BRITISH STANDARD

BS 903-A19: 1986
ISO 188-1982

Methods of testing vulcanized rubber —

Part A19: Heat resistance and accelerated ageing tests —

[ISO title: Rubber, vulcanized — Accelerated ageing or heat-resistance tests]

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Rubber Standards Committee (RUM/-) to Technical Committee RUM/36, upon which the following bodies were represented:

British Railways Board
British Rubber Manufacturers' Association
ERA Technology Ltd.
GAMBICA (BEAMA Ltd.)
Institution of Mechanical Engineers
Institution of Water Engineers and Scientists
Malaysian Rubber Producers' Research Association
Ministry of Defence
National College of Rubber Technology
Rubber and Plastics Research Association of Great Britain
SATRA Footwear Technology Centre

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Contents

National foreword

This revision of BS 903-A19 has been prepared under the direction of the Rubber Standards Committee. It is identical with ISO 188-1982 "*Rubber, vulcanized — Accelerated ageing or heat resistance tests*" published by the International Organization for Standardization (ISO).

The major changes in this revision compared with the 1975 edition are as follows.

a) The oxygen pressure method has been reintroduced in order to satisfy renewed demand (see clause **4**).

b) In the air oven method, the procedures using a cell type oven and a single container oven are no longer referred to as methods A and B respectively (see clause **3**).

c) Also in the air oven method, the list of preferred test temperatures with lower and upper values of 55 ± 1 °C and 250 ± 3 °C respectively has been superseded by a list of specified test temperatures with lower and upper values of 70 ± 1 °C and 300 ± 3 °C respectively (see **3.7**).

NOTE Other test temperatures, from the list given in BS 903-A35, may be used if product specifications require their use.

d) For all methods, the volume occupied by the test pieces is now required to be not more than 10 % of the capacity of the ageing chamber (cell or oven) (see **3.2.1**, **3.2.2** and **4.2.1**).

e) The use of specially prepared test pieces is no longer a requirement but is still recommended in preference to the exposure of complete articles or sample sheets (see **3.3** and **4.3**).

This edition of BS 903-A19 supersedes the 1975 edition, which is withdrawn.

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words "International Standard" appear, referring to this standard, they should be read as "Part of BS 903".

Cross-reference. The Technical Committee has reviewed the provisions of ISO 1826, to which reference is made in **3.4**, and has decided that they are acceptable for use in conjunction with this standard. The relevant requirements of ISO 1826 are reproduced in National appendix A for the convenience of users of this British Standard.

Additional information. In attempting to predict the behaviour of actual rubber articles from the tests described in this standard, consideration should be given to the following effects of thickness.

a) Where rubber articles are a few centimetres thick, the bulk of the material may experience little or no ageing even though the surface and test specimens of the same rubber are very badly aged. For even moderately sized items, most of the bulk property changes are likely to occur under anaerobic conditions and thus accelerated and heat ageing tests on thin samples in the presence of air can give gross underestimates of the useful life of a product.

b) In the air ageing tests, oxygen is at its normal atmospheric concentration and, if oxidation is rapid, may not diffuse into the rubber quickly enough to maintain uniform oxidation unless the test pieces are sufficiently thin. It is not possible to stipulate definitive safe thickness limits since different rubbers can have widely different oxygen diffusion rates and oxidation rates. However, as a working guide for reasonably well protected rubbers that do not age very rapidly, a thickness of 2.0 ± 0.2 mm should prove satisfactory and should be used. At temperatures above 100 °C and for materials that age rapidly, this thickness may be too great. The uniformity of ageing is best checked by determining the aged tensile stress at a given elongation at a number of thicknesses. Tensile strength and hardness, unlike tensile stress at a given elongation, can be considerably affected by surface conditions and the effect of thickness on these two properties may be less pronounced. Comparisons should only be made between test pieces of similar thickness.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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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, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

3 Accelerated ageing by heating in air

3.1 Principle

Tests 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).

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 ± 1 °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 ± 1 °C or ± 2 °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 ± 1 °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 ± 1 °C or ± 2 °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 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 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 interval 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 preheated 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 preheated 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 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 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 antioxidant;

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 ± 1 °C	175 ± 2 °C
85 ± 1 °C	200 ± 2 °C
100 ± 1 °C	250 ± 3 °C
125 ± 2 °C	275 ± 3 °C
150 ± 2 °C	300 ± 3 °C

The product specification should indicate the temperature to be used.

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 appropriate property tests.

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

$$\frac{x_a - x_o}{x_o} \times 100$$

where

x_o is the value of the property before ageing;

x_a is the value of the property after ageing.

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

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 overemphasize 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 medium[1)] surrounding the pressure vessel so that the temperature of the test pieces in the pressure chamber is kept at 70 ± 1 °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 at a gauge pressure of 3,5 MPa.[2)]

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.

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

4.3 Test pieces

It is recommended that the accelerated ageing by heating in oxygen 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 interval 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 ± 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 compound 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 antioxidant;

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

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.

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 ± 1 °C and a gauge pressure of 2,1 ± 0,1 MPa.

4.8 Expression of results

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

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

$$\frac{x_a - x_o}{x_o} \times 100$$

where

x_o is the value of the property before ageing;

x_a is the value of the property after ageing.

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

5 Test report

The test report shall include the following information:

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 regarded as optional, as well as any incidents which may have affected the results.

National appendix A Time-lapse between vulcanization and testing of rubber test pieces and products

A.1 Introduction

This National appendix reproduces the details of ISO 1826 "Rubbers — Time-lapse between vulcanization and testing". Requirements for the time-lapse between vulcanization and testing of rubber test pieces and products are necessary in order to assist in obtaining reproducible results and to minimize disagreement between the customer and the supplier.

A.2 Time-lapse requirements

A.2.1 Unless otherwise specified for technical reasons, the following requirements for time-lapses shall be observed.

A.2.2 For all the test purposes, the minimum time between vulcanization and testing shall be 16 h.

A.2.3 For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and, for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

A.2.4 For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the customer.

Publications referred to

See national foreword.

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