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ISO 188

Sixth edition
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Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests

Caoutchouc vulcanisé ou thermoplastique — Essais de résistance au vieillissement accéléré et à la chaleur

Reference number
ISO 188:2023(E)

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This sixth edition cancels and replaces the fifth edition (ISO 188:2011), which has been technically revised.

The main changes are as follows:

— add oven type with a forced air circulation and high air speed/air exchange rate;

— editorial changes for better understanding.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Accelerated ageing and heat resistance tests are used to determine the change of defined properties of rubber and thermoplastic elastomers over a specified period. These properties are compared before and after the accelerated ageing and heat resistance tests.

In accelerated ageing, the rubber is exposed to an elevated temperature with the intention to simulate the effect of natural ageing in a shorter time. The degree of acceleration depends on the tested material as well as to the property being evaluated.

In the case of heat resistance tests, the rubber is exposed to prolonged periods at the operation temperature of the material.

Two types of ovens are specified in this document, cell ovens and cabinet ovens. Cabinet ovens can be of four types as described in Clause 5.

The duration, temperature, and atmosphere to which the test pieces are exposed and the type of oven to use depends on the purpose of the test and the type of polymer.

The change of properties not only depends on the temperature but can also depend on the air speed. Consequently, even tests at the same temperature but at different air speed (different ovens) may give different results.

Consequences of these effects are

a) accelerated ageing is only a simulation of the natural ageing and can therefore produce different results.

b) If different materials are compared, it is recommended to perform the accelerated ageing tests at more than one elevated temperature as different rubbers might show a different temperature behaviour (change of properties) at certain operation temperatures.

c) It is important to determine the properties of the rubber for the accelerated ageing test, which are used for the intended material application. Only these properties should be used for the evaluation of the test results. If these properties give a different ranking of the materials tested, it is recommended to agree on a lead property for evaluation. It is also recommended, that all evaluated properties are measured according to an international standard or an equivalent test procedure.

Air-oven ageing should not be used to simulate natural ageing under stress (bent or stretched test pieces) and the presence of light or ozone.

To estimate lifetime or maximum temperature of use, tests can be performed at several temperatures and the results can be evaluated by using an Arrhenius plot or the Williams Landel Ferry (WLF) equation as described in ISO 11346.

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Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

1 Scope

This document specifies accelerated ageing or heat resistance tests on vulcanized or thermoplastic rubbers/thermoplastic elastomers. Four methods are possible, they are detailed in Clause 5.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 18899, *Rubber — Guide to the calibration of test equipment*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

4.1 General

Test pieces are exposed to air at a given elevated temperature and at atmospheric pressure with controlled conditions of air circulation.

Physical properties are measured before and after exposure and the results compared.

The physical properties that are important for the intended application of the material should be used to determine the effects of exposure. In the absence of any indication of these properties, it is recommended that tensile strength, stress at intermediate elongation, elongation at break (in accordance with ISO 37) and hardness (in accordance with ISO 48-2) are measured.

4.2 Accelerated ageing test

Test pieces are subjected to a higher temperature than the rubber would experience in its intended application to simulate the effects of natural ageing in a shorter time.

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4.3 Heat resistance test

Test pieces are subjected to the temperature they would experience in their intended application to gain information about their service performance.

5 Apparatus

5.1 Air oven.

The oven shall be of such a size that the total volume of the test pieces does not exceed 10 % of the free space in the oven. Provision shall be made for suspending test pieces so that they are at least 10 mm from each other and, in cabinet ovens and ovens with forced air circulation, at least 50 mm from the sides of the oven.

The temperature of the oven shall be controlled so that the temperature of the test pieces is kept within the specified tolerance for the specified ageing temperature (see Clause 9) for the whole ageing period. A temperature sensor shall be placed inside the heating chamber close to the test pieces to indicate the actual ageing temperature.

No copper or copper alloys shall be used in the construction of the heating chamber.

Method A: using a cell or cabinet oven with low air speed, laminar flow of air past the stationary test pieces and air exchange rate between 3 and 10 changes per hour.

Method B: using a cabinet oven with high air speed, laminar flow of air past the test pieces and air exchange rate between 3 and 10 changes per hour.

Method C: using a cabinet oven with high air speed, turbulent flow of air past the test pieces, rotation of the test piece carrier and air exchange rate between 3 and 10 changes per hour.

For method A, B and method C, provision shall be made for a slow flow of air through the oven of not less than three and not more than ten air changes per hour.

Method D: Using a cabinet oven with high air speed, turbulent flow of air past the stationary test pieces and air exchange rate greater than 30 changes per hour.

For method D, an air flow between 0,25 to 3,0 m/s is necessary as well as an air exchange rate above 30 changes per hour.

Depending on the influence of the air speed/air exchange on the results, to get comparable results always the same method should be used to compare the ageing behaviour of different materials.

The incoming air in the oven should be heated up to the temperature within a ±1 °C tolerance before reaching the test pieces.

The ventilation (or air change rate) can be determined by measuring the volume of the oven chamber and the flow of air through the chamber.

