BRITISH STANDARD

BS ISO 24454:2008

Rubber — Acquisition and presentation of comparable multi-point data

ICS 83.060

British Standards

National foreword

This British Standard is the UK implementation of ISO 24454:2008. It supersedes BS 903-4:2003 which is withdrawn.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.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 August 2008

ISBN 978 0 580 58340 7

Amendments/corrigenda issued since publication

Date	Comments

INTERNATIONAL STANDARD

ISO 24454

First edition
2008-05-01

Rubber — Acquisition and presentation of comparable multi-point data

Caoutchouc — Acquisition et présentation de données multiples comparables

Reference number
ISO 24454:2008(E)

Contents

Page

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 24454 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Rubber — Acquisition and presentation of comparable multi-point data

1 Scope

This International Standard identifies specific test procedures for the acquisition and presentation of comparable multi-point data for selected properties of rubber compounds. The data for each property are generated, using a single test method, as a function of important variables such as time, temperature and environmental effects. An important application of this International Standard consists in helping different suppliers produce material specification sheets in which the same set of properties is measured using the same conditions.

Guidance on the interpretation of results is given in Annex A.

2 Normative references

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

ISO 37, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 815-1, *Rubber, vulcanized or thermoplastic — Determination of compression set at ambient, elevated or low temperatures — Part 1: At ambient or elevated temperatures*

ISO 1431-1, *Rubber, vulcanized and thermoplastic — Resistance to ozone cracking — Part 1: Static and dynamic strain testing*

ISO 1817, *Rubber, vulcanized — Determination of the effects of liquids*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

ISO 3384, *Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and elevated temperatures*

ISO 4664-1, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 4665, *Rubber, vulcanized and thermoplastic — Resistance to weathering*

ISO 6914, *Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation*

ISO 6943, *Rubber, vulcanized — Determination of tension fatigue*

ISO 8013, *Rubber, vulcanized — Determination of creep in compression or shear*

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

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

ISO 24453, *Rubber — Acquisition and presentation of comparable single-point data*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
multi-point data
data characterizing a rubber material by means of a number of test results for a property measured over a range of test conditions

3.2
indicative property
property that has been selected to reveal the influence of an environment on a material through a comparison of measurements of the property before and after exposure

4 Test piece preparation

Where relevant, materials for test shall be prepared, mixed and moulded following the general principles given in ISO 2393.

The detailed conditions used shall be as recommended by the manufacturer of the rubber compound and shall, for each of the processing steps, be the same for each test piece except that different cure times may be used for different sized test pieces.

The equipment, mixing cycle and conditions used for moulding shall be given in the test report.

The final preparation of test pieces (for example stamping from sheet) shall be in accordance with the relevant test method standard.

The properties of a rubber compound can vary depending on the processing procedures used and this should be taken into account when comparing materials.

To maximize the usefulness of results, processing conditions should be representative of those used in production.

5 Conditioning

The time between mixing and moulding shall be between 24 h and one week.

The time between moulding and conditioning shall be between 16 h and four weeks. Recommendations for storage of vulcanized rubber products are given in ISO 2230.

Test pieces shall be conditioned in accordance with the relevant test method standard. Where this refers to standard laboratory conditions of temperature and/or humidity, these are taken to mean (23 ± 2) °C and 50 % RH.

NOTE If tests are carried out at the alternative standard laboratory temperature of (27 ± 2) °C, the results will not be strictly comparable unless adjusted by the known relationship of the property with temperature.

For tests at temperatures other than (23 ± 2) °C, where specific instructions are not included in the test method standard, condition the test pieces at the temperature at which the test is to be conducted for a period sufficient to enable test pieces to attain substantial equilibrium in accordance with ISO 23529.

Details of the conditioning used shall be given in the test report.

6 Test requirements

Obtain multi-point data, as required, in accordance with Clauses 7 to 9.

NOTE Properties of rubbers generally vary significantly with temperature and, because service temperatures can cover a wide range, temperature is an important and commonly used variable for the generation of multi-point data. It is the main variable used in this International Standard for short-term properties.

Short-term mechanical properties also depend on the frequency of the applied stress or strain, and hence data obtained with frequency as the variable are important for dynamic applications. Additionally, dynamic response is generally amplitude-dependent.

Properties will vary over time due to viscoelastic and/or degradation effects, and hence data obtained with time as the variable are essential for understanding the long-term behaviour of rubbers. Generally, the effects of time are required in combination with the effect of temperature and/or other accelerating agents such as fluids, ultraviolet light and repeated mechanical stress. The main variables specified in this International Standard for stress relaxation, creep and degradation tests have been chosen to reflect the data that is generally most useful with respect to service applications.

For all properties, the value of data generated will be increased by extending the levels of the main variable or by testing as a function of additional variables.

7 Mechanical properties

7.1 Tensile stress-strain

Test in accordance with ISO 37 using type 1 or 1A dumbbells at a speed of 500 mm/min.

Obtain stress-strain data at a series of temperatures chosen to be integral multiples of 10 °C, replacing 20 °C by 23 °C. Obtain data at a minimum of six temperatures, one of which shall be 23 °C. The range used shall be representative of the intended service environment.

