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PART 4 ACCELERATED AGEING

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(Second Revision)

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Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART 4 ACCELERATED AGEING

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standard on 7 August 1987, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1965 and revised in 1978.

0.3 In this revision, duration and temperature of test clauses have been modified to align it with latest International Standard on the subject.

0.4 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, 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 is to assess the deterioration of the rubber either during prolonged periods at normal or at high temperature in air, or during use at elevated temperatures and at the elevated oxygen pressure.

0.5 The selection of time, temperature and atmosphere to which the test pieces are exposed shall depend on the purpose of the test and the type of polymer. In the air oven tests, deterioration is accelerated by raising temperature and in the oxygen pressure test, by increasing the oxygen concentration and temperature. The degree of acceleration thus produced varies from one vulcanizate to another and from one property to another. Consequences of this are as follows :

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 temperature.

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 may be measured reasonably accurately.

0.6 This standard is essentially based on ISO 188–1982 'Vulcanized rubbers—Accelerated ageing or heat resistance tests', issued by the International Organization for Standardization.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*

1. SCOPE

1.1 This standard prescribes the methods of test for accelerated ageing or heat resistance on vulcanized rubbers in air or oxygen. Two types of test methods are given :

a) By heating in air, and

b) By heating in oxygen.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in various parts of IS : 7503† shall apply.

3. ACCELERATED AGEING BY HEATING IN AIR

3.1 Principle of the Method

3.1.1 Test pieces are subjected to controlled deterioration by air at a 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, tensile strength, stress at intermediate elongation, breaking elongation and hardness should be measured.

*Rules for rounding off numerical values (*revised*).
†Glossary of terms used in rubber industry.

3.1.2 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 Apparatus

3.2.1 *Cell Type Oven* — The apparatus shall 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 percent of the capacity of the cell. The cells shall be surrounded by a thermostatically controlled good heat transfer medium (aluminium block, liquid bath or saturated vapour). The design of the apparatus shall be such that heated air shall enter the bottom of the cell and be exhausted at the top without being recirculated. Air passing through one cell shall not enter other cells. Provision shall be made for slow circulation of air through the cells of not less than three nor more than ten changes per hour. 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 test pieces are kept within ± 1°C or ± 2°C of the specified test temperature as appropriate for the temperature being used (*see* Note 2). Suitable means shall be provided for controlling and measuring the temperature.

NOTE 1 —No copper and/or copper alloys shall be used in the material of construction of the oven.

NOTE 2 — For temperatures of 125°C and above, the tolerance on the temperature of the test cells shall be maintained within ± 2°C.

3.2.2 *Normal Oven* — The oven shall be of such a size that the total volume of the test pieces does not exceed 10 percent 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 nor more than ten changes per hour. Care shall also be taken 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 thermostatically controlled so that the test pieces are 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* Note 2). A thermometer or thermocouple shall be placed near the centre of the test pieces to record the actual test temperature.

NOTE 1 — No copper and/or copper alloys shall be used in the material of construction of the oven.

NOTE 2 — For temperatures of 125°C and above, the tolerance on the temperature of the test cells shall be maintained within ± 2°C.

3.3 Test Pieces — Ageing test shall be carried out on blemish free, smooth surface test pieces prepared and conditioned, as required; for appropriate test and not on complete articles or sample sheets. The form of the prepared test piece shall be such that no mechanical, chemical or heat treatment is required after ageing. Only those test pieces, which have similar dimensions and approximately the same exposed area, shall be compared with each other. Measurements of the test pieces shall be noted before ageing and reference markings shall be placed on the test pieces after completion of the ageing periods. 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.

