
**Rubber, vulcanized or thermoplastic —
Resistance to ozone cracking —**

**Part 1:
Static and dynamic strain testing**

*Caoutchouc vulcanisé ou thermoplastique — Résistance au craquelage
par l'ozone —*

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Partie 1: Essais sous allongement statique et dynamique
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ISO 1431-1:2004

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 1431-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analyses*.

This fourth edition cancels and replaces the third edition (ISO 1431-1:1989) and, in addition, the second edition of ISO 1431-2 (ISO 1431-2:1994), which have been combined and technically revised.

ISO 1431 consists of the following parts, under the general title *Rubber, vulcanized or thermoplastic — Resistance to ozone cracking*:

- *Part 1: Static and dynamic strain testing*
- *Part 3: Reference and alternative methods for determining the ozone concentration in laboratory test chambers*

Rubber, vulcanized or thermoplastic — Resistance to ozone cracking —

Part 1: Static and dynamic strain testing

WARNING — Persons using this part of ISO 1431 should be familiar with normal laboratory practice. This part of ISO 1431 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 part of ISO 1431 specifies procedures intended for use in estimating the resistance of vulcanized or thermoplastic rubbers to cracking when exposed, under static or dynamic tensile strain, to air containing a definite concentration of ozone and at a definite temperature in circumstances that exclude the effects of direct light.

Great caution is necessary in attempting to relate standard test results to service performance since the relative ozone resistance of different rubbers can vary markedly depending on the conditions, especially ozone concentration and temperature. In addition, tests are carried out on thin test pieces deformed in tension and the significance of attack for articles in service can be quite different owing to the effects of size and of the type and magnitude of the deformation. Explanatory notes on the nature of ozone cracking are given in Annex A.

Reference and alternative methods for determining the ozone concentration are described in ISO 1431-3.

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 1431-3, *Rubber, vulcanized or thermoplastic — Resistance to ozone cracking — Part 3: Reference and alternative methods for determining the ozone concentration in laboratory test chambers*

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

3 Terms and definitions

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

3.1 threshold strain
highest tensile strain at which a rubber can be exposed at a given temperature to air containing a given concentration of ozone without ozone cracks developing on it after a given exposure period

NOTE It is important to distinguish threshold strain from limiting threshold strain defined in 3.2.

3.2 limiting threshold strain
tensile strain below which the time required for the development of ozone cracks increases very markedly and can become virtually infinite

3.3 dynamic strain
strain (normally a tensile strain) varying sinusoidally with time at some selected repetition rate or frequency

NOTE The maximum strain and the repetition rate are used to describe the dynamic strain conditions.

4 Principle

Test pieces are exposed, under static tensile strain, under continuous dynamic strain, or under alternate periods of dynamic and static strain, in a closed chamber at a constant temperature, to an atmosphere containing a fixed concentration of ozone. The test pieces are examined periodically for cracking.

Three alternative evaluation procedures are described for selected values of ozone concentration and exposure temperature:

