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Foreword

This British Standard has been published under the authority of the Rubber Industry Standards Committee.

The method as described in the previous edition of this standard was accepted as the basis for international discussion in Technical Committee ISO/TC 45, Rubber and Rubber Products, and the present revision aligns the detail with international agreement as published in ISO Recommendation 1399 'Determination of the permeability of vulcanized rubber to gases (constant volume method)'.

Consideration is being given to an alternative method based on measuring the increase in volume of gas, at constant pressure, on the low pressure side of a test piece.

British Standard

Methods of testing vulcanized rubber

Part A17. Determination of the permeability of rubber to gases (constant volume method)

1. Scope

This Part of this British Standard describes a method of determining the permeability of rubber to gases based on measurement of the increase in pressure in a space of constant volume on the low pressure side of a test piece subjected to a pressure differential.

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

2. Definitions

For the purposes of this part of this British Standard the following definitions apply.

(1) *Permeability coefficient.* The rate of flow by diffusion of gas between opposite faces of a unit cube of non-porous rubber under unit pressure difference and at a controlled temperature.

NOTE. This definition assumes that the rate of permeation is inversely proportional to the thickness of the rubber. This is substantially true for permanent gases passing through a homogeneous layer of rubber. However, for vapours, or for permeation through heterogeneous materials (e.g. proofed fabrics), there may be considerable deviations from this relationship; in these cases it is therefore better to express the result as transmission rate, i.e. the quantity of vapour or gas passing in unit time through unit area of a material of stated thickness, when the difference of pressure of the diffusing vapour or gas between the two faces of the material is unity.

(2) *Diffusion coefficient.* The rate of flow by diffusion of gas between the opposite faces of a unit cube of non-porous rubber at a controlled temperature when the concentrations of absorbed gas at these faces differ by unity, i.e. 1 ml of gas per ml of rubber.

3. Test piece

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

Suitable dimensions are 50 mm to 65 mm diameter with a free testing surface of 800 mm^2 to 1600 mm^2; thickness is preferably between 0.25 mm and 2.0 mm, the smaller thickness being advantageous for rubber of low permeability, such as *iso*butene-isoprene (butyl). Imperfections and pin-holes shall be absent.

4. Apparatus

The apparatus (see Figs. 1 and 2) shall comprise the items specified in **4.1** to **4.3**.

4.1 Test cell. A means shall be provided 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 force due to the gas pressure so that no gross deformation takes place. For this reason the low pressure side of the test cell shall contain a rigid, easily permeable packing piece which shall fill the cavity completely. An instrument capable of indicating gas pressure up to about 5 bar* with an error of no more than 1 % shall be connected to the high pressure side of the cell.

The internal volume of the high pressure side of the chamber should be at least 25 ml, 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 of small diameter passages through the dismountable coupling and tubing to the pressure measuring device on the low pressure side. A typical volume for the design shown in Fig. 2 is a total volume between test piece and datum mark of between 1 ml and 2 ml. Test cells should be of metal construction with sufficient mass to assist temperature stability and shall be provided with drilled pockets for the selected temperature measuring device.

4.2 Temperature measurement. A means shall be provided for accurate measurement of the temperature to the nearest 0.1 °C.

4.3 Pressure measurement. A suitable means shall be provided on the low pressure side of the cell to measure the pressure increase. A capillary tube of 'U' shape, filled with a non-volatile liquid which does not dissolve the gas (e.g. dioctyl sebacate), graduated on the long vertical portion and provided with a datum mark on the short portion close to the test cell, may be used for this purpose. If such a tube is used, the use of a microscope to observe the liquid level is advantageous. A vertically adjustable reservoir of the liquid shall be connected by a T-piece to the lowest portion of the manometer 'U' tube. A by-pass valve shall be provided between the union and datum mark, to release gas for initial adjustments.

4.4 Temperature control. A constant temperature bath or other means capable of maintaining the test cell at the required test temperature to within ± 0.5 °C shall be provided.

5. Procedure

5.1 Preparation of test piece. The test piece shall be carefully checked for pin-holes or other imperfections within the area of the internal diameter of the cell (which is the effective test area) and shall be free from all surface contamination, such as wax or films of mould lubricant.

The thickness of the test piece in the test area shall be taken to be the average of five measurements, each accurate to ± 0.02 mm, taken at the centre and at four equally spaced points towards the periphery of the test area. After insertion of the permeable packing into the shallow cavity behind the test piece, the test piece shall be securely clamped round its periphery, using a minimum of vacuum grease on the clamping face to ensure gas-tightness. There shall be no grease on the central area of the test piece. With flat test pieces (i.e. without beads) of thickness 0.5 mm or less, washers of soft vulcanized 'butyl' rubber, on both sides of the test piece, may be necessary to ensure a gas-tight seal. The gas chamber (see Fig. 2) shall then be filled with the test gas to the required pressure of test (usually 2 bar* to 4 bar). The pressure shall be kept constant to within ± 2 %. The test cell shall then be brought to the test temperature, and the low pressure measuring device tube coupled to the test cell by means of a union.