3.4 Time Lapse Between Vulcanization and Testing

3.4.1 For all the test purposes, the minimum time lapse between vulcanization and testing shall be 16 hours.

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

3.4.3 Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

3.5 Duration of Test — The period required to obtain any given degree of deterioration of the test pieces depends on the type of polymer, nature of plasticizer, nature of oxidants, etc, used in the vulcanizate under testing. With a view to establishing uniformity of practice, the recommended ageing periods are 24, 72, 168 and 240 hours or multiple of 168 hours. The test periods used shall be such that deterioration of the test pieces may not be so great as to prevent determination of the final values of physical properties.

3.6 Temperature of Test — The oven shall be mataintained at any of the following temperatures as given in the material specification :

70 ± 1°C
85 ± 1°C
100 ± 1°C
125 ± 2°C
150 ± 2°C
175 ± 2°C
200 ± 2°C
250 ± 3°C
275 ± 3°C
300 ± 3°C

The product specification shall indicate the temperature to be used.

NOTE — As oven temperatures are increased, the exposure times may nee to be reduced. Further, it should be recognized that greater the disparit between ageing and service conditions, the less reliable becomes the correlatio between ageing and service life.

3.7 Procedure

3.7.1 *Using Cell Oven* — The test pieces shall be placed in the cell after the oven has been pre-heated to the operating temperature, usin one compound only in each cell. The test pieces shall be stationary, fre from strain, freely exposed to air on all sides and not exposed to light When the heating period is complete, the test pieces shall be remove from the cells and conditioned for not less than 16 hours nor more thai 6 days in a strain-free condition and in accordance with the details of th atmosphere given in the appropriate test method for the particula property being studied.

3.7.2 *Using Normal Oven* — The test pieces shall be placed in th oven after it has been pre-heated to the operating temperature. Th test pieces shall be stationary, free from strain, freely exposed to air o all sides and not exposed to light. When the heating period is complete the test pieces shall be removed from the oven and conditioned for nc less than 16 hours nor more than 6 days in a strain-free condition and i accordance with the details of the atmosphere given in the appropriat test method for the particular property being studied.

Simultaneous heating of different types of compound in the sam oven shall be avoided, in order that migration of sulphur, antioxidant peroxides or plasticizers does not occur, and therefore the use of indiv dual cells is highly recommended. In order, however, to give some guic ance for such cases, where it is not practicable to provide equipment fc individual cells, it is recommended that only the following shall be heate together :

a) polymers of the same general type;

b) vulcanizates containing the same type of accelerator and approx mately 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.

3.8 Expression of Results — The test results of both the unaged (B) an the aged (A) test pieces shall be reported as well as the coefficient deterioration as calculated from the following formula :

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

where

B = the value of the property before ageing; and

A = the value of the property after ageing.

3.8.1 If considered desirable, the results may be reported as coefficient of retention as $\frac{A}{B} \times 100$.

4. AGEING BY HEATING IN OXYGEN

4.1 Principle of the Method

4.1.1 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, tensile strength, stress at intermediate elongation, breaking elongation and hardness should be measured.

4.1.2 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.

4.2 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 total volume of the test pieces does not exceed 10 percent of the free space of the vessel. Copper or brass parts shall be neither within the ageing chamber nor used in the construction of the tubing leading through the oxygen reservoir to the ageing chamber. The heating medium which surrounds the vessel thermostatically controlled and a thermometer shall be immersed in the heating medium. The pressure chamber shall be equipped with a reliable safety valve set at 3·5 MPa (approx 35 kgf/cm^2) and a pressure gauge shall be connected to the apparatus.

NOTE — The heating medium is optional. Water, air or other fluids known to be safe in the presence of oxygen may be used. Water has an advantage because of its rapid heat transfer and non-combustible nature. If air is used, the heated air shall be thoroughly circulated by means of mechanical agitation and baffles shall be used as required to prevent local overheating and dead spots. Oils or other combustible fluids are extremely hazardous in the presence of oxygen and should not be used as a heating medium for this test.

