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Test Methods for Evaluating
Moisture Permeation of Flexible
Unit Dose Packages

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Hugh E Lockhart
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TEST METHODS FOR EVALUATING MOISTURE PERMEATION OF FLEXIBLE UNIT DOSE PACKAGES

By

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ABSTRACT

TEST METHODS FOR EVALUATING MOISTURE PERMEATION OF FLEXIBLE UNIT DOSE PACKAGES

By

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Many hospital pharmacies are involved with the repackaging of drug products. A common type of packaging machine for this purpose is the form-fill-seal. These machines are used to repackage oral solid drugs which are moisture sensitive. A problem that arises is how to insure that the packages formed are good moisture barriers.

This study includes an evaluation of 2 commonly used packages. The effects of external environment, seal discontinuity, and defective material on the permeability of the package were tested. Testing was done according to the United States Pharmacopeial Convention test procedures for permeation of single-unit containers. In addition, a test method is introduced by which an estimation of permeation, USP class, and package integrity can be achieved.

Results indicate that seal discontinuities, pinholes in materials, and increased relative humidity in the storage area all cause increased package permeability. None of these potential problems are readily discernable by the operator without the use of tests. The test procedure developed can detect these conditions before problems arise.

ACKNOWLEDGMENTS

Special thanks to my major professor, Dr. Hugh Lockhart without whose guidance this thesis would not have been possible. His countless hours of hard work, genuine concern, and unselfishness will always be remembered.

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INTRODUCTION

The repackaging of oral solid drugs in single units is widespread in hospitals across the United States (5). One reason for this is that the institutional pharmacist must repackage the drugs obtained in bulk containers into single units to facilitate the use of a unit dose distribution system (7). This process has its shortcomings, however. There have been reports that pharmacists need more information about the permeation characteristics of packages produced in the pharmacy. Reamer and Grady state that not only must moisture permeation of unit dose packages be measured, but a separate verification of the integrity of the heat seal must be done (11).

These problems were presented to the directors of three institutional pharmacies in the Lansing, Michigan area to obtain their views. Again, a concern was expressed by them about the permeation characteristics of the repackaging materials and the shelf life of the product after repackaging. In addition, these pharmacists were also uncertain about the integrity of the finished package. Presently all the information regarding permeation is obtained from the material supplier. Often this data is given for materials only and not for fabricated packages.

The United States Pharmacopeia (USP) (14) provides a method by which packages can be classified based on the amount of moisture gained by a packaged desiccant. Packages are classified from A through D as indicated in Table 1.

TABLE 1

USP Classification of Packages

Class	Moisture Permeation (mg/day) not more than 1 of 10 exceeds:	none exceeds:
A	0.5	1.0
B	5.0	10.0
C	20.0	40.0
D	Above criteria not met	

This method provides a general guide to permeation rates and a package supplier may use this classification when describing a package system. The ranges are quite large within each class as well as from one class to another.

The moisture permeation of the package can also be expressed as a Water Vapor Transmission Rate (WVTR). The procedure for determining WVTR is similiar to the USP method but it allows for measurements of moisture gain at several sequential time intervals. The moisture gain is then plotted versus time and a statistical tool such as linear regression may be applied to calculate the rate of permeation. In comparison, the USP method calls for a measurement of moisture gain at only one time interval. This does not present a good picture of what is occurring before and after that point in time.

There still remains the Lansing pharmacists' concern about package integrity. Even though the supplier provides information on material permeation and package USP classification, how does one know if a good package is being formed? Can this be monitored? If so, how?

This thesis will address the issues concerning the pharmaceutical repackaging operation. Moisture permeation and USP classification of selected packages will be determined. The integrity of the package will be altered to determine the effects on WVTR and USP classification. Finally, a test method will be presented by which the pharmacist can monitor the package system for moisture permeation and package integrity.

THE REPACKAGING OPERATION

Because of the widespread repackaging of drug products, many different machines and materials have become commercially available (10,11). With this variety comes the necessity for the pharmacist to make a decision about which packaging process and material to select to efficiently carry out the repackaging operation. A general overview of a few packaging concepts will help the pharmacist get started in the right direction.

Whenever a package selection process is initiated there are 4 major areas to consider:

1. Package
2. Product
3. Environment
4. Machinery

PACKAGE

When selecting the package, one must have some knowledge about both the product and the environment the product will face after packaging. Once the decision has been made as to which environmental factors the product must be protected from, the process of selecting the proper package can proceed.

PRODUCT

A determination must be made as to whether or not the product needs a package. In the case of an oral solid drug product, it may be necessary to protect the product from certain elements of the environment which may be detrimental to that product. This is where proper packaging and proper package selection comes into play. Drugs may be sensitive to such factors in the environment as oxygen, water vapor, or light or they may lose volatile components (1).

ENVIRONMENT

This study will concentrate on water vapor only since this is the environmental factor causing most problems in oral solid drug shelf life.

Many oral solid drugs are sensitive to water vapor. There is a wide variation in water vapor present in storage areas. In some pharmacies humidity and water vapor are fairly well controlled at 30-50% relative humidity throughout the year. In other pharmacies relative humidity may vary from 15% in winter to 70% in spring or summer.

MACHINERY

In the repackaging operation, selection of material and machinery go hand in hand. Decisions most likely will be based on such factors as cost of machinery, cost of materials, complexity of machinery, and degree of training necessary for the operators. Repackaging in single units may be done with different types of packages, such as strip packages, blister packs, or skin packs (10,11). Strip packages may be formed on horizontal or vertical form-fill-seal machinery. All other things being equal, the aesthetics of the package may be the deciding factor. However, the performance of the package is the key issue that must not be overlooked.

Bacon (4) has stated that there are definite advantages to using form-fill-seal operations. Among them are: low capital investment, low tooling costs, and minimum complexity of equipment. In addition to cost effectiveness, the form-fill-seal package is reduced in size to dimensions which are only slightly larger than the product itself resulting in material savings as well as decreased storage space requirements. This is preferable to using pre-formed pouches or pre-formed blisters. If transparent material is used, product visibility is enhanced. This is important because 85% of hospital nursing supervisors claim that product visibility makes transparent packages preferable (4). Also, specific product information can be imprinted on the package while it is still on the packaging machine (4). The heat sealing operation used to seal the package can be controlled allowing for good package integrity. Thus, the three major advantages of form-fill-seal are cost effectiveness, space efficiency, and package integrity (4).

With the preceeding in mind, the study includes only one type of package: the strip package. The strip package was selected because:

1. it is typical.
2. it is in use in the Lansing area hospitals.
3. local pharmacists expressed concern about whether or not the strip package is adequate as a moisture barrier.
4. these pharmacists also expressed concern about seal integrity and how it could be monitored.
5. the Lansing area hospitals are the basis of this study because they comprise a cooperating group with the School of Packaging for study of these and other hospital packaging questions.

PACKAGE FORMATION

Two methods of package formation will be discussed: horizontal and vertical form-fill-seal strip packages. The methods have some similarities. Among them are:

a) packages are formed from 2 separate webs of material into a strip of 4-side-seal pouches.

b) packages are sealed by a hot bar coming in contact with one side of the package and applying a force to help construct the heat seal bond.

c) one tablet or capsule is placed in each package.

d) temperature of the hot bar is easily controlled by means of a thermostat.

