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Rubber, vulcanized or thermoplastic — Determination of permeability to gases —

Part 1: Differential-pressure methods

Caoutchouc vulcanisé ou thermoplastique — Détermination de la perméabilité aux gaz —

Partie 1: Méthodes à pression différentielle

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 2782-1:2016), of which it constitutes a minor revision.

The main changes are as follows:

— editorially revised to update references.

— editorially revised to take into account the updated ISO/IEC Directives, Part 2.

A list of all parts in the ISO 2782 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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-containing products, as well as 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.

This document proposes three different methods. The first one is the pressure sensor method which allows a complete characterization of a material. The second one is a simplified pressure sensor method which is appropriate when only the gas permeability coefficient is needed (e.g. routine control, specification verification, design). The third one is the gas-chromatographic method.

Rubber, vulcanized or thermoplastic — Determination of permeability to gases —

Part 1: Differential-pressure methods

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 take legal compliance into consideration.

WARNING 2 — Certain procedures specified in this document can involve the use or generation of substances, or the generation of waste, that can constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies three methods for the determination of the permeability to gases of vulcanized or thermoplastic rubber under a differential partial pressure.

The three methods specified are as follows:

— a pressure sensor method (using vacuum): for determining the gas transmission rate, gas permeability coefficient, gas diffusion coefficient and gas solubility coefficient;

— a simplified sensor method (using applied pressure): for determining the gas permeability coefficient only;

— a gas-chromatographic method: for determining the gas transmission rate and gas permeability coefficient.

These methods apply to vulcanized and thermoplastic rubbers of hardness not less than 35 IRHD (international rubber hardness degrees) and to both single gases and mixtures of gases.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2016, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
gas transmission rate
number of moles of test gas passing through a test piece per unit area, per unit time, with unit partial-pressure difference between the two sides of the test piece

3.2
gas permeability coefficient
number of moles of test gas passing through a test piece of unit thickness, per unit area, per unit time, with unit partial-pressure difference between the two sides of the test piece

3.3
gas diffusion coefficient
quantity of test gas passing, by diffusion, through a test piece of unit thickness, per unit area, per unit time, where there is a unit gas concentration gradient across the test piece

3.4
gas solubility coefficient
test gas concentration inside a test piece divided by the partial pressure of the test gas at the surface of the test piece

3.5
gas transmission curve
<pressure sensor method> curve, plotted against time, of the pressure change on the low-pressure side of the test cell until gas transmission reaches a steady state

Note 1 to entry: A gas transmission curve is illustrated in Figure 2.

4 Principle

The cavity of a test cell, maintained at a constant temperature, is divided by a test piece into a high-pressure and a low-pressure side. The high-pressure side of the cell is filled with the test gas. The quantity of gas that diffuses through the test piece to the low-pressure side is determined by a pressure sensor or by a gas chromatograph.

In the pressure sensor method, the gas transmission rate, gas permeability coefficient, gas diffusion coefficient and gas solubility coefficient are measured, producing an average value for a gas mixture. In the simplified pressure sensor method only, the gas permeability coefficient is measured. In the gas-chromatographic method, measurements are possible on test gases containing water vapour, and it is also possible to analyse mixtures of gases to determine the components.

5 Pressure sensor method

5.1 Apparatus

The apparatus consists of the test cell, pressure sensors, a test gas supply reservoir, a vacuum pump and associated tubing and valves. An example of a test apparatus is shown in Figure 1.

5.1.1 Test cell, consisting of a low-pressure side and a high-pressure side, such that, when a test piece is mounted in it, the gas transmission area is clearly defined. The high-pressure side has an inlet port to supply test gas, and a pressure sensor is connected to the low-pressure side to detect the change in pressure caused by the gas transmitted through the test piece. The surfaces of the two halves of the cell which make contact with the test piece shall be smooth and flat to prevent any leakage of gas. A seal such as an O-ring may be used between these areas and the test piece, in which case the gas transmission rate of the seal shall be considerably lower than that of the material being tested so that it does not affect the result of the test. The material of the test cell shall be unreactive with regard to the

test gas and shall not absorb the gas used. The diameter of the gas transmission area shall be within the range 10 mm to 150 mm, depending on the gas transmission rate expected.

The cell shall be equipped with a heating system capable of raising the temperature to 80 °C. The temperature accuracy shall be ± 1 °C for temperatures from 40 °C to 80 °C.

NOTE Examples of a heating system are an electric heating jacket and an oven designed to hold the test cell and test gas supply reservoir.

