International Standard 6914

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, vulcanized — Determination of ageing characteristics by measurement of stress at a given elongation

Caoutchouc vulcanisé — Détermination des caractéristiques de vieillissement par mesurage de la contrainte à un allongement donné

First edition — 1985-04-15

UDC 678.063 : 620.172.21 : 620.193.918 **Ref. No. ISO 6914-1985 (E)**

Descriptors : rubber, vulcanized rubber, tests, stress relaxation tests, determination, aging (materials).

Price based on 4 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6914 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

Printed in Switzerland

Rubber, vulcanized — Determination of ageing characteristics by measurement of stress at a given elongation

0 Introduction

The stress in a rubber test piece at a given elongation changes with time as a result of a combination of simultaneous physical and chemical processes. Chemical processes predominate in the case of thin test pieces exposed to an atmosphere containing oxygen at an elevated temperature for relatively long periods of time. Thus, the ageing characteristics of the rubber vulcanizate may be determined by measurement of the change of stress in a thin test piece deformed in tension after periods of exposure under such conditions.

There are two variants of the technique. Measurements of stress may be made under either

a) continuous strain conditions, or

b) intermittent strain conditions.

In the case of a), continuous strain conditions, the test piece is held in extension throughout the ageing period in the oven. In the case of b), intermittent strain conditions, the test piece is aged in the oven in the unstressed state, but, at periodic intervals, it is stretched to a fixed extended length for a short time in order to determine the stress. Hence, this latter method is a measure of the change in modulus as a function of time.

In a second version of the intermittent test, the test piece is periodically removed from the accelerated ageing atmosphere and the stress is measured under normal laboratory conditions. The advantage of this method is that it does not require the use of special apparatus since a conventional tensile testing machine can be used for the measurement of stress (see note 1).

Measurements made in accordance with the methods described in this International Standard provide information about the structural changes which occur in the rubber during ageing.

Under continuous strain conditions, provided physical relaxation processes are not dominant, the decay of stress provides a measure of the degradative scission reactions in the network.

Any new networks formed as a result of cross-linking reactions are considered to be in equilibrium at the test strain with the main network and therefore do not impose any new stresses (see note 2).

Under intermittent strain conditions, the decay of stress provides a measure of the net effect of both degradative scission and cross-linking reactions.

The validity of the methods described in this International Standard depends on the uniformity of degradation in the rubber. For this reason, the thickness of the test pieces used is 1,0 mm to minimize the effect of oxygen diffusion on ageing.

The change in stress may be directly of interest, but the relative resistance of rubbers to ageing will depend on the properties being measured or required by the application.

This International Standard should therefore be regarded as complementary to ISO 188. In addition, a distinction should be made between this test and the stress relaxation in compression tests specified in ISO 3384[1] and ISO 6056[2], which are primarily intended for the testing of rubbers in applications, for example as seals, where resistance to stress relaxation is a functional property.

NOTES

1 The terms "continuous stress relaxation" and "intermittent stress relaxation" are commonly used to describe the two principal variants of the technique. The latter term, "intermittent stress relaxation", is a misnomer since no true relaxation of stress occurs and indeed the measured stress may increase with time. For this reason, the use of this term has been avoided in this International Standard although it is fairly well established in the literature.

2 Even under conditions conducive to chemical processes, some physical relaxation may occur. The extent to which it does so will depend on the viscoelastic characteristics of the rubber and on the test conditions and care must be exercised in interpretation of the results. Physical relaxation is increased by fillers and will be more evident at short times and at lower temperatures. It is often found to be proportional to logarithmic time and is less temperature sensitive than chemical resistance.

1) ISO 3384, *Rubber, vulcanized — Determination of stress relaxation in compression at normal and at elevated temperatures.*

2) ISO 6056, *Rubber, vulcanized — Determination of compression stress relaxation (rings).*

1 Scope and field of application

This International Standard describes three methods for the measurement of the change of stress in a test piece at a given elongation for the purpose of determining the ageing characteristics of the rubber vulcanizate.

