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Rubber, vulcanized or thermoplastic — Determination of permeability to gases

iTeh STANDARD PREVIEW
Caoutchouc vulcanisé ou thermoplastique — Détermination de la perméabilité aux gaz

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2782 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This second edition cancels and replaces the first edition (ISO 2782:1977), as well as ISO 1399:1982, which have been technically revised.

Introduction

The measurement of the permeability of rubber to gases is important in the evaluation of compounds for products such as inner tubes, tubeless-tyre liners, hoses, balloons and other gas containers, seals and diaphragms. The measurement is also of theoretical importance in the study of the characteristics of gas diffusion and gas solubility in relation to polymer structure.

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Rubber, vulcanized or thermoplastic — Determination of permeability to gases

WARNING — Persons using this International Standard shall be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard describes two procedures for measuring the permeability of rubber to gases under steady-state conditions. One uses a horizontal measuring device for measurements at constant pressure and the other uses a vertical measuring device for measurements at constant volume or constant pressure. The results obtained by these procedures may be extrapolated to thicknesses of material different from that of the test piece providing the rubber is homogeneous and isotropic.

The procedures apply to solid rubber of hardness not less than 35 IRHD and to gases such as air, nitrogen, oxygen, hydrogen, liquified petroleum gas (in gaseous form) and coal gas. Errors may be introduced if the gas used appreciably swells the rubber under test.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 3383:1985, *Rubber — General directions for achieving elevated or subnormal temperatures for test purposes.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 permeability coefficient: The rate of volume flow of gas under steady-state conditions, referred to standard temperature and pressure, between opposite faces of a unit cube of solid rubber when subjected to a unit pressure difference and controlled temperature.

4 Principle

The cavity of a test cell, maintained at a constant temperature, is divided by a disc test piece into a high-pressure and a low-pressure side. The high-pressure side is connected to a constant-pressure gas reservoir or is of such a volume that, once filled, it stays at practically constant pressure. The gas permeates into the low-pressure side, which is of a very low volume and connected to a capillary tube which measures the volume change caused by permeation of the gas.

5 Apparatus

5.1 Test cell, having a means of clamping the test piece round its periphery in a gastight manner so as to expose one surface to gas under pressure. The other surface of the test piece shall be supported against the force due to the gas pressure so that no appreciable deformation takes place. For this reason, the low-pressure side of the test cell shall be filled with a rigid, highly permeable packing piece which may consist of a disc of microporous material such as microporous ebonite, discs of microporous sintered stainless steel or discs of fine wire gauze or filter paper which completely fill the cavity. A means of indicating gas pressure with an error of not more than 1 % shall be connected to the high-pressure side of the cell.

The internal volume of the test cell on the high-pressure side of the test piece shall be at least 25×10^{-6} m^3 (25 cm^3) to minimize the pressure loss due to diffusion during a test, which may last several hours.

The internal volume of the test cell on the low-pressure side of the test piece shall be kept to a minimum by the use of permeable packing as described above and by small-diameter passages through a dismountable coupling and tubing to a capillary tube (5.2). The total free volume between the test piece and the datum mark on the capillary tube shall not exceed 2×10^{-6} m^3 (2 cm^3).

Test cells shall be of metal construction with sufficient mass to ensure temperature stability, and shall be provided with a drilled pocket to hold a temperature-measuring device (5.3). It is essential that the assembled test cell is gastight.

5.2 Capillary tube, for the measurement of permeated-gas volumes, the tube being of known and uniform cross-section over the length used for volume measurements.

Suitable cross-sections are $0{,}7 \times 10^{-6}$ m^2 to $2{,}0 \times 10^{-6}$ m^2 (0,7 mm^2 to 2,0 mm^2). The cross-section shall be uniform to within an accuracy of 1 %.