NOTE To ensure a good precision when doing ageing and heat resistance tests, it is very important to keep the temperature uniform and stable within the oven and during the test. Therefore, it is necessary and to verify that the oven used, is within the temperature limits at all test piece locations and during the complete testing time. Increasing the air speed in the oven improves temperature homogeneity. However, air circulation in the oven and ventilation influences the ageing results. With a low air speed, accumulation of degradation products and evaporated ingredients, as well as oxygen depletion, can happen. A high air speed may increase the rate of deterioration, due to increased oxidation and migration of plasticizers and antioxidants.

NOTE Method D oven can be used for short term testing up to 168 h.

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5.2 Method A cell oven.

The oven shall consist of one or more vertical cylindrical cells having a minimum height of 300 mm. The cells shall be surrounded by a thermostatically controlled good heat transfer medium (aluminium block, liquid bath, or saturated vapour). Air passing through one cell shall not enter other cells.

Provision shall be made for a slow flow of air through the cell. The air speed shall depend on the air change rate only.

5.3 Method A cabinet oven.

The oven should consist of a single chamber without separating walls. Provision shall be made for a slow flow of air through the oven. The air speed shall depend on the air change rate only, and no fans are allowed inside the test chamber.

5.4 Method B, C and D cabinet oven with forced air circulation.

One of the following three types shall be used:

a) Method B cabinet oven with laminar air flow and forced air circulation (see Figure 1).

   The air flow through the heating chamber shall be as uniform and laminar as possible. The test pieces shall be placed so that the smallest surface faces the air flow to avoid disrupting the air flow. The air speed shall be between 0,5 m/s and 1,5 m/s.

   The air speed near the test pieces can be measured by means of an anemometer.

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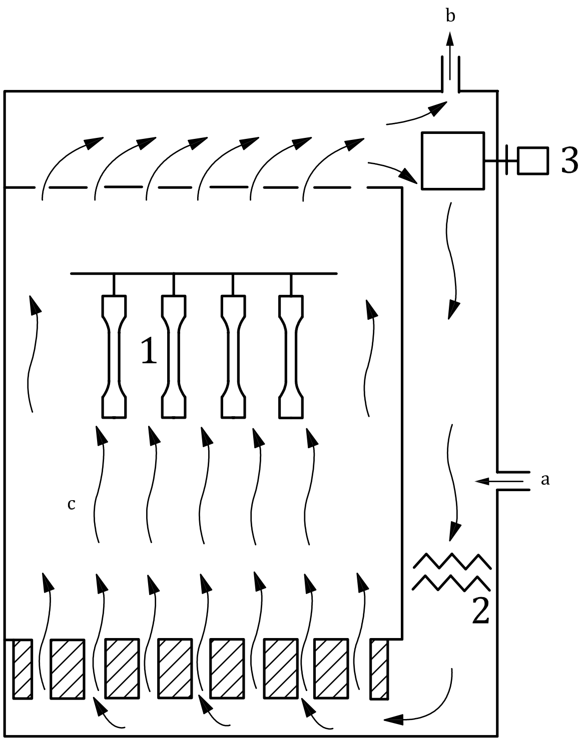

Key

1 test pieces
2 heating element
3 air blower
a Air inlet.
b Air outlet.
c Laminar air flow.

Figure 1 — Example of method B cabinet oven with laminar air flow

b) Method C cabinet oven with forced air circulation, turbulent air flow and provision for rotating the test piece carrier (see Figure 2).

The air entering from a side-wall air-inlet into the heating chamber is turbulent around the test pieces, which are suspended on a carrier rotating at a speed of five to ten rotations per minute so that they are exposed to the heating air as uniformly as possible. The average air speed shall be 0,5 m/s ± 0,25 m/s.

The average air speed near the test pieces can be calculated from measurements made with an anemometer at nine different positions (see Figure A.1). A suitable method of measurement is described in Annex A.

