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Rubber, vulcanized — Accelerated ageing or heat-resistance tests

Caoutchouc vulcanisé — Essais de résistance au vieillissement accéléré ou à la chaleur

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Descriptors : elastomers, vulcanized elastomers, tests, ageing tests (materials), high temperature tests.

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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 45, *Rubber and rubber products*, has reviewed ISO Recommendation R188-1971 and found it technically suitable for transformation. International Standard ISO 188 therefore replaces ISO Recommendation R 188-1971, to which it is technically identical.

ISO Recommendation R 188 had been approved by the member bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Belgium	India	Spain
Brazil	Iran	Sweden
Canada	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep of	Netherlands	United Kingdom
France	New Zealand	U.S.A.
Germany	Poland	U.S.S.R.

No member body had expressed disapproval of the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

Rubber, vulcanized — Accelerated ageing or heat-resistance tests

0 INTRODUCTION

Accelerated ageing or heat-resistance tests are designed to estimate the relative resistance of rubber vulcanizates to deterioration with the passage of time. For this purpose, the rubber is subjected to controlled deteriorating influences for definite periods, after which appropriate properties are measured and compared with the corresponding properties of the unaged rubber.

The purpose of the test may be to assess the deterioration of the rubber either

- a) during prolonged periods at normal or high temperatures in air, or
- b) during use at elevated temperatures and at elevated oxygen pressure.

Two types of test method are given in this International Standard, namely :

- a) air-oven methods (using a cell-type oven or a normal oven);
- b) oxygen pressure method.

The selection of the time, temperature and atmosphere to which the test pieces are exposed will depend on the purpose of the test and the type of polymer.

In the air-oven tests, deterioration is accelerated by raising the temperature and, in the oxygen pressure test, by increasing the oxygen concentration and the temperature. The degree of acceleration thus produced varies from one vulcanizate to another and from one property to another.

Consequences of this are :

- a) Accelerated tests do not truly reproduce under all circumstances the changes produced by natural ageing.
- b) Accelerated tests sometimes fail to indicate accurately the relative natural or service life of different rubbers; thus, tests at temperatures greatly above ambient or service temperatures may tend to equalize the apparent life of rubbers which deteriorate at different rates in storage or service. Tests at one or more intermediate temperatures are useful in assessing the reliability of accelerated ageing at high temperatures.
- c) Accelerated tests involving different properties may not agree in assessing the relative life of different rubbers and may even arrange them in different orders of merit.

Therefore, deterioration should be measured by the changes in property or properties which are of practical importance, provided that they can be measured reasonably accurately.

Attention is drawn to the fact that air-oven and oxygen pressure ageing tests should not be used to simulate natural ageing which occurs in the presence of either light or ozone when the rubbers are stretched.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two types of accelerated ageing or heat-resistance tests on vulcanized rubbers, namely air-oven methods (using either a cell-type oven or a normal oven) and oxygen pressure method.

2 REFERENCE

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

3 ACCELERATED AGEING BY HEATING IN AIR

3.1 Principle

Test pieces are subjected to controlled deterioration by air at an elevated temperature and at atmospheric pressure, after which the physical properties are measured and compared with those of unaged test pieces. The physical properties concerned in the service application are used to measure the deterioration, but in the absence of any statement of these properties, it is recommended that tensile strength, stress at intermediate elongation, breaking elongation and hardness be measured.

In this test, the oxygen concentration is low, and if oxidation is rapid, oxygen may not diffuse into the rubber quickly enough to maintain uniform oxidation. The test is therefore liable to give misleading results with poor-ageing rubbers, when the normal thickness specified in the International Standard appropriate to the test method is used.

3.2 Apparatus

3.2.1 Cell-type oven, consisting of one or more cylindrical vertical cells, having a minimum height of 300 mm and of such dimensions that the space occupied by the test pieces does not exceed 10 % of the capacity of the cell. The cells shall be surrounded by a thermostically controlled good heat transfer medium (aluminium block, liquid bath or saturated vapour).

The design of the apparatus shall be such that heated air will enter the bottom of the cell and be exhausted at the top without being recirculated. Air passing through one cell shall not enter other cells.

Provision shall be made for a slow circulation of air through the cells of not less than three and not more than ten changes per hour.

The temperature of the incoming air shall be within $\pm 1^\circ\text{C}$ of the specified temperature at the point of entry into the cell.

The temperature of the test cells shall be uniform and such that the temperature of the test pieces is kept within $\pm 1^\circ\text{C}$ or $\pm 2^\circ\text{C}$ of the specified test temperature as appropriate for the temperature being used (see 3.7). Suitable means shall be provided for controlling and measuring the temperature.

No copper or copper alloys shall be used in the construction of the heating chamber.

3.2.2 Normal oven, of such a size that the total volume of the test pieces does not exceed 10 % of the free air space of the oven. Provision shall be made for suspending test pieces so that they are at least 10 mm from each other and 50 mm from the sides of the oven.

