BS ISO 6914:2013

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Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation in tension

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National foreword

This British Standard is the UK implementation of ISO 6914:2013. It supersedes BS ISO 6914:2008 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Physical testing of rubber.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation in tension

Caoutchouc vulcanisé ou thermoplastique — Détermination des caractéristiques de vieillissement par mesurage de la contrainte de relaxation en traction

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 6914:2008), which has been aligned with ISO 23529 and completed with details regarding dimensions, test equipment and methods.

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 can 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 can 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.

NOTE 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 can 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.

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.

Measurements made in accordance with the methods described in this International Standard provide information about the structural changes that 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 crosslinking reactions are considered to be in equilibrium at the test strain with the main network and therefore do not impose any new stresses.

NOTE 2 Even under conditions conducive to chemical processes, some physical relaxation can occur. The extent to which it does so will depend on the viscoelastic characteristics of the rubber and on the test conditions and care should be exercised in the 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 relaxation.

Under intermittent strain conditions, the decay of stress provides a measure of the net effect of both degradative scission and crosslinking 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 might be of direct 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 as specified in ISO 3384-1, which is primarily intended for the testing of rubbers in applications, for example as seals, where resistance to stress relaxation is a functional property.

The lifetime of the material, if this is to be investigated, can be determined using the procedures described in ISO 11346.

The most important factor in achieving good repeatability and reproducibility when making these tests is to keep the temperature and the elongation constant during all measurements.

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WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

CAUTION — Certain procedures specified in this International Standard might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

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 a rubber.

— Method A is intended for measurement under continuous strain conditions.

— Method B is the preferred method for measurement under intermittent strain conditions.

In the case of both methods A and B, a stress relaxometer 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 standard laboratory temperature.

The necessary calibration schedule for this type of measurement is given in Annex A.

Measurements at a single elevated ageing temperature can be used for quality control purposes as a measure of heat-ageing resistance. Measurements at a number of temperatures can be used for research and development purposes to estimate long-term ageing characteristics in accordance with the procedures described in ISO 11346.

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 188:2011, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

ISO 5893:2002, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Specification*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Apparatus

3.1 Stress relaxometer, (for method A or B) consisting of two grips which hold the test piece without slipping at a fixed extended length (to within ±1 %) together with a means of measuring and recording the force on the test piece.

The grips shall be arranged such that the test piece can be positioned in an oven. The force-measuring system mayn be, for example, a calibrated spring or electronic load cell, but it shall be accurate and stable to within ±1 % of the force reading throughout the duration of the test.

For method B, the stress relaxometer 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 ±1 % of the elongation.

3.2 Tensile-testing machine, (for method C) using a constant rate of traverse, operating at 50 mm/min and complying with the requirements specified in ISO 5893:2002, force class 1 (measuring force to within ±1 % of the measured value).

The machine shall be capable of cycling between fixed strain limits which are accurate to within ±1 % of the maximum strain. The grips of the tensile-testing machine shall hold the test piece without slippage.

3.3 Oven, complying with the requirements specified for ISO 188:2011, method A (low air speed) or method B (high air speed), for ageing the test piece.

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.

Samples of uniform thickness of less than 1,0 mm or more than 1,0 mm can be used, but these can give different results.

NOTE For temperatures above 125 °C, reducing the test piece thickness to 0,5 mm will be advantageous because of the increased effect of oxygen diffusion at higher temperatures.

Alternatively, product parts or complete products can be used as test pieces taking into account the requirement for thickness.

The other dimensions of the test pieces, i.e. width and length, shall 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 the accuracy of the force and the strain are satisfied.

4.2 Number

The preferred number of test pieces is three for each test temperature, but for routine and screening tests, one or two test pieces are acceptable.

5 Storage and conditioning

The time interval between vulcanization and testing shall be in accordance with ISO 23529.

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 a different composition. This is necessary in order to prevent additives which can affect ageing, such as antioxidants, from migrating from one vulcanizate into other vulcanizates.

Test pieces shall be conditioned for a minimum of 3 h at one of the standard laboratory temperatures specified in ISO 23529 immediately before testing.

6 Test conditions

6.1 Duration of test

The duration of test should preferably be chosen from the following series:

1 h, 2 h, 4 h, 8 h, 24 h, 72 h, 168 h, and multiples of 7 d.

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 the measurement of force.

