INTERNATIONAL STANDARD

ISO 188

Third edition
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Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests

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

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 188 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products,* Subcommittee SC 2, *Physical and degradation tests.*

This third edition cancels and replaces the second edition (ISO 188:1982), which has been technically revised.

Annex A of this International Standard is for information only.

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Introduction

Accelerated ageing and heat resistance tests are designed to estimate the relative resistance of rubber 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 accelerated ageing test may be to assess the deterioration of the rubber

a) either during prolonged periods at normal or elevated temperatures in air;

b) or during prolonged periods at elevated temperatures and at elevated oxygen pressure.

In accelerated ageing, the rubber is subjected to a test environment intended to produce the effect of natural ageing in a shorter time.

In the case of heat resistance tests, the rubber is subjected to prolonged periods at the same temperature as that which it will experience in service.

Three types of method are given in this International Standard, namely an air-oven method using a low air speed, an air-oven method using forced air ventilation and an oxygen pressure method.

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

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

Degradation can also be accelerated by air speed. Consequently, ageing with different ovens can give different results.

Consequences of this are:

a) Accelerated ageing does not truly reproduce under all circumstances the changes produced by natural ageing.

b) Accelerated ageing sometimes fails to indicate accurately the relative natural or service life of different rubbers; thus, ageing 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. Ageing at one or more intermediate temperatures is useful in assessing the reliability of accelerated ageing at high temperatures.

c) Accelerated ageing tests involving different properties may not give agreement in assessing the relative lives 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.

Air-oven and oxygen pressure ageing should not be used to simulate natural ageing which occurs in the presence of either light or ozone when the rubbers are stretched.

To estimate lifetime or maximum temperature of use, tests can be performed at several temperatures and the results can be evaluated by using an Arrhenius plot. This method is described in ISO 11346.

Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests

WARNING – Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, asssociated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies accelerated ageing or heat resistance tests on vulcanized or thermoplastic rubbers. The methods are:

Method A: air-oven method using a cell-type oven or cabinet oven with low air speed and a ventilation of 3 to 10 changes per hour;

Method B: air-oven method using a cabinet oven with forced air circulation by means of a fan and a ventilation of 3 to 10 changes per hour; and

Method C: oxygen pressure method at 2,1 MPa and 70 °C.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties.*

ISO 48:1994, *Rubber, vulcanized or thermoplastic – Determination of hardness (hardness between 10 IRHD and 100 IRHD).*

ISO 471:1995, *Rubber – Temperatures, humidities and times for conditioning and testing*.

ISO 11346:1997, *Rubber, vulcanized or thermoplastic – Estimation of life-time and maximum temperature of use from an Arrhenius plot*.

3 Principle

Test pieces are subjected to controlled deterioration by air at an elevated temperature and at atmospheric pressure (for 3.1 and 3.2) or at an elevated temperature and an elevated oxygen pressure (for 3.3), 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 indication of these properties, it is recommended that tensile strength, stress at intermediate elongation, elongation at break (in accordance with ISO 37) and hardness (in accordance with ISO 48) be measured.

3.1 Accelerated ageing by heating in air

In this method, the oxygen concentration is low and, if oxidation is rapid, oxygen may not diffuse into the rubber quickly enough to maintain uniform oxidation. This ageing method 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 Heat resistance test

In this method, the test pieces are subjected to the same temperature as they would experience in service and, after definite periods, appropriate properties are measured and compared with those of the unaged rubber.

3.3 Accelerated ageing by heating in oxygen

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

4 Apparatus

4.1 **Air oven** (for 3.1 and 3.2)

To achieve a good precision when doing ageing and heat resistance tests, it is very important to keep the temperature uniform and stable during the test and to verify that the oven used is within the temperature limits with regard to time and space. Increasing the air speed in the oven improves temperature homogeneity.

However, air circulation in the oven and ventilation influence the ageing results. With a low air speed, accumulation of degradation products and of evaporated ingredients, as well as oxygen depletion, can take place. A high air speed increases the rate of deterioration, due to increased oxidation and volatilization of plasticizers and antioxidants.

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

The temperature of the oven shall be controlled so that the temperature of the test pieces is kept within the specified tolerance for the specified ageing temperature (see clause 7) for the whole ageing period. A temperature sensor shall be placed inside the heating chamber to record the actual ageing temperature.

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

For the ovens specified in 4.1.1 and 4.1.2, provision shall be made for a slow flow of air through the oven of not less than three and not more than ten air changes per hour. The air speed shall depend on the air change rate only, and no fans are allowed inside the heating chamber.

Care shall also be taken to ensure that the incoming air is heated to within ±1 °C of the temperature of the oven before coming in contact with the test pieces.

The ventilation (or air change rate) can be determined by measuring the volume of the oven chamber and the flow of air through the chamber.

4.1.1 Cell-type oven, consisting of one or more vertical cylindrical cells having a minimum height of 300 mm. The cells shall be surrounded by a thermostatically controlled good-heat-transfer medium (aluminium block, liquid bath or saturated vapour). Air passing through one cell shall not enter other cells.

4.1.2 Cabinet oven, comprising a single chamber without separating walls.

4.1.3 Oven with forced air circulation, with an air speed of 0,5 m/s to 1,5 m/s. The air flow through the heating chamber shall be as uniform and laminar as possible. The test pieces shall be placed with the smallest surface facing towards the air flow direction to avoid disturbing the air flow.

The air shall be changed at a rate of not less than three and not more than ten air changes per hour.

The air speed near the test pieces can be measured by means of an anemometer.