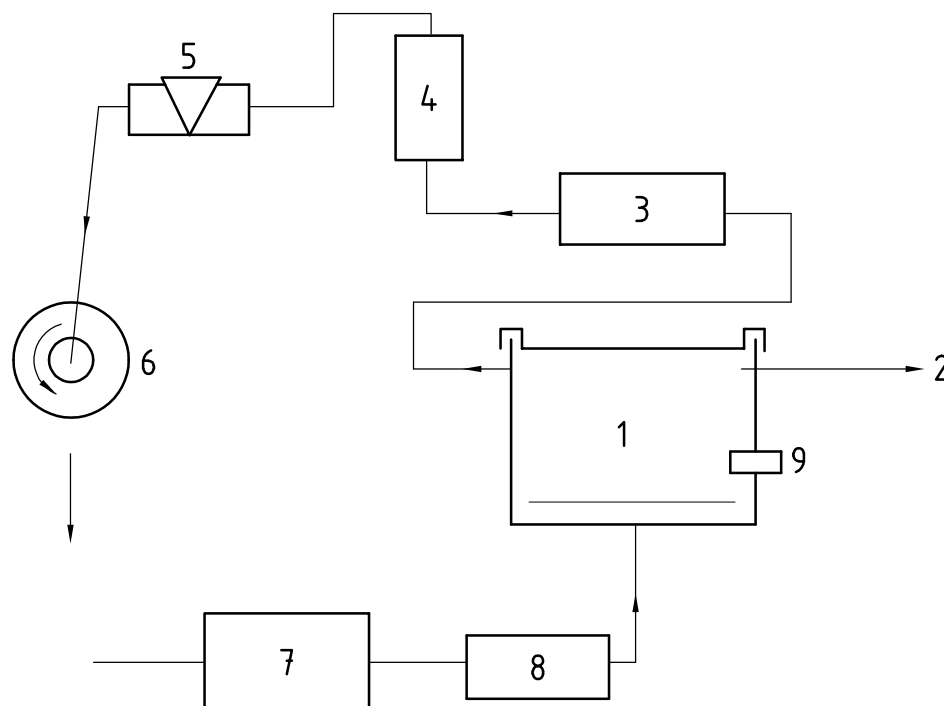
- a) The presence or absence of cracks is determined after exposure for a fixed period of time at a given static strain, dynamic strain or combination of dynamic and static strains. If required, an estimate of the degree of cracking is made.
- b) The time to the first appearance of cracks is determined at any given static strain, dynamic strain or combination of dynamic and static strains.
- c) The threshold strain is determined for any given exposure period (valid only for static tensile-strain tests).

5 Apparatus (see Figure 1)

WARNING — Attention is drawn to the highly toxic nature of ozone. Efforts should be made to minimize the exposure of workers at all times. In the absence of more stringent or contrary national safety regulations in the user's country, it is recommended that 0,1 parts of ozone per million parts of air of the surrounding atmosphere by volume be regarded as an absolute maximum concentration whilst the maximum average concentration should be appreciably lower. Unless a totally enclosed system is being used, an exhaust vent to remove ozone-laden air is advised.

5.1 Test chamber

This shall be a closed, non-illuminated chamber, thermostatically controlled to within $\pm 2^\circ\text{C}$ of the test temperature, lined with, or constructed of, a material (for example aluminium) that does not readily decompose ozone. The dimensions shall be such that the requirements of 5.5 are met. The chamber can be provided with a window through which the surface of the test pieces can be observed. A light to examine test pieces can be installed, but this shall remain switched off at all other times.

**Key**

- | | | | |
|---|---|---|-----------------------|
| 1 | test chamber | 6 | circulation fan |
| 2 | to ozone concentration measurement device | 7 | ozonizer |
| 3 | purifying column | 8 | heat exchanger |
| 4 | flowmeter | 9 | temperature indicator |
| 5 | regulator | | |

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Figure 1 — Example of a test apparatus

5.2 Source of ozonized air

The ozonized air shall be largely free of nitrogen oxides in order to avoid errors in the ozone concentration. One of the following items of apparatus shall therefore be used:

- a) ultra-violet lamp;
- b) silent-discharge tube.

When utilizing the discharge tube, the use of oxygen, as opposed to air, is necessary in order to avoid the formation of nitrogen oxides. The ozonized oxygen or air can subsequently be diluted with air to attain the required ozone concentration. Air used for generation of ozone or for dilution shall first be purified by passing it over activated charcoal and shall be free from any contaminants likely to affect the ozone concentration, the estimation of the ozone concentration or the cracking of the test pieces.

The temperature of the source shall be kept constant to within ± 2 °C.

The ozonized air shall be fed from the source into the chamber via a heat exchanger to adjust its temperature to that required for the test and shall also be brought to the specified relative humidity (see 8.3).

5.3 Means of adjusting the ozone concentration

When an ultra-violet lamp is used, the ozone concentration can be controlled by adjusting either the voltage applied to the tube or the input-gas or diluent-air flow rate, or by shielding part of the tube from the UV light. When a silent-discharge tube is used, the ozone concentration can be controlled by adjusting the voltage applied to the generator, the dimensions of the electrodes, or the oxygen or diluent-air flow rate. Two-stage dilution of the ozonized air can also be used. The adjustments shall be such that they will maintain the concentration within the tolerances given in 8.1. In addition, after each occasion that the test chamber is opened for insertion or inspection of test pieces, the ozone concentration shall return to the test concentration within 30 min. The concentration of the ozone entering the chamber shall at no time exceed the concentration specified for the test.

