BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

PART A 19. ACCELERATED AGEING TESTS

B.S. 903 : Part A 19 : 1956

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BRITISH STANDARDS INSTITUTION

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THIS BRITISH STANDARD, having been approved by the Rubber Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 1st May, 1956.

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The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

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The following B.S.I. references relate to the work on this standard:—
Committee reference RUC/10 Draft for comment CV(RUC) 1058

CO-OPERATING ORGANIZATIONS

The Rubber Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:—

Board of Trade
*British Rubber Producers' Research Association
*Federation of British Rubber and Allied Manufacturers' Associations
*Institution of the Rubber Industry
*Ministry of Supply
Natural Rubber Development Board
*Research Association of British Rubber Manufacturers
*Rubber Growers' Association

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:—

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Association of British Chemical Manufacturers
British Chemical Plant Manufacturers' Association
British Electrical and Allied Industries Research Association
British Railways, The British Transport Commission
British Rubber Adhesive Manufacturers' Association
Department of the Government Chemist
General Post Office
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BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

Part A 19. Accelerated Ageing Tests*

FOREWORD

This British Standard has been published under the authority of the Rubber Industry Standards Committee. In deciding to issue a revision of the 1950 edition, it has also been considered desirable to publish B.S. 903 in separate parts and the present part replaces Part 13 of 1950, from which it differs mainly in the inclusion of a method using the cell type oven and the deletion of the air pressure method.

The group of parts in which the prefix letter ' A ' is used covers methods of testing the physical properties of rubber. Further parts in this group have been issued as follows:—

Part A 1. Determination of density and specific gravity.
Part A 2. Determination of rensile stress-strain.
Part A 3. Determination of tear strength.
Part A 10. Determination of flex cracking.
Part A 11. Determination of resistance to crack growth.
Part A 16. Determination of swelling.
Part A 18. Determination of equilibrium water vapour absorption.

SECTION 1 GENERAL EXPLANATION

Accelerated ageing 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 untreated rubber.

In the forms of ageing test here specified deterioration is accelerated by raising the temperature and, in the oxygen-pressure test, by increasing the oxygen concentration.

The degree of acceleration thus produced varies from one vulcanizate to another and also from one property to another. Consequences of this are:

a. Accelerated tests do not truly reproduce the changes caused by by natural ageing.

* *These methods are substantially those being considered by the technical committee of the International Organization for Standardization (ISO) dealing with rubber.*

b. They do not always predict accurately the relative natural or service life of different rubbers; thus, raising the temperature may tend to equalize the apparent life of rubbers which deteriorate at different rates under natural ageing conditions.

c. Different accelerated tests do not agree in assessing the relative life of different rubbers, and may even arrange them in different orders of merit.

Deterioration caused by accelerated ageing should therefore be measured by the changes in the property or properties which are of practical importance, provided these can be measured accurately, since no one property can be used as a general index of degree of deterioration. *As amended Oct., 1963*

Attention is drawn to the fact that these ageing tests should not be used to simulate natural ageing which occurs in the presence of light or of ozone when the rubbers are stretched.

In this standard three methods for evaluating accelerated ageing are described:

Method A. Cell type oven method.
Method B. Oven method.
Method C. Oxygen pressure method.

In the case of the oven tests Method A is preferred.

Simultaneous ageing in one oven of different types of compound should be avoided in Methods B and C in order that migration of sulphur, antioxidants, peroxides or plasticizers may not occur, and therefore the use of cell type ovens is highly recommended. In order, however, to give some guidance for cases where it is not practicable to provide equipment for individual containers it is recommended that only the following should be aged together:—

1. Polymers of the same general type.
2. Vulcanizates containing the same type of accelerator and approximately the same ratio of sulphur to accelerator.
3. Vulcanizates containing the same type of antioxidant.
4. Vulcanizates containing the same type and amount of plasticizer.

The number of test pieces used for each ageing period, and the method of expressing the results obtained on them, are stated in the appropriate standard method for the property under test.

METHOD A. CELL TYPE OVEN METHOD

SECTION 2 SUMMARY AND EXPLANATORY NOTE

This ageing test of rubber consists in subjecting test pieces 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. This method has the advantage that individual compounds are aged in separate cells which prevents contamination arising from migration of sulphur, antioxidants, peroxides, plasticizers and other volatile products. The physical properties concerned in the service application are used to measure the deterioration, but in the absence of any statement of these it is recommended that tensile strength, modulus, elongation at break and hardness should be measured as specified in the appropriate parts of B.S. 903. 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, unless the test pieces are very thin.

