percent smaller than the total area of the liner as given in formula [11]. Formula [12] describes the entire area of the liner through which enclosed produce must receive its supply of oxygen and lose its undesirable quantities of carbon dioxide and other volatiles. The answer will be in square meters—the more useful form for area data.

Formula [12] includes convolute parts of the liner both at the bottom of the box and at the top. These permeable parts, especially in gusset liners, may contain a variable number of folds of material and their areas will vary with the different box dimensions. It is believed that these folds do not require additional mathematical definition here or additional specification allowance. Folds do not measurably inhibit the flow of gas molecules, and gas pressures probably are the same within folds as in the rest of the liner volume.

With a selected seam allowance added to formulas [9], [10], and [11], the above formulas are sufficient for the fabrication of liners. Specific dimensional allowances for seams were omitted for reasons that follow.

Liner seams

A constant for seams was not given in the liner formulas because seam widths, lengths, and areas are: (a) simple measurements without involved factors; (b) dependent on required box dimensions that the user must select; and (c) dependent on certain choices of the designer or manufacturer of the liner. These choices include whether the liner is to be made of: (a) tube stock hermetically sealed at one end; (b) flat stock folded endwise and sealed along one side and end; (c) either flat or tube

[29] "A bushel of apples" is a standardized volume, but this term is commercially used to mean any container that is approximately a bushel. See *(119, p. 8)*.

stock with gussets (fig. 9, p. 64); and (d) whether the liner seam is sealed folded or flat. (See "Seam Leaks," p. 34.)

In any combination of these choices, the seam allowances (S) is simply the unfolded width of the seam (WS) to and including the seal (s) added to the width (W) or length (L) of the liner as the design requires. The seam width (WS) times the cumulative lengths of the seams, measured in inches and two decimals of an inch and divided by 1,550, gives the seam area in square meters to be added to formula [11]. Neither the seam area nor its design otherwise affects the formulas nor the total permeability of the liner.

Area of liner above produce

A formula for calculation of the area of the liner above the produce is given because fabrication improvements might be made in this area: (a) for considerable net savings for all concerned; and (b) for possible elimination of another variable in the production of specified atmospheres. This area extends above the produce to a height equal to one-half a top-diagonal of the box plus a constant:

$$A_{pnp}=\frac{(\sqrt{w^2+l^2}+6)\,(w+l)}{1{,}550} \qquad [13]$$

The area contains both permeable and nonpermeable sections, in square meters. The part of each that is present depends on the liner design chosen. The generalized formulas calculate this division.

Permeable part of liner top

For the usual tie-off design of liner the permeable part of the liner top is below the tie-off and separately calculated as:

$$A_p=\frac{wl+0.7854\,(w^2+l^2)}{1{,}550} \qquad [14]$$

The answer will be in square meters. The value of this generalized formula is included in formula [12]. It is equal to the area of a circle, having as its radius one-half a top-diagonal plus the length times the width of the box.

Nonpermeable part of liner top

The nonpermeable part of the liner top is easiest computed as the difference between equations [13] and [14], or as

$$A_{np}=\frac{(\sqrt{w^2+l^2}+6)\,(w+l)-wl-0.7854\,(w^2+l^2)}{1{,}550} \qquad [15]$$

The answer will be in square meters. This tie-off part of the liner is a trouble-source variable that accounts for more than 13 percent of the total liner area.

Tolerances

Dimensional tolerances are provided for in the formulas by choice of acceptable *w*, *h*, and *l* measurements. Freight tariffs and other references usually give only the inside measurements of shipping containers; but the use of these for liner dimensions, without tolerances, can slow packing lines and increase costs. The usual remedy is to make the liner "just a little larger," possibly as large as the outside dimensions of the container. The minimum magnitude of this variable was tested by making inside and outside measurements of 15 commercial fiberboard containers of the 113-size from three box manufacturers. It was found that, on the average, liners made on outside box dimensions would have 7.9 to 11.9 percent greater area than if the inside dimensions were used. Measurements of the outside dimensions of the bottom part of the boxes were then compared with the inside dimensions. The differences for these comparisons ranged from 2.8 to 4.4 percent.