Provision shall be made for a slow circulation of air through the oven of not less than three and not more than ten changes per hour. Care shall also be taken to ensure that the incoming air is heated to within $\pm 1^\circ\text{C}$ of the temperature of the oven before coming in contact with the test pieces.

The temperature of the oven shall be thermostically controlled so that the temperature of the test pieces is kept within $\pm 1^\circ\text{C}$ or $\pm 2^\circ\text{C}$ of the specified test temperature, as appropriate for the temperature being used, during the whole of the heating period (see 3.7). A thermometer or thermocouple shall be placed near the centre of the test pieces to record the actual test temperature.

No copper or copper alloys shall be used in the construction of the oven chamber.

3.3 Test pieces

It is recommended that the accelerated ageing or heat-resistance test be carried out on test pieces prepared and conditioned as required for the appropriate property tests, and not on complete articles or sample sheets and that their form be such that no mechanical, chemical or heat treatment will be required after ageing.

Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared. The number of test pieces shall be in accordance with the International Standards for the appropriate property tests. The test pieces shall be measured before heating but, whenever possible, marking should be carried out after heating as some marking inks can affect the ageing of the rubber.

Care shall be taken to ensure that the material used for identifying test pieces is not applied in any critical area of the test piece and is not such as to injure the rubber or become destroyed during heating. Care shall also be taken to ensure that the test pieces have a good smooth finish and are free from blemishes and other flaws.

3.4 Time-lapse between vulcanization and testing

The requirements of ISO 1826 shall be observed.

3.5 Procedure

3.5.1 Using cell-type oven

Place the test pieces in the cells after the oven has been pre-heated to the operating temperature, using one compound only in each cell. The test pieces shall be stationary, free from strain, freely exposed to air on all sides and not exposed to light.

When the heating period is complete, remove the test pieces from the cells and condition them for not less than 16 h and not more than 6 days in a strain-free condition and in accordance with the details of the atmosphere given in the appropriate test method for the particular property being studied.

3.5.2 Using normal oven

Place the test pieces in the oven after it has been pre-heated to the operating temperature. The test pieces shall be stationary, free from strain, freely exposed to air on all sides and not exposed to light.

When the heating period is complete, remove the test pieces from the oven and condition them for not less than 16 h and not more than 6 days in a strain-free condition and in accordance with the details of the atmosphere given in the appropriate test method for the particular property being studied.

Avoid simultaneous heating of different types of compound in the same oven, to prevent the migration of sulphur, antioxidants, peroxides or plasticizer. For this purpose, the use of individual cells is highly recommended. In order, however, to give some guidance for cases where it is not practicable to provide equipment for individual cells, it is recommended that only the following types of material be heated together :

- a) polymers of the same general type;
- b) vulcanizates containing the same type of accelerator and approximately the same ratio of sulphur to accelerator;

- c) vulcanizates containing the same type of anti-oxidant;
- d) vulcanizates containing the same type and amount of plasticizer.

3.6 Duration of test

The period required to obtain any given degree of deterioration of the test pieces will depend upon the type of rubber under examination. It is recommended that the test period be 1, 3, 7, 10 or a multiple of 7 days.

The test periods used shall be such that deterioration of the test pieces will not be so great as to prevent determination of the final values of physical properties.

3.7 Temperature of test

The oven shall be maintained at one of the following temperatures :

$70 \pm 1^{\circ}\text{C}$
$85 \pm 1^{\circ}\text{C}$
$100 \pm 1^{\circ}\text{C}$
$125 \pm 2^{\circ}\text{C}$
$150 \pm 2^{\circ}\text{C}$
$175 \pm 2^{\circ}\text{C}$
$200 \pm 2^{\circ}\text{C}$
$250 \pm 3^{\circ}\text{C}$

The product specification should indicate the temperature to be used.

NOTE — CAUTION : As oven temperatures are increased, exposure times may need to be reduced. Further, it should be recognized that the greater the disparity between ageing and service conditions, the less reliable becomes the correlation between ageing and service life.

3.8 Expression of results

The results shall be expressed in accordance with the International Standard for the relevant property tests.

The test results of both the unaged (*O*) and the aged (*A*) test pieces shall be reported together with the percentage change in the value of the property measured (if appropriate) as calculated from the formula

$$\frac{A - O}{O} \times 100$$

where

O is the value of the property before ageing;

A is the value of the property after ageing.

The change in hardness shall be expressed as a difference calculated from the formula $A - O$.

4 ACCELERATED AGEING BY HEATING IN OXYGEN

4.1 Principle

Test pieces are exposed to an elevated temperature and an elevated oxygen pressure, after which the physical properties are measured and compared with those of unaged test pieces. The physical properties concerned in the service application should be used to determine the degree of deterioration, but in the absence of any statement of these properties, it is recommended that tensile strength, stress at intermediate elongation, breaking elongation and hardness be measured.

