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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 83.060
G 40

GB/T 3512-2014 / ISO 188:2011

Replacing GB/T 3512-2001

Rubber, vulcanized or thermoplastic - Accelerated ageing and heat resistance tests - Air-oven method

(ISO 188:2011, Rubber, vulcanized or thermoplastic -
Accelerated ageing and heat resistance tests, IDT)

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Issued on: December 22, 2014

Implemented on: June 01, 2015

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;**

**Standardization Administration of the People's Republic of
China.**

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Foreword

This Standard is drafted according to the rules provided in GB/T 1.1-2009.

This Standard replaces GB/T 3512-2001 *Rubber, vulcanized or thermoplastic - Accelerated ageing and heat resistance tests - Air-oven method*; compared with GB/T 3512-2001, the main technical changes are as follows:

- Add test method of rubber tensile performance ISO 37 and test method of rubber hardness ISO 48 (as shown in 3.1 of this edition);
- Add test method of multicell ageing oven, cabinet type ageing oven and forced ventilation ageing oven and its wind speed (as shown in 4.1.2, 4.1.3, 4.1.4 of this edition);
- Add provision for equipment verification (as shown in chapter 5 of this edition);
- Use ISO 23529 to replace GB/T 2941 (as shown in chapter 8 of 2001 edition);
- Add results of precision test (as shown in chapter 11 of this edition; chapter 10 of 2001 edition).

Using translation method, this Standard equivalently adopts ISO 188:2011 *Vulcanized rubber or thermoplastic elastomer hot air - Accelerated aging heat resistance test*. The Chinese documents, in this Standard, which have corresponding relationship with the normative international documents are as follows:

- GB/T 528-2009 Rubber, vulcanized or thermoplastic - Determination of tensile stress-strain properties (ISO 37:2005, IDT);
- GB/T 2941-2006 Rubber - General procedures for preparing and conditioning test pieces for physical test methods (ISO 23529:2004, IDT);
- GB/T 6031-1998 Rubber, vulcanized or thermoplastic - Determination of hardness (hardness between 10 IRHD and 100 IRHD) (idt ISO 48:1994).

This Standard was proposed by China Petroleum and Chemical Industry Association.

This Standard shall be under the jurisdiction of Branch of National Rubber and Rubber Goods Technical Committee for Standardization General Test Method (SAC/TC 35/SC 2).

Main drafting organizations of this Standard: Guangzhou Synthetic Material Research Institute Co., Ltd., National Rubber and Latex Products Quality Supervision Test Center, Fengshen Tyre Co., Ltd., Cooper Chengshan (Shandong province) Co., Ltd., Qingdao Yi Kesi New Material Co., Ltd., Shandong Bayi Tyre Manufacture Co., Ltd., Jiangsu Mingzhu Test Machinery Co., Ltd., Beijing Rubber Industry Research and Design Institute, Guizhou Tyre Co., Ltd.

Introduction

Accelerated ageing and heat resistance tests are designed to estimate the relative resistance of rubber to deterioration with the passage of time. For this purpose, the rubber is subjected to controlled deteriorating influences for definite periods, after which appropriate properties are measured and compared with the corresponding properties of the unaged rubber.

In accelerated ageing, the rubber is subjected to a test environment intended to produce the effect of natural ageing in a shorter time.

In the case of heat resistance tests, the rubber is subjected to prolonged periods at the same temperature as that which it will experience in service.

Two types of method are given in this Standard, namely an air-oven method using a low air speed and an air-oven method using forced air ventilation for a high air speed.

The selection of the time, temperature and atmosphere to which the test pieces are exposed and the type of oven to use will depend on the purpose of the test and the type of polymer.

In air-oven methods, deterioration is accelerated by raising the temperature. The degree of acceleration thus produced varies from one rubber to another and from one property to another.

Degradation can also be accelerated by air speed. Consequently, ageing with different ovens can give different results.

Consequences of these effects are:

- a) Accelerated ageing does not truly reproduce under all circumstances the changes produced by natural ageing.
- b) Accelerated ageing sometimes fails to indicate accurately the relative natural or service life of different rubbers; thus, ageing at temperatures greatly above ambient or service temperatures may tend to equalize the apparent lives of rubbers, which deteriorate at different rates in storage or service. Ageing at one or more intermediate temperatures is useful in assessing the reliability of accelerated ageing at high temperatures.
- c) Accelerated ageing tests involving different properties may not give agreement in assessing the relative lives of different rubbers and may even arrange them in different orders of merit. Therefore, deterioration should be measured by the changes in property or properties which are of practical importance, provided that they can be measured reasonably accurately.

Air-oven ageing should not be used to simulate natural ageing which occurs in the

Rubber, vulcanized or thermoplastic - Accelerated ageing and heat resistance tests - Air-oven method

WARNING — Persons using this Standard should be familiar with normal laboratory practice. This 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.