NOTE Such adjustments may be manual or automatic.

5.4 Means of determining the ozone concentration

A means of sampling the ozonized air from the vicinity of the test pieces in the chamber and a means of estimating the ozone content shall be provided.

Reference and alternative methods of determining the ozone concentration are described in ISO 1431-3.

5.5 Means of adjusting the gas flow

A mechanism shall be provided that is capable of adjusting the average velocity of the flow of ozonized air in the test chamber to a value of not less than 8 mm/s and preferably to a value between 12 mm/s and 16 mm/s, calculated from the measured gas flow rate in the chamber divided by the effective cross-sectional area of the chamber normal to the gas flow. In tests intended to be comparable, the velocity shall not vary by more than $\pm 10\%$. The gas flow rate is the volume throughput of ozonized air in unit time, and this shall be sufficiently high to prevent the ozone concentration in the chamber being significantly reduced owing to ozone destruction by the test pieces. The rate of destruction will vary depending on the rubber being used, the test conditions and other details of the test. As a general guide, it is recommended that the ratio of the exposed surface area of the test pieces to the gas flow rate should not exceed 12 s/m² (see Note 1). However, the value of this ratio is not always low enough. In cases where there is doubt, the effects of destruction should be checked experimentally and, if necessary, the test piece area decreased. A diffusing screen or equivalent device shall be used to assist thorough mixing of incoming gas with that in the chamber.

In order to adjust the ozone concentration in the chamber and to exclude the effect of volatile components that are produced by test pieces, air circulation apparatus that leads in fresh ambient air can be used.

If high velocities are desired, a fan can be installed in the chamber to raise the velocity of the ozonized air to 600 mm/s \pm 100 mm/s. If this is the case, it shall be stated in the test report.

NOTE 1 The ratio, expressed in seconds per metre (s/m), is derived from surface area in m² and volumetric flow rate in m³/s.

NOTE 2 Different results may be obtained if different ozonized-air velocities are used.

5.6 Mounting test pieces for static strain testing

Clamps shall be provided to hold the test pieces at the required elongation and with both sides in contact with the ozonized air in such a manner that the longitudinal axis of each test piece is substantially parallel to the direction of gas flow. The clamps shall be made of a material (for example aluminium) which does not readily decompose ozone.

The use of a mechanically rotating carrier mounted in the test chamber and upon which the clamps or frames holding the test pieces are mounted is recommended to equalize the effect of different ozone concentrations in different parts of the chamber. In one example of a suitable carrier, the test pieces move at a speed between 20 mm/s and 25 mm/s in a plane normal to the gas flow and each follows, consecutively, the same path in such a manner that the same position within the chamber is visited by the same test piece every 8 min to 12 min, and the area swept by the test pieces (shown shaded in Figure 2) is at least 40 % of the available cross-sectional area of the chamber.

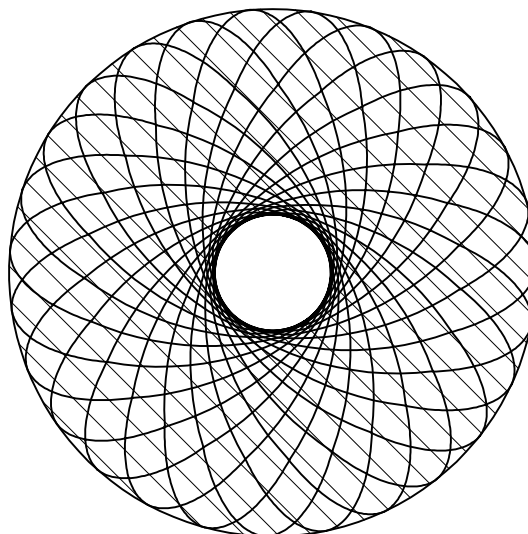


Figure 2 — Path of test pieces and swept area (shaded)

5.7 Mounting test pieces for dynamic strain testing

The apparatus shall be constructed of a material (for example aluminium) that does not readily decompose ozone.