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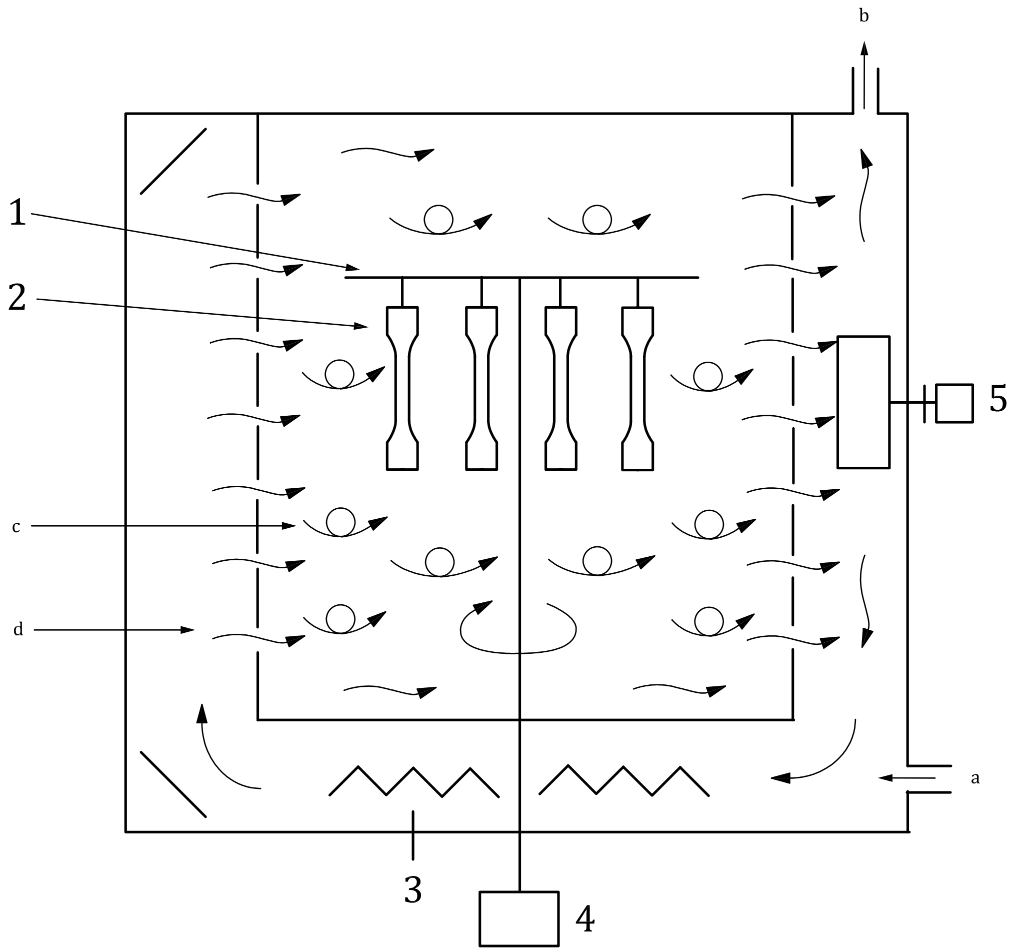

Key

1 test piece carrier

2 test pieces

3 heating element

4 motor

5 air blower

a Air inlet.

b Air outlet.

c Turbulent air flow.

d Laminar air flow (inlet, outlet and near to wall).

Figure 2 — Example of method C cabinet oven with turbulent air flow and provision for rotating the test piece carrier

c) Method D cabinet oven with turbulent air flow (see Figure 3).

The air entering from a back-wall air-inlet into the heating chamber is turbulent around the test pieces, which are suspended inside the oven in such a way that they are exposed to the heating air as uniformly as possible. The average air speed shall be between 0,25 m/s and 3,0 m/s.

The average air speed near the test pieces can be calculated from measurements made with an anemometer at nine different positions (see Figure A.1). A suitable method of measurement is described in Annex A.

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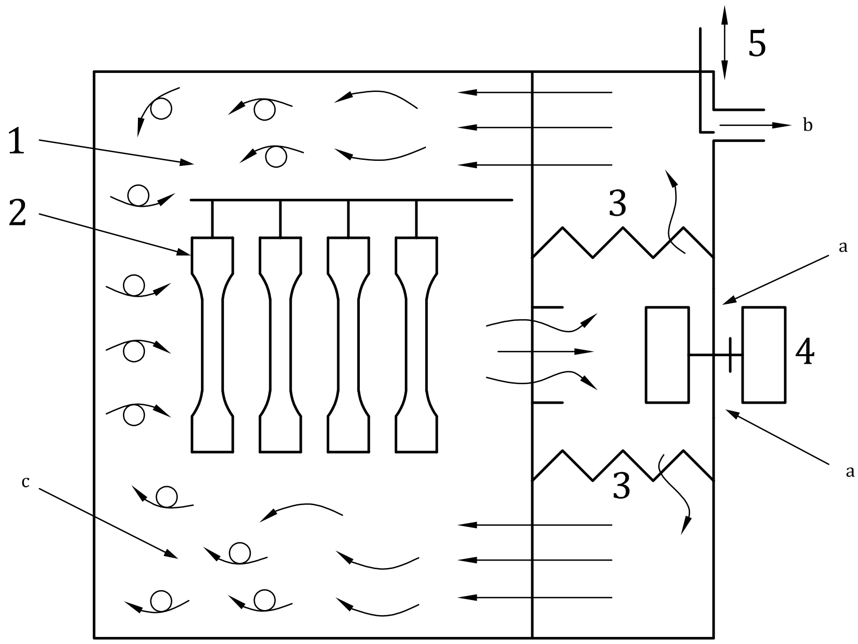

Key

1 test piece carrier
2 test pieces
3 heating element
4 air blower
5 regulator for air exchange rate
a Air inlet.
b Air outlet.
c Turbulent air flow.

Figure 3 — Example of method D cabinet oven with turbulent air flow

NOTE The tolerances for method D are larger which makes it less suitable for exposures longer than 168 h.