Differences also exist and they will be apparent as each method of package formation is separately studied.

The vertical form-fill-seal, as the name implies, involves a process whereby the material is fed into the machine and the package is formed in a vertical orientation. The tablets or capsules are dropped into each package by gravity, usually being fed into the package by means of a slotted, rotating wheel located at the top of the machine (3). The sealing operation consists of 2 steps. The first step seals one end of the package. The tablet then drops into the pouch. In the second step the material is advanced and the other end of the package is sealed, along with the sides and end of the new package. Since one sealing step seals the ends of 2 packages, the tablets will not fall into a previously filled package as the drop. As the packages exit the machine they all may be in one strip with perforations between each package or they may be cut into separate pouches.

The horizontal form-fill-seal package is very similar in formation. The webs of material are fed in and the package is formed horizontally. Since the tablets cannot be gravity fed, an operator will place one tablet or capsule in each package. On the machine used in the study, a press made an indentation in one web so that the tablet would have a place to nest (4). The sealing operation and formation of the strips exiting the machine are the same as described for the vertical form-fill-seal.

MATERIALS AND MACHINERY

Testing for water vapor transmission rates was performed on two package types made of similar material, fabricated on two different packaging machines. For purposes of simplicity in the presentation and discussion of results, the following designations will be used.

Package A:	Materials supplied by Odessa Packaging Services, Inc. Odessa, DE
Machine A:	Mercury U-Pack G-L Industries Mercury Packaging Machine Corp. Philadelphia, PA Sold by Odessa Packaging Services Inc. Type: Vertical F/F/S
Package B:	Materials supplied by In-Pac Systems Inc. Mount Vernon, OH
Machine B:	In-Pac Machine, System III In-Pac Systems, Inc. Mount Vernon, OH Type: Horizontal F/F/S

The materials used in the studies are laminations. Both packages are constructed from two different materials. Each package has a transparent material heat sealed to a non-transparent material containing foil and paper laminated to polyethylene. Information regarding the composition of the materials was obtained from the respective suppliers and is as follows:

Package A:

Transparent material: 250 gauge Saran coated cellophane / 1.5 mil LDPE

Non-transparent material: 25 lb. paper / 0.5 mil LDPE / .35 mil foil / 1 mil LDPE

Package B:

Transparent material: 195 gauge Saran coated cellophane / 1.5 mil LDPE

Non-transparent material: 35 lb. paper / 0.67 mil LDPE / .35 mil foil / 1 mil LDPE

For the tests, packages were fabricated from each pair of materials on that machine sold by the supplier of that material. That is, Package A (Odessa) formed on Machine A (Odessa), and Package B (In-Pac) on Machine B (In-Pac). Then, to measure machine effect, each pair of materials was run on the other suppliers' machine to see if any differences resulted. The combinations were Package A (Odessa)/ Machine B (In-Pac) and Package B (In-Pac)/ Machine A (Odessa).

This approach was taken because each of the suppliers offers a machine/material system of his own selection. The function and geometries of the machines were enough different that we felt the crossover test chould be made.

TEST METHODS

The usual test methods of interest for hospital repackaging are those for moisture permeation and for seal integrity. A descussion of possible methods of measuring these follows:

MOISTURE PERMEATION

Testing of moisture permeation through packages and packaging materials is a very important step to insure that good packages are produced which will protect moisture sensitive products. Procedures are available to test both materials and packages for their ability to serve as moisture barriers. These tests usually involve the use of a desiccant and a gravimetric method for measuring weight gain of the desiccant. The procedure commonly used to measure the rate of transmission of water vapor through materials is ASTM procedure E-96 (Appendix A). Procedures for measuring water vapor transmission into or out of containers include ASTM D 3199 (Appendix A) and USP (671) Containers - Permeation (Appendix A) (1,14).

When water vapor is being transmitted through a given material or package there may be 2 mechanisms involved: leakage and permeation. Leakage can occur when the vapor passes through some discontinuity in a material or between surfaces of materials in contact. Sources of these leaks include pinholes in material or faulty seals. Both are of concern when dealing with strip packages for drugs that are moisture sensitive. Permeation, on the other hand, occurs when the vapor passes through the material itself.

When dealing with moisture sensitive products, the goal is to eliminate leakage and decrease permeation. Some ways to minimize leakage are to (a) decrease the surface area of the seal so the number and size of seal discontinuities are decreased, (b) use a lamination which will help eliminate pinholes since it is very unlikely that pinholes in 2 or more materials will coincide, (c) optimize the sealing conditions of temperature, pressure, and dwell time to gain maximum seal continuity and (d) avoid wrinkles or product particles in the seal area (2).

Permeation rates can be reduced by (a) using thicker material, (b) using a different material with superior barrier properties, or (c) decreasing the surface of the package (2).

SEAL INTEGRITY

Seal integrity can be measured as the strength of the seal, or as the degree of continuity of the seal. In this study, the second of these is treated as the property to be measured.

Various methods of testing seals are cited in the literature. Miltz (8) describes a peel test using a tensile testing machine. Reamer and Grady (12) discuss a method for testing seals using thermal analysis techniques, and Auslander (3) refers to a pressurized ammonia vapor test for evaluating the integrity of package seals. All of these test procedures require special equipment and would be useful in the initial development of optimum machine settings. They could also be used to confirm that optimum sealing conditions have been achieved. These procedures are time consuming as well as costly in terms of machinery and labor and may not be feasible in the institutional pharmacy, since the pharmacy staff is normally totally occupied with dispensing medications and monitoring medication regimens. Furthermore, manufacturers of heat sealing equipment provide reliable information concerning the machine settings necessary to achieve proper seals when using specified material. For this particular application in the pharmacy, seal strength alone is not necessarily a good measure of package performance. The concern is how much moisture enters the packages. A seal that tests as being "strong" by a peel test may indeed leak. This can happen when there is an open channel or discontinuity running through a wide seal which is very strong. The effect of channels will be demonstrated later. A "weak" seal may be continuous and be a good moisture barrier. There should be a simple test by which the pharmacist can monitor the seal integrity with respect to moisture transmission of packages produced on in-house machinery,thus avoiding the costly and painstaking process of conducting a test specifically for seal integrity of either kind.

PERMEATION

The water vapor barrier properties of the two package types were determined using the USP method and a gravimetric method. In both tests the weight gain of a desiccant pellet* was used to measure the moisture permeation of the packages. The pellets were obtained from:

Hunt Sales Co.
13700 Bardon Rd.
Pheonix, MD 21131

These pellets were chosen because of the reference to them in the USP (14). Other desiccants are available, but the one selected was especially well suited to this testing because of its size and shape. A unique feature of the pellets is a change in color from blue to pink when they have absorbed 10% of their weight in moisture. Since the pellets range in weight from 250 to 300 milligrams, the color change occurs when the pellets have gained 25 to 30 milligrams of moisture.

In the determination of moisture permeation, blanks were run with each test to account for any moisture gain by the package.