Its essential features are stationary parts, provided with grips for holding one end of each of the test pieces in a fixed position, and similar but reciprocating parts for holding the other end of each test piece. The travel of the reciprocating parts shall be such that the initial minimum distance between the grips gives zero strain and the maximum distance gives the specified maximum strain.

The reciprocating parts shall be so arranged that their motion is in a straight line and in the direction of the common centreline of each opposing pair of grips. Corresponding planes in the upper and lower grips shall remain parallel to each other throughout the motion.

The eccentric which actuates the reciprocating parts shall be driven by a constant-speed motor to give a frequency of $0,5 \text{ Hz} \pm 0,025 \text{ Hz}$. If necessary, a timing device can be provided which stops the apparatus after a period of dynamic strain exposure and starts it again after a rest period.

The grips shall hold the test pieces firmly, without any slipping or tearing, and shall enable adjustments to be made to the test pieces to ensure accurate insertion. Each test piece shall be held in such a way that both sides are in contact with the ozonized air and the longitudinal axis of the test piece is substantially parallel to the direction of gas flow.

6 Test pieces

6.1 General

Standard test pieces shall be as specified in 6.2 or 6.3.

Test pieces shall be cut from moulded sheet, or, if required, from a finished product, in accordance with ISO 23529. Test pieces shall have an undamaged test surface; ozone resistance shall not be assessed on surfaces that have been cut or buffed. Comparisons of different materials are only valid if the cracking is assessed on surfaces of similar finish produced by the same method.

For each set of test conditions, at least three test pieces shall be used.

NOTE 1 It is recommended that test sheets be moulded between highly polished aluminium foil, which is left on the sheets until the test pieces are prepared. This provides protection against handling and ensures a fresh test surface at the time of testing.

NOTE 2 As an alternative to the exposure of test pieces for static strain tests at several different strains, a test piece in the form of an annulus has been used which is strained to produce a continuous range of extensions. This method has been found to give approximately equivalent results to the standard test pieces when used to determine threshold strain.

NOTE 3 As another alternative to the exposure of test pieces at several different strains, a rectangular test piece in the form of a bent loop can be used to provide a continuous range of extensions with one test piece.

6.2 Wide test piece

This test piece shall consist of a strip of not less than 10 mm in width, of thickness $2,0 \text{ mm} \pm 0,2 \text{ mm}$ and of length not less than 40 mm between the grips before stretching.

The ends of the test piece held in the grips can be protected with an ozone-resistant lacquer. Care shall be taken when selecting the lacquer to ensure the solvent used does not appreciably swell the rubber. Silicone grease shall not be used. Alternatively, the test piece can be provided with modified ends, for example by the use of lugs, to enable it to be extended without causing excessive stress concentration and hence breakage at the grips during ozone exposure.

6.3 Narrow test piece

This test piece shall consist of a strip of width $2,0 \text{ mm} \pm 0,2 \text{ mm}$, thickness $2,0 \text{ mm} \pm 0,2 \text{ mm}$ and length 50 mm, between enlarged tab ends 6,5 mm square (see Figure 3). This test piece shall not be used for procedure A.

NOTE Dumb-bell test pieces in accordance with ISO 37 may also be used.