6 Calibration

The test apparatus shall be calibrated in accordance with Annex D.

7 Test pieces

Select and prepare the test pieces necessary for the tests to be carried out, in accordance with the requirements of ISO 23529.

Only test pieces of the same dimensions, having the same exposed areas shall be compared with each other. The number of test pieces shall be in accordance with the International Standard for the appropriate property tests.

The test pieces shall be capable of being identified after the test, for example by marking. Any method can be used that can withstand the exposure and does not affect the properties of the test piece or change the air flow.

NOTE Heat resistant tags attached with heat resistant string are satisfactory. Some marking inks can affect the ageing of the rubber or wear off during exposure.

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Avoid simultaneous heating of different types of compound in the same oven, to prevent the migration of sulphur, antioxidants, peroxides, or plasticizers. For this purpose, the use of individual cells is highly recommended. If it is not practicable to provide equipment with individual cells, it is recommended that only the following types of material be heated 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) rubbers containing the same type of antioxidant;

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

8 Time interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements, in accordance with ISO 23529 for time intervals, shall be observed.

For test purposes in cases of arbitration, the minimum time shall be 72 h.

9 Ageing conditions (duration and temperature)

9.1 General

Unless otherwise specified for technical reasons, the following requirements, in accordance with ISO 23529 for ageing time and temperatures, shall be observed.

Different type of rubbers and thermoplastic elastomers may require different periods of testing to observe any changes in properties. The ageing duration should not lead to a damage of the test pieces so that it is impossible to measure the required properties.

The use of high ageing temperatures may cause different degradation mechanisms than at operating temperatures, which invalidates the results.

To get the temperature as accurate as possible a calibrated temperature sensor shall be placed close to the test pieces and use this to set the oven to the required temperature. Use the correction factor from the calibration certificate of the oven to get as close as possible to the true temperature.

For methods A, B and C the ovens shall have a set point resolution of 0,1 °C. For Method D 1 °C resolution is acceptable.

It is crucial for comparable and reproducible results that the temperature is kept as stable as possible. Temperature tolerances stated in ISO 23529 are ±1 °C (up to and including 100 °C) and ±2 °C (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.

9.2 Accelerated ageing test

If the duration of ageing and the ageing temperature are not in accordance with ISO 23529, they can be agreed between the interested parties based on the product specification. The ageing shall be performed at atmospheric pressure.

9.3 Heat resistance test

If the duration of ageing and the ageing temperature are not in accordance with ISO 23529, they can be agreed between the interested parties based on the product specification. The temperature shall

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be representative of the operating temperature and the heating shall be performed at atmospheric pressure.

10 Procedure

Heat the oven to the required temperature and place the test pieces in it. When using a cell-type oven, only one rubber or compound shall be placed in each cell. The test pieces shall be free from strain, freely exposed to air on all sides and not exposed to light.

When the heating period is complete, remove the test pieces from the oven and condition them for no less than 16 h and no more than 6 days, strain-free in the atmosphere as required in the appropriate test method for the property being measured.

11 Expression of results

The results shall be expressed in accordance with the International Standard for the appropriate property tests.

The test results for both the unaged and the aged test pieces shall be reported together, as well as the percentage change in the value of the property measured as calculated by Formula (1):

$$\frac{x_a - x_0}{x_0} \times 100 \qquad (1)$$

where

x_0 is the value of the property before ageing;

x_a is the value of the property after ageing.

Exception: express changes in hardness always as the difference $x_a - x_0$.

NOTE The rubber industry uses the term equation for the relationships herein termed formula. The term formula is used to describe the table of ingredients in a rubber compound.

12 Precision

See Annex B.

13 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 188:2023;

b) the sample details:

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

   2) details of the compound and its condition of cure, if known,

   3) the time interval between forming and testing,

   4) the method used to prepare the test pieces (e.g. moulding, cutting from the sample) and the location where the test pieces were taken from the sample;

c) the test method:

   1) a reference to this document,

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   2) the method used (A, B, C or D),
   3) the properties determined and the type of test piece used;

d) the test details:
   1) the type of oven used,
   2) the number of test pieces used,
   3) whether accelerated ageing or a heat resistance test was carried out,
   4) the temperature and duration of ageing,
   5) details of any procedures not specified in this document;

e) the test results:
   1) the individual values before and after ageing, expressed in accordance with the International Standards for the appropriate property tests,
   2) the changes in the property values, expressed as a percentage or, for hardness, as the difference between the values;

f) the date of the test.

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Annex A
(informative)

Determination of the air speed inside the ovens with forced air circulation

A.1 Scope

This annex describes a method for determining the air speed method B, method C and method D type ovens.

A.2 Apparatus

A portable anemometer can be used.

A.3 Procedure

A.3.1 The air speed should be measured at nine positions at the level of the centre of a suspended test piece. For this purpose, prepare an at least 2 mm thick transparent plastic plate made of PVC [(poly(vinyl chloride)] or PMMA [(poly(methyl methacrylate)], of the same size as the door of the oven chamber. Drill three holes into the plate, each big enough to allow an anemometer to be inserted through it, two located 70 mm from the left and right edge, respectively, and one centred between the other two (see Figure A.1).