Packages were tested under three storage conditions, given in Table 2.

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* Grade 944 Indicating Pellets, P/N 944-0%-X1746 from Davison Chemical Division of W.R. Grace

Table 2

Storage Conditions for Package WVTR Measurements

Temperature	Relative Humidity
72°F	50%
72°F *	75% *
100°F	85%

* USP Test Conditions

Conditions were chosen to represent ambient and accelerated storage environments as well as the USP test conditions. The USP classification of both Package A and B appears in Table 3.

Next the materials were run on the other supplier's machine. The combinations were Package A (Odessa)/Machine B (In-Pac) and Package B (In-Pac)/Machine A (Odessa). These results are presented in Table 4.

To compare the effects of different external environments on the moisture permeation of the package, Package A was tested at the 3 storage conditions listed in Table 2. These results are summarized in Tables 5,6, and 7 and presented graphically in Figure 1.

LEAKAGE

Occasionally a package may be formed which is less than acceptable. That is, the material may be defective or the seal may be non-continuous. This should result in a higher WVTR. To verify this assumption, packages were intentionally damaged.

First, packages were formed having non-continuous seals

by inserting a common paper clip wire (.032 inches in diameter) into the seal area while the seal jaw closed. The result was a channel through the seal area. The packages were then tested by the USP method. The data appears in Table 9. Both materials were run on Machine A due to difficulty in creating the seal defect on Machine B. Because of the geometry of Machine B, there were physical obstructions making it impossible to create the defect. Also, Package A was tested with two channels to determine what degree of seal discontinuity was needed to produce a Class C package.

Secondly, to simulate defects in material, a hole was made in the transparent side of Package A. The packages were then stored at 72°F, 50% RH and weighed periodically. The results are in Table 10.

For ease of comparison between intact packages and those with defects, refer to Figure 2.

TABLE 3

USP Classification of Packages
(Values represent moisture gain in mg/day)
Storage Conditions: 72°F, 75% RH

	Package A/Machine A	Package B/Machine B
	0.7	3.3
	0.7	4.1
	0.8	6.3
	1.3	3.1
	0.7	4.4
	5.8*	3.0
	1.0	3.2
	0.7	2.9
	0.7	3.0
	0.9	3.3
Average	0.8	3.7
Std Dev	0.2	1.1
USP Class	B	B

* bad seal - value not included in average

TABLE 4

USP Classification of Packages
(Values represent moisture gain in mg/day)
Storage Conditions: 72°F, 75% RH

	Package A/Machine B	Package B/Machine A
	0.4	3.6
	0.7	3.6
	0.4	3.4
	0.4	3.4
	0.4	15.2*
	0.6	3.5
	0.4	3.6
	0.4	3.6
	0.3	3.8
	0.4	3.4
Average	0.4	3.5
Std Dev	0.1	0.1
USP Class	B	B

* bad seal - value not included in average

TABLE 5

Water Vapor Transmission Rates (mg/pkg)
Package A/Machine A
72°F 50% RH

	Time (days)					
	1	3	5	7	12	17
Average weight gain*	0.04	1.4	1.9	2.6	4.6	6.2
Std Dev	0.07	0.1	0.2	0.2	0.2	0.2
Desiccant color:	Blue	Blue	Blue	Blue	Blue	Blue

TABLE 6

Water Vapor Transmission Rates (mg/pkg)
Package A/Machine A
72°F 75% RH

	Time (days)					
	1	2	3	4	6	13
Average weight gain*	1.7	1.0	2.4	3.0	4.2	9.9
Std Dev	0.2	0.4	0.7	0.9	1.5	2.9
Desiccant color:	Blue	Blue	Blue	Blue	Blue	Blue

* average of 10 samples

TABLE 7

Water Vapor Transmission Rates (mg/pkg)
Package A/Machine A
100°F 85% RH

	Time (days)			
	1	2	3	4
Average weight gain*	3.2	8.9	12.4	19.2
Std Dev	0.4	0.4	0.3	0.4
Desiccant color:	Blue	Blue	Blue	Pink

* average of 10 samples

TABLE 8

Permeation Rates at Various Storage Conditions

Storage Condition		Permeation Rate (mg/day)	USP Class	$\bar{P}$*
72°F	50% RH	0.4	B	0.040
72°F	75% RH	0.7	B	0.046
100°F	85% RH	5.2	B	0.124

* $\bar{P}$ is the permeability constant expressed in the units:

