KENYA STANDARD

STRETCH CLING FILM — SPECIFICATION

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REVISION OF KENYA STANDARDS

In order to keep abreast of progress in industry, Kenya Standards shall be regularly reviewed. Suggestions for improvements to published standards, addressed to the Managing Director, Kenya Bureau of Standards are welcome.

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STRETCH CLING FILM—SPECIFICATION

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Foreword

This first edition of this Kenya Standard was prepared by the technical committee on Household Products and endorsed by the Standards Projects Committee under the authority of the National Standards Council.

Cling film is used in sealing food items in containers to keep them fresh (for short time preservation).The standard addresses quality, safety and clinging capability and environmental and plasticizer issues. This standard has therefore covered the material requirements, clinging and stretching properties, environmental and safety concerns. The standard covers both perforated and non-perforated cling films.

During the preparation of this standard, reference was made to the following:

- IS 14995:2001 Stretch Cling Films – Specification
- GB 10457:2008 Plastic Cling Wrap Film for keeping food fresh
- ISO 2528:1995 Sheet materials - Determination of water vapour transmission rate - Gravimetric (dish) method
- ISO 15105-2:2003 Plastics — Film and sheeting — Determination of gas-transmission rate — Part 2: Equal-pressure method
- ASTM D882:2010 Standard Test Method for Tensile Properties of Thin Plastic Sheeting1

Acknowledgement is hereby made for the assistance derived from these sources.

KENYA STANDARD

Stretch Cling Film —— SPECIFICATION

1. Scope

This Kenya Standard specifies requirements, methods of sampling and test for stretch cling films. This standard covers stretch cling film for keeping food fresh which is made of resin such as polyethylene, polyvinylchloride, polyvinyl dichloride, processed by the method of monolayer or multilayer extrusion. It will cover perforated and non-perforated cling films.

2. Definitions

2.1 Cling film- Plastic cling wrap with ability to adhere to itself and made of resin to keep food fresh.
2.2 Polyethylene cling film - A type of food-use cling film made of polyethylene.
2.3 Polyvinyl chloride cling film - A type of food-use cling film made of PVC.
2.4 Polyvinyl dichloride cling film- A type of food-use cling film made of PVDC.
2.5 Multilayer extrusion cling film – A type of cling film processed by the method of multilayer extrusion.
2.6 Self cling – The self cling property of the cling film itself; also called shear and peel strength.
2.7 Open wrapping – the degree of difficulty of removing the film from the paper core during use.
2.8 Anti-fogging- The property of preventing a cling wrap film from forming any water drops or water fogging on the film surface.

3. Classification

Cling film shall be classified as follows:
a) On the basis of material where we have Polyethylene, Polyvinylchloride and Polyvinyl dichloride
b) Perforations: Perforated and non-perforated.

4. Requirements

4.1 Materials
4.1.1 Resin- The resin selected should be of food grade material and shall comply where necessary with KS 2322.
4.1.2 Additives: The additives selected and their dosages should meet the requirements set out in KS 2322
4.2 Colour- Cling film shall be transparent, normal natural colour
4.3 Dimension:
Thickness for

- non-perforated: 0.01mm0± 0.002
- perforated:

Width = 200 to 1000mm
4.4   Appearance: The material shall be uniform in colour, texture and finish. The material shall be free from streaks and foreign particles. There shall be no other visible defects such as perforations/holes (for non-perforated), tears or blisters. The edges shall be free from nicks and cuts visible to unaided eye, free from wrinkles, fold-overcreases and gels. There shall be no splices or air bubbles.
4.5   Odor, the film shall be free from any objectionable odor.
4.6   Tensile strength:

Table 1 Requirements

Item	Index		
	PE	PVC	PVDC
Tensile Strength (longitudinal, transverse) MPa	$\geq$ 10	$\geq$ 15	$\geq$ 60
Nominal Tensile strain at break (longitudinal, transverse) %	$\geq$ 120	$\geq$ 150	$\geq$ 50
Light Transmissivity, %	$\geq$ 90	$\geq$ 92	$\geq$ 90
Haze	$\leq$ 3	$\leq$ 2	$\leq$ 3
Deviation of gas transmission rate – Oxygen, %	$\pm$ 20		
Deviation of gas transmission rate – Carbon dioxide, %	$\pm$ 20		
Deviation of water vapour permeance, %	$\pm$ 20		
Self Cling (shear and peel strength), N/cm^2	$\geq$ 0.5		
Open wrapping	The sample shall be completely peeled off within 5 second		
Anti fogging	Under test conditions, no water drops shall adhere to the film surface.		

5.   Packaging

The material shall be packaged to provide adequate protection during normal handling and transportation and each package shall contain only one size of material The packaging shall be made of material free from contaminants, such as to maintain the integrity of the cling film. Film bags or boxes shall be used as sealed packaging.

6. Marking

Cling Film shall be marked legibly and indelibly with the following information:

i) Product name
ii) The name and/or registered trade mark of the manufacturer.
iii) Address of manufacturer or importer.
iv) "Made in Kenya" or country of origin where applicable
v) Recommended use
vi) Instructions for disposal
vii) Size in length by width
viii) Standardization mark
ix) Safety precautions e.g 'microwave safe' or 'microwave unsafe'

Annex A
Determination of Tensile Properties

Apparatus

A.1 *Testing Machine*—A testing machine of the constant rate-of-crosshead-movement type and comprising essentially the following:

A.1.1 *Fixed Member*—A fixed or essentially stationary member carrying one grip.

A.1.2 *Movable Member*—A movable member carrying a second grip.

A.1.3 *Grips*—A set of grips for holding the test specimen between the fixed member and the movable member of the testing machine; grips can be either the fixed or self-aligning type. In either case, the gripping system must minimize both slippage and uneven stress distribution.

A.1.3.1 Fixed grips are rigidly attached to the fixed and movable members of the testing machine. When this type of

grip is used, care must be taken to ensure that the test specimen is inserted and clamped so that the long axis of the test specimen coincides with the direction of pull through the center line of the grip assembly.

A.1.3.2 Self-aligning grips are attached to the fixed and movable members of the testing machine in such a manner that they will move freely into alignment as soon as a load is applied so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly. The specimens must be aligned as perfectly as possible with the direction of pull so that no rotary motion will cause slippage to occur in the grips; there is a limit to the amount of misalignment self-aligning grips will accommodate.

A.1.3.3 The test specimen shall be held in such a way that slippage relative to the grips is prevented insofar as possible. Grips lined with thin rubber, crocus-cloth, emery cloth, or pressure-sensitive tape as well as file-faced or serrated grips have been successfully used for many materials. The choice of grip surface will depend on the material tested, thickness, etc. Line grips padded on the round face with 0.75-1.00 mm (0.030-0.040 in.) blotting paper or filter paper have been found superior. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to "neck" into the grips, since pressure is maintained at all times (see Notes 1-7). In cases where samples frequently fail at the edge of the grips, it could be advantageous to slightly increase the radius of

curvature of the edges where the grips come in contact with the test area of the specimen.