Alternatively, the test can be stopped when the stress indicator, expressed as the ratio of the force, F_t, at time t to the initial force, F_0 (see Clause 8), 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 at least 10 °C. If the test pieces are exposed at only one temperature, this shall be chosen from the series of temperatures given in ISO 23529.

The temperature shall be kept as constant as possible during the test, with a tolerance of ±2 °C for standard laboratory temperature, ±1 °C for all elevated temperatures up to 100 °C, and ±2 °C for all elevated temperatures above 100 °C.

It is crucial for the best results that the temperature be kept as stable as possible during the test for two reasons.

— Temperature tolerances in ISO 23529 are ±1 °C up to and including 100 °C and ±2 °C for 125 °C up to and including 300 °C. However, studies have shown that a 1 °C change in temperature corresponds to a 10 % difference in ageing time at an Arrhenius factor of 2, or 15 % at a factor of 2,5. This means that two laboratories carrying out ageing at 125 °C can have ageing times which differ by 60 % from each other and still be within the specification. To get accurate results, keep the temperature as accurate as possible by placing a calibrated temperature sensor close to the test pieces and use this to set the oven so that the temperature at this position is correct. Use the correction factor from the calibration certificate to get as close as possible to the true temperature.

— The volume expansion of rubber is 10 to 20 times greater than that of steel, and a temperature variation will cause a variation in the force reading.

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

7 Procedure

7.1 Method A

Method A can be performed either with a tensile-testing machine equipped with a temperature cabinet or with a specific testing device. Method A is carried out in the following manner.

a) Mount the test piece in the preheated grips in the unstrained condition.

b) Position the grips and test piece in the oven preheated to the test temperature.

c) (5 ± 0,5) min after the temperature measured close to the test piece has reached the test temperature, stretch the test piece, in not more than 1 min, to an elongation between 45 % and 55 % and hold it to within 1 % of that elongation. A smaller elongation can be used, for instance in the case of rupture of the test pieces, when (20 ± 2) % is preferred. The initial force, F_0, is taken to be that (5 ± 0,5) min after stretching the test piece.

d) Record the force, F_t on the test piece as a function of time for the duration of the test.

e) At the end of the test, examine the surfaces of the stretched test piece for signs of cracking using a lens with about × 7 magnification. If cracking is found, it shall be reported in the test report.

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

NOTE At higher temperatures, 5 min will not be enough to reach the test temperature because of the cooling of the rig during mounting of the test piece. In this case, it is advised to stretch the test pieces 5 min after the temperature close to the test piece has reached the desired value. This should be noted in the report.

7.2 Method B

Method B can be performed either with a tensile-testing machine equipped with a temperature cabinet or with a specific testing device. Method B is carried out in the following manner.

a) Mount the test piece in preheated grips in the unstrained condition.

b) Position the grips and test piece in the oven preheated to the test temperature.

c) (5 ± 0,5) min after the temperature measured close to the test piece has reached the test temperature, 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 1 % of that value for (10 ± 1) s. A smaller elongation can be used, for instance in the case of rupture of the test pieces, when (20 ± 2) % is preferred.

d) Note the force and return the test piece to the unstrained condition, in not more than 2 s.

e) Repeat the measurement of force every hour, after having restretched the test piece to within 1 % of the initial elongation, computed from the initial length of the test piece.

Other time intervals between measurements of force can be used, provided they are reported in the test report.

NOTE At higher temperatures, 5 min will not be enough to reach the test temperature because of the cooling of the rig during mounting of the test piece. In this case, it is advised to stretch the test pieces 5 min after the temperature close to the test piece has reached the desired value. This should be noted in the report.

7.3 Method C

Method C can be performed either with a tensile-testing machine equipped with a temperature cabinet or with a specific testing device. Method C is carried out in the following manner.

a) Mount the test piece in the grips of the tensile-testing machine in the unstrained condition.

b) Set the machine to operate at a rate of grip separation of 50 mm/min.

c) Stretch the test piece to a fixed length corresponding to an elongation between 45 % and 55 %, the actual elongation being known to within 1 % of that elongation, and then relax the test piece. A smaller elongation can be used, for instance in the case of rupture of the test pieces, when (20 ± 2) % is preferred.

d) Without delay, repeat the straining cycle five times. The initial force, F_0, is taken as that on the fifth cycle.

e) Remove the test piece from the tensile-testing machine.

f) Place the unstrained test piece in the oven at the required test temperature.

g) After 24 h, remove the test piece and allow it to cool at standard laboratory temperature for (30 ± 5) min.

h) Mount the test piece in the tensile-testing machine and repeat the straining cycle to within 1 % of the initial elongation five times (the elongation is that used in item c above). For the purpose of the determination, changes in the length of the test piece as a result of cycling or ageing shall be ignored.

i) Note the force, F_t, on the fifth cycle.

j) Replace the test piece in the oven within 2 h of removing it for testing.

k) Repeat the measurement at intervals of 24 h. If the change in stress is small, increase the time interval between the measurements in accordance with the series 3 d, 7 d, and multiples of 7 d.