$$\frac{\text{mg } H_2O \text{ x pkg.}}{\text{day x mmHg}}$$

Figure 1

Moisture Gain at Various Storage Conditions

Package A/Machine A

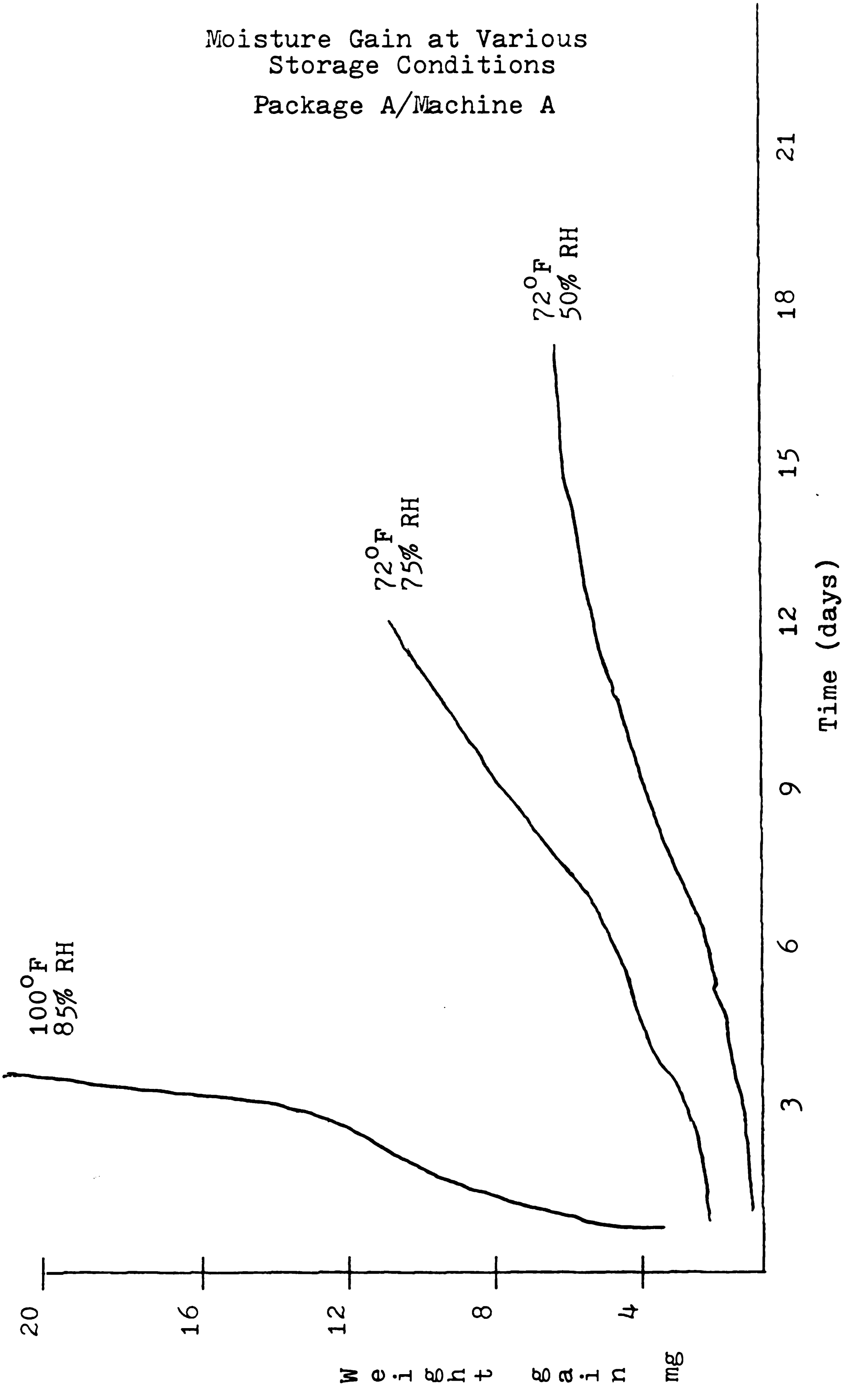


TABLE 9

USP Classification of Packages with Seal Defects
(Values represent moisture gain in mg/day)
72°F 75% RH

	Package A/Machine A (one channel)	Package A/Machine A (two channels)	Package B/Machine A (one channel)
	4.2	6.0	5.4
	3.7	5.0	6.1
	4.0	11.4	8.5
	4.2	12.2	6.7
	4.0	13.0	11.8
	3.9	6.3	7.6
	4.1	12.5	6.8
	4.6	6.7	6.8
	3.5	13.0	8.9
	2.8	14.2	7.6
Average	3.9	10.1	7.6
Std Dev	0.5	3.7	1.8
USP Class	B	C	C

TABLE 10

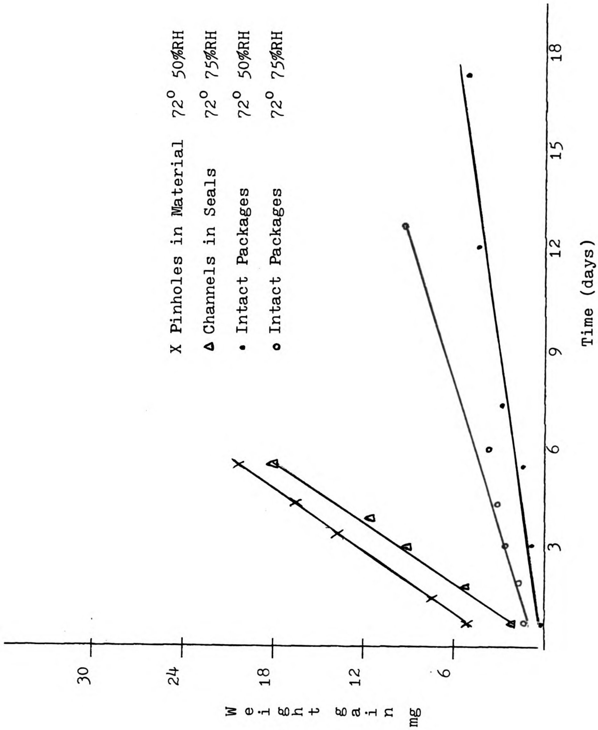
Moisture Gain (mg) in Package A with Pinhole
72°F 50% RH

	Time (days)				
	1	2	4	5	7
Average weight gain*	3.6	7.4	13.2	15.7	19.2
Std Dev	0.3	0.8	1.1	1.4	1.5
Desiccant color:	Blue	Blue	Blue	Blue	Pink

* average of 10 samples

Figure 2

Weight Gain of Defective
and Intact Packages
Package A/Machine A



DISCUSSION OF RESULTS

USP CLASSIFICATION VS WVTR

According to data in Table 3, and the classification in Table 1, packages A and B have the same USP classification in spite of the fact that Package B has four and one half times greater rate of gain than Package A. This difference is not explained by differences in material unless Package A has a thicker coating of Saran on the 250 cellophane. The hydroscopic nature of cellophane is such as to indicate that thicker cellophane alone should not account for this large difference. Note that packages A and B have the same USP classification, but Package A will provide a shelf life perhaps 4 times longer than Package B.

In order to gain an insight into what moisture permeation occurs due to the environment, Package A was stored at the three conditions presented in Table 2. The results (Table 8) show that as external relative humidity increases, so does the permeation rate. At the constant temperature of 72°F, the increase in relative humidity from 50% to 75% causes a small rise in permeation rate from 0.4 to 0.7 mg/day/package. Taking into account that the balance used can measure only to the nearest 0.1 mg, these results are very close. However, when the package is stored in an extremely severe environment, 100°F and 85% RH, the permeation rate increases more than five-fold. This demonstrates the importance of monitoring or controlling storage conditions.

CROSSOVER TEST OF MATERIAL ON OPPOSITE MACHINE

From Table 4, Package B has about the same WVTR on either machine. Package A has an average WVTR about one half the value obtained on Machine A. However, if the standard deviation is taken into account, it must be recognized that Package A is probably about the same when produced on either machine.

LEAKAGE INDUCED BY DEFECT IN PACKAGE OR MATERIAL

While package permeability will vary based on the external environment, defects in materials or seal areas will also cause changes in permeability. The data in Table 9 helps to illustrate this statement. Package A has a permeability of 0.8 mg/day/package under USP test conditions. With the introduction of one channel in the seal area the permeability increases by 3.1 mg/day, or nearly four-fold. Two channels caused an increase of 9.3 mg/day (twelve-fold) and the package is no longer a Class B but a Class C package. Meanwhile, Package B with one channel has an increase in permeability of 3.9 mg/day and also becomes a Class C package. Material defects cause the same effect. Package A has an increase of 2.2 mg/day when one hole is introduced into the material and the package is stored at ambient conditions (72°F, 50% RH). Package A has a permeability of 2.6 mg/day as determined by linear regression of the data in Table 10, whereas from Table 8 one can see that the same intact package has a permeation rate of 0.4 mg/day.

The interesting aspect of the previous discussion is that these defects are not readily visible. Even upon careful inspection of each package the defect is difficult if not impossible to spot. Since the machines used in this study operate at 50-60 packages per minute, the operator could not possibly inspect each package. Even if 100% inspection were desirable it wouldn't be practical. Thus, the need is established for a way to identify defects and consequently prevent potential problems.

CONCLUSION AND RECOMMENDATIONS

Many problems may arise when attempting to package a moisture sensitive product. The problems could result in a more than desirable amount of moisture entering the package and affecting the product. The pharmacist has guidelines available to assist in the repackaging operation (9,13). These guidelines do not provide a means for evaluating the integrity of packages produced in the pharmacy. The pharmacist can also obtain information from his machine and material supplier about machine settings and material USP classification or WVTR. However, these do not provide a means to monitor the integrity of packages as they are produced.