Method A is intended for measurement under continuous strain conditions while Method B is the preferred method for measurement under intermittent strain conditions. In both cases, a stress tester is used to record the stress at the temperature of ageing.

Method C is an alternative to method B for measurement under intermittent strain conditions in which the test piece is removed from the ageing environment for measurement of the stress at the standard laboratory temperature.

Measurements at a single elevated ageing temperature may be used for quality control purposes as a measure of heat-ageing resistance. Measurements at a number of temperatures may be used for research and development purposes to estimate long-term ageing characteristics.

No agreement between the three methods should be inferred. The method to be used will depend on the purpose of the test.

2 References

ISO 188, *Rubber, vulcanized — Accelerated ageing or heat-resistance tests.*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubber — Time-interval between vulcanization and testing — Specification.*

ISO 5893, *Plastics and rubber — Tensile, flexural and compression testing machines (constant rate of traverse).* [1)]

3 Apparatus

3.1 Stress relaxometer (for methods A or B)

The stress tester shall consist of two grips which hold the test piece without slipping at a fixed extended length together with a means of measuring and recording the force in the test piece. The grips shall be arranged such that the test piece can be positioned in an oven.

The force measuring system may be, for example, a calibrated spring or electronic load cell, but it shall be accurate and stable to within 2 % of the force reading throughout the duration of the test.

For method B, the stress tester shall, in addition, be equipped with a device such that the test piece can be extended and relaxed at intervals. Repeated extension of the test piece shall be constant to within 2 % of the applied elongation.

3.2 Tensile machine (for method C)

The force shall be measured using a constant rate of traverse tensile machine complying with the requirements of ISO 5893, grade B, operating at 50 mm/min. The machine shall be capable of cycling between fixed strain limits which are accurate to within 2 % of the maximum strain. The grips of the tensile machine shall hold the test piece without slippage.

3.3 Oven

The test piece shall be aged in an oven complying with the requirements specified in ISO 188. Care shall be taken to ensure that the oven is clean prior to each test and that the walls of the oven are not contaminated by volatile substances.

4 Test pieces

4.1 Dimensions

Test pieces shall be parallel sided strips, cut from a sheet. For the tests described in this International Standard, it is vital to ensure uniform degradation in the rubber. For this reason, the thickness of the test pieces shall be 1,0 ± 0,05 mm in order to minimize the effect of oxygen diffusion on ageing.

NOTE — Samples for uniform thickness of less than 1,0 mm may be used, but these may give different results.

The other dimensions of the test pieces, i.e. width and length, should be chosen to suit the sensitivity of the load measuring device and the precision of the mechanism used for adjusting the strain, in order that the requirements of 3.1 and 3.2 relating to accuracy in force and strain are satisfied. In the absence of other considerations, the preferred dimensions are :

a) for methods A and B : width 4 ± 0,1 mm, length 80 ± 1 mm;

b) for method C : width 10 ± 0,2 mm, length 150 ± 1 mm.

4.2 Number

For each material, a minimum of three test pieces shall be used for each test temperature.

5 Storage and conditioning

The time-interval between vulcanization and testing shall be in accordance with ISO 1826.

Material and test pieces shall be protected from light as much as possible during the interval between vulcanization and testing. They shall not be allowed to come into contact with test sheets and test pieces of different composition. This is necessary in order to prevent additives which may affect ageing, such as antioxidants, from migrating from one vulcanizate into adjacent vulcanizates.

Cut test pieces shall be conditioned immediately before testing for a minimum of 3 h at a standard laboratory temperature.

1) At present at the stage of draft.

6 Test conditions

6.1 Duration of test

The duration of test should preferably be chosen from the series 1, 2, 4, 8, 24, 72 and 168 h and multiples of 7 days. For methods A and B, the test period shall be considered to commence when the initial force measurement is made. For method C, the test period shall be considered to be the time in the oven, excluding the time for cooling and measurement of force.

Alternatively, the test may be stopped when the stress, expressed as the ratio f_t/f_o, reaches a predetermined value (e.g. 0,5).