NOTE 1—Caution needs to be taken when choosing the type of grips and the type of grip surfaces to use for testing specimens films composed of high strength LLDPE and VLDPE resins. Test results tend to differ more when comparing these types of specimens films tested with the grips lined with different materials.

NOTE 2—The gage of pressure sensitive tape, thin rubber, crocus-cloth, and emery cloth needs to be adequate enough to prevent slipping and premature failures of the test specimens (for example, pressure

sensitive tape is used on the surface of the grips: the test specimen can may begin to tear at the edge of the grips during the test if the tape is too thin.).

NOTE 3—The grit size of crocus-cloth and emery cloth is suggested to be at least 800. The use of these materials helps to prevent test specimens from slipping in the grips. One must be cautious when using these materials so that premature failures of the test specimens do not occur.

A.1.4 *Drive Mechanism*—A drive mechanism for imparting to the movable member a uniform, controlled velocity with respect to the stationary member.

A.1.5 *Load Indicator*—A suitable load-indicating mechanism capable of showing the total tensile load carried by the

test specimen held by the grips. This mechanism shall be essentially free of inertial lag at the specified rate of testing (see Note 4). Unless a suitable extensometer is used , the motion of the weighing system shall not exceed 2 % of the

specimen extension within the range being measured. The load indicator shall determine the tensile load applied to the specimen with an accuracy of 61 % of the indicated value, or better. The accuracy of the testing machine shall be verified.

A.1.6 *Crosshead Extension Indicator*—Asuitable extensionindicating mechanism capable of showing the amount of

change in the separation of the grips, that is, crosshead movement. This mechanism shall be essentially free of inertial lag at the specified rate of testing (see Note 4) and shall indicate the crosshead movement with an accuracy of 61 % of the indicated value, or better.

A.2 *Extensometer (Optional)*—A suitable instrument used for determining the distance between two designated points on the test specimen as the specimen is stretched. The use of this type of instrument is optional and is not required in this test method. This apparatus, if employed, shall be so designed as to minimize stress on the specimen at the contact points of the specimen and the instrument . It is desirable that this instrument automatically record the distance, or any change in it, as a function of the load on the test specimen or of the elapsed time from the start of the test, or both. If only the latter is obtained, load-time data must also be taken. This instrument must be essentially free of inertial lag at the specified speed of testing (see Note 4).

A.2.1 *Modulus of Elasticity and Low-Extension Measurements*—Extensometers used for modulus of elasticity

and low-extension (less than 20 % elongation) measurements shall, at a minimum, be accurate to 61 % and comply with the requirements set forth in Practice E83 for a Class C instrument.

A.2.2 *High-Extension Measurements*—Instrumentation and measuring techniques used for high-extension (20 % elongation or greater) measurements shall be accurate to 610 % of the indicated value, or better.

NOTE 4—A sufficiently high response speed in the indicating and recording system for the load and extension data is essential. The response speed required of the system will depend in part on the material tested(high or low elongation) and the rate of straining.

A.3 *Thickness Gauge*—A dead-weight dial or digital micrometer appropriate for the material or specimen geometry being tested.

A.4 *Width-Measuring Devices*—Suitable test scales or other width measuring devices capable of measuring 0.25 mm (0.010 in.) or less.

A.5 *Specimen Cutter*

A.5.1 Devices that use razor blades have proven especially suitable for materials having an elongation-at-fracture above 10 to 20 %.

A.5.2 The use of a punch press or a striking die is not recommended because of their tendency to produce poor and inconsistent specimen edges.

A.5.3 The use of a cutting template and a single razor blade is not recommended as it will affect the parallelism of the test specimen.

A.6 Test Specimens

The test specimens shall consist of strips of uniform width and thickness at least 50 mm (2 in.) longer than the grip separation used. The nominal width of the specimens shall be not less than 5.0 mm (0.20 in.) or greater than 25.4 mm (1.0 in.). A width-thickness ratio of at least eight shall be used. Narrow specimens magnify effects of edge strains or flaws, or both. 6.4 The utmost care shall be exercised in cutting specimens to prevent nicks and tears that cause premature failures (seeNote 5). The edges shall be parallel to within 5 % of the width over the length of the specimen between the grips.

NOTE 5—Microscopical examination of specimens can be used to detect flaws due to sample or specimen preparation. Test specimens shall be selected so that thickness is uniform to within 10 % of the thickness over the length of the specimen between the grips in the case of specimens 0.25 mm (0.010 in.) or less in thickness and to within 5 % in the case of specimens greater than 0.25 mm (0.010 in.) in thickness but less than 1.00 mm (0.040 in.) in thickness.

NOTE 6—In cases where thickness variations are in excess of those recommended above, results tend not to be characteristic of the material under test.

If the material is suspected of being anisotropic, two sets of test specimens shall be prepared having their long axes respectively parallel with and normal to the suspected direction of anisotropy.

For tensile modulus of elasticity determinations, a specimen gage length of 250 mm (10 in.) shall be considered as standard. This length is used in order to minimize the effects of grip slippage on test results. When this length is not feasible, test sections as short as 100 mm (4 in.) can be used if it has been shown that results are not appreciably affected. However, the 250-mm (10-in.) gage length shall be used for reference purposes. The speed of testing of shorter specimens must be adjusted in order for the strain rate to be equivalent to that of the standard specimen.

NOTE 7—Two round robin tests4 have shown that, for materials of less than 0.25-mm (0.010 in.) in thickness, line grips padded on the round side with 1.0-mm (0.040-in.) blotting paper give the same results with a 100-mm (4-in.) test section as a 250-mm (10-in.) test section produces with flat-face grips.

NOTE 8—Excessive jaw slippage becomes increasingly difficult to overcome in cases where high modulus materials are tested in thicknesses greater than 0.25 mm (0.010 in.).

A.7. Conditioning

A.7.1Conditioning—Condition the test specimens at 23 ± 2℃ (73.4 ± 3 .6℉) and 50 6 10 % relative humidity for not less than 40 h prior to test. In cases of disagreement, the tolerances shall be 61℃ (61.8℉) and 65 % relative humidity.

A.7.2 *Test Conditions*—Conduct the tests at 23 ± 2℃ (73.4 ± 3.6℉) and 5 0 ± 10 % relative humidity unless otherwise specified by agreement or the relevant ASTM material specification. In cases of disagreement, the tolerances shall be ±1℃ (±1.8℉) and ±5 % relative humidity.

A.8. Number of Test Specimens

A.8.1 In the case of isotropic materials, at least five specimens shall be tested from each sample.

A.9. Procedure

A.9..1 Select a load range such that specimen failure occurs within its upper two thirds. A few trial runs could be necessary to select a proper combination of load range and specimen width.

A.9.2 Measure the cross-sectional area of the specimen at several points along its length. Measure the width to an accuracy of 0.25 mm (0.010 in.) or better. Measure the thickness to an accuracy of 0.0025 mm (0.0001 in.) or better for specimens less than 0.25 mm (0.010 in.) in thickness and to an accuracy of 1 % or better for specimens greater than 0.25 mm (0.010 in.) but less than 1.0 mm (0.040 in.) in thickness.