Based on data taken, and observations made during testing, two methods have been devised for the purpose of evaluating the integrity of packages as produced in the pharmacy. Furthermore, they provide a means for estimating the shelf life (or monitoring the approach of the end of shelf life) of products packaged in the hospital pharmacy. These methods have the advantage that they do not require the pharmacist to apply time to learn and to use skills which are not a part of his primary duty. These methods, properly applied, should allow the monitoring of package integrity and shelf life on a routine basis, using only a minimum of time.

PACKAGE TEST METHOD

With the possibility of problems due to changing environment, non-continuous seals, or defective material, it is necessary to have a way to determine what kind of moisture protection the package will provide. There is no replacement for the standard water vapor transmission testing of packages in determining moisture barrier properties. In the hospital pharmacy, however, this process can be time consuming and costly, especially if done on a frequent basis. A simple test to give an estimation of WVTR and/or package USP classification would be beneficial.

The desiccant pellets used in these studies would be ideal for such a test. Since the pellets turn from blue to pink as moisture is absorbed, a simple visual test could be used to monitor package permeability. Such a test has been devised using the results of this study. This test provides an on-line check without extensive use of weighing devices on a regular basis, thus saving time and money.

The test procedure is as follows:

1. Place one desiccant pellet in a package at the beginning, middle, and end of each run.
2. Identify each of these three packages and record its weight to the nearest 0.1 mg (1.0 mg is acceptable but 0.1 is preferred.)
3. Identify and weigh a blank package without a pellet.
4. Store the packaged desiccant and blank in the same area where packaged drug products are stored.
5. Daily observe the pellets until a pink color is apparent. NOTE: Controls must also be set up; that is, one pink and one blue pellet to help distinguish minor color changes.

6. Weigh the package and blank.

Once these weights have been determined, the following calculation will estimate the moisture permeation.

P_I = Initial weight of package and desiccant (mg)

P_F = Final weight of package and desiccant (mg)

B_I = Initial weight of blank (mg)

B_F = Final weight of blank (mg)

D = Weight gain of desiccant (mg)

T = Time (days) from initial to final weighing

$$(P_F - B_F) - (P_I - B_I) = D$$

$$\frac{D}{T} = \text{moisture permeation (mg/day)}$$

This is an estimate of permeation since only an initial and final weighing are used. Ideally, several weighings should be done and moisture gain versus time plotted. Linear regression of these points would provide a more realistic WVTR (slope of the line) (1).

An example can be taken from Table 10. The pellets stored at 72°F and 50% RH turned pink after 7 days. The calculations yield a moisture gain of 19.2 mg over this period. Dividing 19.2 by 7 gives a permeation rate of about 2.7 mg/day/package. The same package with no pinholes (Table 8) had a permeation rate of 0.4 mg/day/package. This would alert the pharmacist to a possible problem for the drugs packaged the same day as the pellets. Obviously some initial studies must be done to establish baselines for non-defective packages. Once the time has been established for the desiccant in a good package to turn pink, the weighing may be eliminated or reduced.

METHOD FOR USP CLASSIFICATION

A simple test method can be used to get a rough estimate of USP classification. By noting the time required for a packaged desiccant pellet to turn pink, a chart such as Table 11 can be used to estimate the USP classification.

TABLE 11

Estimation of USP Classification Based on Desiccant Color Change
72°F 75% RH

Time (days)	USP Class
40 or more	A
4 - 40	B
1 - 4	C
Less than 1	D

The assumptions made in constructing Table 11 are:

1. The desiccant pellet weighs close to 200 mg.
2. The desiccant turns from blue to pink when it gains 20 mg of moisture (10% by weight). This is true for the desiccant used in this study.
3. Any testing to verify USP classification or WVTR will be conducted according to the specific procedure.

APPENDIX A

Designation: D 3199 - 73$^{\epsilon}$

Standard Test Method for
WATER VAPOR TRANSMISSION THROUGH SCREW-CAP CLOSURE LINERS[1]

This Standard is issued under the fixed designation D 3199; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

$^{\epsilon}$NOTE—The title was changed editorially in January 1977.

1. Scope

1.1 This method covers the measurement of the barrier efficiency of screw-cap liners against water vapor transmission either into or outward from a container capped under standard conditions.

2. Summary of Method

2.1 The method involves periodic weighing of containers sealed with screw caps containing the liners to be evaluated to determine moisture gain or loss under controlled conditions of temperature and humidity.

Significance

3.1 This test may be used to compare or screen new or existing closure liner materials by their performance in a standard test.

3.2 It also may be used to indicate whether the liner material will be satisfactory for use with moist products, dry products, or both types of products. When this method is used to assist in the choice of liner material for a specific use, the choice of containers and closures should simulate the conditions of use as closely as practicable.

3.3 The method may be used to establish performance specifications.

4. Apparatus

4.1 *Bottles*, ½ oz (approximately 15 cm^3) with a 28 mm Glass Container Manufacturers Institute (GCMI) 400 or 405 finish. Select test bottles for uniformity of flatness of finish, width of lip sealing surface, and freedom from splits, chips, or cracks.

4.2 *Screw Caps*, 28 mm continuous thread (CT) of either metal or plastic.

4.3 *Torque Tester*[2], to determine application torque, accurate to within ±½ lbf-in. (0.06 N·m).

4.4 *Humidity Cabinet*, capable of control to ±1°F (±0.6°C) and ±2% relative humidity at 100°F (38°C). Ranges required are 25 and 75% relative humidity.

4.5 *Analytical Balance*, with an accuracy to at least 0.001 g.

4.6 *Pipete or Graduate*, capable of delivering 10 ml of water with an accuracy of ±0.1 ml.

5. Reagent

5.1 Activated silica gel (Grade H, Type 46, 16 mesh) anhydrous calcium chloride, or other suitable desiccant material.

6. Test Specimens

6.1 The closure-liner test specimen should be die-cut by either standard manufacturers' screw-cap lining equipment or by a single punch, using care that the liner is made uniform and without distortion of the liner facing.

6.2 No torn liners or wrinkled liners should be used. White, virgin pulp backing should be used for all tests. Prior to use, the liner backing combination must be laminated together and

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[1] This method is under the jurisdiction of ASTM Committee D-10 on Packaging.

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[2] Owens-Illinois torque tester, available from Owens-Illinois, Inc., Glass Container Div., P.O. Box 1035, Toledo, Ohio 43601, or equivalent, has been found satisfactory for this method.

adhered to the cap, if plastic; or forced into the cap, if metal. At least ten test specimens and ten control specimens of each liner to be evaluated should be used for this test.

7. Procedure

7.1 *Water Vapor Transmission Outward From a Container:*

7.1.1 Tare weigh each ½-oz (28-mm) glass container together with its respective lined closure. Use five containers for this portion of the test.

7.1.2 Place 10.0 ml of distilled water in each tared glass container.

7.1.3 Apply the screw caps carrying the test liners with an application of 15 lbf-in. (approximately 1.7 N·m).

7.1.4 Assemble with the caps as in accordance with 7.1.1 and 7.1.3 and weigh five empty bottles for use as controls. Correct the test results by subtracting the average mass loss observed in the control containers from the average mass loss in the test containers.