4.2 Apparatus for heating in oxygen (for 3.3)

4.2.1 Oxygen pressure chamber, consisting of a vessel, made of stainless steel or another suitable material, designed to contain an 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 space in the vessel.

No copper or copper alloy parts shall be inside the pressure chamber or in the construction of the tubing leading from the oxygen reservoir to the pressure chamber.

4.2.2 Thermostat, for controlling the temperature of the heating medium[1] surrounding the pressure vessel so that the temperature of the test pieces in the pressure chamber is kept at 70 °C ±1 °C.

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

4.2.4 Reliable safety valve, set at a gauge pressure of 3,5 MPa [2].

4.2.5 Pressure gauge.

5 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 products 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 shall be carried out after heating as some marking inks can affect the ageing of the rubber.

Care shall be taken to ensure that the markings used to identify the test pieces are not applied in any critical area of the test piece and are not such as to damage the rubber or to disappear 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.

Avoid simultaneous heating of different types of compound in the same oven, to prevent the migration of sulfur, antioxidants, peroxides or plasticizers. 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 with 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 sulfur to accelerator;

c) rubbers containing the same type of antioxidant;

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

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

[2] 1 MPa = 1 MN/m^2

6 Time interval between vulcanization and testing

The requirements of ISO 471 shall be observed.

7 Ageing conditions (duration, temperature and pressure)

7.1 General

The period required to obtain a given degree of deterioration of the test pieces will depend upon the type of rubber under examination.

The ageing period 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.

The use of high ageing temperatures may result in different degradation mechanisms than those which occur at service temperatures, thus invalidating the results.

7.2 Accelerated ageing

The duration of ageing and the ageing temperature shall be chosen in accordance with ISO 471 (including tolerances), as stated in the product specification or as agreed between the interested parties. The ageing shall be performed at atmospheric pressure.

7.3 Heat resistance test

The test duration and the temperature of test shall be chosen in accordance with ISO 471 (including tolerances), as stated in the product specification or as agreed between the interested parties. The temperature shall be representative of the service temperature and the heating carried out at atmospheric pressure.

7.4 Accelerated ageing in oxygen

The duration of ageing shall be chosen in accordance with ISO 471 (including tolerances). The test pieces shall be aged at a temperature of 70 °C ±1 °C and at an oxygen pressure of 2,1 MPa ± 0,1 MPa. Other ageing conditions may be used as stated in the product specification or as agreed between the interested parties.

8 Procedure

8.1 Accelerated ageing in air and heat resistance test

Heat the oven to the operating temperature and place the test pieces in it. When using a cell-type oven, only one rubber or compound shall be placed 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 oven and condition them for not less than 16 h and not more than 6 days in a strain-free condition in the atmosphere given in the appropriate test method for the particular property being studied.

8.2 Accelerated ageing in oxygen

Heat the pressure chamber to the operating temperature and suspend the test pieces in it. Before starting ageing, flush the air out of the chamber by pressurizing with oxygen and releasing the pressure several times. The test pieces in the chamber 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 MPa ± 0,1 MPa at 70 °C ±1 °C; the exposure shall be continous for the specified time, without any pressure reduction or opening of the chamber.

When the ageing period is complete, release the pressure in the pressure chamber slowly and uniformly over a period of at least 5 min. Remove the test pieces from the chamber and condition for not less than 16 h and not

more than 6 days in a strain-free condition in the atmosphere given in the appropriate test method for the particular property being studied.

CAUTION – 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.

9 Expression of results

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

The test results for both the unaged and the aged test pieces shall be reported together, as well as, when appropriate, the percentage change in the value of the property measured as calculated from the formula:

$$\frac{x_a - x_0}{x_0} \cdot 100$$

where

x_0 is the value of the property before ageing;

x_a is is the value of the property after ageing.

Express changes in hardness as the difference $x_a - x_0$.

10 Precision

10.1 General

The interlaboratory test program (ITP) and the precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272:1986, *Rubber and rubber products – Determination of precision for test method standards*. Consult this for precision concepts and nomenclature. Annex A gives guidance on the use of repeatability and reproducibility results.

10.2 Precision details

10.2.1 The ITP was organized in 1996 and the results analysed in 1997. Prepared test pieces were sent out to all participating laboratories using four compounds (of types NR, NBR, EPDM and AEM). Ageing was carried out by method A and method B.

The ageing time was 168 h for all compounds at 70 °C for NR materials, 100 °C for NBR materials, 125 °C for EPDM materials and 150 °C for AEM materials.

10.2.2 A total of 16 laboratories participated in this ITP. Eleven of the laboratories carried out the ageing by method A and ten laboratories by method B. Five of the laboratories used both method A and B. For certain of the tests carried out after ageing, values were missing from the compiled data, and for these tests less than these numbers of laboratories were involved. The actual number for each test is listed in the precision tables.

10.2.3 The hardness was measured in accordance with ISO 48, method M, on three-ply dumbells before and after ageing. The three tensile strength properties were measured in accordance with ISO 37 on five test pieces before and after ageing. Type 1 and type 2 test pieces were used.

10.2.4 The performance parameter for hardness was taken as the difference in the IRHD values before and after ageing. The performance parameter for the three tensile properties was taken as the percentage change in each property during ageing.

10.2.5 The precision determined in this ITP is a type 1 precision, i.e. fully prepared test pieces were submitted to all laboratories. The precision is also an intermediate-term or intermediate time period precision, with a time of 2 to 3 weeks between the two replicate determinations. This is n distinction to the more usual day 1 to day 2 replication with a few days between the determinations.