A.9.3 Set the rate of grip separation to give the desired strain rate, based on the initial distance between the grips, Zero the calibrated load weighing system, extension indicator(s) and recording system.

NOTE 9—Extensometers can be used for modulus of elasticity determinations with the expectation of obtaining more accurate values than can be obtained using grip separation as the effective gage length. Precautions must be taken to ensure that extensometer slippage and undue stressing of the specimen do not occur.

A.9.4 In cases where it is desired to measure a test section other than the total length between the grips, mark the ends of the desired test section with a soft, fine wax crayon or with ink. Do not scratch these marks onto the surface since such scratches can act as stress raisers and cause premature specimen failure. Extensometers can be used if available; in this case, the test section will be defined by the contact points of the extensometer.

NOTE 10—Measurement of a specific test section is necessary with some materials having high elongation. As the specimen elongates, the accompanying reduction in area results in a loosening of material at the inside edge of the grips. This reduction and loosening moves back into the grips as further elongation and reduction in area takes place. In effect, this causes problems similar to grip slippage, that is, exaggerates measured extension.

A.9.5 Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize slipping of the specimen during test.
A.9.6 Start the machine and record load versus extension. When the total length between the grips is used as the test area, record load versus grip separation. When a specific test area has been marked on the specimen, follow the displacement of the edge boundary lines with respect to each other with dividers or some other suitable device. If a load-extension curve is desired, plot various extensions versus corresponding loads sustained, as measured by the load indicator.
A.9.7 When an extensometer is used, record load versus extension of the test area measured by the extensometer.
A.9.8 If modulus values are being determined, select a load range and chart rate to produce a load-extension curve of between 30 and 60° to the X axis. For maximum accuracy, use the most sensitive load scale for which this condition can be met. The test may be discontinued when the load-extension curve deviates from linearity.
A.9.9 In the case of materials being evaluated for secant modulus, the test can be discontinued when the specified extension is reached. 10.10 If tensile energy to break is being determined, some provision must be made for integration of the stress-strain curve. This can be either an electronic integration during the test or a subsequent determination from the area of the finished stress-strain curve
A.10. Calculation
A.10.1 *Breaking Factor* (nominal) shall be calculated by dividing the maximum load by the original minimum width of the specimen. The result shall be expressed in force per unit of width, usually newtons per metre (or pounds per inch) of width, and reported to three significant figures. The thickness of the film shall always be stated to the nearest 0.0025 mm (0.0001 in.).

Example—Breaking Factor = 1.75 kN/m (10.0 lbf/in.) of width for 0.1300-mm (0.0051-in.) thickness.

NOTE 11—This method of reporting is useful for very thin films (0.13 mm (0.005 in.) and less) for which breaking load is not proportional to cross-sectional area and whose thickness is difficult to determine with precision. Furthermore, films which are in effect laminar due to orientation, skin effects, nonuniform crystallinity, etc., have tensile properties disproportionate to cross-sectional area.

A.10.2 *Tensile Strength* (nominal) shall be calculated by dividing the maximum load by the original minimum crosssectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures. The maximum load can occur at the yield point, the breaking point, or in the area between the yield point and the breaking point.

NOTE 12—When tear failure occurs, so indicate and calculate results based on load and elongation at which tear initiates, as reflected in the load-deformation curve.

A.10.3 *Tensile Strength at Break* (nominal) shall be calculated in the same way as the tensile strength except that the load at break shall be used in place of the maximum load (Note 12 and Note 13).
NOTE 13—In many cases tensile strength and tensile strength at break are identical, but not always.
A.10.4 *Percent Elongation at Break* shall be calculated by dividing the extension at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation; otherwise the distance between the grips shall be used. The result shall be expressed in percent and reported to two significant figures (see Note 12).
A.10.5 *Tensile Yield Strength*, where applicable, shall be calculated by dividing the load at the yield point by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures. Alternatively, for materials that exhibit Hookean behavior in the initial part of the curve, an offset yield strength shall be obtained. In this case the value shall be given as "yield strength at —% offset."
A.10.6 *Percent Elongation at Yield*, where applicable, shall be calculated by dividing the extension at the yield point by the initial gage length of specimen and multiplying by 100. When gauge marks or extensometers are used to define a specific test section, only this length shall be used in the calculation. Before calculating, correct the extension for "toe compensation". The results shall be expressed in percent and reported to two significant figures. When offset yield strength is used, the elongation at the offset yield strength shall be calculated.
A.10.7 *Elastic Modulus* shall be calculated by drawing a tangent to the initial linear portion of the load-extension curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. Before calculating, correct the extension for "toe compensation" . For purposes of this determination, the tensile stress shall be calculated by dividing the load by the average original cross section of the test section. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch), and reported to three significant figures.
A.10.8 *Secant Modulus*, at a designated strain, shall be calculated by dividing the corresponding stress (nominal) by the designated strain. Elastic modulus values are preferable and shall be calculated whenever possible. However, for materials where no proportionality is evident, the secant modulus values shall be calculated. The stress to be used in the calculation is then determined by dividing the load at the designated strain on the load-extension curve by the original average cross-sectional area of the specimen.

Annex B
Determination of Self Cling (Shear and Peel Strength)

B.1   Sample preparation
Cut 10 sheets of sample 50 mm long and 25 mm wide, with every two sheets forming a set. Place the adhesive sides of each set of samples face to face in a lap joint, the length of a lap joint being 15mm and

the width of the lap joint being 25mm. Lay the samples on a smooth surface, use a rubber roller (diameter of 40mm, length of 100mm, mass of 300g) and carry out reciprocation rolling and press on the lap joint three times, until there is no air left between the lap jointed layers of cling wrap film. Leave the prepared samples in an environment under test conditions for 20 minutes and then conduct the test.

B.2 Procedure

Stretch out each set of the samples on a tension machine and determine the force needed to separate the two sheets of sample. The arithmetic mean value of the five sets shall be regarded as the result. The stretching velocity, using an appropriate test instrument, shall be 250± 50 mm/min. The self-cling (shear and peel strength) shall be determined by the formula

$$T = \frac{P}{a \times b}$$

Where
T – The self cling (shear and peel strength), in Newton/centimetre^2 (N/cm^2)
P - The force needed to separate the sample, in Newtons N
b -The width of the lap joint in cm
a - The length of the lap joint in cm

Annex C
Determination of the open-wrapping

C.1 Sample preparation

Cut 6 sheets of sample 150mm long and 50mm wide, with every two sheets forming a set. Place the adhesive sides of each set of samples face to face in a lap joint, the length of the lap joint being 100mm.

C.2 Procedure

Fix one end of the sample and at the other end of the sample fix a 4g weight using the self-cling of the cling wrap film or a piece of adhesive paper. Let the weight down slowly and allow the sample to peel naturally. All three sample sets shall meet the property requirements.

