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**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 — Partie 1: Essais sous allongement statique et dynamique*





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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 analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 1431-1:2004), which has been technically revised, mainly by addition of a calibration schedule (Annex B) and an annex proposing a simple rating scale (Annex C).

It also incorporates the Amendment ISO 1431-1:2004/Amd.1:2009.

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*

Part 2 was combined with Part 1 at the previous revision of Part 1.

# 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 18899:2004, *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

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:

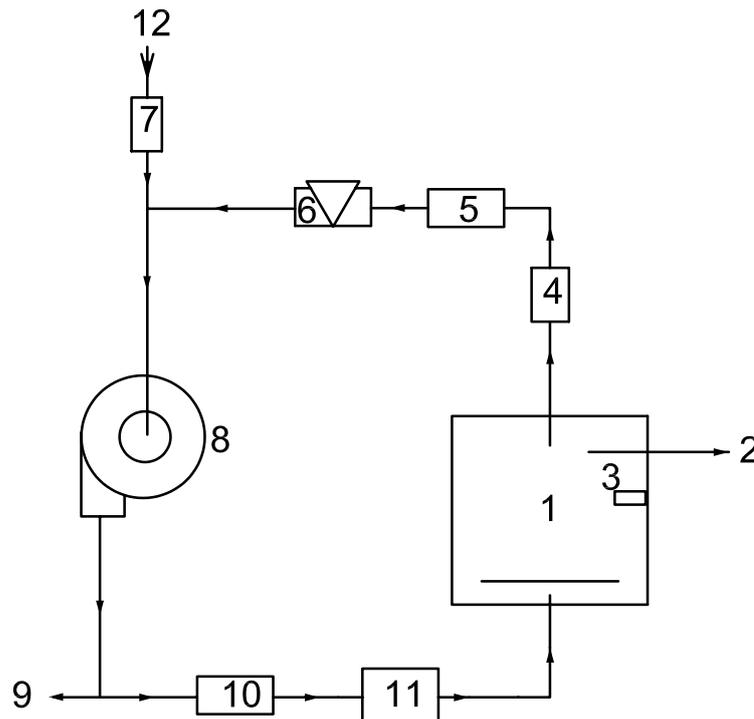
- a) The presence or absence of cracks is determined after exposure for a fixed period of time at a given static strain, dynamic strain or combination of dynamic and static strains. If required, an estimate of the degree of cracking is made.
- b) The time to the first appearance of cracks is determined 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  $\pm 2$  °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 may be provided with a window through which the surface of the test pieces can be observed. A light to examine test pieces may be installed, but this shall remain switched off at all other times.

**Key**

1 test chamber	7 air filter
2 to ozone concentration measurement device	8 circulation fan
3 temperature indicator	9 air outlet
4 purifying column	10 heat exchanger
5 flowmeter	11 ozonizer
6 regulator	12 air inlet

**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.

Air used for the generation of ozone or for dilution of ozonized air 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.

**NOTE** Interference by oxides of nitrogen, which theoretically can be produced in a silent-discharge tube using air, is not expected at the low ozone concentrations specified.

The temperature of the source shall be kept constant to within  $\pm 2$  °C.

The ozonized air shall be fed from the source into the chamber via heat exchanger to adjust its temperature to that required for the test and shall also be brought to the specified relative humidity (see 9.3).

### 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 may also be used. The adjustments shall be such that they will maintain the concentration within the tolerances given in 9.1. 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.

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 not exceed 12 s/m (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 draws in fresh ambient air may be used.

If high velocities are desired, a fan may 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 m<sup>2</sup> and volumetric flow rate in m<sup>3</sup>/s.

NOTE 2 Different results might 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.

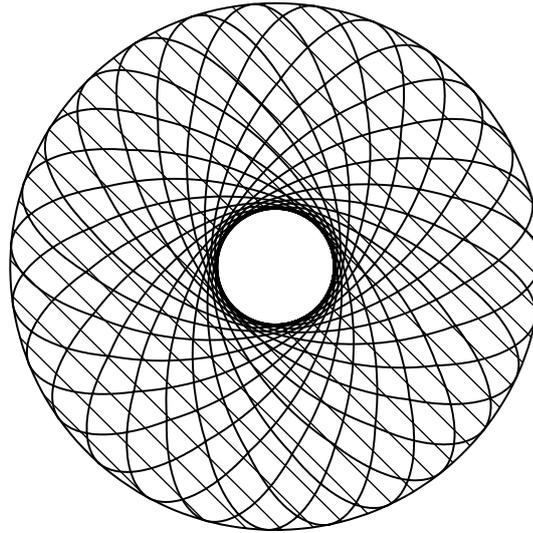


Figure 2 — Path of test pieces and swept area (shaded)

## 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 may 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 Calibration

The requirements for calibration of the test apparatus are given in Annex B.