In the accelerated oven test 70°C is the highest temperature that can be regarded as providing satisfactory uniform ageing. Accordingly, oven tests at temperatures of above 70°C should not be used except as tests for products that are subjected to such temperatures during use. These tests are used with many synthetic rubbers and temperatures such as 100°C, 150°C, 200°C and 250°C are commonly used.

SECTION 3 TEST PIECE

As amended Oct., 1963

The ageing shall be carried out on the test pieces, prepared and conditioned as required for the appropriate test method, and not on complete articles or sample sheets, and their form shall be such that no mechanical, chemical or heat treatment will be required after ageing. The test pieces shall be measured before and marked after ageing. Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared with each other. Care shall be taken to ensure that the test pieces have a good, smooth finish and are free from blemishes and other flaws. Care should be taken that the material used for identifying test pieces is not applied in any critical area of the test piece (e.g. the test length of a dumb-bell or the place where a hardness test is to be made) and is such as not to injure the rubber or become destroyed during ageing.

SECTION 4 APPARATUS

The apparatus shall consist of one or more cylindrical vertical cells having a minimum height of 30 cm. The cells shall be surrounded by a thermostatically controlled good heat-transfer medium (aluminium block, liquid bath, saturated vapour).

No copper or copper alloy shall be within the ageing chamber. Provision shall be made for suspending test pieces so that they are not within 1 cm of each other or of the sides of the oven. Glass or aluminium supports are recommended for use with ring test pieces.

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

Provision shall be made for a circulation of air through the cells of not less than 3 and not more than 10 changes in one hour. The incoming air shall be heated by the same heat-transfer medium to the temperature of the oven before entering the cell.

The temperature of the test cells shall be uniform in time and space within the limits specified in Section 6.

Suitable means shall be provided for controlling and measuring the temperature and air flow.

SECTION 5 PROCEDURE

5.1 Conditioning of samples and test pieces. When the test is not to be carried out immediately following vulcanization, all samples and test pieces shall be stored in the dark for a period not exceeding 14 days prior to commencement of the ageing test. The temperature of storage before subjection to an accelerated ageing test shall not exceed 30°C. Prior to testing, the test pieces shall be prepared and conditioned according to the details given in the appropriate test method for the particular property being studied. The unaged test pieces shall be tested within 24 hours of the start of the ageing period. When the ageing period is complete the test pieces shall be removed from the oven and stored for 16 to 96 hours in conditions which are in accordance with the details given in the appropriate test method for the particular property being studied.

As amended Dec., 1965

5.2 Ageing procedure. The test pieces shall be placed in the cells which have 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. The exposure shall be continuous for the specified time. The volume of the test pieces shall not exceed 10 per cent of the volume of the cell.

5.3 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 and the temperature of test.

For tests at 70°C and 100°C it is recommended that the ageing period should be 3, 7, 10 or some multiple of 7 days. As 7 days at 70°C is too short a period to produce a marked deterioration in most rubbers of good quality, one of the longer periods is preferred. The ageing periods used, however, shall be such that deterioration will not be so great as to prevent the determination of final physical properties.

5.4 Number of test pieces and averaging of results. The number of test pieces and the method of averaging the results shall be in accordance with the requirements of the particular test method.

SECTION 6 TEMPERATURE OF TEST

As amended Oct., 1963

The test pieces shall be aged at one of the following temperatures:—

70±1°C, 100±1°C, 125±1°C, 150±2°C, 175±2C°, 200±2°C, 250±2°C.

The ageing test should be carried out at the lowest standard temperature above the service temperature.

SECTION 7 CALCULATION OF RESULTS AND REPORT

Calculation of results and report. The properties of test pieces aged for different periods shall be determined as the intervals terminate in the progress of ageing.

The test results of both the unaged (O) and the aged (A) test pieces shall be reported as well as the percentage deterioration as calculated from the following formula:—

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

The ageing period or periods, the temperature of test, and the properties determined shall be stated.

METHOD B. OVEN METHOD

SECTION 8 SUMMARY AND EXPLANATORY NOTE

This ageing test of rubber consists in subjecting test pieces 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 it is recommended that tensile strength, modulus, elongation at break and hardness should be measured as specified in the appropriate parts of B.S. 903. 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, unless the test pieces are very thin.

