International Standard 1399

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

Caoutchouc vulcanisé — Détermination de la perméabilité aux gaz — Méthode à volume constant

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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.

International Standard ISO 1399 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 1399-1976), which had been approved by the member bodies of the following countries :

Australia	Hungary	Spain
Austria	India	Sweden
Canada	Iran	Switzerland
Colombia	Israel	Thailand
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	New Zealand	USSR
Germany, F. R.	Poland	

No member body had expressed disapproval of the document.

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

0 Introduction

The measurement of the permeability of rubber to permanent gases is important in the evaluation of rubbers for such products as inner tubes, tubeless tyre liners, hoses, balloons or other gas containers, or seals. The measurement is also of theoretical importance in the study of characteristics of diffusion and gas solubility in relation to polymer structure. The fundamental requirements of a permeability test for industrial use are accuracy, rapidity and good temperature control, combined with maximum simplicity in the assembly of the equipment.

1 Scope and field of application

This International Standard specifies a method for the determination of the permeability of vulcanized rubbers to gases.

2 Definition

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

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.

3 Apparatus (See figures 1 and 2)

3.1 Test cell, in which the test piece may be clamped 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 deformation takes place. For this reason, the low-pressure side of the test cell shall be filled with a rigid, easily permeable packing piece which may consist of a disk of microporous ebonite or disks of fine wire gauze which completely fill the cavity. A means of indicating gas pressure up to about 500 kPa[1)] 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 test cell shall be at least 25 cm^3 to minimize the pressure loss due to diffusion during a test which may last several hours.

The internal volume of the low-pressure (atmospheric) side of the test cell shall be kept to a minimum by the use of permeable packing as described above and by small diameter passages through the dismountable coupling and tubing to the manometer. For the design shown in figure 2, a total volume between test piece and datum mark of 1 to 2 cm^3 is typical.

The test cell shall be made of metal and shall be of sufficient mass to facilitate temperature stability; it shall be provided with a drilled pocket to hold a suitable temperature-measuring device.

3.2 Temperature-measuring device, accurate to 0,2 °C.

3.3 Manometer, consisting of a capillary tube of U shape, filled with a non-volatile liquid such as dioctyl sebacate which does not dissolve the gas, graduated on the long, straight, vertical portion and provided with a datum mark on the short portion close to the test cell.

The use of a microscope to observe the liquid level is advantageous.

A vertically adjustable reservoir of liquid shall be connected by a T-piece to the lowest portion of the manometer U-tube. A bypass valve shall be provided between the union and datum mark, to release gas for initial adjustments.

NOTE — 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.

3.4 Constant-temperature bath, or other means capable of maintaining the test cell at the required test temperature to within ± 0,5 °C. The wall of the bath shall be arranged so that the outlet from the test cell will project through the side, leaving the dismountable coupling accessible. A number of test cells containing different test pieces may then be connected in turn to a single manometer apparatus.

1) 1 kPa = 1 kN/m^2

4 Test piece

The test piece shall consist of a disk 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 disk 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 5 to 6,5 cm diameter with a free testing surface of 8 to 16 cm^2. The thickness may be between 0,25 and 3,0 mm, the smallest thickness being advantageous for rubbers of low permeability, such as butyl. Imperfections and pinholes shall be absent.

5 Time interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements for time intervals shall be observed.

5.1 For all test purposes, the minimum time between vulcanization and testing shall be 16 h.

5.2 For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

5.3 For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt by the purchaser.

6 Temperature of test

For normal comparison of permeability of different rubber vulcanizates, the test temperature shall be a standard laboratory temperature (23 ± 2 °C or 27 ± 2 °C), but higher temperatures may be used where conditions are required to approximate to the service temperature of rubber products. Such higher temperatures should be selected from the following list of preferred temperatures :

40, 55, 70, 85, 125, 150, 175, 200, 225 and 250 °C

The tolerance on the temperature shall be ± 0,5 °C in any given test or series of tests intended to be comparable.

7 Procedure

7.1 Preparation of test piece

Check the test piece for pinholes or imperfections within the area of the internal diameter of the test cell (which is the effective test area) and free it from all surface contamination, such as wax or films of mould lubricant.

Measure the thickness of the test piece in the test area at six different points to an accuracy of 0,02 mm. Take the average of these measurements as the thickness of the test piece.

After insertion of the permeable packing in the shallow cavity behind the test piece, clamp the test piece securely round its periphery, using a minimum of vacuum grease on the clamping faces to secure gas-tightness. No grease shall be allowed to appear 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.