7.2 Other properties

Test other mechanical properties listed in ISO 24453 as a function of temperature in the same manner as tensile stress-strain properties.

8 Viscoelastic properties

8.1 Dynamic properties

Test in accordance with ISO 4664-1 in shear mode.

NOTE 1 These conditions are most conveniently achieved by using a dynamic mechanical thermal analyser (DMTA) with automatic temperature and frequency scanning.

Obtain dynamic property data at a series of temperatures chosen to be integral multiples of 10 °C, replacing 20 °C by 23 °C. Obtain data at a minimum of six temperatures, one of which shall be 23 °C. At each temperature obtain data at frequencies of 0,1 Hz, 1 Hz, 10 Hz and 100 Hz using a strain amplitude of ± 2 % and zero prestrain.

NOTE 2 For DMTA apparatus used in shear mode, the lowest temperature will usually need to be above the glass transition temperature of the rubber.

To investigate the effect of strain, carry out amplitude tests over a series of strains selected from those given in ISO 4664-1.

8.2 Stress relaxation

Test in accordance with ISO 3384, Method A, with the standard cylinder test piece.

Obtain measures of the stress relaxation after 167 h at a series of temperatures chosen to be integral multiples of 10 °C, replacing 20 °C by 23 °C. Obtain data at a minimum of four temperatures, starting at 23 °C.

NOTE Further data may be provided by continuing the relaxation for longer than 167 h.

8.3 Creep

Test in accordance with ISO 8013 using the type A test piece and applying mechanical conditioning.

Obtain measures of the creep increment after 167 h at a series of temperatures chosen to be integral multiples of 10 °C, replacing 20 °C by 23 °C. Obtain data at a minimum of four temperatures, starting at 23 °C.

NOTE Further data may be provided by continuing the creep for longer than 167 h.

ISO 8013 specifies choosing a stress to give 20 % initial strain. If required, data may also be obtained at additional stress levels.

9 Degradation and environmental tests

9.1 Exposure to heat

Test in accordance with ISO 11346, selecting indicative properties relevant to the intended application. The basic indicative properties commonly used are hardness, in accordance with ISO 48, tensile stress-strain properties, in accordance with ISO 37, and compression set, in accordance with ISO 815-1. The preferred procedure for obtaining data on the change in tensile “modulus” (stress relaxation) is to test in accordance with ISO 6914 using Method B or C. The choice of test piece and test conditions for indicative properties shall be, where relevant, in accordance with ISO 24453 (single-point data), except that for hardness the micro-method should be used.

Obtain measures of the indicative properties as a function of time at a series of temperatures and process these data as specified in ISO 11346 using either the Arrhenius or WLF procedures. Derive the maximum temperature of use after 20 000 h [T(20000)].

Because the form of the relation between property and time will vary with the material and property in question, the threshold value to be used has not been specified. Where appropriate, the time to 50 % change in the property should be used.

9.2 Resistance to liquids

Test in accordance with ISO 1817, selecting indicative properties relevant to the intended application. The basic indicative properties commonly used are volume change, hardness, in accordance with ISO 48 and tensile stress-strain properties, in accordance with ISO 37. The choice of test piece and test conditions for indicative properties shall be, where relevant, in accordance with ISO 24453, except that for hardness, the micro-method should be used.

Select test liquids relevant to the intended application. For most purposes it will be appropriate to select from those listed in ISO 1817. All relevant liquids shall be tested. The test temperature will depend on the material and the intended use. For most general purpose rubbers, 70 °C or 100 °C is appropriate for tests in oils and 23 °C is appropriate for fuels. Obtain measures of the change in indicative properties after 100 h and 1 000 h.

9.3 Accelerated weathering

Test in accordance with ISO 4665 using a xenon arc apparatus filtered to approximate terrestrial sunlight with an irradiance of 550 W/m^2 in the wavelength range of 290 nm to 800 nm, a black panel temperature of (65 ± 3) °C, a humidity of 50 % and a spray cycle of 18 min on-102 min off. Monitor the irradiation dose. Select indicative properties relevant to the intended application; the basic indicative properties commonly used are hardness, in accordance with ISO 48, and tensile stress-strain properties, in accordance with ISO 37. The choice of test piece and test conditions for indicative properties shall be, where relevant, in accordance with ISO 24453, except that for hardness, the micro-method should be used.

Obtain measures of the change in indicative properties after a minimum of five exposure times.

9.4 Exposure to ozone

In accordance with ISO 1431-1, test static strain, at 50 pphm ozone and 40 °C, using a minimum of five strains between 5 % and 80 %.

Obtain the times to first visible cracks.

9.5 Fatigue

In accordance with ISO 6943, test fatigue at (23 ± 2) °C using standard type 2 dumbbell test pieces at a minimum of five strain amplitudes.

Determine the median fatigue life at each strain.

10 Presentation of results

Test the minimum number of test pieces that is specified for each property in the associated test method standard to obtain each experimental point. Present the results in the formats shown in Tables 1 to 10. Where the test method specified allows a choice of conditions or procedure, these details shall be reported. Precede the data with information that identifies the material together with the information required by Clauses 4 and 5.