It is believed that outside dimensions for the bottom part of boxes will ordinarily provide a sufficient tolerance and yet will provide an acceptable limit for this variable for permeability specification purposes. The use of fruit trays with rounded corners will provide an additional tolerance that may speed packing operations and cut packing costs.

The total effect of liner design in RSAV△ applications, based on the above formulas, has been combined into a single adjustment, which will be given in the next section.

PRACTICAL APPLICATION AND IMPORTANCE

Formula adaptation

Within the foregoing sections results of experiments, observations, analyses, and suggestions on the control of packaging variables were presented to aid in obtaining specific modified atmospheres in packages. These are believed to enhance the RSAV△ formula for required film permeabilities for storage of apples and perhaps other fresh produce.

The RSAV△ formula was presented by the writer in 1962 (*119*) as a practical tool for estimating required film permeabilities for apple storage. Where a certain film has been found through repeated experience to produce a beneficial modified atmosphere that is satisfactory under commercial conditions, it becomes academic to know the permeability of that film. It is important, however, to know that the film requested is the film received and to know how to describe the film. For new or different film-packaging proposals, factual values that have been determined certainly should be substituted in any adaptation of the RSAV△ formula. It is believed this bulletin has reasonably demonstrated what might be accomplished when such substitutions are possible.

This study has not demonstrated a need for any revision of the basic concepts for the RSAV△ formula; but it has emphasized that the factors of the formula need to be determined as accurately as possible and that they accurately describe the conditions they are presumed to represent. The following paragraphs direct specific attention to certain admonitions that concern applications of the formula.

R-factor

Estimated *R* values for 20 principal varieties of apples at five common storage temperatures have been presented (*119, table 1*). They were intended to be used where actual values were not available; and formulas were given for factual substitutions by the user. The two variables forming each *R* value are the mean net weight of the usual box of apples and the mean respiration rate of the apples under the stated circumstances. If the user reliably knows these two variables for his set of circumstances, the first value in pounds and the second in milligrams of carbon dioxide per kilogram per hour, he then needs only to multiply the product of these two figures by 5.5422 to derive his own *R* value. If the two variables are not known, current estimates of *R* may be obtained by multiplying values in (*119, table 1*) by 1.0317. Should the user need temperature adjustments, (*119, table 8*) provides means of finding these.[30]

S-factor

The use of the suggested dimensions for liners as developed in the preceding section will affect the box-liner surface constants, *S*, given in (*119, table 2*). The constants there given were theoretical estimates for custom-fit liners without folds, a fabrication discussed in this bulletin but yet to be accomplished in commercial practice. Until such liners are available, more acceptable *S* factors for liners with folds may be obtained by multiplying the first 15 constants in (*119, table 2*) by 0.6476. The last eight constants require an average multiplier of 0.6493. Coefficients of variation for these multipliers, respectively, were 4.2 and 3.4 percent.

A-factor

Formerly (*119*) *A* values were separately computed and used for each test gas. The present study showed that closer approximations to the interaction of carbon dioxide and oxygen upon permeabilities could be obtained by the use of a single *A* value for both gases—that value to be a product of constants newly derived by given formulas. This was found possible through only slight changes in the original values (*119, table 3*). (See p. 60.)

[30] The constant for 40° F. (*119, table 8*) should read 1.469; the footnote formula should read (0.02089× ° F.) − 0.66848.

V-factor

The present study demonstrated the validity of this factor. No change in computation appears to be necessary.

△-factor

The true value of this factor may need revision to mean local barometric variations. Variations at the test site of this study approximated the formula conditions, and thus the data were too meagre for any study of variance traceable to this source. The △ values obtained by the present formulas were satisfactory. See (*15, 52*).