Fill the gas chamber (see figure 2) with the test gas to the required pressure of test, usually between 200 and 400 kPa. Then bring the test cell to the test temperature, and couple the manometer tube by means of the union.

7.2 Conditioning of test piece

Maintain the assembled apparatus at the test temperature for a minimum of 1 h or, where the approximate value of the diffusivity is known, for a minimum time, t, given by the equation

$$t = \frac{d^2}{2\,Q} \times V = \frac{d^2}{2\,D}$$

where

t is the conditioning time, in seconds;

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

Q is the permeability coefficient;

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

V is the gas volume, in cubic centimetres, absorbed by 1 cm^3 of the test piece at a pressure of 1 Pa.

This minimum time t ensures that the diffusion of gas through the test piece, and hence the pressure gradient through the test piece, may reach the steady state corresponding to the right-hand (straight) portion of the curve shown in figure 3. The left-hand portion of this curve indicates the initial approach to steady conditions due to diffusion through the test piece. The strictly linear portion of the curve only shall be used for permeability measurement.

7.3 Determination of permeability to gas

With the bypass valve open to the atmosphere, adjust the liquid reservoir to bring the liquid level above the datum mark. If a gas other than air is used, the gas chamber of the test cell shall be flushed out with the test gas prior to commencement of test. Then close the bypass valve. As the gas diffuses through the test piece, the meniscus descends; when it crosses the datum mark, start the stop-watch (i.e. zero time).

Raise the reservoir again to bring the meniscus above the datum mark, and note the time and manometer reading when the meniscus again passes the mark. Repeat this procedure until sufficient readings have been obtained to establish with adequate accuracy the slope of the straight part of the

time/manometer reading curve (see figure 3). About 12 readings are required. Record the temperature in the test cell at intervals throughout the test. If the time/manometer reading curve shows any appreciable departure from linearity in the sense of curvature towards the right (i.e. a reduction in slope with increasing time), this shows that there is leakage from the low-pressure side of the apparatus. If this occurs, the test result shall be rejected, and the apparatus dismantled and re-assembled.

7.4 Duration of test

The duration of test of a single test piece should usually be of the order of 15 to 30 min, readings being taken about every 2 min and plotted on a graph, such as that shown in figure 3, to check that steady conditions have been reached.

8 Expression of results

The measurement of rate of gas flow shall be carried out by noting the rate of increase of pressure at constant volume. It is therefore important to calibrate the apparatus by an accurate determination of the gas volume between the test piece and the datum mark, by measuring the dimensions of the cavity and adding the volume of the passages and tubing.

The effective volume of the permeable packing which is inserted in the test cell may be calculated by dividing the mass of the packing by the density of the material from which it is made; for example, if copper gauze is used for the packing material, the total mass of the copper gauze shall be divided by the density of solid copper, i.e. 8,80 Mg/m^3. The effective volume of the packing shall then be deducted from the internal volume of the low-pressure side of the apparatus up to the datum mark.

The units in which permeability should be measured are $m^2 \cdot s^{-1} \cdot Pa^{-1}$,[1] and typical figures for a natural rubber gum stock are of the order of 9×10^{-17}. The apparatus described has a useful working range of $0{,}1 \times 10^{-17}$ to 15×10^{-17} $m^2 \cdot s^{-1} \cdot Pa^{-1}$.

The permeability, expressed in metres squared per second per pascal, is given by the formula

$$\frac{dh}{dt} \times \frac{V_0 \times d \times \varrho \times 10^3 \times 9{,}81 \times 273}{A \times \Delta p \times T \times 10^5}$$

where

$\frac{dh}{dt}$ is the manometer rise, expressed in metres per second;

V_0 is the effective gas volume, in cubic metres, of the low-pressure side of the test cell;

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

ϱ is the density, expressed in megagrams per cubic metre, of the manometer liquid;

A is the area, in square metres, of the test piece (neglecting the clamped area);

Δp is the pressure difference, in pascals, of the gas diffusing across the test piece;

T is the test temperature, in kelvins;

10^5 is the approximately normal atmospheric pressure, in pascals.

9 Reproducibility of results

The accuracy of test by the method specified should usually be of the order of ± 5 % (coefficient of variation) for repeat tests on a single test piece. The coefficient of variation between repeat vulcanizates of the same compounding formula may give rise to a further error of ± 3 %.

10 Test report

The test report shall include the following information :

a) a reference to this International Standard;

b) the identification of the sample;

c) the permeability;

d) the gas used in the test;

e) the temperature of the test;

f) the thickness of the test piece.