Annex D
Determination of the anti-fogging

Add 200ml of water at a temperature of 23°C ± 2^{0}C into each of three beakers with a volume of 1000ml. Use three equal sized pieces of cling wrap film, with the adhesive side facing down, to completely cover each of the beaker mouths. Ensure the film surfaces are flat, and put the three beakers into a low temperature tank at 3°C to maintain a constant temperature. After 10 minutes, check the adhesive state of the water drops on the surface of the cling wrap film. All three samples shall meet the property requirements.

Annex E
Determination of water vapour transmission rate – Gravimetric (dish) method

Water Vapour Transmission Rate (WVTR) is the mass of water vapour transmitted through a unit area in a unit time under specified conditions of temperature and humidity. It is expressed in grams per square metre per 24 h [g/(m^2 ·d)].

NB; The WVTR depends upon the thickness, composition and permeability of the constituent material or materials and upon the conditions of temperature and relative humidity under which the test is carried out

E.1 Principle
Dishes containing a desiccant and closed by the material to be tested are placed in a controlled atmosphere These dishes are weighed at suitable intervals of time and the WTR is determined from the increase in mass when this increase has become proportional to the time interval.

E.2 Apparatus and material
Figure 1 shows examples of equipment which has proved satisfactory in use, but other equipment may be equally satisfactory.

E.2.1 **Test dishes**, shallow, of glass, aluminium or stainless steel and of as large a diameter as can be accommodated on the balance to be used. The dishes should be light, but rigid and resistant to corrosion under the test conditions. Dishes made from aluminium, grade Al 99,5 and protected by chemical or anodic oxidation have been found suitable. Each dish has a groove around the rim for sealing the test piece with wax. This groove has a profile such that the test piece can be sealed over the opening of the dish and no water vapour can escape at or through the edges of the test piece. The internal diameter of the dish shall be equal to or very slightly larger than the diameter of the waxing templates. The internal depth of the dish below the plane of the test piece should be not less than 15 mm (deep dish) or 8 mm (shallow dish) and there shall be no obstruction within the dish that might interfere with the flow of water vapour between the test piece and the desiccant. The surface area of the bottom of the dish where it is filled with desiccant shall be similar to that of the exposed surface of the test piece. Each dish shall be assigned a different number.

E.2.2 **Lids**, each numbered to correspond with a dish and made from the same material as the dish, with an outer rim designed to fit neatly over the outside of the dish so that there is negligible loss of water vapour when the dishes are removed from the test atmosphere for weighing.

E.2.3 **Waxing templates**, to place the wax easily and to allow the test area to be defined sealant exactly. Their diameter, D, should preferably be 79,8 mm + 0,4 mm (an area of - 50 cm*). If any other diameter of template is used, this fact shall be mentioned in the test report. In no case shall the diameter be less than 56,l mm, and shall be known to an accuracy better than 1 %. These templates may be either:
a) Cross-braced ring templates, which remain in place during the test. Their diameter, D, is the internal diameter of the ring. As many ring templates as dishes are required;
or
b) Cover templates, which must be taken off when the applied wax has cooled, comprising a disc with a central handle, drilled with a small hole at a suitable point (see figure I), and having the edge chamfered at an angle of approximately 45". Their diameter, D, is the diameter of this smaller circle. Small guides can be fixed to the template to centre it automatically. A few templates are sufficient.

E.2.4 **Sealant**, a wax mixture (see annex C) which adheres strongly to both the dish and the test piece and is not brittle at ordinary temperature, not hygroscopic and not susceptible to oxidation. A surface of 50 cm* of freshly melted wax when exposed for 24 h in condition B (see annex B) shall not change in mass by more than 1 mg.

E.2.5 **Water bath**, for melting the wax.

E.2.6 **Device for distributing the wax**, of at least 25 ml capacity and a rapid rate of discharge, such as a pipette with a discharge tube of about 3 mm i.d. or a metal pourer with an insulated handle.

E.2.7 **Cutting template** or test-piece cutter, of a size suitable for cutting circular test pieces of a diameter suitable for the dishes in use (see figure 1). This diameter is slightly less than the inside diameter of the top of the dish (see figure2).

E.2.8 **Desiccant**, silica gel or anhydrous calcium chloride (CaCl,), in the form of granules l,6 mm to 4 mm in size or alternatively in the form of a friable flaked product l,5 mm to 2,0 mm in size.

NB: The limiting saturation of 1 g of calcium chloride is 0,l g of water. The limiting saturation of 1 g of silica gel is 0,04 g of water.
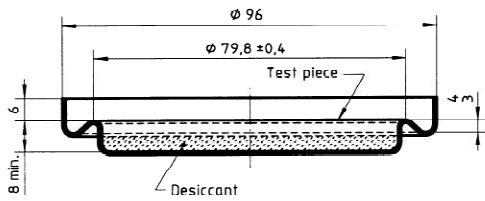
E.2.9 **Balance**, for determining the mass of each dish, lid and contents to 0,l mg
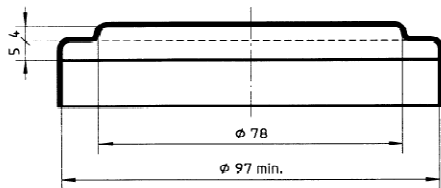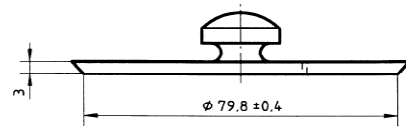
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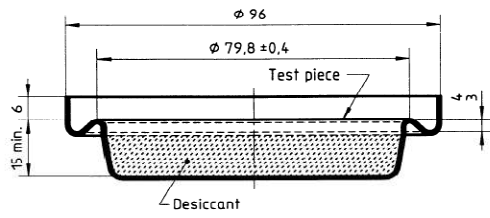
Deep dish (5.1) for materials with transmission rate greater than 100 g/(m²·d), (tapered to nest)
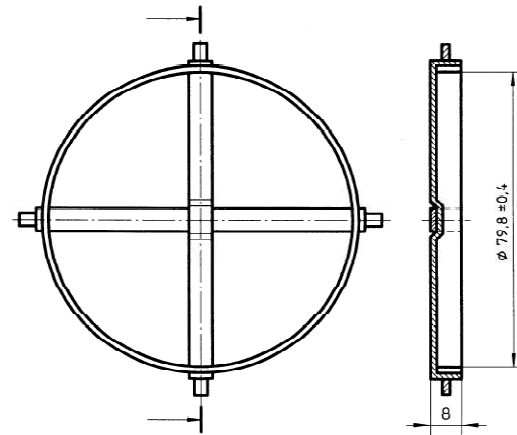
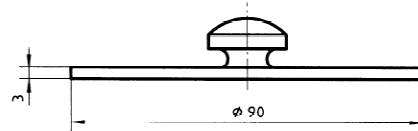Shallow dish (5.1) for materials with normal transmission rate

Lid (5.2) with rim to fit outside dish

Cover template (5.3 b)]

Ring template (5.3 a)]

Cutting template (5.7)

NOTES

1 Dimensions are shown for test areas of 50 cm². Values for dishes and lids show inside dimensions, except the overall diameter of the dishes, which is an outside dimension.