In this test, the increased oxygen concentration promotes rapid diffusion and so helps to ensure uniform oxidation. On the other hand, the artificial promotion of oxidation may over-emphasize oxidative changes relative to those caused by after-vulcanization, so that the total effect may not resemble that of natural ageing.

4.2 Apparatus

4.2.1 Oxygen pressure chamber, consisting of a vessel of stainless steel or other suitable material designed to retain an internal atmosphere of oxygen under pressure, with provision for placing rubber test pieces within it and subjecting them to a controlled uniform temperature. The size of the vessel is optional, but shall be such that the total volume of the test pieces does not exceed 10 % of the free gas space of the vessel.

No copper or brass parts shall be within the ageing chamber or used in the construction of the tubing leading from the oxygen reservoir to the ageing chamber.

4.2.2 Thermostat, for controlling the temperature of the heating medium¹⁾ surrounding the pressure vessel so that the temperature of the test pieces in the pressure chamber is kept at $70 \pm 1^{\circ}\text{C}$.

4.2.3 Thermocouple or other suitable device, placed near the centre of the ageing test pieces to record the actual test temperature.

4.2.4 Reliable safety valve, set a gauge pressure of 3,5 MPa*.

4.2.5 Pressure gauge.

1) For safety reasons a combustible fluid such as mineral oil should not be used as a heating medium in an apparatus such as this which employs oxygen.

* 1 MPa = 1 MN/m²

4.3 Test pieces

It is recommended that the accelerated ageing or heat-resistance test be carried out on test pieces prepared and conditioned as required for the appropriate property tests, and not on complete articles or sample sheets, and that their form be such that no mechanical, chemical or heat treatment will be required after ageing.

Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared with each other. The number of test pieces shall be in accordance with the International Standard for the appropriate property tests. The test pieces shall be measured before heating but, whenever possible, marking should be carried out after heating as some marking inks can affect the ageing of the rubber.

Care shall be taken to ensure that the material used for identifying test pieces is not applied in any critical area of the test piece and is not such as to injure the rubber or become destroyed during ageing. Care shall also be taken to ensure that the test pieces have a good smooth finish and are free from blemishes and other flaws.

4.4 Time-lapse between vulcanization and testing

The requirements of ISO 1826 shall be observed.

4.5 Procedure

Suspend the test pieces vertically in the pressure chamber after it has been heated to the operating temperature. Before commencing the test, flush the air out of the vessel by releasing the oxygen pressure and refilling. The test pieces in the vessel shall be stationary, free from strain and freely exposed to the oxygen on all sides.

Pass oxygen into the pressure chamber to give a gauge pressure of $2,1 \pm 0,1$ MPa at 70°C ; the exposure shall be continuous for the specified time, without pressure reduction or opening of the chamber.

When the ageing period is complete, relieve the pressure in the pressure chamber slowly and uniformly, during at least 5 min. Remove the test pieces from the vessel and condition for not less than 16 h and not more than 6 days in a strain-free condition and in accordance with the details of the atmosphere given in the appropriate test method for the particular property being studied.

Avoid simultaneous ageing of different types of compounds to prevent migration of sulphur, antioxidants, peroxides or plasticizers. For this purpose, the use of individual pressure chambers is highly recommended. In order, however, to give some guidance for such cases where it is not practicable to provide equipment for individual pressure chambers, it is recommended that only the following types of material be aged together :

- a) polymers of the same general type;
- b) vulcanizates containing the same type of accelerator and approximately the same ratio of sulphur to accelerator;

c) vulcanizates containing the same type of anti-oxidant;

d) vulcanizates containing the same type and amount of plasticizer.

NOTE — Adequate safety precautions are important when heating oxidizable organic materials in oxygen under pressure, since the rate of oxidation may, in some cases, become very rapid, particularly if a large surface area of material is exposed.

4.6 Duration of test

The period required to obtain any given degree of deterioration of the test pieces will depend on the type of rubber under examination. With a view to establishing uniformity of practice, it is recommended that the ageing period be 24 h or a multiple thereof.

4.7 Temperature and pressure of test

The test pieces shall be aged at a temperature of $70 \pm 1^\circ\text{C}$ and a gauge pressure of $2,1 \pm 0,1$ MPa.

4.8 Expression of results

The results shall be expressed in accordance with the International Standards for the appropriate property tests.

The test results of both the unaged (*O*) and the aged (*A*) test pieces shall be reported together with the percentage change in the value of the property measured (if appropriate) as calculated from the formula :

$$\frac{A - O}{O} \times 100$$

where

O is the value of the property before ageing;

A is the value of the property after ageing.

The change in hardness shall be expressed as a difference calculated from the formula $A - O$.

5 TEST REPORT

The test report shall include the following particulars :

- a) a reference to this International Standard;
- b) whether an air-oven method (cell oven or normal oven) or the oxygen pressure method was used;
- c) the duration and temperature of ageing;
- d) the properties determined, with their individual values before and after ageing and, if appropriate, the percentage change;
- e) any test conditions and operations not provided for in this International Standard or which are optional, as well as any incidents which may have affected the results.