## 7 Test pieces

### 7.1 General

Standard test pieces shall be as specified in 7.2 or 7.3.

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.

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.

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.

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.

### 7.2 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 may 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 may 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.

### 7.3 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.

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

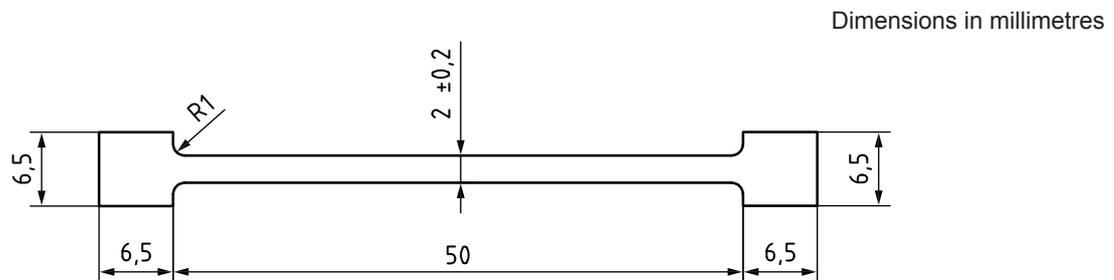


Figure 3 — Narrow test piece

## 8 Conditioning

### 8.1 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 may 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 may 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.

## 8.2 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 may 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.

## 9 Test conditions

### 9.1 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 can 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.

### 9.2 Temperature

The preferred test temperature is 40 °C  $\pm$  2 °C. Other temperatures, such as 30 °C  $\pm$  2 °C or 23 °C  $\pm$  2 °C, may be used if they are more representative of the anticipated service environment, but the results obtained will differ from those obtained at 40 °C  $\pm$  2 °C.

For applications where markedly varying temperatures might be encountered, it is recommended that two or more temperatures, covering the service range, be used.

### 9.3 Relative humidity

The relative humidity of the ozonized air shall normally be not more than 65 % at the test temperature.

Very high humidity can influence the results; when applicable, for products intended for use in damp climates, the test shall be carried out at a relative humidity in the range 80 % to 90 %, if this is practicable.

### 9.4 Maximum elongation

Tests shall normally be carried out using one or more of the following strain levels:

(5 ± 1) %, (10 ± 1) %, (15 ± 2) %, (20 ± 2) %, (25 ± 2) %, (30 ± 2) %, (40 ± 2) %, (50 ± 2) %, (60 ± 2) %, (80 ± 2) %.

The elongation(s) used should be similar to those anticipated in service.

## 10 Static strain testing

### 10.1 General

Adjust the rate of flow and temperature of the ozonized gas and its ozone concentration to that required and place the strained test pieces, suitably conditioned, in the test chamber. Maintain the test conditions at the required levels.

Periodically examine the test pieces for the development of cracking by means of a lens of magnification between 5 and 10, the test pieces being illuminated at the time of examination by a suitably arranged light source. Either the lens may be mounted in a window in the chamber wall, or the test pieces may be removed from the chamber for a short period, in their clamps. The test pieces shall not be handled or knocked against anything when carrying out the examination.

Cracking on surfaces which have been cut or buffed shall be ignored.

The following three alternative procedures for exposure of test pieces are permissible.

### 10.2 Procedure A

Unless otherwise specified, strain the test pieces at 20 % elongation, condition them in accordance with 8.2, and examine them after 72 h in the test chamber for the development of cracking (an alternative elongation and an alternative exposure period may be given in the appropriate material specification).

### 10.3 Procedure B

Strain the test pieces at one or more of the elongations given in 9.4 and condition them in accordance with 8.2. If only one elongation is used, this shall be 20 %, unless otherwise specified. Examine the test pieces after 2 h, 4 h, 8 h, 24 h, 48 h, 72 h and 96 h and, if necessary, at suitable intervals thereafter in the test chamber and note the time until the first appearance of cracks at each elongation.