1 Scope

This Standard specifies accelerated ageing or heat resistance tests on vulcanized or thermoplastic rubbers. Two methods are given:

Method A: air-oven method using a cell-type oven or cabinet oven with low air speed and a ventilation of 3 to 10 changes per hour;

Method B: air-oven method using a cabinet oven with forced air circulation by means of a fan and a ventilation of 3 to 10 changes per hour.

2 Normative references

The articles contained in the following documents have become part of this document when they are quoted herein. For the dated documents so quoted, all the modifications (including all corrections) or revisions made thereafter shall be applicable to this document.

GB/T 25269-2010 Rubber - Guide to the calibration of test equipment (ISO 18899:2004, IDT)

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

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

ISO 23529 Rubber - General procedures for preparing and conditioning test pieces for physical test methods

3 Principle

3.1 General

keep the temperature uniform and stable during the test and to verify that the oven used is within the temperature limits with regard to time and space. Increasing the air speed in the oven improves temperature homogeneity. However, air circulation in the oven and ventilation influence the ageing results. With a low air speed, accumulation of degradation products and evaporated ingredients, as well as oxygen depletion, can take place. A high air speed increases the rate of deterioration, due to increased oxidation and volatilization of plasticizers and antioxidants.

4.1.2 Cell-type 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.

4.1.3 Cabinet oven

This shall comprise 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 heating chamber.

4.1.4 Oven with forced air circulation

Either of the following two types shall be used:

- a) Type 1 oven with laminar air flow (see Figure 1).

The air flow through the heating chamber shall be as uniform and laminar as possible. The test pieces shall be placed with the smallest surface facing towards the air flow to avoid disturbing 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.

- b) Type 2 oven with turbulent air flow (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 $0.5 \text{ m/s} \pm 0.25 \text{ m/s}$.

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 in Annex A). A suitable method of measurement is described in Annex A.

8 Ageing conditions (duration and temperature)

8.1 General

The period required to obtain a given degree of deterioration of the test pieces will depend upon the type of rubber under examination.

The ageing period used should preferably be such that deterioration of the test pieces will not be so great as to prevent determination of the final values of physical properties.

The use of high ageing temperatures may result in different degradation mechanisms than those which occur at service temperatures, thus invalidating the results.

It is crucial for the best results that the temperature be kept as stable as possible. Temperature tolerances in ISO 23529 are $\pm 1^\circ\text{C}$ up to and including 100°C and $\pm 2^\circ\text{C}$ 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.

8.2 Accelerated ageing

The duration of ageing and the ageing temperature shall be chosen in accordance with ISO 23529, as stated in the product specification or as agreed between the interested parties. The ageing shall be performed at atmospheric pressure.

8.3 Heat resistance test

The test duration and the temperature of test shall be chosen in accordance with ISO 23529, as stated in the product specification or as agreed between the interested parties. The temperature shall be representative of the service temperature and the heating shall be carried out at atmospheric pressure.

9 Procedure

Heat the oven to the operating 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 not less than 16 h and not more than 6 days in a strain-free condition in the atmosphere given in the appropriate test method for the particular property being studied.

- 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 of the test pieces in the sample.
- b) test method:
- 1) a reference to this Standard,
 - 2) the method used (A or B),
 - 3) the properties determined and the type of test piece used.
- c) 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 the procedures not specified in this Standard.
- d) 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.
- e) the date of the test.

Annex A

(Informative)

Determination of the air speed in ovens with forced air circulation

A.1 Scope

This annex describes a method for determining the air speed in both type 1 and type 2 ovens.

A.2 Apparatus

A portable anemometer can be used.

A.3 Procedure

A.3.1 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, and drill three apertures, each big enough to allow an anemometer to be inserted in it, two located 70 mm from the left and right edge, respectively, and one at the mid-point between the 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.

A.3.4 Operate the oven and, inserting the anemometer sensor through each aperture in turn, measure the air speed at all nine positions indicated in Figure A.1. Keep the gap between the plate and the stem of the anemometer airtight.

A.3.5 Read the maximum value of the air speed at each position so as 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.

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. The first ITP was organized in 1996 and the results analysed in 1997, and the second one in 2005. Consult ISO/TR 9272 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 48:1994 , 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;

Annex D

(Normative)

Calibration schedule

D.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration is carried out 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 as approximate and 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.

D.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using GB/T 25269-2010 for guidance.

The calibration schedule given in Table D.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, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to GB/T 25269-2010, to another publication or to a procedure particular to the test method which is detailed (whenever a more specific or detailed calibration procedure than in GB/T 25269-2010 is available, it shall be used in preference).

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

- P particular procedure;
- C requirement to be confirmed, but no measurement;
- N initial verification only;
- S standard interval as given in GB/T 25269-2010;
- U in use.

Bibliography

- [1] ISO/TR 9272, Rubber and rubber products - Determination of precision for test method standards (GB/T 14838-2009, ISO/TR 9272:2005, IDT)
- [2] ISO 11346, Rubber, vulcanized or thermoplastic - Estimation of life-time and maximum temperature of use (GB/T 20028-2005, ISO 11346:1997, IDT)

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