The capillary tube may be either horizontally or vertically mounted. It shall be filled with a non-volatile liquid which does not dissolve the gas [suitable liquids are di-(2-ethylhexyl) sebacate or tritolyl phosphate, coloured with Sudan red]. If the capillary is mounted horizontally, a drop of liquid is used to indicate variations in volume. If the capillary tube is mounted vertically, a vertically adjustable reservoir of liquid is connected by a T-piece to the bottom of the capillary tube. The capillary tube shall be graduated or a graduated scale shall be fixed close to the long straight section. Alternatively, a microscope or cathetometer may be used to observe the position of the liquid. If the capillary tube is mounted vertically, a by-pass valve shall be fitted between the point at which the capillary tube leaves the cell and the datum mark on the scale, to allow gas to be released and the liquid in the capillary brought back to the datum mark after the conditioning period.

NOTE 1   When making measurements at constant volume, an alternative means of measuring pressure, for example a transducer, may be used, provided it is suitably calibrated and enables the procedure to be carried out in essentially the same manner.

Examples of suitable apparatus are shown in figures 1 and 2.

5.3 Temperature-measuring device, readable to within ± 0,2 °C.

5.4 Barometer.

5.5 Device for controlling the test temperature, consisting of a constant-temperature bath or other device capable of maintaining the test cell at the required test temperature to within the specified tolerance (see clause 8). If a bath is used and the capillary tube is mounted vertically, the walls of the bath shall be arranged so that the outlet from the test cell projects through the side, leaving the connection point for the capillary tube accessible. A number of test cells containing different test pieces may then be connected in turn to the same manometer.
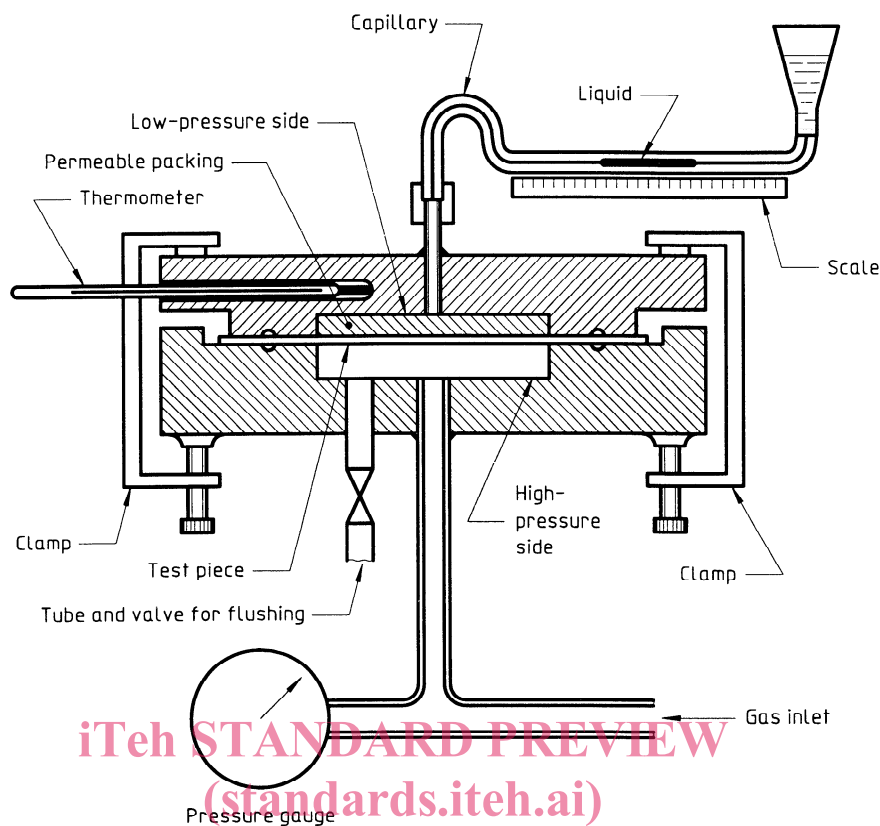
Figure 1 — Apparatus for constant-pressure method with horizontal capillary

6 Test pieces

6.1 Shape and dimensions

The test piece shall be a disc of uniform thickness and of dimensions matching those of the test cell. The disc may be either moulded or cut from a sheet or product. It is preferable to use a moulded disc having on each face a circumferential bead which fits into grooves in the two halves of the test cell (see figure 3). Test pieces of rubber of both high hardness and high thickness shall be prepared with such beads. When the test piece is a flat sheet, suitable O-rings shall be fitted in the grooves in the test cell. The overall variation in thickness of the test piece (excluding the bead) shall not exceed 10 % of the mean thickness.