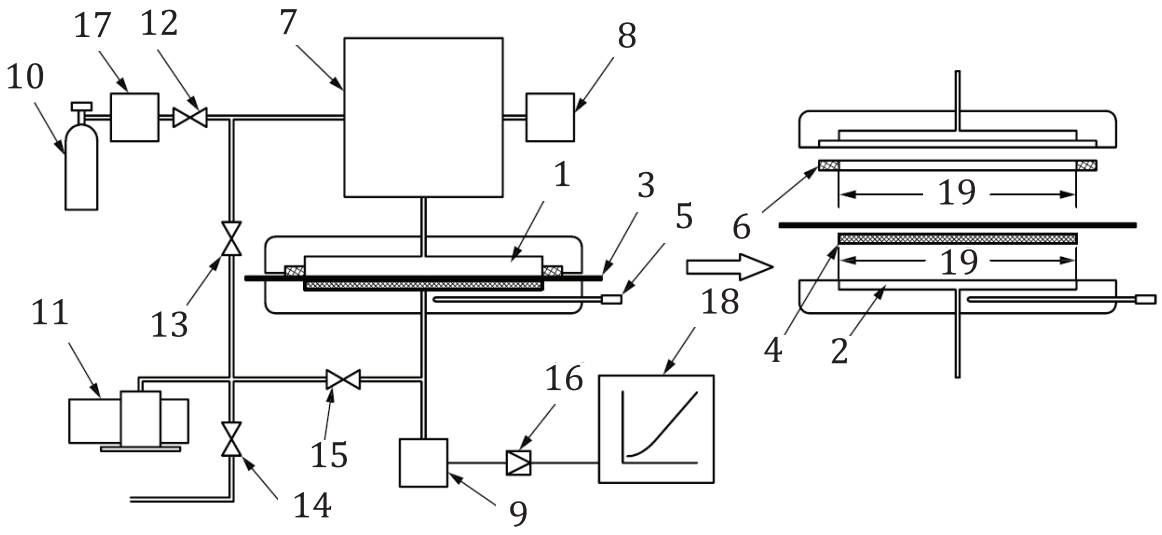
5.1.2 Test piece support, installed on the low-pressure side of the test cell in order to prevent deformation of the test piece due to the pressure difference between the high- and low-pressure sides. Any material, such as filter paper or wire mesh, that does not affect the result of the test may be used. When using filter paper, paper such as that used in chemical analysis is recommended, of thickness 0,1 mm to 0,3 mm, depending on the depth of the low-pressure side of the cell.

5.1.3 Two pressure sensors, the first, capable of reading to within 5 Pa or better, to measure the change in pressure on the low-pressure side of the test cell. A vacuum gauge with no mercury, an electronic diaphragm-type sensor or other suitable sensor shall be used as this pressure sensor. The second, capable of reading to within 1 % or better, is used to measure the pressure of the test gas supply reservoir.

5.1.4 Test gas supply reservoir, for supplying test gas at a constant pressure to the high-pressure side of the test cell. The volume of the reservoir shall be sufficient to ensure that the pressure drop on the high-pressure side, due to transmission of the test gas through the test piece to the low-pressure side during the test, does not exceed 1 % of the test pressure.

5.1.5 Vacuum pump, capable of evacuating the test cell to a pressure of 10 Pa or lower.

5.1.6 Temperature sensor, fitted in the test cell, for measuring the test temperature, and capable of reading to within 0,1 °C or better.



Key

1 high-pressure side of test cell
2 low-pressure side of test cell
3 test piece
4 test piece support
5 temperature sensor
6 sealing ring
7 test gas supply reservoir
8 pressure gauge for test gas supply reservoir
9 pressure sensor for low-pressure side of test cell
10 test gas cylinder
11 vacuum pump
12 valve 1
13 valve 2
14 valve 3
15 valve 4
16 signal amplifier
17 pressure-reducing valve
18 data-processing unit
19 diameter of gas transmission area

NOTE Diagram at right shows an exploded view of the test cell.

Figure 1 — Example of apparatus for gas permeability measurement (pressure sensor method)

5.2 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

5.3 Test gas

Use a single gas, such as nitrogen, oxygen or hydrogen, or a mixture of gases, such as air, liquefied petroleum gas (in gaseous form) or coal gas. The purity of a single gas or the purity of each component in a gas mixture shall be 99,5 % by volume or higher, unless otherwise agreed between the interested

parties, in which case a gas of lower purity may be used. The test gas shall not include any impurity that might affect the measurement.

When using a gas mixture, the purity of each component shall be verified in advance with a suitable instrument, such as a gas chromatograph.

When using a toxic and/or flammable gas, all necessary precautions should be taken in its use and in its recovery or disposal.

5.4 Test pieces

5.4.1 Shape and dimensions

The test pieces shall be of uniform shape and have a thickness of more than 0,1 mm but less than 2,2 mm, unless otherwise agreed between the interested parties. The test pieces shall be large enough to extend across the full width of the test cell and to be clamped between the flat edges of the two halves of the test cell (see 5.1.1).

5.4.2 Preparation

The test pieces shall be cut out and prepared in accordance with ISO 23529. Any test piece containing foreign matter, air bubbles, scratches or holes shall be discarded.

5.4.3 Number of test pieces

Three or more test pieces shall be used except when testing for quality control purposes, in which case the number of test pieces may be reduced.

5.4.4 Measurement of thickness

Measure the thickness of each test piece at five or more points, including the centre of the gas transmission area, to the nearest 0,01 mm using method A specified in ISO 23529:2016, and take the arithmetic mean of the measurements. No single measurement on a test piece shall deviate by more than 10 % from the mean for that test piece, and the mean value for any one test piece shall not deviate by more than 10 % from the mean value for all the test pieces measured.

5.4.5 Time interval between forming and testing

The time interval between forming and testing shall be in accordance with ISO 23529.

Samples and test pieces shall be stored in accordance with ISO 23529 during the interval between forming and testing.

5.5 Conditioning

The minimum time between forming and the commencement of conditioning of the test pieces shall be 16 h.

Unless otherwise required in the material specification or agreed between the interested parties, the material shall be conditioned before testing for 16 h to 24 h at a standard laboratory temperature and humidity specified in ISO 23529. When using a test piece that can be easily affected by moisture, dry it for more than 48 h at the test temperature in a desiccator containing a suitable drying agent, such as anhydrous calcium chloride.

5.6 Test conditions

5.6.1 The laboratory conditions shall be in accordance with ISO 23529.