7.1.5 Place the containers upright in the humidity chamber at 100±1°F (38±0.6°C) and 25 ±2% relative humidity.

7.1.6 Seven days after starting the test, begin weighing each test container to the nearest 0.001 g at weekly intervals for 4 weeks. Allow the containers to come to room temperature before weighing.

7.1.7 Replace the test containers in the humidity cabinet immediately after each weighing.

7.2 *Water Vapor Transmission Into a Container:*

7.2.1 Tare weigh each ½-oz (28-mm) glass container together with its respective lined closure. Use five containers for this portion of the test.

7.2.2 Place 10.0 g of activated silica gel or other suitable desiccant in each container.

7.2.3 Apply the screw caps carrying the test liners with an application torque of 15 lbf-in. (approximately 1.7 N·m).

7.2.4 Assemble with the caps in accordance with 7.2.1 and 7.2.3 and weigh five empty bottles for use as controls. Correct the test results by subtracting the average mass gain observed in control containers from the average mass gain in the test containers.

7.2.5 Place the containers upright in the humidity chamber at 100 ± 1°F (38 ± 0.6°C) and 75 ±2% relative humidity.

7.2.6 Seven days after starting the test, begin weighing each test container at weekly intervals for 4 weeks. Allow the packages to come to room temperature before weighing.

7.2.7 Replace the test containers in the humidity cabinet immediately after each weighing.

8. Report

8.1 The report shall include the following:

8.1.1 Material of construction of the liner and whether adhered to or only forced into the cap,

8.1.2 Material of construction of the cap,

8.1.3 Name of the desiccant used,

8.1.4 Number of replicates and controls,

8.1.5 Time interval or intervals of the test,

8.1.6 Temperature and humidity of the test,

8.1.7 Mass losses and gains in tabular form, and their averages,

8.1.8 Average mass gain and loss and the range, in milligrams corrected for the average mass gain and loss of the controls,

8.1.9 Calculation of daily gain or loss (optional),

8.1.10 Graph on arithmetical paper of mass gain or loss versus time (optional),

8.1.11 Evaluation of comparative results if more than one liner construction was tested,

8.1.12 Statement that this test was done in accordance with ASTM Method D 3199.

Designation: E 96 – 66 (Reapproved 1972)

Standard Test Methods for
WATER VAPOR TRANSMISSION OF MATERIALS IN SHEET FORM[1]

This Standard is issued under the fixed designation E 96; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 These methods cover determination of the rate of water vapor transmission (WVT) of materials in sheet form. The methods are applicable to materials such as paper, plastic films, and sheet materials in general. The methods are most suitable for specimens $^1/_8$ in. (3.18 mm) or less in thickness, but may be used with caution for somewhat thicker specimens. For specimens of materials used in building construction and similar materials greater than $^1/_8$ in. in thickness, see ASTM Methods C 355, Test for Water Vapor Transmission of Thick Materials.[2] Six procedures are provided for the measurement of transmission under different test conditions, as follows:

1.1.1 *Procedure A*—For use when the materials to be tested are employed in the low range of humidities.

1.1.2 *Procedure B*—For use when the materials to be tested are employed in the high range of humidities, but will not normally be wetted.

1.1.3 *Procedure BW*—For use when materials to be tested may in service be wetted on one surface but under conditions where the hydraulic head is relatively unimportant and moisture transfer is governed by capillary and water vapor diffusion forces.

1.1.4 *Procedure C*—Conducted at an elevated temperature for use with materials employed in the low range of humidities, and intended to shorten the time of testing of highly impermeable materials. This procedure eliminates, in most cases, the need for refrigeration.

1.1.5 *Procedure D*—Conducted at an elevated temperature for use with materials employed in the high range of humidities, but not normally wetted, and intended to shorten the time of testing of highly impermeable materials employed in this range. This procedure also eliminates, in most cases, the need for refrigeration.

1.1.6 *Procedure E*—For use in measuring the WVT at an elevated temperature, with a very low humidity on one side of the sheet and a high humidity on the other side.

NOTE 1—The values stated in U.S. customary units are to be regarded as the standard. The metric equivalents of U.S. customary units may be approximate.

2. Summary of Methods

2.1 The material to be tested is fastened over the mouth of a dish, which contains either a desiccant or water. The assembly is placed in an atmosphere of constant temperature and humidity, and the weight gain or loss of the assembly is used to calculate the rate of water vapor movement through the sheet material under the conditions prescribed in Table 1.

3. Definitions

3.1 *rate of water vapor transmission, WVT*—of a body between two specified parallel surfaces— the time rate of water vapor flow normal to the surfaces, under steady conditions, through unit area, under the conditions of

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[1] These methods are under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials.

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[2] *Annual Book of ASTM Standards*, Part 18.

test. An accepted unit of WVT is 1 g/24 h · m.^{2} The test conditions must be stated.

3.2 *water vapor permeance*—of a body between two specified parallel surfaces—the ratio of its WVT to the vapor pressure difference between the two surfaces. An accepted unit of permeance is a metric perm, or 1 g/24 h · m^2 · mm Hg. Since the permeance of a specimen is generally a function of relative humidity and to a lesser extent temperature, the test conditions must be stated.

3.3 *water vapor permeability*—the product of its permeance and thickness. This property may vary with conditions of exposure. An accepted unit of permeability is a metric perm-centimeter, or 1 g/24 h · m^2 · mm Hg · cm of thickness. Since the permeability of most materials is a function of relative humidity and to a lesser extent temperature, the test conditions must be stated.

NOTE 2—The definition does not imply that permeance is inversely proportional to thickness when the material is not fully homogeneous.

3.4 The following U.S. customary units are also widely used:

For WVT: 1 grain/h · ft^2.

For permeance: 1 perm, or 1 grain/h · ft^2 in. Hg.

For permeability: 1 perm-inch, or 1 grain/h · ft^2 in. Hg · in. of thickness.

Metric-U.S. customary conversion factors are stated in Table 2.

4. Significance

4.1 The purpose of these tests is to obtain, by means of simple apparatus, reliable values for the rate of water vapor transmission of sheet materials, expressed in suitable units. Correlation of test values with any given use must be determined by experience.

4.2 In any application, the vapor transmission of a sheet is determined by its permeance under the given conditions. With many materials the permeance of a sheet increases markedly when the relative humidity on either or both sides is raised. In contrast, the effect of a moderate change of temperature is quite small. Consequently, Procedures A and C, each with an average relative humidity of 25 percent, yield about the same permeance. Procedures B and D, with 75 percent average relative humidity, yield a different pair of similar values. The broad range of relative humidity provided by these two pairs of procedures (sometimes referred to as the desiccant and water methods, respectively) indicates in a practical way the permeance of a given sheet in many applications.

4.3 Procedure E provides the highest WVT, but the permeance generally falls between the values obtained by the desiccant and water methods. Although it has special applications, it is generally not as revealing as the water method for severe relative humidity exposures.

4.4 In Procedure BW, the water cup is inverted to wet the specimen, but otherwise it is the same as Procedure B. For some specimens, capillary transfer will be increased. It is intended specifically for those applications where wetting of one surface over extended periods is anticipated.