In the accelerated oven test 70°C is the highest temperature that can be regarded as providing satisfactory uniform ageing. Accordingly oven tests at temperatures above 70°C should not be used except as tests for products that are subjected to such temperatures during use. These tests are used with many synthetic rubbers and temperatures such as 100°C, 150°C, 200°C and 250°C are commonly used.

SECTION 9 TEST PIECE

As amended Oct., 1963

The ageing shall be carried out on the test pieces, prepared and conditioned as required for the appropriate test method, and not on complete articles or sample sheets, and their form shall be such that no mechanical, chemical or heat treatment is required after ageing. The test pieces shall be measured before and marked after ageing. Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared with each other. Care shall be taken to ensure that the test pieces have a good, smooth finish and are free from blemishes and other flaws. Care should be taken that the material used for identifying test pieces is not applied in any critical area of the test piece (e.g. the test length of a dumb-bell or the place where a hardness test is to be made) and is such as not to injure the rubber or become destroyed during ageing.

SECTION 10 APPARATUS

As amended Nov. 1964

The air oven shall be of such a size that the total volume of the test pieces does not exceed 10 per cent of the free air space of the oven. Provision shall be made for suspending test pieces so that they are not within 1 cm of each other or of the sides of the oven. Glass or aluminium supports are recommended for use with ring test pieces.

Provision shall be made for a slow circulation of air through the oven of not less than three or more than ten, changes per hour. The incoming air shall be heated to the temperature of the oven before coming in contact with the test pieces. Suitable means shall be provided for controlling and measuring the rate of air flow.

The temperature of the oven shall be thermostatically controlled so that the test pieces are kept within the limits specified in Section 12. The walls of the oven shall not be substantially different in temperature from the test temperature. A thermometer shall be placed amongst the test pieces to record the actual ageing temperature.

SECTION 11 PROCEDURE

11.1 Conditioning of samples and test pieces. When the test is not to be carried out immediately following vulcanization, all samples and test pieces shall be stored in the dark for a period not exceeding 14 days prior to commencement of the ageing test. The temperature of storage before sub-

jection to an accelerated ageing test shall not exceed 30°C. Prior to testing, the test pieces shall be prepared and conditioned according to the details given in the appropriate test method for the particular property being studied. The unaged test pieces shall be tested within 24 hours of the start of the ageing period. When the ageing period is complete the test pieces shall be removed from the oven and stored for 16 to 96 hours in conditions which are in accordance with the details given in the appropriate test method for the particular property being studied.

11.2 Ageing procedure. The test pieces shall be placed 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. The exposure shall be continuous for the specified time.

11.3 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 and temperature of test.

For tests at 70°C and 100°C it is recommended that the ageing period should be 3, 7, 10 or some multiple of 7 days. As 7 days is too short a period to produce a marked deterioration in most rubbers of good quality, one of the longer periods is preferred. The ageing periods used, however, shall be such that deterioration will not be so great as to prevent determination of final physical properties.

11.4 Number of test pieces and averaging of results. The number of test pieces and the method of averaging the results shall be in accordance with the requirements of the particular test method.

SECTION 12 TEMPERATURE OF TEST

As amended Oct., 1963

The test pieces shall be aged at one of the following temperatures:

70±1°C, 100±1°C, 125±1°C, 150±2°C, 175±2C°, 200±2°C, 250±2°C.

The ageing test should be carried out at the lowest standard temperature above the service temperature.

SECTION 13 CALCULATION OF RESULTS AND REPORT

The properties of test pieces aged for different periods shall be determined as the intervals terminate in the progress of ageing.

The test results of both the unaged (*O*) and the aged (*A*) test pieces shall be reported as well as the percentage deterioration as calculated from the following formula:—

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

The ageing period or periods, the temperature of test, and the properties determined shall be stated.

SECTION 14 REPRODUCIBILITY OF RESULTS

The reproducibility of test results after oven ageing will depend greatly upon the type of rubber, and it is therefore difficult to give a value which will be a reliable guide to the accuracy of ageing results. Experience shows, however, that mean tensile strength results calculated as percentage deterioration by the method given in Section 13, generally have standard errors in the range 8–15 units. These errors represent the combined variation due to mixing, vulcanization, ageing and testing.

METHOD C. OXYGEN PRESSURE METHOD

SECTION 15 SUMMARY AND EXPLANATORY NOTE

This ageing test of rubber consists in exposing test pieces to an elevated temperature and elevated oxygen pressure, after which the physical properties are determined and compared with those of unaged test pieces. The physical properties concerned in the service application are used to determine the degree of deterioration, but in the absence of any statement of these it is recommended that tensile strength, modulus, elongation at break and hardness should be measured as specified in the appropriate parts of B.S. 903.