In order that the value recorded for each property be as representative as possible of the material tested, the measurements are preferably made on test pieces obtained from a number of batches of material blended together. Alternatively, the average of results from several sets of test pieces produced from different batches may be presented.

Table 1 — Tensile stress-strain data as a function of temperature

Test method details:							
Temperature, °C							
TS_b, MPa							
E_b, %							
S_{100}, MPa							
S_{300}, MPa							

Table 2 — Other mechanical property data as a function of temperature

Test method details:							
Temperature, °C							
Property 1							
Property 2							
Property 3							
Property 4							

Table 3 — Dynamic properties

Test method details:							
Frequency, Hz							
Temperature, °C							
Elastic shear modulus (in-phase modulus), MPa							
Loss shear modulus (out-of-phase modulus), MPa							
Absolute value of complex shear modulus, MPa							
tan δ							

Table 4 — Stress relaxation as a function of temperature

Test method details:							
Temperature, °C							
Relaxation R(167), %							
NOTE Preferably, also present a plot of relaxation against time so that there is the opportunity for extrapolation or interpolation of data.							

Table 5 — Creep as a function of temperature

Test method details:							
Temperature, °C							
Creep increment after 167 h							
NOTE Preferably, also present a plot of creep against time so that there is the opportunity for extrapolation or interpolation of data.							

Table 6 — Exposure to heat

Test method details:		
	T(20000) °C	**Threshold value used**
Hardness		
Tensile strength		
Elongation at break		
Stress at 100 % elongation		
Compression set		
Stress relaxation		
Property		
NOTE Preferably, also present the graphs of property against time and the Arrhenius plot or the WLF master curve.		

Table 7 — Change in indicative properties after exposure to liquids

Test method details:		
Liquid:	**Exposure temperature**, ºC:	
	100 h	**1 000 h**
Hardness, IRHD		
Volume change, %		
Tensile strength, %		
Elongation at break, %		
Stress at 100 % elongation, %		
Property		

Table 8 — Resistance to weathering

Test method details:							
Exposure time, h							
Hardness, IRHD							
Tensile strength, %							
Elongation at break, %							
Stress at 100 % elongation, %							
Property							
NOTE Preferably, also present the graphs of property against time.							

Table 9 — Ozone resistance

Test method details:							
Strain, %							
Time to first cracks, h							

Table 10 — Fatigue life

Test method details:							
Strain, %							
Fatigue life cycles							
NOTE Preferably, also present the graph of fatigue life against strain.							

Annex A
(informative)

Interpretation of results

The basic value of data generated in accordance with this International Standard is that it extends the usual single-point data to cover a number of levels of one or more variables such as temperature and time. The limitations of these data are the same as for the single-point data and depend on the property test method used. For example, most standard methods for short-term mechanical properties produce data under arbitrary conditions of test piece geometry which cannot readily be applied to other geometries. However, the relative changes in properties with levels of variable are generally more widely applicable.

For creep and stress relaxation tests, the levels of property measured will be due to both physical and chemical effects. At lower temperatures and shorter times, the physical relaxation will dominate, but at longer times and higher temperatures, irreversible degradation effects will increasingly become important. One consequence is that caution has to be exercised in extrapolating results to longer times.

There are severe limitations to using accelerated durability data to estimate lifetimes because the behaviour of the material with time may be complicated, reactions can occur at accelerated levels of the degradation agent which are not present at ambient conditions, and because of uncertainties in the extrapolation protocol used.

For accelerated heat-ageing data, the maximum temperature of use derived from the results is likely to depend on the method of analysis and the threshold criterion used. It is necessary to inspect the plots of property against time to judge the applicability of the results for a particular situation. Extrapolation of the results will increasingly be subject to large uncertainty and is not recommended for more than 30 °C from the last data point.

The effects of liquids on rubbers can be a combination of physical swelling and chemical degradation. Physical absorption will reach an equilibrium level (generally within the time scale of 1 000 h specified), but chemical effects and extraction of constituents of the material can continue. This will be particularly important at elevated temperatures when thermal degradation will occur and, hence, caution should be applied to extrapolation of results.

Similar restrictions apply to weathering data as to accelerated heat ageing. Service conditions are very variable in respect of irradiance, spectral distribution, temperature and precipitation; hence, it is recommended that the accelerated data be used only to compare materials.

Results of accelerated ozone exposure often show an apparent threshold strain below which no cracking occurs. However, this threshold might be less apparent in natural exposure at lower concentrations and it might be dangerous to use threshold strain as a simple approach to prediction.

If a relation is fitted to a plot of fatigue life against strain, a limiting strain below which the fatigue life is very long may be found. This is often used as a means of predicting fatigue life at lower strains; however, it should be appreciated that at longer times and higher temperatures there will be ageing effects which generally reduce fatigue resistance. Similarly, cracking can be induced more quickly if fatigue occurs in the presence of ozone.

More detailed discussions of the limitations of test methods and the data obtained can be found in the bibliography.

Bibliography

[1] ISO 2230, *Rubber products — Guidelines for storage*

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