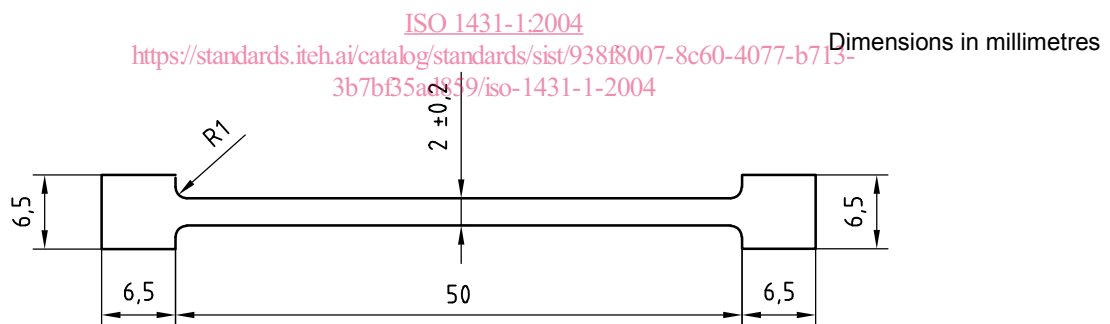


Figure 3 — Narrow test piece

7 Conditioning

7.1 Conditioning in the unstrained state

For all tests, the minimum time between vulcanization and straining of the test pieces shall be 16 h.

For non-product tests, the maximum time between vulcanization and straining of the test pieces shall be 4 weeks.

For product tests, wherever possible, the time between vulcanization and straining of the test pieces shall not be more than 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the customer.

Test pieces and test sheets shall not, between the time of vulcanization and insertion in the test chamber, be allowed to come into contact with rubbers of a different composition. This is necessary to prevent additives which can affect the development of ozone cracks, such as anti-ozonants, from migrating by diffusion from one rubber into adjacent rubbers.

It is recommended that aluminium foil be placed between test pieces and sheets of different compositions, but other methods which prevent migration of additives can also be used.

Samples and test pieces shall be stored in the dark, in an essentially ozone-free atmosphere, during the period between vulcanization and testing; the storage temperature shall normally be a standard laboratory temperature (see ISO 23529), but other, controlled, temperatures can be used if appropriate for particular applications. The same storage conditions shall also be used, as far as possible, for products. For evaluations intended to be comparative, the storage time and conditions shall be the same.

For thermoplastic rubbers, conditioning and storage shall begin immediately after shaping.

7.2 Conditioning in the strained state (for static strain testing only)

After stretching, the test pieces shall be conditioned for a period of between 48 h and 96 h in an essentially ozone-free atmosphere in the dark; the temperature for this conditioning shall normally be a standard laboratory temperature (see ISO 23529), but other temperatures can be used if appropriate for particular applications. The test pieces shall not be touched or otherwise disturbed in any way during the conditioning period. For tests intended to be comparative, the conditioning time and temperature shall be the same.

8 Test conditions

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8.1 Ozone concentration

The test shall be carried out at one of the following ozone concentrations, expressed in parts of ozone per billion of air by volume (ppb) and, in brackets, parts per hundred million (pphm) (see Note 1):

- 250 ppb \pm 50 ppb (25 pphm \pm 5 pphm)
- 500 ppb \pm 50 ppb (50 pphm \pm 5 pphm)
- 1 000 ppb \pm 100 ppb (100 pphm \pm 10 pphm)
- 2 000 ppb \pm 200 ppb (200 pphm \pm 20 pphm)

Unless otherwise specified, the test shall be carried out at an ozone concentration of 500 ppb \pm 50 ppb (50 pphm \pm 5 pphm). If a lower concentration is required for testing rubbers known to be used at low ambient ozone concentrations, an ozone concentration of 250 ppb \pm 50 ppb (25 pphm \pm 5 pphm) is recommended. If highly resistant polymers are being tested, a concentration of 1 000 ppb \pm 100 ppb (100 pphm \pm 10 pphm) or 2 000 ppb \pm 200 ppb (200 pphm \pm 20 pphm) is recommended.

NOTE 1 ppb is used in environmental science for atmospheric pollutants, while pphm has been the traditional unit for ozone concentration in the rubber industry.

NOTE 2 It has been found that differences in atmospheric pressure can influence the effective ozone concentration, and hence the result, when the ozone concentration is expressed in parts per billion (or parts per hundred million) by volume. This effect may be eliminated by expressing the ozone content of the ozonized air in terms of the partial pressure of ozone, i.e. in millipascals, and making comparisons at constant ozone partial pressure. Under standard conditions of atmospheric pressure and temperature (101 kPa, 273 K), an ozone concentration of 10 ppb is equivalent to an ozone partial pressure of 1,01 mPa. Further guidance is given in ISO 1431-3.