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Rubber, vulcanized — Determination of permeability to gases — Constant pressure method

Caoutchouc vulcanisé — Détermination de la perméabilité aux gaz — Méthode de la pression constante

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries :

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Rubber, vulcanized — Determination of permeability to gases — Constant pressure method

0 INTRODUCTION

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

1 SCOPE AND FIELD OF APPLICATION

This International Standard gives basic directions for measuring the permeability of rubber to gases at steady state conditions, when a constant pressure is maintained on each of the end faces of the rubber to be tested. Results obtained by this test procedure should not be extrapolated to thicknesses of material considerably different from that of the test piece.

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

2 REFERENCES

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubbers — Time-lapse between vulcanization and testing.*

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

3 DEFINITION

permeability of rubber to gases : 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 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 (atmospheric) 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 a practically constant pressure. The gas permeates into the low pressure side, which is of a very low volume and connected to a capillary tube; this is provided for measuring the permeated volume, while keeping or restoring the same low pressure within this side.

Absolute pressure for the high pressure side is maintained in the range 0,3 to 1,5 MPa* and for the low pressure side it is maintained at the prevailing atmospheric pressure.

For normal comparison of permeability of different rubber vulcanizates, the test temperature is a standard laboratory temperature but higher preferred temperatures may be used where conditions are required to approximate to the service temperature of rubber products. Standard laboratory temperature and higher preferred temperatures shall be selected from ISO 471. When higher preferred temperatures are used, directions given in ISO 3383 shall be observed.

WARNING — Because of the dangers connected with high pressure and flammability, handling of gases should be done by experienced personnel only.

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* 1 MPa = 1 N/mm^2

5 APPARATUS

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

5.1 Test cell having a means of clamping the test piece round its periphery in a gas-tight manner so as to expose one surface to gas under pressure. The other surface of the test piece shall be supported against the load 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, very 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 volume of the high pressure side of the cell 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 (atmospheric) 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 the capillary tube. The total free volume between test piece and zero mark shall not exceed 2×10^{-6} m^3 (2 cm^3).

Test cells shall be of metal construction with sufficient mass to assist temperature stability, and shall be provided with a drilled pocket to hold the temperature measuring device (5.2).

5.2 Temperature measuring device, to read accurately to within ± 0,2 °C.

5.3 Barometer.

5.4 Device for the measurement of permeated gas volumes, consisting fundamentally of a capillary tube of known and uniform cross-section over the length used for volume measurements.

Suitable cross-sections are $0{,}7 \times 10^{-6}$ to 2×10^{-6} m^2 (0,7 to 2 mm^2), with an accuracy of within 1 % in uniformity.

The capillary tube shall be graduated or a graduated scale shall be held close to it on the long straight portion.

It shall be suitably 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].

The capillary tube may be mounted on the cell layer or may be provided with a by-pass valve in order to start the volume measurements after a conditioning period.