1) $1\ m^2 \cdot s^{-1} \cdot Pa^{-1} = 1\ m^4 \cdot s^{-1} \cdot N^{-1}$

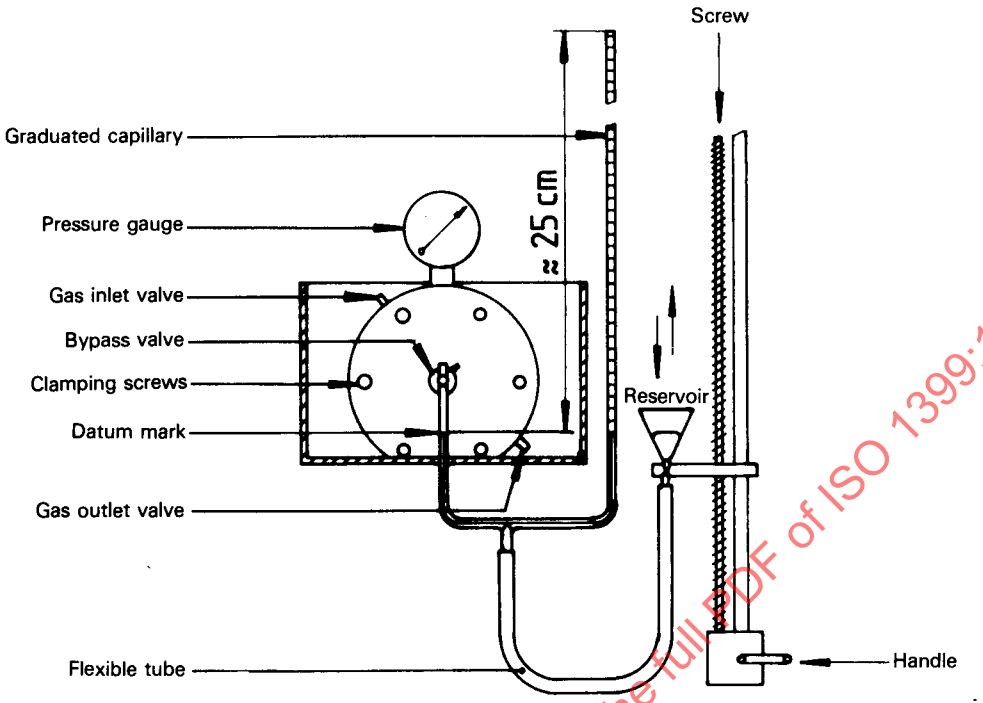

Figure 1 — Permeability apparatus

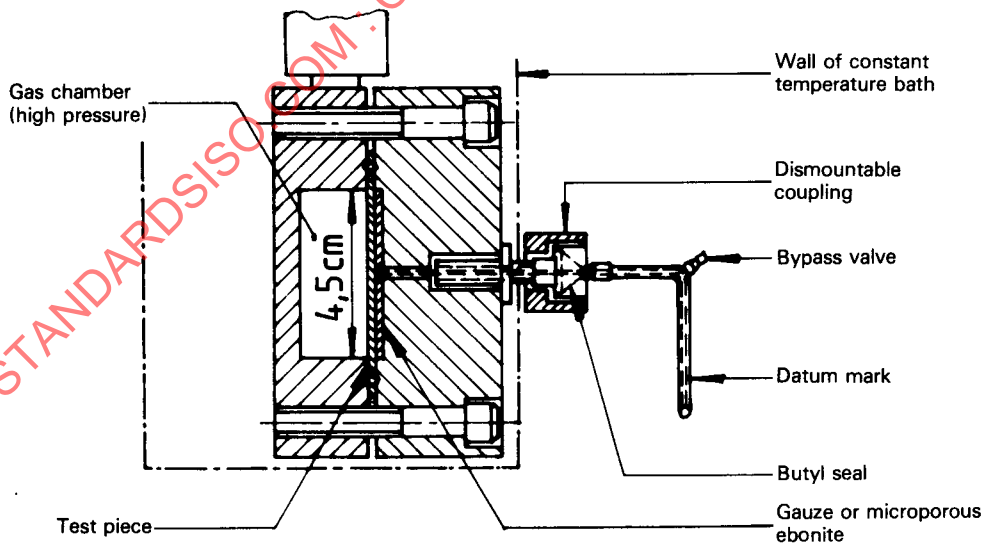

Figure 2 — Section showing detail of dismountable coupling