4.5 The report of a test result as a permeance value (rather than WVT) largely eliminates a fault in the test space conditions allowed in the tolerances stated in 5.2, provided the actual vapor pressure in the space is properly determined and used in the calculation as required in 15.2. Also, the permeance value of a sheet is a rational basis for evaluating its performance and comparing various sheets for a given application. Therefore, values of vapor permeance both in specifications and in reporting test results are to be encouraged.

4.6 The calculation of permeability (permeance × thickness) is also logical when the sheet is unquestionably homogeneous, with smooth surfaces and uniform thickness. The value so obtained can be used for calculating the permeance of a different thickness of like material in the same exposure. In Table 2 permeability units and metric-U.S. customary conversion factors are shown, but no report of the permeability is required.

5. Apparatus

5.1 *Test Dishes*—Open-mouth dishes shall be of such size or shape that they can be accommodated readily on the pan of an analytical balance. The dishes shall be constructed of a noncorroding, nonpermeable material, and shall be as light as is consistent with the necessary rigidity. The area of the opening shall be as large as practical and at least 30 cm^2. The area of the desiccant or water in the dish shall be at least equal to the exposed area of the specimen (see Procedures A, B, BW, C, D, and E, Sections 9 to 14). There shall be no obstructions within the dish that would restrict the flow of water vapor between the specimen area and the water or desiccant in the dish. For examples of suitable dishes and methods of sealing the specimen to the test dishes, see the Appendix.

5.2 *Test Chambers:*

5.2.1 The room or cabinet where the assembled test dishes are to be placed shall have controlled temperature and humidity which are measured frequently or preferably recorded continuously. The air conditions at any test location shall be one of those shown in Table 1 and shall be held constant within temperature limits of ±0.6 C (1 F) and relative humidity limits of ±2 percemt.

NOTE 3—These tolerances permit a range of vapor pressure up to 11 percent of nominal (in Procedures A and B) when temperature and relative humidity peaks occur together. This variation can be accepted only when control cycles are short and the mean vapor pressure is measured with due regard for such variation.

5.2.2 Air shall be continuously circulated throughout the chamber with a velocity sufficient to maintain uniform conditions at all test locations. Its velocity over the specimen in meters per minute shall be (numerically) not less than five times the permeance of the specimen, in metric perms (fpm = 10 times perms), but not over 150 m (500 ft)/min. Suitable racks shall be provided on which to place the test dishes within the test chamber.

NOTE 4—If the dishes are to be placed on the racks in an inverted position, care must be taken that the specified air velocity over the surfaces of the specimens is maintained.

5.3 *Balance and Weights*—The sensitivity of the balance shall be not less than 1 percent of the weight change during the period when a steady state is considered to exist. The weights used shall be accurate to 1 percent of the weight change during the steady-state period. A light wire sling may be substituted for the usual pan to accommodate a larger and heavier load.

5.4 *Weighing Covers*—Weighing covers for the test dishes shall be provided if the dishes have to be removed from the test room or cabinet for weighing (see the Appendix).

5.5 *Template*—A template may be used for defining the test area and effecting the wax seal (see the Appendix).

6. Materials

6.1 *Sealant*—The sealant used for attaching the specimen to the dish, in order to be suitable for this purpose, must be highly resistant to the passage of water vapor (and water). It must not lose weight to, or gain weight from, the atmosphere in an amount, over the required period of time, that would affect the test result by more than 2 percent. It must not affect the vapor pressure in a water-filled dish. Molten wax or asphalt is required for specimens having a permeance of less than 2.5 metric perms. The sealant shall be so applied that the test area of the specimen is well-defined and the same on both sides, and that no leakage of water vapor can occur at or through the edges of the specimen.

6.2 *Desiccant (for Procedures A, C, and E)*—A desiccant having a high affinity for water vapor and a high drying efficiency, that is, giving a low water vapor pressure after absorbing a large amount of water. The desiccant shall remain essentially unchanged in physical condition and exert no chemical or physical action, other than dehydration effects, on test specimens with which it is in contact. A suitable desiccant is anhydrous calcium chloride in the form of small lumps that will pass a No. 8 (2.36-mm) sieve, and free of fines that will pass a No. 30 (600-μm) sieve.[3] It shall be dried at 200 C (392 F) before use and the moisture gain during the test shall be limited to 10 percent of its starting weight. If $CaCl_2$ will react chemically on the specimen, an absorbing desiccant such as silica-gel, activated at 200 C, may be used but the moisture gain by this desiccant during the test shall be limited to 4 percent.

7. Sampling

7.1 The material shall be sampled in ac-

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[3] Detailed requirements for these sieves are given in ASTM Specification E 11, for Wire-Cloth Sieves for Testing Purposes, which appears in the *Annual Book of ASTM Standards*, Parts 14, 18, 26, 30, and 41.

cordance with standard methods of sampling applicable to the material under test. The sample shall be selected so as to represent the material to be tested, and shall be of uniform thickness. If the material is of nonsymmetrical construction, the two faces shall be designated by distinguishing marks (for example, on a one-side-coated sample, "I" for the coated side and "II" for the uncoated side).

8. Test Specimens

8.1 Test specimens shall be representative of the sample. When the faces of a specimen are indistinguishable, at least three specimens shall be tested by the same method. When the material is designed for use in one position only, at least three specimens shall be tested by the same method with the vapor flow in the designated direction. When the material is in ready-to-use form and may be used with either face toward the vapor source, at least four specimens shall be tested by the same method, two being tested with the vapor flow in each direction and so reported. Great care shall be taken not to contaminate the test area of the specimen.

NOTE 5—When testing a low-permeance material that may be expected to lose or gain weight throughout the test (because of its evaporation or oxidation), it may be advisable to provide an additional specimen or "dummy" tested exactly like the other except that no desiccant or water is put in the dish.

8.2 The thickness of each specimen shall be measured at the center of each quadrant to the nearest 0.025 mm (0.001 in.) and the results averaged.

9. Procedure A. Desiccant Method at 23 C (73.4 F)

9.1 Fill the dish with desiccant to within 6 mm of the specimen. Leave enough space so that shaking of the dish, which must be done at each weighing with the dish in the upright position, will mix the desiccant. The depth of the desiccant shall be at least 12 mm. Seal the specimen to the opening of the dish in such a manner that leakage of water vapor at and through the edges is prevented. (See the Appendix for examples of suitable methods of sealing.)

9.2 Weigh the assembly on the analytical balance. Then place the assembly on a rack in a test chamber, as described in 5.2, and locate so that the conditioned air circulates over the exposed surface of the specimen with the specified velocity. The specimen may be placed in either the upright position or inverted so that the desiccant is in direct contact with the specimen. This latter position is preferred for specimens having a high rate of water vapor transmission, but care must be taken to ensure that the seal is not broken or the surface of the specimen damaged.

9.3 Make successive weighings of the assembly at suitable intervals until a constant rate of gain is attained. Weighing should be accomplished without removal of the test dishes from the controlled atmosphere; if removal is necessary, the specimen must be tightly covered, and the weighing made immediately after removal with the assembly returned to the test chamber immediately after each weighing. Plot the results as prescribed in 15.1. Terminate the test or change the desiccant before the water added to the desiccant exceeds the limits specified in 6.2.