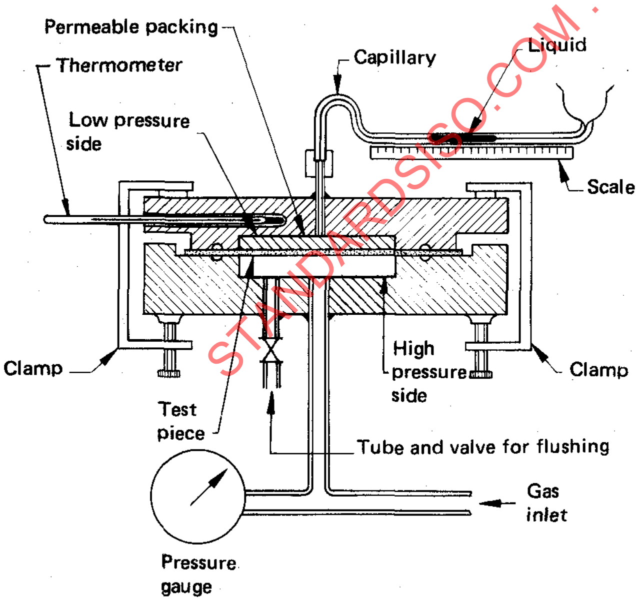

FIGURE 1 — **Apparatus with horizontal measuring device**

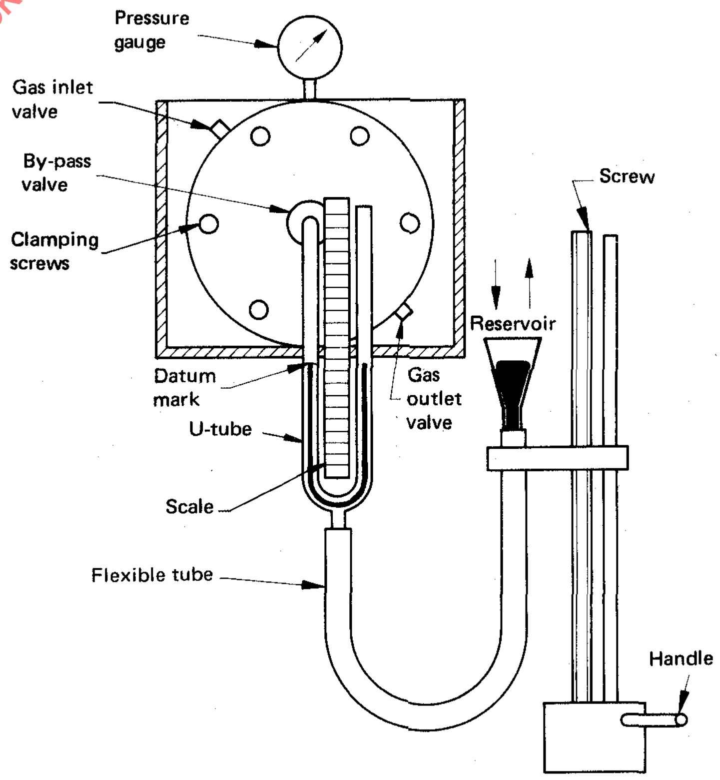

FIGURE 2 — **Apparatus with vertical measuring device**

The capillary tube may be either horizontally or vertically mounted. In the first case only, a drop of liquid may be used as index of volume variations. In the second case, a vertically adjustable reservoir of liquid connected by a T-piece to the lowest portion of the capillary tube shall be provided to restore the low side pressure.

A microscope or a cathetometer may be used to observe the liquid position.

5.5 Constant temperature bath or other means capable of maintaining the test cell at the required test temperature to within ± 1 °C.

The wall of the bath shall be arranged so that the outlet from the test cell will project through the side, leaving the capillary dismountable coupling accessible.

6 TEST PIECES

6.1 Shape and dimensions

The test piece shall be a disc of uniform thickness and of dimensions to suit those of the test cell, and may be either moulded or cut from a test sheet or a product. It is preferable to use a moulded disc having on each face a circumferential rib or bead to fit into corresponding grooves in the clamping members. Where the test piece is a flat sheet, suitable "O" rings shall be used to fit into grooves in the test cell.

The overall variation in thickness (excluding beads) shall not exceed 10 % of the mean thickness.

Suitable dimensions are 50 to 155 mm diameter with a free testing surface of 800×10^{-6} to $7\,000 \times 10^{-6}$ m^2 (8 to 70 cm^2); thickness may be between 0,25 and 3 mm, the smallest thickness being advantageous for rubbers of low permeability, such as isobutene-isoprene rubber. Imperfections and pinholes shall be absent.

6.2 Number

Duplicate test pieces of each rubber shall be tested.

7 TIME-LAPSE BETWEEN VULCANIZATION AND TESTING

The requirements of ISO 1826 apply.

8 TEMPERATURE OF TEST

For normal comparisons of permeability of different rubber vulcanizates, the test temperature shall be a standard laboratory temperature as given in ISO 471. Higher temperatures may be used where conditions are required to approximate to service temperatures of rubber products. Such higher temperatures shall be selected from the list of preferred temperatures, as 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 it shall be ± 2 °C for temperatures of 200 °C and above.

9 PROCEDURE

9.1 Preparation of test piece

Examine the test piece carefully for pinholes or imperfections within the area of the internal diameter of the cell (which is the effective test area). Care should be taken to avoid contamination of the surface between manufacture and test.

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. Insert the permeable packing in the shallow cavity behind the test piece on the low pressure side. Clamp the test piece securely round its periphery.

NOTE — It is permitted to use a minimum of vacuum grease on the clamping surfaces to ensure gas-tightness. No grease shall be allowed to appear on the central area of the test piece.

Place the test cell in the constant temperature bath and connect to the test gas reservoir at the required pressure of test (0,3 to 1,5 MPa depending on the permeability of the test piece).

Maintain the pressure of gas to within ± 2,5 % during the test.

9.2 Conditioning of the test piece

The assembled apparatus shall be allowed to remain at the test temperature for a minimum of 16 h, or, where the approximate value of the diffusivity is known, for a minimum time t, in seconds, derived from the following equation :

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

where

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

Q is the permeability in metres squared per pascal second $[m^2/(Pa\cdot s)]$;

D is the diffusion coefficient in metres squared per second;

S is the solubility constant (the volume of the gas, dissolved per unit volume of the test piece at unit pressure) in reciprocal pascals $\left(\frac{1}{Pa}\right)$.

This minimum time t ensures that the diffusion of gas through the test piece, and hence the gas flow throughout the test piece, may reach the steady state corresponding