A.3.2 The measurement of the air speed should be carried out at a standard laboratory temperature.

A.3.3 Open the door of the chamber and fix the plastic plate in the door opening. Make sure that the oven can be operated even the door is open.

A.3.4 Operate the oven and insert the anemometer sensor through each aperture. Measure the air speed at all nine positions as indicated in Figure A.1. Keep the gap between the plate and the handle of the anemometer airtight. The two not used apertures shall be closed during measurement.

A.3.5 For method D type ovens make sure that the anemometer is inserted in the direction of the main airflow (back to front) of the oven.

A.3.6 Read the maximum value of the air speed at each position to avoid any effect due to the directionality of the sensor.

A.4 Calculation of result

A.4.1 Calculate the mean value of the air speed measured at the nine measurement positions.

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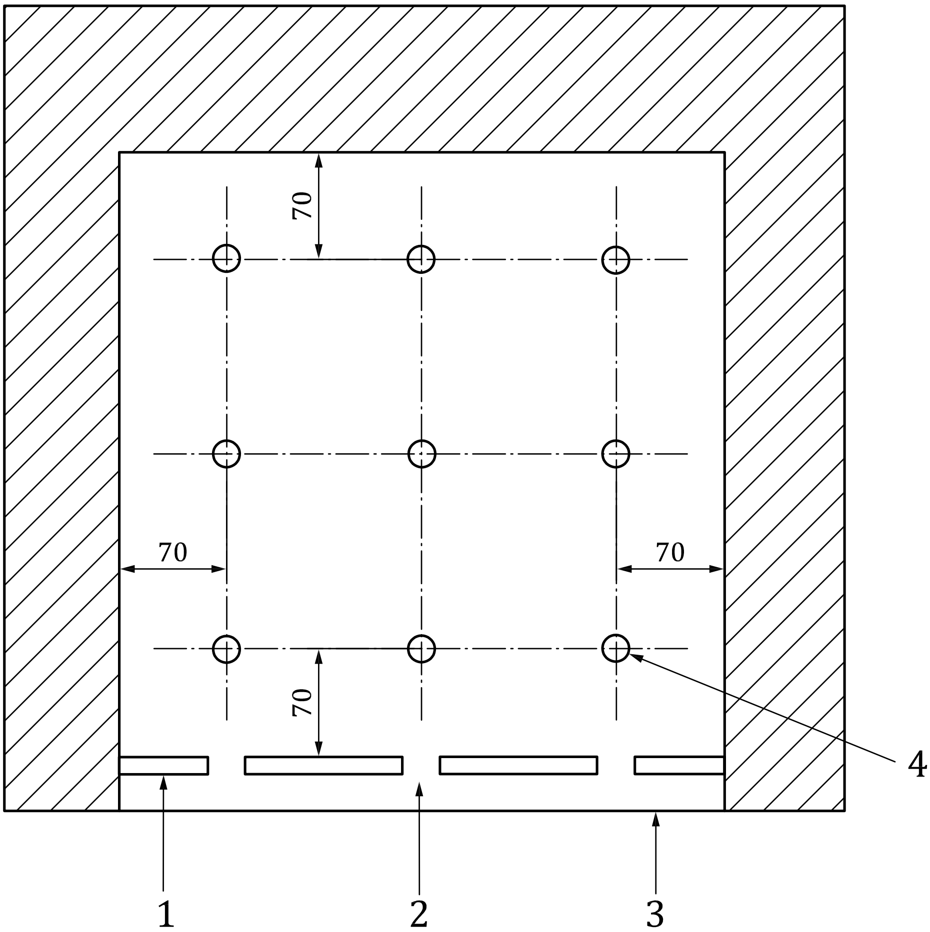
Dimensions in millimetres



Key

1 plastic plate

2 aperture

3 door opening

4 measurement position

Figure A.1 — Positions for measuring air speed inside the oven

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Annex B
(informative)

Precision

B.1 General

Two interlaboratory test programmes (ITPs) and the precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272[1]. The first ITP was organized in 1996 and the results analysed in 1997, and the second one in 2005. Consult ISO 19983 for precision concepts and nomenclature. Annex C gives guidance on the use of repeatability and reproducibility results.

B.2 Precision details of the first ITP

B.2.1 Prepared test pieces were sent out to all participating laboratories using four compounds (of types NR, NBR, EPDM and AEM). Ageing was carried out by both method A and method B.

The ageing time was 168 h for all compounds, at 70 °C for NR, 100 °C for NBR, 125 °C for EPDM and 150 °C for AEM.

B.2.2 A total of 16 laboratories participated in this ITP. Eleven of the laboratories carried out the ageing by method A and ten laboratories by method B. Five of the laboratories used both method A and B. For certain of the tests carried out after ageing, values were missing from the compiled data, and for these tests fewer than these numbers of laboratories were involved. The actual number for each test is listed in the precision tables.