Suitable dimensions are 50 mm to 155 mm in diameter with an exposed (test) surface of 800×10^{-6} m^2 to $7\,000 \times 10^{-6}$ m^2 (8 cm^2 to 70 cm^2); the thickness may be between 0,25 mm and 3,0 mm, the smallest thickness being suitable for rubber of low permeability, such as isobutene-isoprene rubber. The test piece shall have no pinholes or other imperfections.

6.2 Number of test pieces

Duplicate test pieces of each rubber material shall be tested.

7 Time between vulcanization and testing

The time between vulcanization and testing shall be in accordance with the requirements of ISO 471.
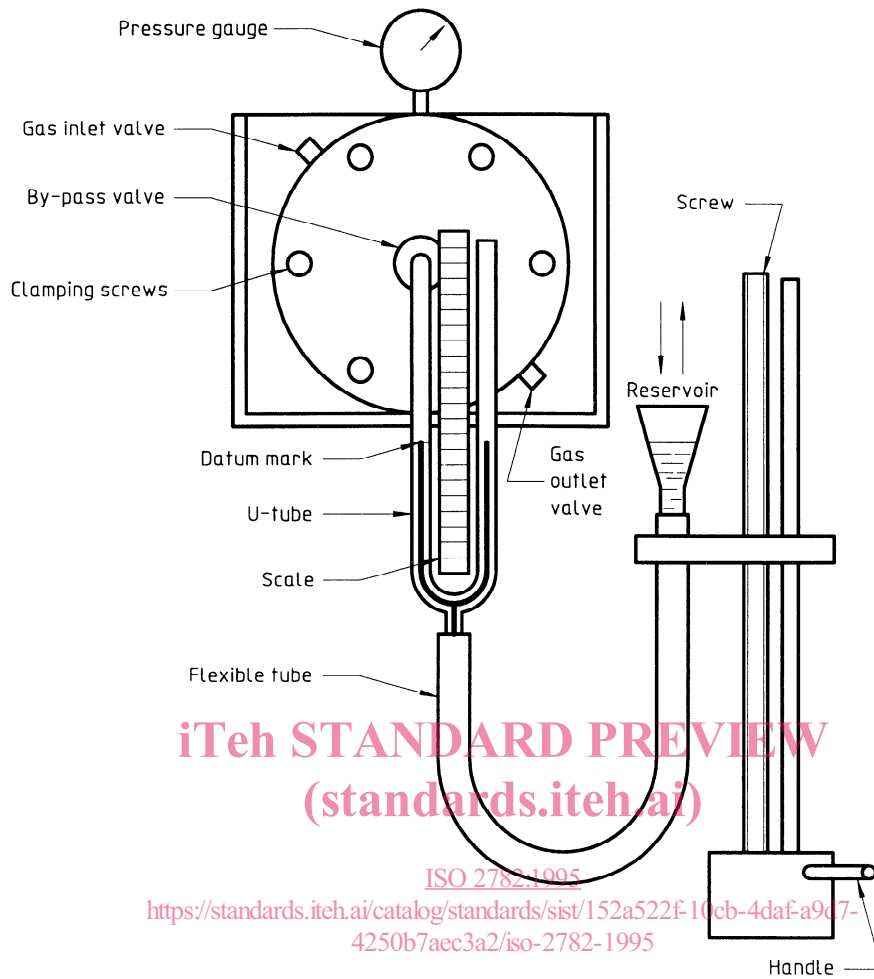
Figure 2 — Apparatus for constant-pressure and constant-volume methods with vertical capillary
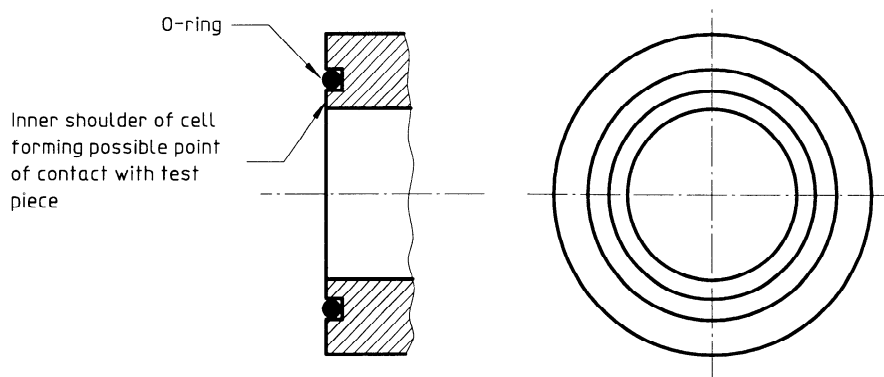