In the oxygen pressure 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.

SECTION 16 TEST PIECE

As amended Oct., 1963

The ageing shall be carried out on the test pieces prepared and conditioned as required for the appropriate test method, and not on complete articles or sample sheets, and their form shall be such that no mechanical, chemical or heat treatment will be required after ageing. The test pieces shall be measured before and marked after ageing. Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared with each other. Care shall be taken to ensure that the test pieces have a good smooth finish and are free from blemishes and other flaws. Care should be taken that the material used for identifying test pieces is not applied in any critical area of the test piece (e.g. the test length of a dumb-bell or the place where a hardness test is to be made) and is such as not to injure the rubber or become destroyed during ageing.

SECTION 17 APPARATUS

The oxygen pressure chamber shall 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 shall be such that the total volume of the test pieces does not exceed 10 per cent of the internal volume of the vessel.

No copper or copper alloy shall be within the ageing chamber. Provision shall be made for suspending test pieces so that they are not within 1 cm of each other or of the sides of the oven. Glass or aluminium supports are recommended for use with ring test pieces.

The heating medium surrounding the vessel shall be thermostatically controlled so that the test pieces are kept at a temperature of 70 ± 1°C. A thermometer shall be immersed in the heating medium.

The pressure chamber shall be equipped with a reliable safety valve set at 500 lb/sq. in., and a pressure gauge shall be connected to the apparatus.

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

SECTION 18 PROCEDURE

As amended Dec., 1965

18.1 Conditioning of samples and test pieces. When the test is not to be carried out immediately following vulcanization, all samples and test pieces shall be stored in the dark for a period not exceeding 14 days prior to commencement of the ageing test. The temperature of storage before subjection to an accelerated ageing test shall not exceed 30°C. Prior to testing, the test pieces shall be prepared and conditioned according to the details given in the appropriate test method for the particular property being studied. The unaged test pieces shall be tested within 24 hours of the start of the ageing period. When the ageing period is complete the test pieces shall be removed from the vessel and stored for 16 to 96 hours in conditions which are in accordance with the details given in the appropriate test method for the particular property being studied.

18.2 Ageing procedure. The test pieces shall be suspended in the pressure chamber after it has been dried and heated to the operating temperature. Before commencing the test the air shall be flushed out by filling the vessel with oxygen to 300 ± 10 lb/sq. in. pressure and then relieving the pressure. The test pieces in the vessel shall be stationary, free from strain, and freely exposed to the oxygen on all sides.

Oxygen shall be introduced into the pressure chamber until a pressure of 300 ± 15 lb/sq. in. gauge (21 ± 1 kgf/sq. cm gauge) is reached; the exposure shall be continuous for the specified time, without pressure reduction or opening of the chamber. When the ageing period is complete the oxygen pressure shall be released gradually, this operation taking at least five minutes.

18.3 Duration of test. The period required to obtain any given degree of deterioration of the test piece will depend on the type of rubber under examination; with a view to establishing uniformity of practice it is recommended that the ageing period should be 24 hours or a multiple thereof.

18.4 Number of test pieces and averaging of results. The number of test pieces and the method of averaging the results shall be in accordance with the requirements of the particular test method.

SECTION 19 TEMPERATURE AND PRESSURE OF TEST

The test pieces shall be at a temperature of 70 ± 1°C, and in oxygen at a pressure of 300 ± 10 lb/sq. in. (21 ± 0·7 kg/sq. cm).

SECTION 20 CALCULATION OF RESULTS AND REPORT

The properties of test pieces aged for different periods shall be determined as the intervals terminate in the progress of ageing.

The test results of both the unaged (O) and the aged (A) test pieces shall be reported as well as the percentage deterioration as calculated from the following formula:

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

The ageing period or periods and the properties determined shall be stated.

SECTION 21 REPRODUCIBILITY OF RESULTS

The reproducibility of test results after oxygen-pressure ageing will depend greatly upon the type of rubber, and it is therefore difficult to state a value which will be a reliable guide to the accuracy of ageing results. Experience shows, however, that mean results, calculated as percentage deterioration by the method given in Section 20, generally have standard errors in the following ranges:—

Tensile strength after ageing	5–10 units
Elongation at break after ageing	2–5 units

These errors represent the combined variation due to mixing, vulcanization, ageing and testing.

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