B.2.3 The hardness was measured in accordance with ISO 48-2, method M, before and after ageing. The three tensile strength properties were measured in accordance with ISO 37 on five test pieces before and after ageing. Type 1 and type 2 dumb-bell test pieces were used.

B.2.4 The precision determined in this ITP is a type 1 precision, i.e. fully prepared test pieces were submitted to all laboratories. The precision is also an intermediate-term or intermediate time period precision, with a time of two to three weeks between the two replicate determinations. This is in distinction to the more usual day 1 to day 2 replication with a few days between the determinations.

The symbols used in the tables are as follows:

- *r* repeatability, in measurement units;
- (*r*) repeatability, expressed as a percentage of the average;
- *R* reproducibility, in measurement units;
- (*R*) reproducibility, expressed as a percentage of the average.

(*r*) and (*R*) have only been calculated for all the materials together.

1) Withdrawn and replaced by ISO 19983.

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B.3 Precision results from the first ITP

B.3.1 The precision results are given in Tables B.1 to B.4 for method A (low air speed) and in Tables B.5 to B.8 for method B (high air speed). In these tables, no values of the relative precision (*r*) and (*R*) are given for the individual materials because many of the mean values of the performance parameters are near zero and this gives very large (*r*) and (*R*) values that have little meaning. The tables do give a mean value (similar but not equal to a pooled value) for all four materials together. These overall means are useful in comparing the relative precision of the four types of test performed. The relative precision for these overall means enables the two methods (A and B) to be compared.

B.3.2 On reviewing the tables, it will be observed that there is only a small difference between the repeatability *r* and the reproducibility *R*, and in several cases the two are equal. This phenomenon has been observed in the previous edition of this document ageing-precision testing. This demonstrates that a very large component of the variation observed in this type of testing is not due to differences between laboratories but is due to some inherent source of variation that is just as likely to occur "within" a laboratory as on a "between"-laboratory basis. This unknown source is connected with the ageing process.

Table B.1 — Ageing precision determined from change in hardness (IRHD) (method A: low air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	3,1	3,10		3,63		11
NBR	4,4	2,08		3,68		11
EPDM	22,0	5,50		10,30		11
AEM	3,9	6,78		7,78		11
Absolute mean (without regard to sign)	8,3	4,4		6,3		
Relative precision			53		76	

Table B.2 — Ageing precision determined from change in tensile strength (TS_b) (method A: low air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	−8,7	8,43		9,34		11
NBR	6,6	9,26		11,83		11
EPDM	4,1	8,24		14,92		11
AEM	−9,3	8,13		10,71		11
Absolute mean (without regard to sign)	7,2	8,5		11,7		
Relative precision			118		162	

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Table B.3 — Ageing precision determined from change in stress at 100 % elongation (S_{100}) (method A: low air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	25,2	13,4		16,0		11
NBR	38,4	26,8		26,8		11
EPDM	247,1	78,9		135,3		11
AEM	0,4	15,4		22,7		11
Absolute mean (without regard to sign)	77,7	33,6		50,2		
Relative precision			43		65	

Table B.4 — Ageing precision determined from change in elongation at break (E_b) (method A: low air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	−13,3	10,36		10,36		11
NBR	−17,7	14,00		14,00		11
EPDM	−66,5	4,85		7,44		11
AEM	0,8	7,72		17,12		11
Absolute mean (without regard to sign)	24,2	9,2		12,2		
Relative precision			38		50	

Table B.5 — Ageing precision determined from change in hardness (IRHD) (method B: high air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	4,1	5,14		5,14		10
NBR	8,7	3,20		5,29		10
EPDM	35,9	3,89		9,67		10
AEM	8,0	5,04		8,00		10
Absolute mean (without regard to sign)	14,2	4,3		7,0		
Relative precision			30		49	

Table B.6 — Ageing precision determined from change in tensile strength (TS_b) (method B: high air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	−8,5	7,07		9,23		10
NBR	12,3	12,88		12,88		10
EPDM	7,9	11,88		11,88		10
AEM	−4,4	8,93		10,73		10

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Table B.6 *(continued)*

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
Absolute mean (without regard to sign)	8,3	10,2		11,2		
Relative precision			122		134	

Table B.7 — Ageing precision determined from change in stress at 100 % elongation (S_{100}) (method B: high air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	24,3	10,3		14,0		10
NBR	54,4	25,0		26,7		10
EPDM	392,1	62,5		194,0		10
AEM	19,3	12,0		14,1		10
Absolute mean (without regard to sign)	122,5	27,4		62,2		
Relative precision			22		51	

Table B.8 — Ageing precision determined from change in elongation at break (E_b) (method B: high air speed)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR	−14,8	6,86		9,65		10
NBR	−19,3	9,41		13,14		10
EPDM	−73,0	5,76		8,89		10
AEM	−3,3	9,39		11,80		10
Absolute mean (without regard to sign)	27,6	7,9		10,9		
Relative precision			29		39	

B.4 Precision details of the second ITP

B.4.1 Prepared test pieces were sent out to all participating laboratories using three compounds (of types of NR, NBR and EPDM). Ageing was carried out in type 1 and type 2 ovens using method B (method B cabinet ovens and method C cabinet ovens).