5.2 Conditioning of test piece. The assembled apparatus shall be allowed to remain at the test temperature for a period of at least 1 hour, or, where the approximate value of the diffusion coefficient is known, for a minimum time t (in seconds) given by the formula:

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

where b = thickness (m)
Q = permeability coefficient ($m^4/(s\ N)$),
D = diffusion coefficient (m^2/s),
and S = quantity of gas (ml) absorbed by 1 ml of the test piece at gas pressure of 1 Pa.

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* $1\ bar = 10^5\ Pa$

6. Conditioning and temperature of test

For all test purposes the minimum time between vulcanization and testing shall be 16 hours.

For non-product tests the maximum time between vulcanization and testing shall be 28 days and for evaluations intended to be comparable the tests shall, as far as possible, be carried out after the same time interval.

For product tests the maximum time between vulcanization and testing shall be 90 days if the date of vulcanization is known. If the date of vulcanization is not known, the maximum time before testing shall be 60 days from the date of receipt by the customer unless otherwise specified.

For normal tests the temperature shall be 23 ± 0.5 °C, but higher temperatures may be used where conditions are required to approximate the service temperature of rubber products, and such temperatures should be selected from the following series of preferred temperatures: 40 °C, 50 °C, 70 °C, 85 °C, 100 °C, 125 °C, 150 °C, 175 °C, 200 °C, 225 °C, 250 °C.

NOTE. Permeability of vulcanized rubbers increases by 5 % to 8 % per 1 °C rise of temperature; close temperature control is therefore necessary to achieve reproducible results.

7. Test report

The test report shall include at least the following information:

(1) permeability calculated as in **5.5**, or transmission rate calculated as in **5.5** and test piece thickness;
(2) gas used in the test;
(3) temperature of test;
(4) identification of the sample;
(5) date of test.