4.3 Test Pieces — Ageing test shall be carried out on blemish-free, smooth surface test pieces prepared and conditioned as required for appropriate

est and not on complete articles or sample sheets. The form of th prepared test piece shall be such that no mechanical, chemical or hea reatment is required after ageing. Only those test pieces, which hav similar dimensions and approximately the same exposed area, shall b compared with each other. Measurements of the test pieces shall b noted before ageing and reference markings shall be placed on the tes pieces after completion of the ageing periods. Care shall be taken t nsure that that the material used for identifying test pieces is no pplied in any critical area of the test piece and is not such as t njure the rubber or become destroyed during heating.

.4 Time Lapse Between Vulcanization and Testing

4.4.1 For all test purposes, the minimum time lapse between vulcanization and testing shall be 16 hours.

4.4.2 For product tests, whenever possible, the time lapse between ulcanization and testing shall not exceed four months. In all other cases, sts shall be made within two months of the date of receipt of the roduct by the customer.

4.4.3 Samples and test pieces shall be protected from light as comple- ly as possible during the interval between vulcanization and testing.

.5 Duration of Test — The period required to obtain any given degree f deterioration of the test pieces depends on the type of rubber under xamination. With a view to establishing uniformity of practice, ageing eriod of 24 hours or some multiples thereof is recommended.

6 Temperature and Pressure of Test — Subject the test pieces to a mperature of 70 ± 1°C and a pressure of 2·1 ± 0·1 MPa (approx ± 1 kgf/cm²).

7 Procedure

4.7.1 The test pieces shall be suspended vertically in the pressure amber after it has been heated to the operating temperature. Before mmencing the test, air shall be flushed out of the vessel by releasing e oxygen pressure and refilling. The test piece shall be stationary, free om strain and freely exposed to the oxygen on all sides. Oxygen shall be ssed into the pressure chamber to give a pressure of 2·1 ± 0·1 MPa approx 21 kgf/cm²) at 70°C; the exposure shall be continuous for the ecified time, without pressure reduction or opening of the chamber.

4.7.2 When the ageing period is complete, the pressure in the pressure chamber shall be relieved slowly and uniformly, requiring at least 5

minutes. The test pieces shall be removed from the chamber and condi tioned for not less than 16 hours nor more than 6 days in a strain-fre condition and in accordance with the details of the atmosphere given i appropriate test method for the particular property being studied.

4.7.3 Simultaneous ageing of different types of compound in the sam oven shall be avoided, in order that migration of sulphur, antioxidants peroxides or plasticizers does not occur, therefore the use of indivi dual pressure chambers is highly recommended. In order, however, t give some guidance for such cases where it is not practicable to provid equipment for individual pressure chambers, it is recommended that onl the following shall be aged together :

a) Polymers of the same general type,

b) Vulcanizates containing the same type of accelerator and approxi- mately the same ratio of sulphur to accelerator,

c) Vulcanizates containing the same type of antioxidant, and

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

NOTE — Adequate safety precautions are important when heating oxidizable organic materials in oxygen under pressure, since the rate of oxidation may, in some case, become very rapid, particularly if a large surface area of material is exposed. Ageing of cellular vulcanizates is not recommended as there is danger of explosion, if not properly carried out.

4.8 Expression of Results — The test results of both the unaged (B) and the aged (A) test pieces shall be reported as well as the coefficient of deterioration calculated from the following formula :

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

where

B = the value of the property before ageing, and

A = the value of the property after ageing.

4.8.1 If considered desirable, the results may be reported as coefficient of retention as $\frac{A}{B} \times 100$.

5. TEST REPORT

5.1 The test report should include the following particulars

a) Whether an air oven method (cell oven or normal oven) or the oxygen pressure method was used;

) The duration and temperature of ageing;

;) The properties determined, with their individual values before and after ageing and, if appropriate, the coefficient of deterioration;

j) Any test conditions and operations not provided for in this standard or which are optional as well as any incidents which may have affected the results;

:) If agreed to between the parties, coefficient of retention shall also be reported; and

f) Reference to this Indian Standard.