2 Only the dimension 79,8 mm + 0,4 mm shall be strictly respected; the other dimensions are approximate.
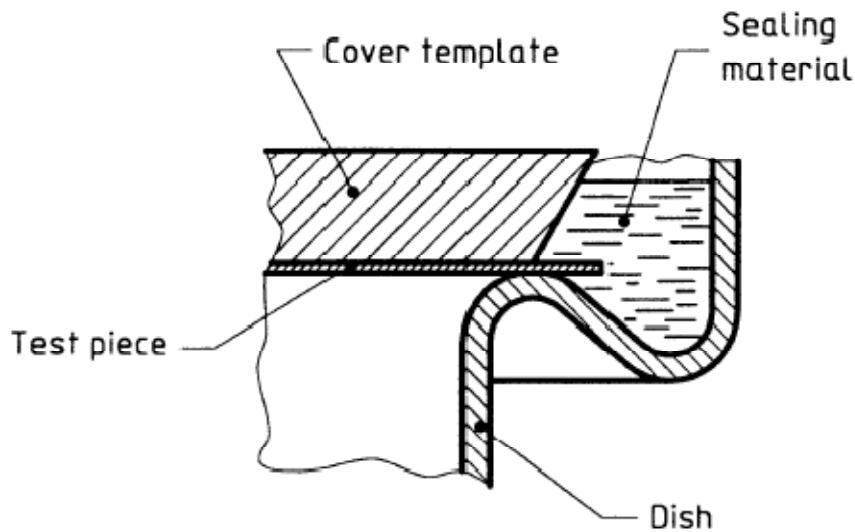
Figure 1 — Examples of test dishes and templates

Figure 2 — Detail of sealing of test piece

E.2.10 **Tongs**, holders or other means of manipulating the dishes.

E.2.11 **Enclosure**, in which the required controlled atmosphere can be set (see annex B) and with air continuously circulated. The control shall be such that the specified conditions are re-established not more than 15 min after the door of the enclosure has been closed.

E.3 Preparation of test pieces

Avoiding all damaged areas, cut from the sample, with the aid of the cutting template or test piece cutter at least three circular test pieces of the appropriate diameter, normally 90 mm (see figure I), for each face to be tested. Mark the test pieces in some way so that the side to be exposed to the test atmosphere can be readily identified.

E.4 Preparation of dishes

The method of preparation of the dishes differs slightly according to whether a cover or ring template is used. Always begin by carefully cleaning and drying the dishes and the templates. Introduce the desiccant into the dish then put the test piece (clause 8) on the dish with the required face upwards and then the waxing template, and make a vapour-tight wax seal between the test piece and the dish. The work must be done rapidly in order to keep the absorption of water vapour by the desiccant to a minimum. WARNING - Care should be taken when handling hot wax, as serious burns could occur if the wax is spilled or splashed. Suitable protective equipment such as glasses, gloves, etc. should be worn.

E.4.1 **Use of wax and a cover template**

Fill each dish with desiccant up to 3 mm to 4 mm below the final position of the test piece and level by tapping. Melt the wax on the water bath and fill the dispensing device. Place the test piece centrally in position, followed by the waxing template. Run the molten wax into the groove until it reaches the level of the upper surface of the waxing template and, after cooling, complete the joint by removing air bubbles and hair cracks with a small gas flame. A warm spatula may be run over the wax to assist in this process, so that shrinkage cracks that may have developed during cooling will be closed. Remove the waxing template and examine the assembly to make sure that the joint is satisfactory. To ensure that the waxing template comes away easily, it is advisable first to smear a thin film of petroleum jelly around the edge and to wipe away any excess which could contaminate the test piece.
Cover the assembly with a lid numbered to correspond with the number of the dish.

E.4.2 **Use of wax and a ring template**
Fill each dish with desiccant up to a level of 3 mm to 4 mm below the final position of the test piece and level by tapping. Melt the wax on the water bath and fill the dispensing device. Run the molten wax into the circular groove round the dish until a slight meniscus is produced above the inner edge of the groove. Place the test piece centrally in position on the dish, followed by the ring template, and load it with a 1 kg weight. Run more wax into the annular space so formed and, after cooling, complete the joint by emoving any air bubbles and hair cracks with a small gas flame. A warm spatula may be run over the wax to assist in this process, so that shrinkage cracks that may have developed during cooling will be closed. Remove the weight and leave the ring in place. Cover th e assembly with a lid numbered to correspond with the number of the dish.

E.5 **Procedure**

E.5.1 **General method**
Weigh all the prepared dishes, with their lids, on the balance to the nearest 0,l mg. Place them upright in the enclosure controlled to the conditions of the test after removing the lids. Carry out successive weighings of the dishes, with their lids, at suitable intervals of time. The weighings shall be carried out as follows:
Cover the dishes with their respective lids and remove them from the controlled enclosure using the tongs or holders and leave them for 15 min to reach ambient temperature. Weigh the assemblies to the nearest 0,l mg, and return them to the enclosure after again taking off the lids. Take care to work rapidly, taking the dishes in small groups always containing the same number, so that the whole weighing operation always lasts about the same time (not exceeding 30 min). It is also possible to work without the lids, but in this case it is advisable to use blank assemblies and transport and cooling of the dishes must be done in a closed vessel with calcium chloride desiccant.
The interval between weighings should preferably be 24 h, 48 h or 96 07, but shorter time intervals (for example 3 h, 4 h or 8 h) may be necessary for materials with a high transmission rate. The choice depends on the transmission rate of the sheet being tested; the gain in mass between two successive weighings should be at least 5 mg. The choice of time interval is to be made at the beginning of the test. If the first weighing shows a gain in mass too large or too small, the subsequent time intervals for weighing may be modified. Continue the weighings until the increase in mass of two successive weighings per unit time of exposure to the selected atmosphere becomes constant to within 5 %.
The test must be completed before the efficiency of the desiccant is appreciably reduced. (In practice, the total increase in mass should not exceed I,2 g for shallow dishes and 3,2 g for deep ones.)

E.5.2 **Use of blank assemblies**
If the sample is of low transmission rate and considerable thickness, for example rubber, plastics or polyethylene-coated board, or is appreciably hygroscopic, it is advisable to test two or more blank assemblies, prepared in the same manner but without desiccant, in addition to the three normal test assemblies. All the measured masses are then corrected at each time interval by subtracting the mean change in mass of the blank assemblies which undergo the same treatment.

E.6 Expression of results
 For each dish, represent the total increase in mass graphically as a function of time of exposure, the test being completed when three or four points lie on a straight line showing a constant rate of passage of water vapour. Using this straight line, the WVTR for each test piece is then calculated, in grams per square metre per 24 h, from the formula

(i)

$$\frac{240 \times m_1}{S}$$

where
m_1 is the rate of increase in mass, in milligrams per hour, determined from the graph;

S is the area, known to within 1 %, in square There is insufficient data available at this time to allow centimetres (normally 50 cm'), of the tested surface of the test piece.