Figure 3 — Surface of test cell

8 Temperature of test

For normal comparisons of permeability of different rubber materials, the test temperature shall be one of the standard laboratory temperatures defined in ISO 471. Higher temperatures may be used where the conditions are required to approximate to the service temperatures of rubber products. Such higher temperatures shall be selected from the list of preferred temperatures given in ISO 471.

In any given test or series of tests intended to be comparable, the tolerance on the temperature shall be ± 1 °C for temperatures up to 175 °C and ± 2 °C for temperatures of 200 °C and above.

9 Test procedures

9.1 General

9.1.1 Preparation of test piece

Prepare the test piece by moulding or by cutting from sheet or by cutting from a rubber product.

Examine the test piece carefully for pinholes or other imperfections in the area which will be exposed inside the test cell (which is effectively the test area). Take care to avoid contamination of the surface between manufacturing and testing.

Determine the thickness of the test piece in the test area as the average of six measurements, each made to an accuracy of 0,02 mm.

Determine the area of the test piece exposed to the high-pressure gas by measuring the internal diameter of the test cell.

NOTE 2   There may be some uncertainty as to the exact size of the exposed area due to contact between the specimen and the two halves of the cell other than at the O-ring or bead as shown in figure 3.

9.1.2 Setting up the apparatus

Insert the permeable packing in the cavity behind the test piece on the low-pressure side of the test cell. Clamp the test piece securely round its edge. If necessary, apply the minimum amount of vacuum grease to the clamping surfaces to ensure a gastight seal. No grease shall be allowed to contact or contaminate the central exposed area of the test piece.

Connect the test cell to the test-gas reservoir at the required test pressure (0,3 MPa to 0,5 MPa, depending on the permeability of the test piece).

NOTE 3   If a constant-temperature bath is being used, it may be necessary to place the test cell in the bath prior to connecting to the test-gas reservoir.

9.1.3 Conditioning the test cell

Allow the assembled apparatus to remain at the test temperature for a minimum of 16 h or, if the approximate value of the diffusion coefficient is known, for a minimum time t, in seconds, derived from the following equation:

$$t = \frac{b^2}{2Q} \times S = \frac{b^2}{2D}$$

where

b is the thickness, in metres, of the test piece;

Q is the permeability, in metres squared per pascal second;

D is the diffusion coefficient, in metres squared per second;

S is the solubility constant (the volume of gas which dissolves in unit volume of the test piece at unit pressure), in reciprocal pascals.

This minimum time t ensures that the diffusion of gas through the test piece, and hence the gas flow, reaches the steady state corresponding to the right-hand (straight) part of the curve in figure 4. The left-hand part of this curve indicates the non-steady-state conditions which exist initially due to diffusion of the gas through the test piece. Only the strictly linear part of the curve is used for permeability measurements.

Theoretically, a straight line is obtained only with constant-pressure measurements, but in practice constant-volume measurements may also give a linear plot if the pressure build-up is very small compared with the applied pressure.

NOTE 4   The expression governing the time taken to reach the steady state is a special case of a more general equation. The boundary conditions prevailing in the two methods (constant pressure and constant volume) may have an effect on the non-steady-state period.