Importance to plastics industry

The results of this study in no way intend to discredit or to favor any of the five types of films chosen for these experiments. These experimental films were chosen to illustrate and provide a basic study of variables. They did this job well. The film industry has greatly progressed in the last 30 years. Packagers and suppliers currently are investing nearly $1 billion a year to achieve better, more economical packaging (*3*). It is estimated that each dollar spent in basic research will ultimately mean $1,000 in new plants and equipment. In the future, packaging doubtless will include a far greater usage of improved plastic films.

In 1964, Brown (*17*) wrote: "A number of articles have been published which deal with permeability and shelf life, but few predictions with later confirmation have been made. The factors affecting shelf life include moisture, temperature, light, air, product/package interaction, microbiological contamination, instability of package ingredients, change of chemical systems, absorption of off-flavors from the packaging material, loss of volatile constituents and so on." His conclusions imply that biological results, rather than mechanical ones, should be the objective of packaging research. It indeed has been the objective of this current study.

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APPENDIX:

EXAMPLES OF FORMULAS APPLICATIONS

(1) A worker finds that an apple liner contained the following percentages of carbon dioxide (c): first day, 0.0; sixth day, 2.2; 12th day, 4.3; 18th day, 5.6; 24th day, 3.5; and 30th day, 3.6. What was the probable effective carbon dioxide component of the atmosphere during this period?

Solution: Use formula [1], page 6.

$$G_c = \frac{2(2.2+4.3+5.6+3.5)+0.0+3.6}{2(6)-2} = 3.5 \text{ percent} \qquad [16]$$

(2) The corresponding oxygen percentages in this liner were: 21.0, 16.6, 12.4, 9.2, 13.6, 13.4. The worker is concerned that the liner may have been punctured during his fourth inspection. Is this probable?

Solution: Apply formula [2], page 31, to the fourth, fifth, and sixth inspections.

$$\frac{21.0-9.2}{5.6} = 2.1 \qquad \frac{21.0-13.6}{3.5} = 2.1 \qquad \frac{21.0-13.4}{3.6} = 2.1 \qquad [17]$$

Based on Tomkins (*121*) observations, the 2.1 ratio is greater than 1.5 and thus the liner probably is intact. The cautious worker probably would further check this liner similarly for at least two-thirds of the inspections.

(3) A film sample was measured for its carbon dioxide permeability at 5° C. The test conditions (see p. 49) were: An estimated diffusible sample area of 13.6221cm.^{2}, for a constant of $(2.73\times10^2\times1.44\times10^3\times10^1)\div(1.36221\times10^1)=2.89\times10^8$; $C=0.0305$; $L=14.3$; $P_b=756$; $K=(273+5)$; $t=8.34$; and $P_g=(50\ \text{PSIG}\times760)\div14.695=2{,}586$. What was the permeability at the actual thickness of the sample? What would it be "per mil"?

Solution: Use formula [3], page 49,

$$\frac{2.89\times10^8\times3.05\times10^{-2}\times1.43\times10^1\times7.56\times10^2}{2.78\times10^2\times8.34\times2.586\times10^3}=1.5893\times10^4. \quad [18]$$

The permeability is 15,893 $ml._{(STP)}$/m.^{2}/24 hr./760 mm.△/5° C. for the sample thickness. Multiply this result by the actual thickness in mils to obtain the permeability "per mil."

(4) The determined oxygen permeabilities of a film at 5° and 20° C. are, respectively, 500 and 1,600 $ml._{(STP)}$/mil/m.^{2}/atmos./day. What would be a probable estimate of the permeability of this film at 7°?

Solution: Use the right-hand half of formula [4], page 58, to find abscissa values for 5°, 7°, and 20°.

$$5^\circ=\frac{1{,}000}{(273+5)}=3.60;\ 7^\circ=\frac{1{,}000}{(273+7)}=3.57;\ 20^\circ=\frac{1{,}000}{(273+20)}=3.41 \quad [19]$$

On a semilog chart, plot horizontally 3.60 and 3.41 against their corresponding permeabilities located on the vertical log scale, and connect the loci by a straight line. From the 3.57 intercept, read 590 ml. on the log scale. (Any extrapolations, especially for lower temperatures, may be invalid.)