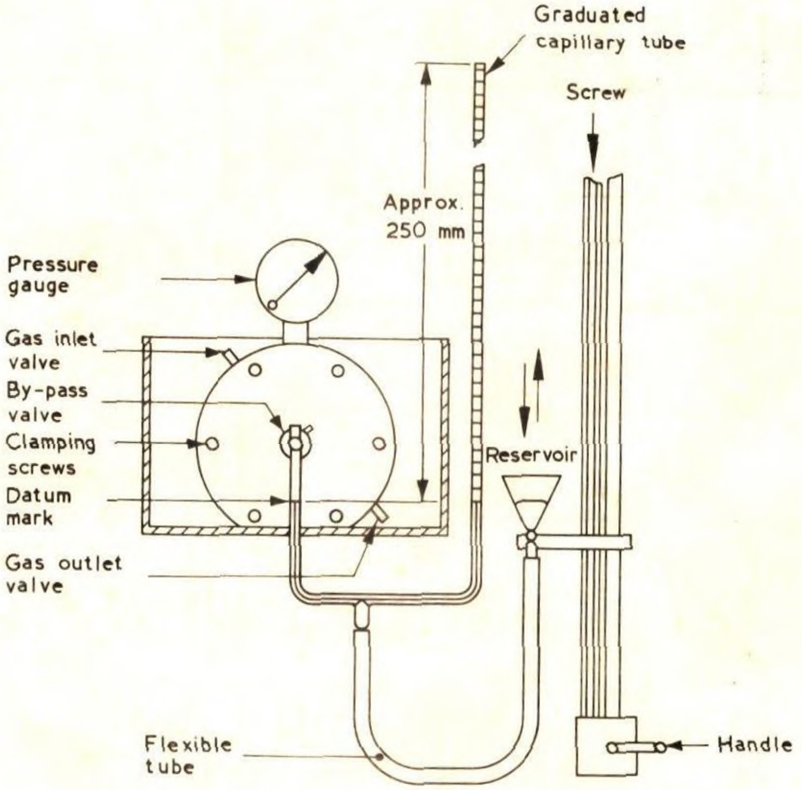

Fig. 1. Permeability apparatus. Front elevation

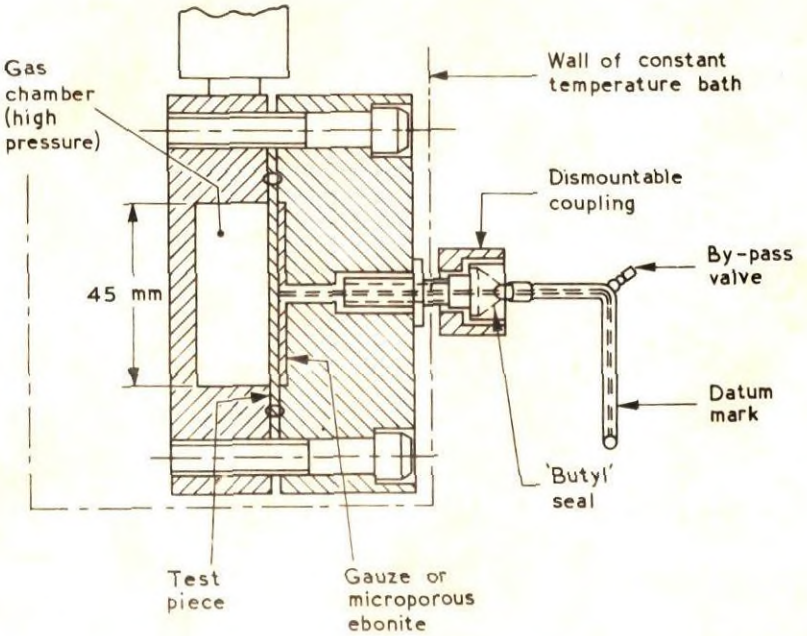

Fig. 2. Section showing detail of dismountable coupling

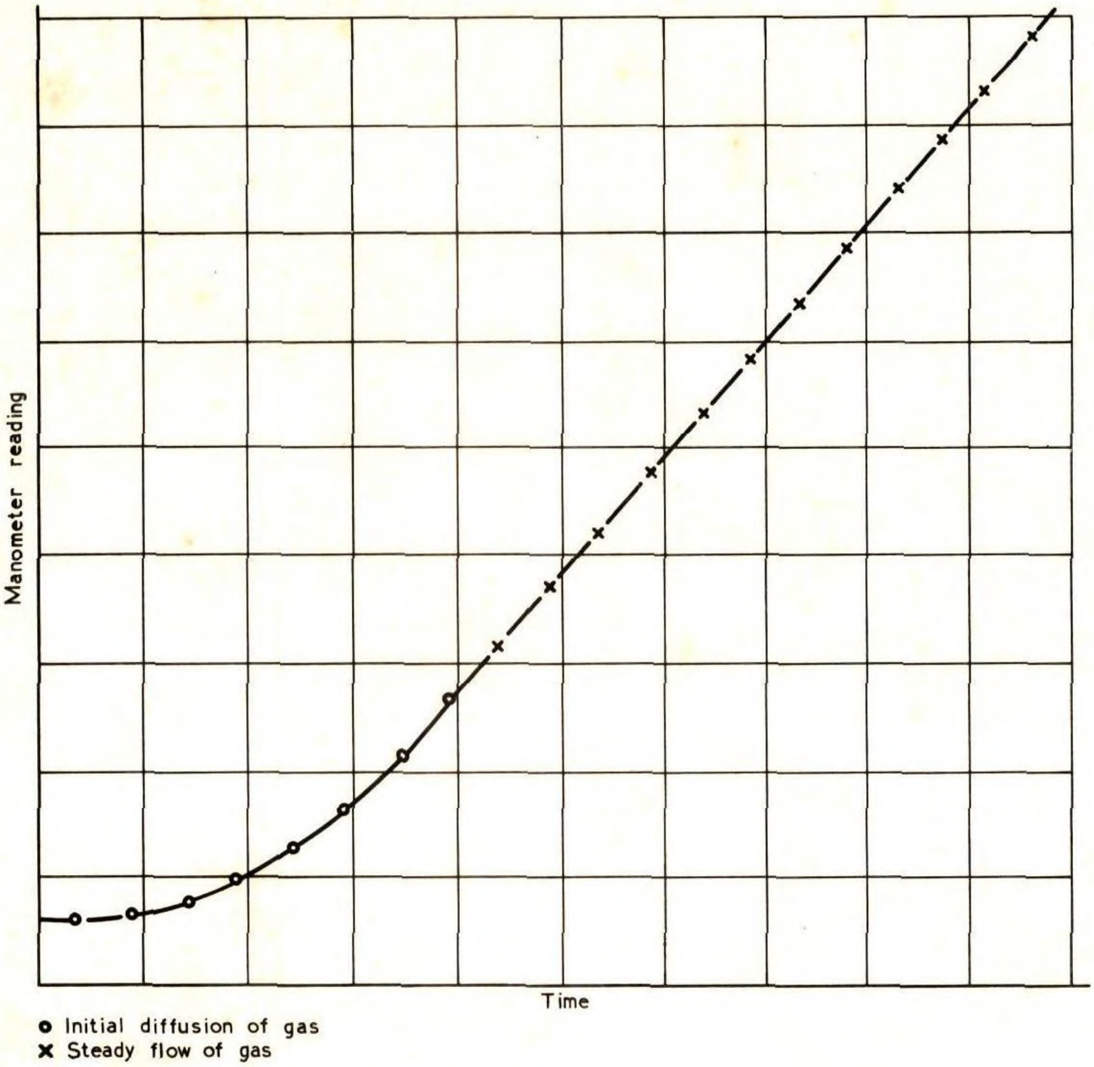

Fig. 3. Typical time/manometer reading curve