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ACCELERATED AGEING OR SIMULATED SERVICE TESTS ON VULCANIZED NATURAL OR SYNTHETIC RUBBERS

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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 (B.S.I.).

Work on this matter which the Technical Committee had begun since 1948, came to an end in 1957, with the adoption of a proposal as a Draft ISO Recommendation.

On 17 July 1957, the Draft ISO Recommendation (No. 171) was distributed to all the ISO Member Bodies and was approved by the following Member Bodies:

Australia	Hungary	Romania
Austria	India	Spain
Burma	Ireland	Sweden
Canada	Italy	Switzerland
Czechoslovakia	Japan	Union of South Africa
Denmark	New Zealand	United Kingdom
Finland	Pakistan	U.S.A.
Germany	Poland	U.S.S.R.
Greece	Portugal	

One Member Body opposed the approval of the Draft: France.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1961, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 188 February 1961

ACCELERATED AGEING OR SIMULATED SERVICE TESTS ON VULCANIZED NATURAL OR SYNTHETIC RUBBERS

1. INTRODUCTION

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 its properties are measured and compared with the corresponding properties of the untreated rubber.

The purpose of the test may be to assess the deterioration of the rubber either

(*a*) during prolonged periods at normal temperatures, or

(*b*) during use at high temperatures or at elevated oxygen pressure.

Three ageing tests are given in the following sections:

The selection of the conditioning time and temperature will depend on the purpose of the test and the type of polymer. This selection decides whether an accelerated ageing or simulated service (heat) test is to be used.

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

In the two first forms of ageing test specified above, deterioration is accelerated by raising the temperature and, in the third form of test, by increasing the oxygen concentration. The degree of acceleration thus produced varies from one vulcanizate to another and from one quality to another. Consequences of this are:

(*a*) Accelerated tests do not truly reproduce the changes produced by natural ageing.

(*b*) They do not indicate 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.

(*d*) Ageing should 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.

Attention is drawn to the fact that oven and bomb 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.

2. CELL TYPE OVEN METHOD

2.1 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, 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, unless the test pieces are very thin.

2.2 Test pieces. It is recommended that the ageing should be carried out on the test pieces prepared and conditioned as required for the appropriate 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 ageing.

Only test pieces of similar dimensions and having approximately the same exposed areas are compared with each other. The test pieces should be measured before and marked after ageing.

Care should be taken that the material used for identifying test pieces should not be applied in any restricted area of the test piece and should be such as not to injure the rubber or become destroyed during ageing. Care should be taken to ensure that the test pieces have a good smooth finish and are free from blemishes and other flaws.

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

The design of the apparatus should be such that heated air will enter the bottom of the cell and be exhausted out at the top of it without being recirculated. Air passing through one cell should not enter other cells. Provision is made for a slow circulation of air through the cells of not less than three changes per hour. The incoming air should be within 1 °C of the specified temperature at the point of entry into the cell.

The temperature of the test cells should be uniform in time and space within $\pm$ 1.0 °C of the specified ageing temperature. Suitable means should be provided for controlling and measuring the temperature.

2.4 Procedure. When the test is not to be carried out within a few hours following vulcanization, all test pieces are stored in the dark for a period not exceeding 14 days prior to commencement of the ageing test. The maximum temperature of storage before subjection to an accelerated ageing test should not exceed 30 °C.

The testing of unaged test pieces is carried out within 14 days from the commencement of the ageing period; this will enable unaged test pieces and test pieces aged for periods of not more than 14 days to be tested together.

The test pieces are placed in the oven after it has been pre-heated to the operating temperature. The test pieces are stationary, free from strain, freely exposed to air on all sides and not exposed to light. The test pieces should take up not more than 10 per cent in volume of the space of each cell.

When the ageing period is complete, the test pieces are removed from the oven and conditioned in accordance with the details given in the appropriate test method for the particular property being studied.

2.5 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 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, should be such that deterioration of the test pieces will not be so great as to prevent determination of final physical properties.

2.6 Temperature of oven. The oven should be at one of the following temperatures:

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

The product specification should indicate the temperature to be used.

2.7 Expression of results. The number of test pieces and the method of expressing the results are in accordance with the recommendations already made for the particular test being carried out.

The properties of the test pieces aged for different periods are determined as the intervals terminate in the process of ageing.

The test results of both the unaged (O) and the aged (A) test pieces are reported, as well as the coefficient of deterioration as calculated from the following formula:

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

The properties determined should be stated.

3. OVEN METHOD

3.1 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, 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, unless the test pieces are very thin.