(5) The only known oxygen permeability of a certain film is 7,900 ml./mil/m.^{2}/atmos./day, at 25° C. However, the manufacturer has supplied for this particular film a P_o constant of 0.031 and an activation-energy constant of 9.6 kilogram-calories. What would be the estimated permeability of this film at 0°?

Solution: Use formula [5], page 58, with $P_o=3.1\times10^{-2}$ and $E_p=9.6$.

$$\text{Log } P=\log(3.1\times10^{-2})-\left[\frac{9.6\times10^3}{4.58(273+0)}\right] \quad [20]$$

$$=(\log 3.1-2\log 10)-7.67791$$

$$=0.49136-2.0-7.67791=-9.18655,\text{ or } +0.81345-10$$

$$\therefore P=6.5080\times10^{-10}ml._{(STP)}/\text{sec.}/\text{cm.}^2/\text{mm.}/\text{cm.Hg, at } 0^\circ\text{ C.}$$

To convert this system of units to $ml._{(STP)}$/mil./m.^{2}/24 hr./760 mm. Hg.△, the above result is now multiplied by 258.51917×10^{10}. The calculated estimate is 1,682 ml.[31] (See cautions on p. 58.)

(6) What constant in a RSAV△ specification would one use for an approximate 4.5 percent oxygen?

[31] An estimated permeability of this film has been reported by others to be 1,797, a difference of 115 ml.

Solution: Use formula [8], page 60.

$$\text{Log } (10Y_o) = 0.54899 + 0.02147(4.5) = 0.64560 \quad [21]$$

$\therefore 10Y_o = 4.4218$, and $Y_{4.5} = 0.442$, the value of the constant.

(7) A liner is needed for an 1/8-inch fiber-stock apple container whose inside dimensions are 11 3/4 inches high, 12 inches wide, and 19 3/4 inches long. The corresponding outside dimensions of the bottom part of the box are 12 inches, 12 1/4 inches, and 20 inches. The liner is to be provided with adequate gussets, is to have a 3/4-inch seam, is to have a double-tie twist closure, and is to be made from tube stock. What should be the dimensions of the liner; and how much material will each liner require?

Solutions:

(a) The length, by formula [9], page 64, would be

$$L = \left[\frac{2(12) + 12.25 + \sqrt{12.25^2 + 20^2} + 6}{2}\right] + 0.75 = 33\tfrac{5}{8} \text{ inches.} \quad [22]$$

(b) The width of tube stock, by formula [10], would be

$$W = 12.25 + 20.0 = 32\tfrac{1}{4} \text{ inches.} \quad [23]$$

(c) The depth of the gussets, by figure 9, page 64, should be one-half the width of the box, or 6 1/8 inches each. [24]

(d) The single-sheet material per liner would be, by formula [11],

$$A_t = \frac{32.25 \times 2 \times 33.625}{1{,}550} = 1.4 \text{ sq. meters, or } 1.67+ \text{sq. yds.} \quad [25]$$

Dimensions may be checked by substitution in figure 9. For all liners, the minimum value of *PA* should be 3 inches. In practice, this liner would be given a pilot-plant test to be sure its tolerances would be satisfactory for the range of variances to be found in the factory of the user.

(8) For the purpose of calculating the *S* factor for an RSAV△ specification, what would be the total permeable area of a liner of the above dimensions?

Solution: Use formula [12], page 66, and the answers obtained by formulas [9] and [10] in example 7.

$$A_{tp} = \left\{ \frac{2(32.25 \times 33.625) - [(\sqrt{12.25^2 + 20^2} + 6)(12.25 + 20)] + (12.25 \times 20) + 0.7854(12.25^2 + 20^2)}{1{,}550} \right\}$$

$$= 1.223 \text{ m.}^2 \quad [26]$$

This answer is only 73 square inches less than the *S* constant previously given for this box (*119, table 2*). The careful designer probably would calculate dimensions for both sizes of liners, then make careful samples of each and test them for the preferable fit under the conditions and variances in the plant of the user.