6.2 Temperature of exposure

The material being tested should preferably be examined at a series of temperatures at intervals of approximately 10 °C. If the test pieces are exposed at one temperature only, this shall be chosen from the following preferred temperatures listed in ISO 471 :

70 ± 1 °C	175 ± 2 °C
85 ± 1 °C	200 ± 2 °C
100 ± 1 °C	225 ± 2 °C
125 ± 2 °C	250 ± 2 °C
150 ± 2 °C	

NOTE — As the temperature is increased, the exposure time may need to be reduced. Further, it should be recognized that the greater the disparity between ageing and service conditions, the less reliable is the correlation between ageing and service life.

7 Procedure

NOTE — For all three methods, a smaller elongation of 20 ± 2 %, may be used in place of 50 ± 5 %.

7.1 Method A

Mount the test piece in the preheated grips in the unstrained condition. Position the grips and test piece in the oven preheated to the test temperature. After 5 ± 0,5 min, stretch the test piece, in not more than 1 min, to an elongation between 45 and 55 % and hold it to within 2 % of that elongation. The initial force (f_o) is taken to be that 5 ± 0,5 min after stretching the test piece. Record the force on the test piece as a function of time for the duration of the test. At the end of the test examine the surfaces of the stretched test piece for signs of cracking using a lens of magnification about X 7. If cracking is found, it shall be mentioned in the test report.

NOTE — With certain types of rubber, stress relaxation additional to that caused by oxygen and heat may occur as a result of surface attack by traces of atmospheric ozone. Cracking may invalidate the test and be the cause of variations between measurements.

7.2 Method B

Mount the test piece in preheated grips in the unstrained condition. Position the grips and test piece in the oven preheated to the test temperature. After 5 ± 0,5 min, measure the initial force by stretching the test piece, in not more than 2 s, to an elongation between 45 and 55 % and hold it to within 2 % of that value for 10 ± 1 s. Note the force and return the test piece to the unstrained condition. Repeat the measurement of force every hour, after having restretched the test piece to within 2 % of the initial applied elongation.

NOTE — Other time-intervals between measurements of force may be used provided that they are reported in the test report.

7.3 Method C

Mount the test piece in the grips of the tensile machine in the unstrained condition. Set the machine to operate at a rate of grip separation of 50 mm/min. Stretch the test piece to a fixed length corresponding to an elongation between 45 and 55 %, the actual elongation being known to within 2 % of that elongation and then relax the test piece. Without delay, repeat the straining cycle five times. The initial force (f_o) is taken as that on the fifth cycle. Remove the test piece from the tensile machine.

Place the unstrained test piece in an ageing oven at the required test temperature. After 24 h, remove the test piece and allow it to cool at standard laboratory temperature for 30 ± 5 min. Mount the test piece in the tensile machine and repeat the straining cycle to within 2 % of the initial elongation five times. For the purpose of the determination, changes in the length of the test piece as a result of cycling or ageing should be ignored. Note the force (f_t) on the fifth cycle. Replace the test piece in the oven within 2 h of removing it for testing. Repeat the measurement at intervals of 24 h. If the change of stress is low, take the time for measurement from the series 1, 3, 7 days and multiples of 7 days.

8 Expression of results

The retention of stress after time t is given by the formula

$$\frac{f_t}{f_o}$$

The results should preferably be presented in the form of a graph of f_t/f_o against time t.

The value of f_t/f_o may also be given for a specified time, or, alternatively, the time for a specified value of f_t/f_o to be reached may be given.

9 Test report

The test report shall include the following information :

a) sample details :

1) a full description of the sample and its origin,

2) compound details, cure-time and temperature, when appropriate,

3) method of preparation of test pieces from the sample;

b) test method and test details :

1) the number of this International Standard,

2) the method used (A, B or C),

3) the standard laboratory temperature,

4) the duration and temperature of exposure,

5) the type of test pieces and their dimensions,

6) the elongation of the test piece,

7) a description of the principles of the testing device including the oven,

8) details of any procedures not specified in this International Standard;

c) test results :

1) the number of test pieces tested,

2) the median value of the test results, expressed in accordance with clause 8;

d) date of test.

This page intentionally left blank

This page intentionally left blank