3.2 **Test pieces.** It is recommended that the ageing should be carried out on the test pieces prepared and conditioned as required for the appropriate 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 ageing.

Only test pieces of similar dimensions and having approximately the same exposed areas are compared with each other. The test pieces should be measured before and marked after ageing.

Care should be taken that the material used for identifying test pieces should not be applied in any restricted area of the test piece and should be such as not to injure the rubber or become destroyed during ageing. Care should be taken to ensure that test pieces have a good smooth finish and are free from blemishes and other flaws.

3.3 **Apparatus.** The air oven is 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 is made for suspending test pieces so that they are not within 10 mm of each other or the sides of the oven.

Provision is made for a slow circulation of air through the oven of not less than three changes per hour. Care should also be taken that the incoming air is heated to the temperature of the oven before coming in contact with the test pieces.

The temperature of the oven is thermostatically controlled so that the test pieces are kept within $\pm$ 1 °C of the specified ageing temperature. A thermometer should be placed near the centre of the ageing test pieces to record the actual ageing temperatures.

3.4 **Procedure.** When the test is not to be carried out within a few hours following vulcanization, all test pieces are stored in the dark for a period not exceeding 14 days prior to commencement of the ageing period. The maximum temperature of storage before subjection to an accelerated ageing test should not exceed 30 °C.

The testing of unaged test pieces is carried out within 14 days from the commencement of the ageing period; this will enable unaged test pieces and test pieces aged for periods of not more than 14 days to be tested together.

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

When the ageing period is complete, the test pieces are removed from the oven and conditioned in accordance with the details given in the appropriate test method for the particular property being studied.

Simultaneous ageing of different types of compound should be avoided, in order that migration of sulphur, antioxidants, peroxides or plasticizers may not occur, and therefore the use of individual containers is highly recommended. In order, however, to give some guidance for such cases where it is not practicable to provide equipment for individual containers, it is recommended that only the following should 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.

3.5 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 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, should be such that deterioration of the test pieces will not be so great as to prevent determination of final physical properties.

3.6 Temperature of oven. The oven should be at one of the following temperatures:

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

The product specification should indicate the temperature to be used.

3.7 Expression of results. The number of test pieces and the method of expressing the results are in accordance with the recommendations already made for the particular test being carried out.

The properties of test pieces aged for different periods are determined as the intervals terminate in the process of ageing.

The test results of both the unaged (O) and the aged (A) test pieces are reported, as well as the coefficient of deterioration as calculated from the following formula:

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

The properties determined should be stated.

4. OXYGEN PRESSURE METHOD

4.1 Summary and explanatory note. This ageing test of rubber consists in exposing test pieces to an elevated temperature and an 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, breaking elongation and hardness should be measured.

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.

4.2 Test pieces. It is recommended that the ageing should be carried out on the test pieces prepared and conditioned as required for the appropriate 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 ageing.

Only test pieces of similar dimensions and having approximately the same exposed areas are compared with each other. The test pieces should be measured before and marked after ageing.

Care should be taken that the material used for identifying test pieces should not be applied in any restricted area of the test piece and should be such as not to injure the rubber or become destroyed during ageing. Care should be taken to ensure that the test pieces have a good smooth finish and are free from blemishes and other flaws.

4.3 Apparatus. The oxygen pressure chamber consists 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 per cent of the free gas space of the vessel. No copper or brass parts should be within the ageing chamber.

The heating medium which surrounds the vessel is thermostatically controlled and a thermometer should be immersed in the heating medium.

The pressure chamber is equipped with a reliable safety valve set at 500 lbf/in² (35 kgf/cm²) and a pressure gauge is connected to the apparatus.

4.4 Procedure. When the test is not to be carried out within a few hours following vulcanization, all test pieces are stored in the dark for a period not exceeding 14 days prior to commencement of the ageing test. The maximum temperature of storage before subjection to an accelerated ageing test should not exceed 30 °C.

The testing of unaged test pieces is carried out within 14 days from the commencement of the ageing period; this will enable unaged test pieces and test pieces aged for periods of not more than 14 days to be tested together.

The test pieces are suspended vertically in the pressure chamber after it has been dried and heated to the operating temperature. Before commencing the test, the air is flushed out of the vessel by releasing the oxygen pressure and refilling. The test pieces in the vessel are stationary, free from strain and freely exposed to the oxygen on all sides.

Oxygen is passed into the pressure chamber at a pressure of 300 $\pm$ 15 lbf/in² (21 $\pm$ 1 kgf/cm²); the exposure is continuous for the specified time, without pressure reduction or opening of the chamber.