8 Expression of results

The tensile stress relaxation, *R(t),* after a specified duration, *t*, of testing, expressed as a percentage of the initial force, is given by the equation

$$R(t)=\frac{F_0-F_t}{F_0}\times 100=\frac{\sigma_0-\sigma_t}{\sigma_0}\times 100 \quad (1)$$

where

F_0 is the initial tensile force, in newtons;

F_t is the tensile force, in newtons, measured after the specified duration, *t*, in newtons;

σ_0 is the initial tensile stress, in megapascals;

σ_t is the tensile stress, in megapascals, measured after the specified duration, *t*, in mégapascals.

Choose the median value of the results for the test pieces. The individual values for the test pieces shall be within 10 % of the median value. If they are not, repeat the test. Plot the stress relaxation values measured after different times of exposure as a function of time on a logarithmic scale to facilitate the interpretation of the test data. For some applications, it is more useful to calculate tensile stress ratio values, i.e. F_t/F_0, after different time of exposure, rather than stress relaxation values. In this case, present the tensile stress ratio values graphically as a function of logarithmic time.

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

b) Test method and test details:

1) a reference to this International Standard, i.e. ISO 6914-2013;

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

3) a description of the principles of the testing device, including the oven (3.3);

4) the standard laboratory temperature used for conditioning;

5) the duration and temperature of exposure;

6) the type and dimensions of the test pieces;

7) the elongation of the test piece;

8) the results of the examination of the surface for cracking (method A only);

9) the time between force measurements if different from the times stated in 7.2 (method B only);

10) details of any deviations from the procedures specified in this International Standard;

c) Test result:

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.

Annex A
(normative)

Calibration

A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is made in the "as-received" condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified in the test method for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

A.2 Calibration schedule

The calibration schedule given in Table A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, part of that apparatus or to an ancillary apparatus necessary for the test. In some cases, tolerances not given in the test method standard have been added.

Table A.1 — Calibration schedule

Parameter	**Requirement**	**Subclause of** ISO 18899:2013	**Verification frequency**[a]
Stress relaxometer (3.1)			
Grips	Non-slip, can be positioned in an oven		C
Accuracy of force-measuring system	within ± 1 % of reading	21.2	S
Accuracy of elongation-measuring system	within ± 1 % of reading	21.1	S
Tensile-testing machine (3.2)	As in ISO 5893:2002	21.1	S
Accuracy, force	Grade 1	21.2	S
Accuracy, elongation	Grade 1	21.1	S
Speed	50 mm/min	23.4	S
Grips	Non-slip	—	C
Oven (3.3)	Parameters as in ISO 188	18	S
Temperature	As in 6.2 of this International Standard		
	Tolerances:		
	±1 °C for elevated temperatures		
	±2 °C at 23 °C and 27 °C		
Thickness of test pieces	(1 ± 0,05) mm	15.1	S

[a] The following code letters are used in the schedule:

C requirement to be confirmed but no measurement;

S standard interval as given in ISO 18899.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a detailed procedure particular to that test method (whenever a calibration procedure more specific or detailed than in ISO 18899 is available, preference shall be given to the more detailed procedure).

The verification frequency for each parameter is given by a code letter (see Table A.1).

In-use checks additional to the schedule shall be listed separately.

A list shall be given of reference materials either required by the calibration schedule or otherwise required by the test method.

Most standards for the testing of materials require the test piece to be conditioned. This implies the use of a thermometer and possibly a hygrometer, both of which will require calibration. Standard conditioning and test temperatures and humidities cited in the relevant standard have not been repeated.

Test-piece dimensions are usually specified. This implies the use of dimension-measuring instruments, which will require calibration. Test-piece dimensions cited in the relevant standard have not been repeated.

Bibliography

[1] ISO 3384-1, *Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression — Part 1: Testing at constant temperature*

[2] ISO 11346, *Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use*

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