If weighings are made at identical time intervals, it is possible to calculate the transmission rate for each test piece directly from the results, without preparing a graph, by using the formula below

(ii)

$$\frac{240 \times m_2}{s \times t}$$

where
t is the total duration, in hours, of the last two exposure periods
m_2 is the increase in mass, in milligrams, of the assembly during the time t.
For several assemblies corresponding to a single sample of test material and to a single face, calculate the arithmetic mean of the results obtained in accordance with either formula (i) or (ii) above in E.6.
Report the mean WTR by rounding values over 100 g/(m^2.d): to the nearest 10 g/(m^2.d);
values from 10 g/(m^2.d) to 100 g/(m^2.d): to the nearest whole number; values less than 10 g/(m^2.d): to the first decimal place.

Annex F
Determination of gas-transmission rate —
Equal-pressure method (Oxygen and carbon dioxide)

F.1 Principle

A test specimen is mounted in a gas-transmission cell (see Figures 3 and 4) so as to form a sealed barrier between two chambers. One chamber is slowly swept with a carrier gas. The second chamber is fed with the test gas. The total pressure is identical (atmospheric) in each chamber but, since the partial pressure of the test gas is higher in the second chamber, the test gas permeates through the barrier into the carrier gas in the first chamber.

The test gas which permeates through the specimen is carried by the carrier gas to a sensor the nature of which will depend on the material under test and the test gas used.

F.2 Test specimens

Test specimens shall be representative of the material under investigation, free from shrivelling, folds and pinholes, and of uniform thickness. They shall be larger than the gas-transmission area of the measurement cell and be capable of being mounted airtight. Use three specimens unless otherwise specified or agreed upon among the interested parties. Mark the side of the material facing the permeating gas.

NOTE In principle, the test should replicate the actual conditions of use, with the permeating gas passing from the inside to the outside of e.g. packaging material, or *vice versa*.

Measure the thickness of each specimen in to the nearest 1 µm, at at least
five points distributed over the entire test area, and record the minimum, maximum and average values.

F.3 Conditioning and test temperature
F.3.1 Conditioning
The specimens shall be
☐☐☐either dried for not less than 48 h at the same temperature as that at which the test is to be carried out,
using calcium chloride or another suitable drying agent in a desiccator;
☐☐☐or conditioned at the temperature and humidity specified for the test for a length of time appropriate to the nature of the material under test.
F.3.2 Test temperature
Carry out the test in a room kept at 23 ℃ ☐☐2 ℃, unless otherwise specified.

F.4 Apparatus and materials
F.4.1 General
Figures 3 and 4 show examples of apparatus for determining gas-transmission rate. The apparatus consists of a gas-transmission cell designed to allow a test gas to permeate through a specimen, a sensor to measure the amount of test gas which has permeated through the specimen, and gas conditioning devices and flowmeters for the test gas and carrier gas.

F.4.2 Test gas
The test gas may be either a single gas, or a mixture of gases. In the first case, the pressure of the gas shall be equal to atmospheric pressure.
In the second case, the pressure of the gas shall also be atmospheric and the partial pressure of each individual component will be determined by its concentration in the mixture.

F.4.3 Transmission cell
The transmission cell is divided into two chambers by a test piece, cut from the sheet of material under test, to allow permeation of the test gas (see 1 in Figures 3 and 4).

The test gas flows, under defined conditions, into chamber A and leaves this chamber through a suitably dimensioned outlet at ambient atmospheric pressure.

A carrier gas flows through the other chamber (chamber B) of the cell, under defined conditions, such that the pressure difference between the two chambers is as low as possible to prevent distortion of the test piece.

The shape of the cell shall be such that the test specimen is swept on both sides by laminar gas flows. In particular, the ratio of the effective permation area to the volume of each chamber shall be such that the velocity of each gas is lower than a specified limit, depending on the shape of the cell.

The effective permation area shall be adapted according to the range of values to be determined. It is generally between 1 cm^2 and 150 cm^2.

The effective permation area of the test specimen may be reduced by means of a mask placed between the two chambers. In this case, the mask shall be glued on the test specimen and carefully checked for leaks, which would affect the measurements.

When the cell is fitted with gaskets, the material used for the gaskets shall have a gas-transmission rate which is negligible compared to that expected for the material under test.

F.5 Detection and measurement

There are two methods for measuring amount of gas permeating through a specimen.

A: coulometric sensor (for oxygen-transmission rate);
B: chromatographic sensor.

NOTE The method used will depend on:
- the permeability of the material to the test gas;
- the sensitivity required.

F.6 Diffusion conditions

Unless otherwise specified, the test gas and the carrier gas shall be conditioned under one of the sets of conditions of temperature and relative humidity given in Table 2.

Table 2 — Conditions for diffusion

Set of conditions	Temperature °C	Relative humidity %
1	23	0
2	23	50
3	23	60
4	23	75
5	23	85
6	10	85

F.7 Procedure

Remove a test specimen from the conditioning atmosphere. Insert the specimen in the cell.
Inspect the specimen for visual defects such as creases made during insertion. Connect the cell to the sensor. Using the two valves immediately upstream of the cell, allow the carrier gas to flow through both chambers, A and B. The gas flow rate is generally set at between 5 ml/min and 100 ml/min. Inspect the apparatus for leaks, then purge the apparatus completely, taking into account possible desorption from the specimen. Continue to purge the apparatus until a constant signal is received from the sensor.
Once a constant signal has been obtained, record this value as the zero value.

NOTE The time taken for the apparatus to stabilize may vary from less than one hour to several hours, depending on the material under test, its thickness and the test conditions.

Allow the test gas to flow into chamber A, under the specified conditions of flow, temperature and humidity. The gas flow rate is generally set at between 5 ml/min and 100 ml/min. Proceed until a constant signal is obtained. Record this signal. Repeat with the remaining specimens.

F.8 Expression of results

See Clause F.8 or F.10 depending on the method used.

F.9   Method for the determination of the oxygen-transmission rate using a coulometric sensor

F.9.1 General

This method is used to determine the amount of oxygen permeating through the material, using a coulometric sensor which measures the amount of oxygen which has permeated through the material and been carried out of the cell by the carrier gas.
The sensor generates an electric current proportional to the amount of oxygen flowing through the sensor per unit time.

F.9.2 Apparatus

Figure 3 shows a typical apparatus.

Valve 12 bypasses the sensor (9), thus avoiding the cell (1) being swamped with air when the test specimen (2) is being placed in the cell. The valves (7) immediately upstream of the transmission cell allow either carrier gas or test gas to be passed into chamber A of the cell. The catalyst bed (11) is intended to eliminate any traces of oxygen which might be present in the carrier gas.

NOTE Any other suitable means of removing oxygen from the carrier gas is acceptable.

F.9.3 Carrier gas and test gas

The carrier gas shall be a mixture of dry nitrogen with 0,5 % to 3 % by volume of hydrogen. The volume fraction of oxygen in the carrier gas shall not exceed 100 µl/l.

