INTERNATIONAL STANDARD

ISO 188

Fifth edition
2011-10-01

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

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

Reference number
ISO 188:2011(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 188 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 188:2007), of which it constitutes a minor revision to include an annex (Annex D) specifying a calibration schedule for the apparatus used.

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.

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.

Two types of method are given in this International Standard, namely an air-oven method using a low air speed and an air-oven method using forced air ventilation for a high air speed.

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 air-oven methods, deterioration is accelerated by raising 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 these effects 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 lives 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 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 or the Williams Landel Ferry (WLF) equation as described in ISO 11346[2].

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, associated 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. Two methods are given:

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.

2 Normative references

The following referenced documents are indispensable for the application 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 37, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

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

3 Principle

3.1 General

Test pieces are subjected to controlled deterioration by air at an elevated temperature and at atmospheric pressure, after which specified 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.

3.2 Accelerated ageing by heating in air

In this method, the test pieces are subjected to a higher temperature than the rubber would experience in service in order to produce the effects of natural ageing in a shorter time.

3.3 Heat resistance test

In this method, the test pieces are subjected to the same temperature as they would experience in service.

4 Apparatus

4.1 Air oven

4.1.1 General

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 8) for the whole ageing period. A temperature sensor shall be placed inside the heating chamber close to the samples to record the actual ageing temperature.

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

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.

Care shall 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.

NOTE To ensure 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 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.

4.1.2 Cell-type oven

The oven shall consist 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.

Provision shall be made for a slow flow of air through the cell. The air speed shall depend on the air change rate only.

4.1.3 Cabinet oven

This shall comprise a single chamber without separating walls. Provision shall be made for a slow flow of air through the oven. The air speed shall depend on the air change rate only, and no fans are allowed inside the heating chamber.

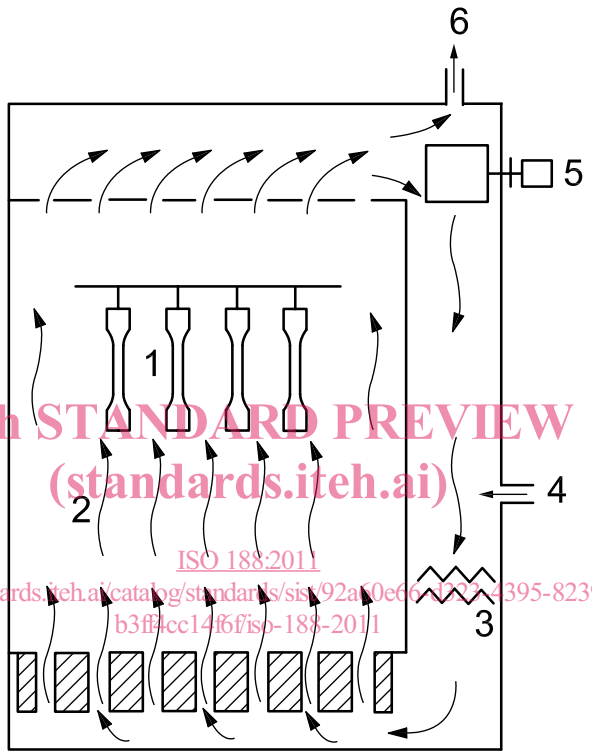
4.1.4 Oven with forced air circulation

Either of the following two types shall be used:

a) Type 1 oven with laminar air flow (see Figure 1).

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 to avoid disturbing the air flow. The air speed shall be between 0,5 m/s and 1,5 m/s.