The ageing time was 72 h and 168 h for all compounds at 85 °C for NR, 100 °C for NBR and 125 °C for EPDM.

B.4.2 A total of 11 laboratories participated in this ITP. Five of the laboratories carried out the ageing in type 1 ovens (method B cabinet ovens) and six laboratories in type 2 ovens (method C cabinet ovens). The actual number of laboratories for each test is listed in the precision tables.

B.4.3 The three tensile strength properties were measured in accordance with ISO 37 on five test pieces before and after ageing. Type 1A test pieces were used. Hardness was omitted from the analysis because there were insufficient test results.

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B.4.4 The precision determined in this ITP is a type 1 precision, i.e. fully prepared test pieces were submitted to all laboratories. The precision is also an intermediate-term or intermediate time period precision, with a time of two to three weeks between the two replicate determinations. This is in distinction to the more usual day 1 to day 2 replication with a few days between the determinations.

The symbols used in Tables B.9 to B.14 are the same as those for the first ITP.

B.5 Precision results from the second ITP

B.5.1 The precision results are given in Tables B.9 to B.11 for type 1 ovens (method B cabinet ovens) and in Tables B.12 to B.14 for type 2 ovens (method C cabinet ovens). In these tables, the values for the two ageing times, 72 h and 168 h, are included, but no values of the relative precision (*r*) and (*R*) are given for the individual materials, as in the first ITP. The relative precision for these overall means enables the two types of oven to be compared in the same way as in the first ITP.

B.5.2 On reviewing the tables, it can be seen that the type 1 and type 2 ovens (method B cabinet ovens and method C cabinet ovens) give almost the same precision. The type 2 oven (method C cabinet oven) in fact gives slightly more uniform ageing and a slightly larger change in the properties on ageing.

Table B.9 — Ageing precision determined from change in tensile strength (TS_b) (type 1 oven) (method B cabinet oven)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR, 72 h	−3,2	4,2		8,7		5
NR, 168 h	−11,5	6,7		15,7		5
NBR, 72 h	0,5	6,0		13,8		5
NBR, 168 h	−4,0	11,6		11,3		5
EPDM, 72 h	−6,0	7,7		10,3		5
EPDM, 168 h	−7,8	14,9		19,0		5
Absolute mean (without regard to sign)	5,5	8,5		13,1		
Relative precision			155		238	

Table B.10 — Ageing precision determined from change in stress at 100 % elongation (S_{100}) (type 1 oven) (method B cabinet oven)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR, 72 h	26,6	30,0		30,8		5
NR, 168 h	45,6	54,1		45,7		5
NBR, 72 h	39,5	7,4		48,5		5
NBR, 168 h	52,1	8,2		59,7		5
EPDM, 72 h	78,3	44,5		58,0		5
EPDM, 168 h	102,5	48,0		78,2		5
Absolute mean (without regard to sign)	57,4	32,0		53,5		
Relative precision			56		93	

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Table B.11 — Ageing precision determined from change in elongation at break (E_b) (type 1 oven) (method B cabinet oven)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR, 72 h	−12,5	11,9		9,6		5
NR, 168 h	−19,3	1,4		13,8		5
NBR, 72 h	−23,0	4,7		15,6		5
NBR, 168 h	−29,3	9,1		13,1		5
EPDM, 72 h	−42,8	5,9		4,2		5
EPDM, 168 h	−49,3	13,3		11,4		5
Absolute mean (without regard to sign)	29,4	7,7		11,3		
Relative precision			26		38	

Table B.12 — Ageing precision determined from change in tensile strength (TS_b) (type 2 oven) (method C cabinet oven)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR, 72 h	−4,4	7,5		5,5		6
NR, 168 h	−16,1	9,4		9,5		6
NBR, 72 h	−6,7	7,8		17,2		6
NBR, 168 h	−9,6	7,3		8,2		6
EPDM, 72 h	−9,5	7,2		10,3		6
EPDM, 168 h	−9,6	12,7		14,0		6
Absolute mean (without regard to sign)	9,3	8,7		10,8		
Relative precision			94		116	

Table B.13 — Ageing precision determined from change in stress at 100 % elongation (S_{100}) (type 2 oven) (method C cabinet oven)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR, 72 h	38,4	31,5		24,5		6
NR, 168 h	59,1	36,7		29,8		6
NBR, 72 h	53,7	10,4		24,7		6
NBR, 168 h	75,0	28,5		28,9		6
EPDM, 72 h	88,2	27,4		32,2		6
EPDM, 168 h	112,1	39,6		59,6		6
Absolute mean (without regard to sign)	71,1	29,0		33,3		
Relative precision			41		47	

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Table B.14 — Ageing precision determined from change in elongation at break (E_b) (type 2 oven) (method C cabinet oven)

Material	Mean change	Within laboratory		Between laboratories		Number of labs
	%	*r*	(*r*)	*R*	(*R*)	
NR, 72 h	−15,6	13,8		10,2		6
NR, 168 h	−26,1	13,0		10,2		6
NBR, 72 h	−29,6	9,1		12,7		6
NBR, 168 h	−36,0	1,6		3,9		6
EPDM, 72 h	−47,9	14,5		14,7		6
EPDM, 168 h	−53,2	10,4		16,9		6
Absolute mean (without regard to sign)	34,7	10,4		11,4		
Relative precision			30		33	

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Annex C
(informative)

Guidance for using precision results

C.1 The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

C.2 Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the “test” data average under consideration. This line will give the applicable *r*, (*r*), *R* or (*R*) for use in the decision process.