The test gas shall be dry oxygen containing at least 99,5 % by volume of O_2.

Gas-conditioning devices (see 4 in Figure 3) shall be placed upstream of the cell in order to achieve one of the sets of conditions given in Table 2. Devices for monitoring the humidity of the gases may be fitted in the carrier and/or test gas circuits.

Key
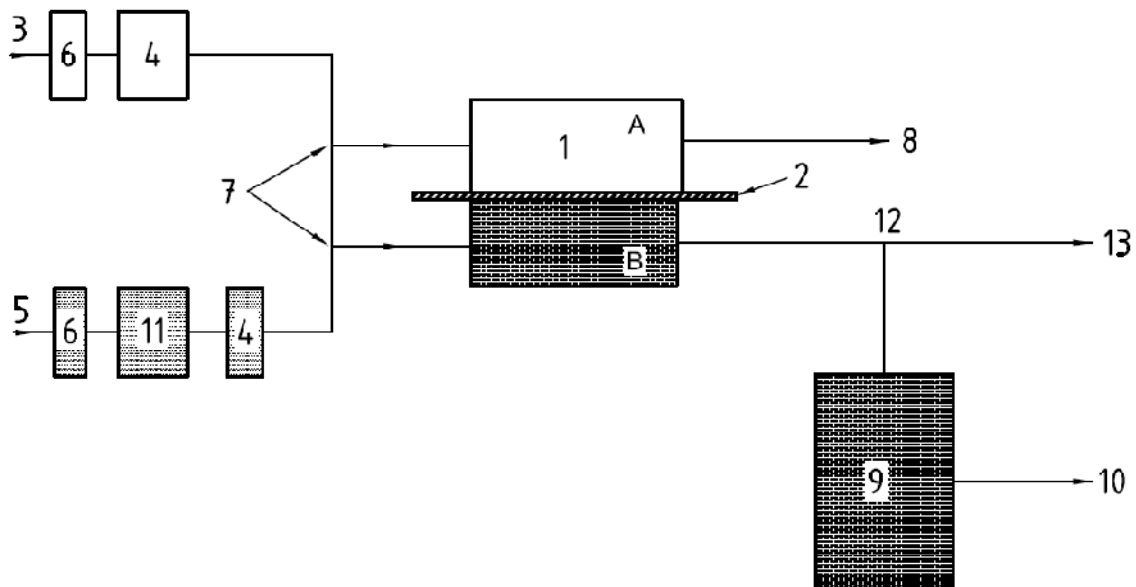
1	transmission cell with chambers A and B	8	oxygen outlet (or carrier gas outlet when purging)
2	specimen	9	coulometric sensor for measurement of O_2 in carrier gas
3	oxygen inlet	10	carrier gas outlet
4	gas-conditioning device	11	catalyst bed, to eliminate traces of O_2 in carrier gas
5	carrier gas inlet	12	3-way valve
6	flowmeter	13	carrier gas outlet when purging the chambers
7	3-way valves		

Figure 3 — Typical example of apparatus for determination of oxygen-transmission rate using a coulometric sensor

F.9.4 Procedure

Insert a test specimen, already conditioned, into the transmission cell, and adjust the test conditions to the values applicable to the material and the specification.
Purge both chambers (A and B), setting the carrier gas flow rate at the following value:
5 ml/min to 25 ml/min for 30 min.For materials of very low permeability, increase the purge time, setting the carrier gas flow rate at the following values: 25 ml/min to 50 ml/min for the first 3 min to 4 min; then 5 ml/min to 25 ml/min for the next 30 min. Inspect the apparatus for leaks.
The electric current generated by the sensor is measured by passing it through a shunt fitted in parallel with a voltage-measuring device. Wait until the sensor signal stabilizes and record this value as the zero value. Reset the two valves (7) immediately upstream of the cell to allow oxygen to pass through chamber A and wait until a constant signal is obtained. Record the value of this signal.
While some thin films with high transmission rates may reach equilibrium in 30 min to 60 min, thicker or more complex materials may require several hours to reach a steady state of oxygen transmission. Record the time required in the test report. Repeat the procedure with the remaining specimens.

F.9.5 Expression of results
Oxygen-transmission rate

The oxygen-transmission rate is calculated using the following equation:

$$O_2GTR = \frac{k(U - U_0)}{A} \times \frac{p_a}{p_0}$$

where

O_2GTR is the oxygen-transmission rate, expressed in moles per square metre second pascal [mol/(m^2.S.Pa)];

U is the voltage for the specimen, expressed in volts;
U_0 is the zero-value voltage, expressed in volts;
k is the calibration constant for the apparatus;

p_a is the ambient atmospheric pressure, expressed in pascals;
p_O is the partial pressure of oxygen in the test gas, expressed in pascals;
A is the effective permeation area, expressed in square metres.

NOTE In practice, the voltages U and U_0 include the calibration constant for the apparatus.

Oxygen permeability, or coefficient of oxygen permeability
The gas permeability, or coefficient of permeability, P is a physical characteristic of a polymer material, only depending on the test gas and the test conditions.

The theoretical value of P is given by the equation:

$$P = O_2GTR \times d$$

Where

P is the oxygen permeability, or the coefficient of oxygen permeability, expressed in mole metres per square metre second pascal $[(mol.m)/(m^2.s.Pa)]$;
O_2GTR is the oxygen-transmission rate, expressed in moles per square metre second pascal $[mol/(m^2.s.Pa)]$;

d is the thickness of the specimen, expressed in metres.
O_2GTR is generally expressed in cubic centimetres per square metre 24 h $[cm^3/(m^2.24\ h)]$, the volume of the gas being converted to standard conditions.

P is often expressed in cubic centimetre millimetres per square metre 24 h atmosphere $[(cm^3.mm)/(m^2.24\ h.atm)]$
d used to be expressed in millimetres.

F.10 Detection by gas chromatography
F.10.1 General
This method is used for the determination of the gas-transmission rate of various gases or mixtures of gases using a gas chromatograph with columns suited to the nature of the gas or gases concerned.

F.10.2 Principle
Gas molecules which permeate through the test specimen in the transmission cell are carried by the carrier gas to a sampling loop which allows the total contents of the sampling loop to be injected repeatedly into a gas chromatograph. The peaks in the chromatogram obtained are compared with a calibration graph prepared from chromatograms produced using calibration standards.

NB Automatic injection valves may induce a pressure back-up and hence an overpressure in the transmission cell. In such cases, it is necessary to compensate for this overpressure to avoid the test specimen being distorted.

Figure 4 shows a typical apparatus.

F.10.3 Chromatograph
F.10.4 General
Various gas-chromatographic columns and detectors are suitable, depending on the test gas involved. Some are described here. Other columns and detectors may be used for specific test gases or to ensure the required sensitivity.
### F.10.5 Packed column used with thermal-conductivity detector (TCD)
This equipment is applicable to: O_2, CO_2 and/or N_2; mixtures of these gases or other gases.
NB Gas conditioning (see Table 2) may be carried out upstream of the transmission cell, but it is not advisable to condition the carrier gas when using a TCD sensor.