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



Key

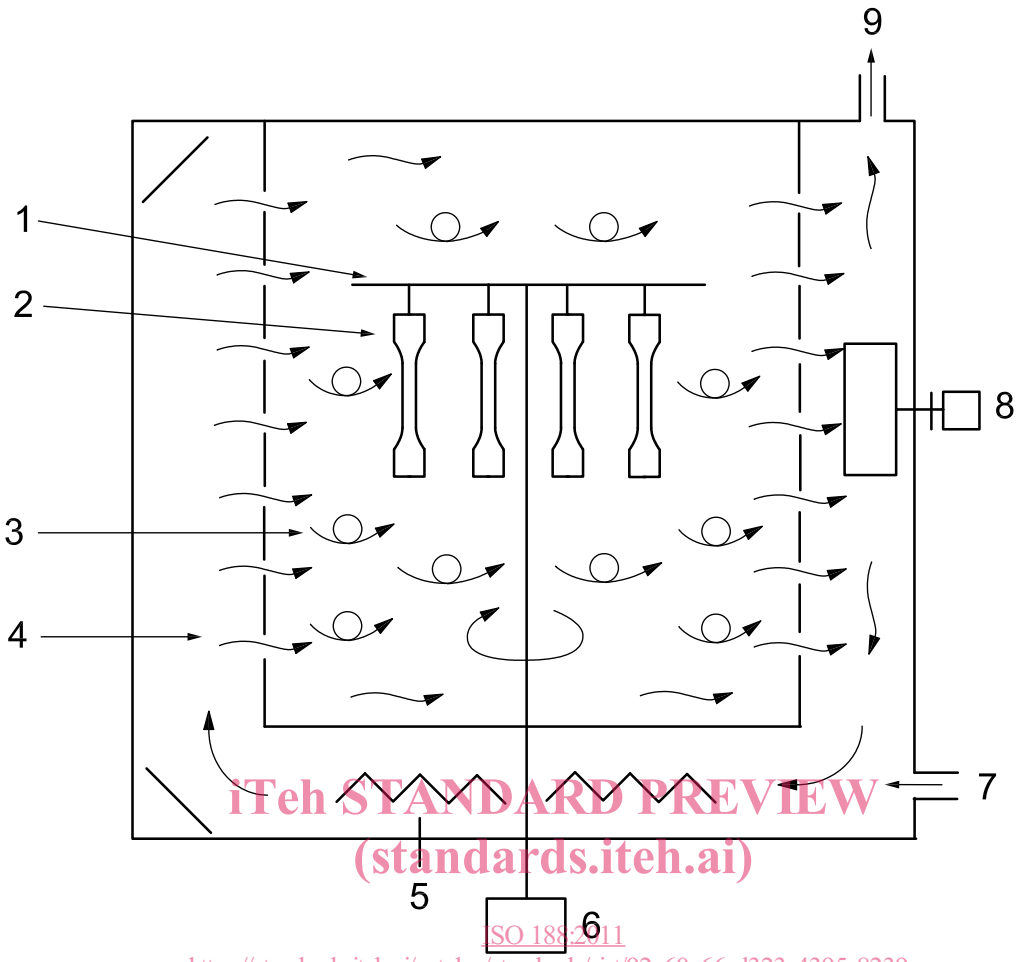
1 test pieces
2 laminar air flow
3 heating element
4 air inlet
5 air blower
6 air outlet

Figure 1 — Type 1 oven with laminar air flow

b) Type 2 oven with turbulent air flow (see Figure 2).

The air entering from a side-wall air-inlet into the heating chamber is turbulent around the test pieces, which are suspended on a carrier rotating at a speed of five to ten rotations per minute so that they are exposed to the heating air as uniformly as possible. The average air speed shall be 0,5 m/s ± 0,25 m/s.

The average air speed near the test pieces can be calculated from measurements made with an anemometer at nine different positions (see Figure A.1 in Annex A). A suitable method of measurement is described in Annex A.



Key

1 test piece carrier
2 test pieces
3 turbulent air flow
4 laminar air flow (inlet, outlet and near to wall)
5 heating element
6 motor
7 air inlet
8 air blower
9 air outlet

Figure 2 — Type 2 oven with turbulent air flow

5 Calibration

The test apparatus shall be calibrated in accordance with Annex D.

6 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 will not damage the rubber or disappear during heating.

Avoid simultaneous heating of different types of compound in the same oven, to prevent the migration of sulphur, 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 sulphur to accelerator;

c) rubbers containing the same type of antioxidant;

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

7 Time interval between vulcanization and testing

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

For all normal test purposes, the minimum time between vulcanization and testing shall be 16 h. In cases of arbitration, the minimum time shall be 72 h.

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

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

8 Ageing conditions (duration and temperature)

8.1 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 should preferably 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.

It is crucial for the best results that the temperature be kept as stable as possible. Temperature tolerances in ISO 23529 are ±1 °C up to and including 100 °C and ±2 °C for 125 °C up to and including 300 °C. However, studies have shown that a 1 °C change in temperature corresponds to a 10 % difference in ageing time at an Arrhenius factor of 2, or 15 % at a factor of 2,5. This means that two laboratories carrying out ageing at 125 °C can have ageing times which differ by 60 % from each other and still be within the specification. To get accurate results, keep the temperature as accurate as possible by placing a calibrated temperature sensor close to the test pieces and use this to set the oven so that the temperature at this position is correct. Use the correction factor from the calibration certificate to get as close as possible to the true temperature.