

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 188

VULCANIZED RUBBERS

ACCELERATED AGEING OR HEAT RESISTANCE TESTS

2nd EDITION

November 1971

This second edition supersedes the first edition

COPYRIGHT RESERVED

The copyright of ISO Recommendations and ISO Standards belongs to ISO Member Bodies. Reproduction of these documents, in any country, may be authorized therefore only by the national standards organization of that country, being a member of ISO.

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 188, *Accelerated ageing or simulated service tests on vulcanized natural or synthetic rubbers*, 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. 171, which was circulated to all the ISO Member Bodies for enquiry in July 1957.

The Draft was approved by 26 Member Bodies. One Member Body opposed the approval of the Draft : France.

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

BRIEF HISTORY RELATING TO THE SECOND EDITION

Work started on the revision of ISO Recommendation R 188 soon after its publication in 1961 as there appeared to be certain inadequacies. Draft ISO Recommendation No. 1630 was adopted as a result and was circulated to all the ISO Member Bodies for enquiry in July 1968.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

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 the second edition of ISO Recommendation R 188.

The title of this ISO Recommendation has been modified as follows : *Vulcanized rubbers – Accelerated ageing or heat resistance tests*.

This edition (second edition) cancels and replaces the first edition of ISO Recommendation R 188.

VULCANIZED RUBBERS

ACCELERATED AGEING OR HEAT RESISTANCE TESTS

INTRODUCTION

Accelerated ageing tests or exposure to heat 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 at high temperature in air, or
- (b) during use at elevated temperatures and at elevated oxygen pressure.

Two types of test method are given in this ISO Recommendation, 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) They 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 using 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 the property or properties which are of practical importance, provided 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

This ISO Recommendation describes accelerated ageing or heat resistance tests on vulcanized rubbers. Two types of test method are recommended : air oven methods (using either a cell type oven or a normal oven) and the oxygen pressure method.

2. ACCELERATED AGEING BY HEATING IN AIR

2.1 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 should 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 ISO Recommendation appropriate to the test method is used.

2.2 Apparatus

2.2.1 Cell type oven. The apparatus should consist 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 should be surrounded by a thermostatically controlled good heat transfer medium (aluminium block, liquid bath or saturated vapour).

The design of the apparatus should 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 should not enter other cells.

Provision should be made for a slow circulation of air through the cells of not less than three nor more than ten changes per hour.

The incoming air should 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 should be uniform and such that the test pieces are 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 clause 2.7). Suitable means should be provided for controlling and measuring the temperature.

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

2.2.2 Normal oven. The oven should be 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 should 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 should be made for a slow circulation of air through the oven of not less than three nor more than ten changes per hour. Care should also be taken 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 should be thermostatically controlled so that the test pieces are 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 clause 2.7). A thermometer or thermocouple should be placed near the centre of the test pieces to record the actual test temperature.

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

2.3 Test pieces

It is recommended that the ageing or heat resistance test should 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 their form should be such that no mechanical, chemical or heat treatment will be required after the ageing or heat resistance test.

Only test pieces of similar dimensions and having approximately the same exposed areas should be compared. The number of test pieces should be in accordance with the ISO Recommendations made for the appropriate property tests. The test pieces should 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 should be taken 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 should be taken also to ensure that the test pieces have a good smooth finish and are free from blemishes and other flaws.

2.4 Time lapse between vulcanization and testing

The requirements of ISO Recommendation R 1826, *Time lapse between vulcanization and testing of rubbers*, should be observed.

2.5 Procedure

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

When the heating period is complete, the test pieces should be removed from the cells and conditioned for not less than 16 hours nor 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.

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

When the heating period is complete, the test pieces should be removed from the oven and conditioned for not less than 16 hours nor 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.

Simultaneous heating of different types of compound in the same oven should be avoided, in order that migration of sulphur, antioxidants, peroxides or plasticizer does not occur, and therefore the use of individual cells is highly recommended. In order, however, to give some guidance for such cases where it is not practicable to provide equipment for individual cells, it is recommended that only the following should 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 antioxidant;
- (d) vulcanizates containing the same type and amount of plasticizer.

2.6 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 should be, 1, 3, 7, 10 or some multiple of 7 days.

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

2.7 Temperature of test

The oven should be at one of the following temperatures :

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

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.

2.8 Expression of results

The results should be expressed in accordance with the ISO Recommendations made for the appropriate property tests.

The test results of both the unaged (O) and the aged (A) test pieces should be reported as well as the coefficient of deterioration (if appropriate for the property measured) as calculated from the following formula :

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

where

O is the value of the property before ageing and

A is the value of the property after ageing.

3. AGEING BY HEATING IN OXYGEN

3.1 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 should be measured.

In this test, the increased oxygen concentration promotes rapid diffusion and so helps to give 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.

3.2 Apparatus

The oxygen pressure chamber should consist 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 should 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 should be within the ageing chamber nor used in the construction of the tubing leading from the oxygen reservoir to the ageing chamber.

The temperature of the heating medium* which surrounds the pressure vessel should be thermostatically controlled so that the temperature of the test pieces in the pressure chamber is kept at $70 \pm 1 \text{ }^{\circ}\text{C}$. A thermocouple or other suitable device should be placed near the centre of the ageing test pieces to record the actual test temperature.

The pressure chamber should be equipped with a reliable safety valve set at a gauge pressure of 3.5 MN/m^2 and a pressure gauge should be connected to the apparatus.

* 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.