F.10.6 Packed column used with flame-ionization detector (FID)
This equipment is applicable to: CO_2 where high sensitivity is required, the chromatograph being fitted with an upstream methanization oven; various organic gases.

Key
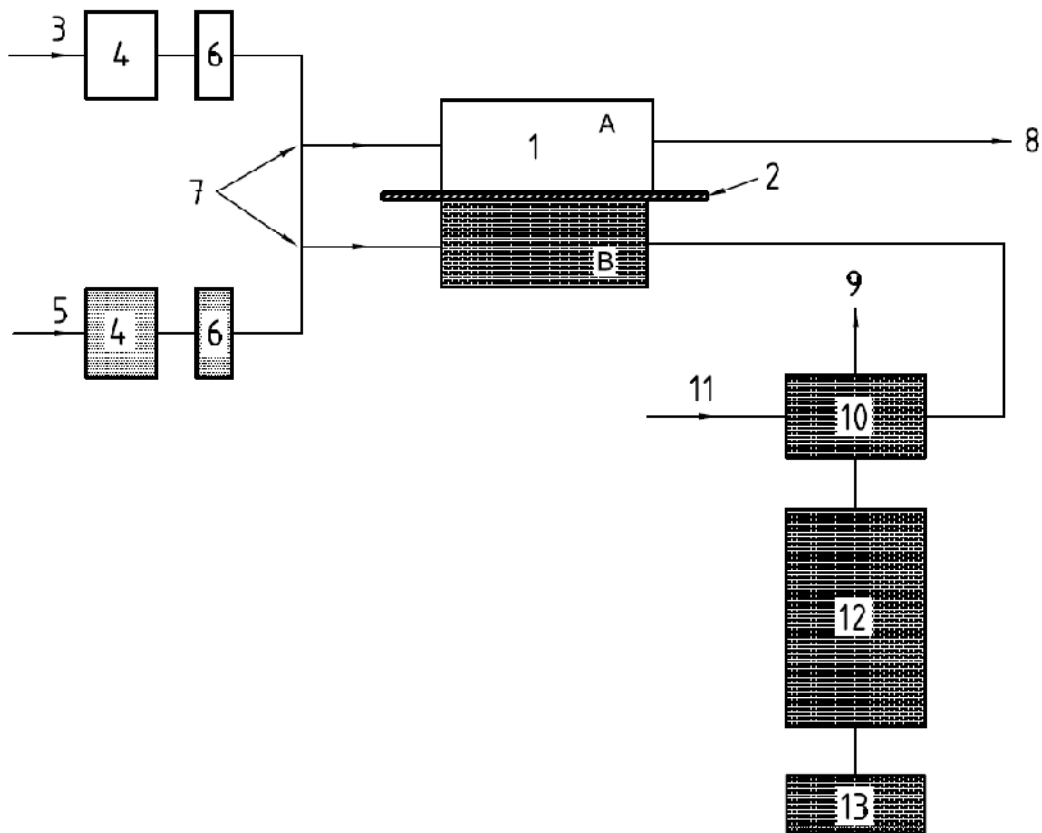
1	transmission cell with chambers A and B	8	test gas outlet (or carrier gas outlet when purging)
2	specimen	9	carrier gas outlet
3	test gas inlet	10	sampling loop
4	gas-conditioning device	11	chromatography carrier gas inlet
5	carrier gas inlet	12	chromatographic column
6	flowmeter	13	TCD or FID detector
7	3-way valves		

Figure 4 —Typical example of apparatus for determination of gas-transmission rate using

gas-chromatographic detection

F.10.7 Capillary column used with ionization detector (FID)
This equipment is applicable to high-molecular-mass organic vapours, such as odours or aromas.
In such cases, care shall be taken to avoid condensation of the vapour as it passes through the transmission
cell and the chromatographic column and detector.

F.10.8 Carrier gas and test gas
The carrier gas shall be compatible with the detection equipment. The concentration of each gas in a mixture of test gases and the purity of the test gas shall be known to within ± 1 %.
Gases shall not contain impurities which may affect the measurements.

F.10.9 Calibration graph
F.10.9.1 General
A calibration graph may be obtained for the chromatographic column.
Order, from specialized suppliers, gas cylinders containing gas mixtures with known concentrations of the gas of interest and covering a concentration range straddling the expected values of the future measurements.
Connect each gas cylinder in turn upstream of the sampling loop, and record the chromatogram for each under the same conditions.

F.10.9.2 Preparation of calibration graph
For either method, the chromatograms recorded can be used to produce a graph relating the detector signal to the concentration of the gas of interest in the carrier gas.
F.10.9.3 Procedure
Feed the gas which permeates through the test specimen to the sampling loop and injecting samples onto the column in accordance with the chromatograph manufacturer's instructions. Monitor the detector response for the gas of interest, continuing until the detector signal has reached a steady value. Record the time required in the test report. Using the integrator of the chromatograph, determine the area of the peak in the chromatogram corresponding to the gas of interest.
Determine, from the calibration graph the concentration of the gas of interest in the carrier gas.
In parallel, using a calibrated flowmeter (e.g. a soap-bubble flowmeter) determine the flow rate of the carrier gas through the column. This measurement may be performed at the carrier gas outlet (9) after diverting the gas from the column by closing off the sampling loop (10) (see Figure 4).

F.10.9.4 Expression of results

F.10.9.4.1 Gas-transmission rate
The gas-transmission rate is calculated using the following equation:

$$GTR = 1\,440 \times D \times C \times \frac{10^4}{A} \times \frac{p_a}{p_0}$$

where
GTR is the gas-transmission rate of the gas of interest, expressed in moles per square metre second pascal [mol/(m^2.s.Pa)];
D is the carrier gas flow rate, expressed in cubic centimetres per minute;
C is the concentration, by volume, of the gas of interest in the carrier gas, as measured from the chromatogram;

A is the effective permeation area of the test specimen, expressed in square metres;

p_a is the ambient atmospheric pressure, expressed in pascals;

p_0 is the partial pressure of the gas of interest in the test gas, expressed in pascals.

F.10.9.4.2 Gas permeability, or coefficient of gas permeability

The gas permeability, or coefficient of permeability, *P* is calculated using the following equation:

P=GTR × d

where

P is the gas permeability, or coefficient of gas permeability, expressed in mole metres per square metre second pascal [(mol.m)/(m^2.s.Pa)];

GTR is the gas-transmission rate, expressed in moles per square metre second pascal [mol/(m^2.s.Pa)];

d is the average specimen thickness, expressed in metres.

NB: GTR is generally expressed in cubic centimetres per square metre 24 h [cm^3/(m^2.24 h)], the volume of the gas
being converted to standard conditions under a pressure of one atmosphere.

P is often expressed in cubic centimetre millimetres per square metre 24 h atmosphere [(cm^3.mm)/(m^2.24 h.atm)] and *d* used to be expressed in millimetres.