10. Procedure B. Water Method at 23 C (73.4 F)

10.1 In this method it is intended to provide 100 percent relative humidity in the dish but to avoid direct wetting of the surface facing the water. This requires that the test be carried out with the dish in the upright position and that a certain distance be maintained between water and specimen. As a result, there will exist a layer of air between the surface of the water and the under surface of the specimen through which water vapor will diffuse at a rate that cannot be measured conveniently. The influence of this air layer will become apparent only on specimens of a high rate of water vapor transmission; in this case the relative humidity on the under side of the specimen will be less than 100 percent; and the measured WVT will be slightly too low.

10.2 Place distilled water in the dish to bring the level of the water to such a height that the distance between the surface of the water and the under surface of the specimen is 20 ± 5 mm. A depth of 5 mm of water will adequately take care of losses by evaporation through the most permeable specimens. The depth shall be not less than 3 mm to ensure coverage of the dish bottom throughout the test. The possibility of splashing may be reduced by the use of a noncorroding grid in the dish to break the water surface. This grid shall be at least 6 mm below the specimen and it shall not reduce the water surface by more than 10 percent.

NOTE 6—For any of the water methods, baking the empty dish and promptly coating its mouth with sealant before assembly is recommended. The water may be added most conveniently after the specimen is attached, through a small sealable hole in the dish above the water line.

10.2 Attach the specimen to the dish, and proceed with the weighing as prescribed for Procedure A in 9.2 and 9.3, except place the assembly on the test racks in the upright position. Small variations in the temperature to which the dish and specimen might be exposed can result in condensation of water vapor on the underside of the specimen. Such condensation can significantly affect the measured water vapor transfer rate of some materials and is to be avoided. For this reason weighing should be carried out within the test chamber. If removal is necessary, the specimen must be tightly covered. The dishes shall not be exposed to a temperature that differs more than 3 C from the cabinet temperature to minimize condensation on the specimen. Plot results as prescribed in 15.1.

11. Procedure BW. Inverted Water Method at 23 C (73.4 F)

11.1 This is similar to Procedure B except that the dish is inverted, so that the water is in contact with the specimen surface at all times during the test.

11.2 Place distilled water in the dish to a depth of not less than 5 mm and not greater than 10 mm.

11.3 Attach the specimen to the dish and proceed with the weighing as prescribed for Procedure A in 9.2 and 9.3, placing the dish on the test racks in an inverted position. The dish must be sufficiently level so that water covers the inner surface of the specimen despite any distortion of the specimen due to the weight of the water. With highly permeant specimens it is especially important to locate the test dish so that air circulates over the exposed surface at the specified velocity. The test dishes may be placed on the balance in the upright position for weighing, but the period during which the wetted surface of the specimen is not covered with water must be kept to a minimum.

12. Procedure C. Desiccant Method at 32.2 C (90 F)

12.1 Follow Procedure A (Section 9), except place the assembly in a chamber maintained at 32.2 C (90 F) as prescribed in 5.2.2.

13. Procedure D. Water Method at 32.2 C (90 F)

13.1 Follow Procedure B (Section 10), except place the assembly in a chamber maintained at 32.2 C (90 F) as prescribed in 5.2.2.

14. Procedure E. Desiccant Method at 37.8 C (100 F)

14.1 Follow Procedure A except place the assembly in a chamber maintained at 37.8 C (100 F) and 90 percent relative humidity, as prescribed in 5.2.2.

NOTE 7—The use of weighing covers is recommended, particularly with this procedure, since weighings will have to be made in a room whose humidity and temperature will differ from the conditions in the cabinet.

15. Plotting and Calculation

15.1 Plot the results of successive weighing against elapsed time and draw a smooth curve through the plotted points. Judgment is required here and therefore numerous points are helpful. When a straight line adequately fits the plot of four properly-spaced, successive points (with due allowance for weighing errors), a nominally steady state exists, and the slope of the straight line is the rate of vapor transmission for the test area.

NOTE 8—The time required to reach the steady state will depend upon the permeance, thickness, hygroscopicity, and initial moisture content of the specimen. For specimens of very low permeance, buoyancy effects due to changes in barometric pressure may cause a greater change in measured weight between weighings than that due to moisture transfer. Long periods of test are required to obtain significant results in these instances.

15.2 Calculate the water vapor transmission (WVT) and permeance as follows:

$$\text{Water vapor transmission (WVT)} = (g \times 24)/(t \times a)$$

where:

g = weight gain or loss, g,

t = time, during which gain or loss, g, was observed, h,

a = exposed area of specimen, m^2, and

WVT = rate of water vapor transmission, $g/m^2 \cdot 24$ h.

$$\text{Permeance} = \text{WVT}/\Delta P = \text{WVT}/[S(R_1 - R_2)]$$

where:

ΔP = vapor pressure difference, mm Hg,

S = saturation vapor pressure, at test temperature, mm Hg,

R_1 = relative humidity at the source,

R_2 = relative humidity at the sink, and

Permeance = WVT/mm Hg expressed in metric perms.

In the controlled chamber the relative humidity and temperature are the average values actually measured during the test and (unless continuously recorded) these measurements shall be made as frequently as the weight measurements. In the dish the relative humidity is nominally 0 percent for the desiccant and 100 percent for the water. These values are usually within 3 percent relative humidity of the actual relative humidity for specimens below 2.5 metric perms when the required conditions are maintained (no more than 10 percent moisture in $CaCl_2$ and no more than 25-mm air space above water).

15.3 When the test specimen is homogeneous (not laminated) and thick relative to pore dimensions, the average water vapor permeability, in metric perm-centimeters, may be calculated as follows:

$$\text{Average permeability} = \text{permeance (metric perms)} \times \text{thickness (centimeters)}$$

For thin specimens the accuracy of the results will depend greatly on the precision with which the average thickness is determined.

15.4 Other units and conversion factors are given in Table 2.

16. Report

16.1 The report shall include the following:

16.1.1 A description of the sheet tested, including identification of side I and side II. If indistinguishable, so state.

16.1.2 Thickness of the specimen tested and exposed area.

16.1.3 The following actual conditions of the test:

16.1.3.1 Procedure, whether A, B, BW, C, D, or E,

16.1.3.2 Measured temperature,

16.1.3.3 Measured relative humidity in the test spaces, and

16.1.3.4 State side of sheet, I or II, exposed to the test space.

16.1.4 WVT of each specimen in stated units as tested.

16.1.5 Average WVT of all specimens tested in each direction of vapor flow.

16.1.6 Permeance of each specimen in stated units under the test conditions.

16.1.7 The average permeance of all specimens tested in each direction of vapor flow.

17. Precision

17.1 Results obtained by any one procedure on several specimens from the same sample may differ as much as 10 percent from their average. Two significant figures in water vapor transmission or permeance will, therefore, usually suffice to characterize the sample. Nevertheless, careful attention to all aspects of the procedure is required in order to obtain test results of acceptable precision.

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