The essential difference, in this illustration of application, is not which size of liner is chosen but that the permeable area of that liner shall have a known value that will be meaningful in the permeability specification of the liner. On the other hand, it also is important to the user that the selected liner is not of such dimensions that his labor costs would be increased. (See paragraphs for calculation of *S*-factors, p. 69 and below).

(9) Rome Beauty apples are to be stored at 32° F. in liners that are to attain an effective atmosphere of 3 percent oxygen and 4 percent carbon dioxide. The mean net weight of the apples has been reliably determined to be 42 pounds per box. The boxes to be used are closest in dimensions to those of tariff box 6056. The liners are to have double-sealed seams and to be double-tie sealed. What estimated film permeabilities should be specified? Solutions:

R-factor.—Since the true respiration rate of the apples is unknown, an estimated rate must be used (*119, table 7*). Multiply this rate by the box weight and the adjustment factor explained on page 69.

$$R = 42.00 \times 2.63 \times 5.5422 = 6.1219 \times 10^2 \qquad [27]$$

S-factor.—There are two methods available for computing this factor. For the first method, compute the A_{tp} value (formula 12, p. 66) and use it in following formula

$$S_1 = \frac{1}{A_{tp}} = \frac{1}{1.223} = 8.1766 \times 10^{-1} \qquad [28]$$

For the second method, multiply constant *S* (1.270) (*119, table 2, tariff number 6056*) by adjustment factor 0.6476 (p. 69).

$$S_2 = 1.270 \times 0.6476 = 8.2245 \times 10^{-1} \qquad [29]$$

The S_1 value is preferred. It is a little more accurate than S_2.

A-factor.—Multiply a_c value for 4 percent carbon dioxide ((0.915+0.866)÷2, or 0.89050) (*119, table 3, footnote 1*) by constant a_o, as revised, for 3 percent oxygen (0.411) (p. 60).

$$A = 0.8905 \times 0.411 = 3.6600 \times 10^{-1} \qquad [30]$$

V-factor.—See (*119, formula 1*).

$$\left[V = \frac{0.79}{1.00 - (0.04 + 0.03)} \right] = 8.4946 \times 10^{-1} \qquad [31]$$

RSAV-factor.—This is the product of the preceding four factors. It is the same for carbon dioxide and oxygen; it saves time to compute it once for both.

$$= 6.1219 \times 10^2 \times 8.1766 \times 10^{-1} \times 3.66 \times 10^{-1} \times 8.4946 \times 10^{-1}$$
$$= 1.55626 \times 10^2 \qquad [32]$$

Λ-factor.—(a) carbon dioxide requires formula 2 (*119*), and (b) oxygen requires formula 3 of the same reference.

$$\text{(a)}\quad \triangle_c = \left[\frac{760}{(0.04\times 760\times 0.84946)-0.228}\right] = 2.96926\times 10^{1} \qquad [33]$$

$$\text{(b)}\quad \triangle_o = \left[\frac{760}{159.52-(0.03\times 760\times 0.84946)}\right] = 5.42267\times 10^{0} \qquad [34]$$

If the local mean barometric pressure during the usual storage period is known, substitute this value for 760 in the denominators of [33] and [34]. In (a), multiply the barometric mean by 0.0003 to obtain the replacement factor for 0.228. In (b), multiply the barometric mean by 20.99 to obtain the replacement for 159.52.

RSAV$\triangle_c$.—The carbon dioxide permeability specification would be [32] × [33], or

$$=1.55626\times 10^{2}\times 2.96926\times 10^{1} = 4.6209\times 10^{3} \qquad [35]$$

RSAV$\triangle_o$.—The oxygen permeability specification would be [32] × [34], or

$$=1.55626\times 10^{2}\times 5.42267\times 10^{0} = 8.43908\times 10^{2} \qquad [36]$$

Both [35] and [36] are: $ml._{(STP)}$/mil/m.^{2}/24 hr./760 mm. Hg.$\triangle$/ at 32° F. The required permeabilities thus are about 4,600 ml. of carbon dioxide and 850 ml. of oxygen, as the nearest practical values.

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