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DETERMINATION OF THE PERMEABILITY OF VULCANIZED RUBBERS TO GASES

(CONSTANT VOLUME METHOD)

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BRIEF HISTORY

The ISO Recommendation R 1399, *Determination of the permeability of vulcanized rubbers to gases (Constant volume method)*, was drawn up by Technical Committee ISO/TC 45, *Rubber*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1399, which was circulated to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Sweden
Austria	Iran	Switzerland
Canada	Israel	Thailand
Colombia	Italy	Turkey
Czechoslovakia	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Poland	U.S.S.R.
Hungary	Spain	

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1399 January 1971

DETERMINATION OF THE PERMEABILITY OF VULCANIZED RUBBERS TO GASES (CONSTANT VOLUME METHOD)

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

This ISO Recommendation describes a method for the determination of the permeability of vulcanized rubbers to gases.

2. DEFINITION

Permeability of rubber to gases. The rate of flow by diffusion of gas between opposite faces of a unit cube of non-porous rubber, for unit difference in pressure, when tested under controlled pressure and temperature.

3. APPARATUS

(See Fig. 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 should 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 should be filled with a rigid, easily permeable, packing piece which may consist of a disc of microporous ebonite or discs of fine wire gauze which completely fill the cavity. A means of indicating gas pressure up to about 5×10^5 N/m^2, with an error of no more than 1 %, should be connected to the high-pressure side of the cell.

The internal volume of the high-pressure side of the test cell should be at least 25 cm^3 to minimise 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 should 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 should be of metal construction with sufficient mass to assist temperature stability, and should 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 di-octyl 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 should be connected by a T-piece to the lowest portion of the manometer U tube. A by-pass valve should be provided between the union and datum mark, to release gas for initial adjustments.

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 should 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.

4. TEST PIECE

The test piece should consist of 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) should 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 must be absent.

5. TIME LAPSE BETWEEN VULCANIZATION AND TESTING

Unless otherwise specified for technical reasons the following requirements for time lapses should be observed.

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

5.2 For non-product tests the maximum time between vulcanization and testing should 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 should be made within 2 months of the date of receipt by the customer of the product.

6. TEMPERATURE OF TEST

For normal comparison of permeability of different rubber vulcanizates the test temperature should be a standard laboratory temperature (20 ± 2 °C, 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, 50, 70, 85, 125, 150, 175, 200, 225 and 250 °C.

The tolerance on the temperature should 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 should 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 Fig. 2) with the test gas to the required pressure of test, usually 2×10^5 to 4×10^5 N/m^2. 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 hour or, where the approximate value of the diffusivity is known, for a minimum time t derived from the following equation :

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

where

t is the conditioning time in seconds;

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

Q is the permeability coefficient;

D is the diffusion coefficient, in metres per second;

S is the gas volume, in cubic centimetres, absorbed by 1 cm^3 of the test piece at a pressure of 1 N/m^2.

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 should be used for permeability measurement.

7.3 Determination of permeability to gas

With the by-pass valve open to 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 should be flushed out with the test gas prior to commencement of test. Then close the by-pass 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 Fig. 3). About twelve 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 should 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 minutes, readings being taken about every 2 minutes and plotted on a graph, such as shown in Figure 3, to check that steady conditions have been reached.

8. EXPRESSION OF RESULTS

The measurement of rate of gas flow should 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 should be divided by the density of solid copper, i.e. 8.80 Mg/m^3. The effective volume of the packing should then be deducted from the internal volume of the low pressure side of the apparatus up to the datum mark.

The SI units in which permeability is measured are $m^4\ s^{-1}\ N^{-1}$ 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 0.1×10^{-17} to 15×10^{-17} units.

The permeability in SI units should be calculated from the following formula :

$$\frac{dh}{dt} \times \frac{V_0 \times b \times \rho \times 10^3 \times 9.81 \times 273}{A \times P \times T \times 10^5}$$

where

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

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

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

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

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

P is the pressure difference, in newtons per square metre, of the diffusing gas across the test piece;

T is the test temperature, in kelvins;

10^5 is approximately normal atmospheric pressure, in newtons per square metre.

9. REPRODUCIBILITY OF RESULTS

The accuracy of test by the method described 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 should include the following particulars :

(*a*) the permeability;

(*b*) the gas used in the test;

(*c*) the temperature of the test;

(*d*) the thickness of the test piece.