C.3 With these *r* and (*r*) values, the following general repeatability statements may be used to make decisions:

a) For an absolute difference: the difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material test pieces under normal and correct operation of the test procedure, will exceed the tabulated repeatability *r* on average not more than once in twenty cases.

b) For a percentage difference between two test (value) averages: the percentage difference is given by Formula (C.1):

$$\frac{|x_1 - x_2|}{\frac{1}{2}(x_1 + x_2)} \times 100 \quad \text{(C.1)}$$

between two test values, found on nominally identical material test pieces under normal and correct operation of the test procedure, will exceed the tabulated repeatability (*r*) on average not more than once in twenty cases.

C.4 With these *R* and (*R*) values, the following general reproducibility statements may be used to make decisions:

a) For an absolute difference: the absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material test pieces, will exceed the tabulated reproducibility *R* not more than once in twenty cases.

b) For a percentage difference between two test (value) averages: the percentage difference is given by Formula (C.2):

$$\frac{|x_1 - x_2|}{\frac{1}{2}(x_1 + x_2)} \times 100 \quad \text{(C.2)}$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material test pieces, will exceed the tabulated reproducibility (*R*) not more than once in twenty cases.

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Annex D
(normative)

Calibration schedule

D.1 Inspection

Before any calibration is conducted, the condition of the items to be calibrated shall be determined and documented in a calibration report or certificate. It shall be indicated whether calibration is carried out before the first operation or after a repair or correction of the item.

It shall be insured that the item is generally suitable for the intended purpose, including all needed specifications even they are not necessary for calibration. If such parameters can change, the need for periodic checks shall be included in the detailed calibration procedures.

D.2 Schedule

Verification/calibration of the test device is a mandatory part of this document. The frequency of calibration and procedures used are at the discretion of the individual laboratory, unless otherwise stated, using ISO 18899 as a guide.

The calibration schedule given in Table D.1 has been compiled by listing all the parameters given in the test method along with the specified requirement. A parameter and requirement can relate to the main test device, to part of this device or to an additional device required for the test.

For each parameter, a calibration procedure is given with reference to ISO 18899, to another publication or to a procedure specific to the detailed test method. If a more specific or detailed calibration procedure is available than given in ISO 18899 it shall be used in preference.

The checking frequency for each parameter is indicated by a code-letter. The code-letters used in the calibration schedule are:

P particular procedure;

C requirement to be confirmed, but not measured;

N initial check only;

S standard interval as given in ISO 18899;

U in use.

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Table D.1 — Calibration frequency schedule

Parameter	Requirement	Clause or subclause in ISO 18899:2013	Verification frequency guide	Notes
Oven volume: Sample Volume relationship	Such that volume of test piece does not exceed 10 % of free space	C	U	
Provision for suspending test pieces	At least 10 mm apart and (for cabinet and forced air circulation ovens) at least 50 mm from sides	15.2	U	
Temperature	Within tolerances specified in Clause 8	18 (procedure B)	S	
Material of oven chamber	No copper or copper alloys used in construction of heating chamber	C	N	
Air flow	Between 3 and 10 changes per hour (method A, B, C) >30 changes per hour (Method D)	16.3	S	
Method A cell oven Height Air speed Design	 300 mm minimum Shall depend only on exchange rate Cells surrounded by good heat transfer medium and air passing through one cell shall not enter other cells	 15.2 C C	 N N N	
Method A cabinet oven	No fans in heating chamber	C	N	
Method B, C, D cabinet oven with forced air circulation Layout Air speed	 See 5.4 0,5 m/s to 1,5 m/s (method B) or 0,5 ± 0,25 m/s (method C) or 0,25 m/s to 3,0 m/s (method D)	 C P	 N S	See Annex A for a method of verification

In addition to the items listed in Table D.1, the use of the following elements is implied, all of which shall be calibrated in accordance with ISO 18899:

— a timer;

— a thermometer for monitoring the conditioning and test temperatures.

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Bibliography

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

[2] ISO 48-2, *Rubber, vulcanized or thermoplastic — Determination of hardness — Part 2: Hardness between 10 IRHD and 100 IRHD*

[3] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*[2)]

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

[5] ISO 19983, *Rubber — Determination of precision of test methods*

2) Withdrawn and replaced by ISO 19983.

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