NOTE Examination after 16 h might also be desirable, even though it is not convenient in practice.

### 10.4 Procedure C

Strain the test pieces at no fewer than four of the elongations given in 9.4 and condition them in accordance with 8.2. Examine the test pieces after 2 h, 4 h, 8 h, 24 h, 48 h, 72 h and 96 h and, if necessary, at suitable intervals thereafter in the test chamber and note the time until the first appearance of cracks at each elongation so that the threshold strain can be estimated.

NOTE See Note to 10.3.

## 11 Dynamic strain testing

### 11.1 General

Adjust the rate of flow and temperature of the ozonized gas and its ozone concentration to that required. Place each test piece, mounted at zero strain, in the dynamic testing apparatus, and by moving the reciprocating part of the apparatus adjust the maximum travel between the grips to give the required maximum elongation. Move the reciprocating part to the position of minimum travel and check that the test piece has returned to zero strain.

After inserting in the test chamber, start the dynamic testing apparatus. Maintain the test conditions at the required levels. No adjustment shall be made during the test to the minimum and maximum travel between the grips. Thus, no adjustment shall be made for any changes in zero and maximum strain caused by development of set in the test piece.

Periodically stop the machine with the test piece held at the maximum elongation and examine for the development of cracking by means of a lens of magnification between 5 and 10, the test pieces being illuminated at the time of examination by a suitably arranged light source. Either the lens may be mounted in a window in the chamber wall, or the test pieces may be removed in their clamps from the chamber for a short period. The test pieces shall not be handled or bumped when carrying out the examination.

Cracking on surfaces which have been cut or buffed shall be ignored.

There are essentially two permissible types of dynamic exposure — continuous and intermittent. In the first type, the test pieces are continuously cycled between zero and maximum strain whilst, in the second type, periods of dynamic cycling are interspersed with periods of static strain exposure.

### 11.2 Continuous dynamic exposure

#### 11.2.1 Choice of procedure

Two alternative procedures for continuous dynamic exposure of test pieces are permissible, as indicated in 11.2.2 and 11.2.3.

#### 11.2.2 Procedure A

Unless otherwise specified, cycle the test pieces between zero and 10 % elongation at 0,5 Hz and examine them after 72 h for the development of cracking (alternative maximum elongation and alternative exposure periods might be given in the material specification).

#### 11.2.3 Procedure B

Cycle the test pieces between zero and one or more of the maximum elongations given in 9.4 at 0,5 Hz. If only one elongation is used, this shall be 10 % unless otherwise specified. Examine the test pieces after 2 h, 4 h, 8 h, 24 h, 48 h, 72 h and 96 h and, if necessary, at suitable intervals thereafter and note the time until the first appearance of cracks at each elongation.

NOTE See Note to 10.3.

### 11.3 Intermittent dynamic exposure

#### 11.3.1 Exposure procedure

Cycle the test pieces between zero and the specified maximum elongation for the specified period. With the test pieces held at the maximum elongation, continue exposure in the static condition in the same ozonized atmosphere. Repeat the sequence of alternate dynamic and static periods as necessary.

Unless otherwise specified, the maximum elongation shall be 10 %. For certain products, intermittent dynamic exposure tests can show better correlation with service performance than continuous dynamic exposure tests. The timing of the dynamic and static exposure periods shall be as given in the product specification.

Two alternative procedures for evaluation are permissible, as indicated in 11.3.2 and 11.3.3.

### 11.3.2 Procedure A

Examine the test pieces at the end of the specified number of dynamic and static exposure periods. Note the presence or absence of cracks.

### 11.3.3 Procedure B

Examine the test pieces at the end of each pair of dynamic and static exposure periods and, if necessary, at suitable intervals during the exposure periods. Note the total time until the first appearance of cracks.

## 12 Expression of results

### 12.1 Procedure A

Report the results as “no cracking” or “cracking”. If cracking has occurred and an estimate of the degree of cracking is required, a description of the cracks (for example, the appearance of the individual cracks, the number of cracks per unit area and the average length of the 10 largest cracks) may be given, or a photograph of the cracked test piece may be taken. The description scheme used shall be described in detail in the test report. See also Annexes A and C.

### 12.2 Procedure B

Take the time to the first appearance of cracks as the measure of ozone resistance at the specified strain.

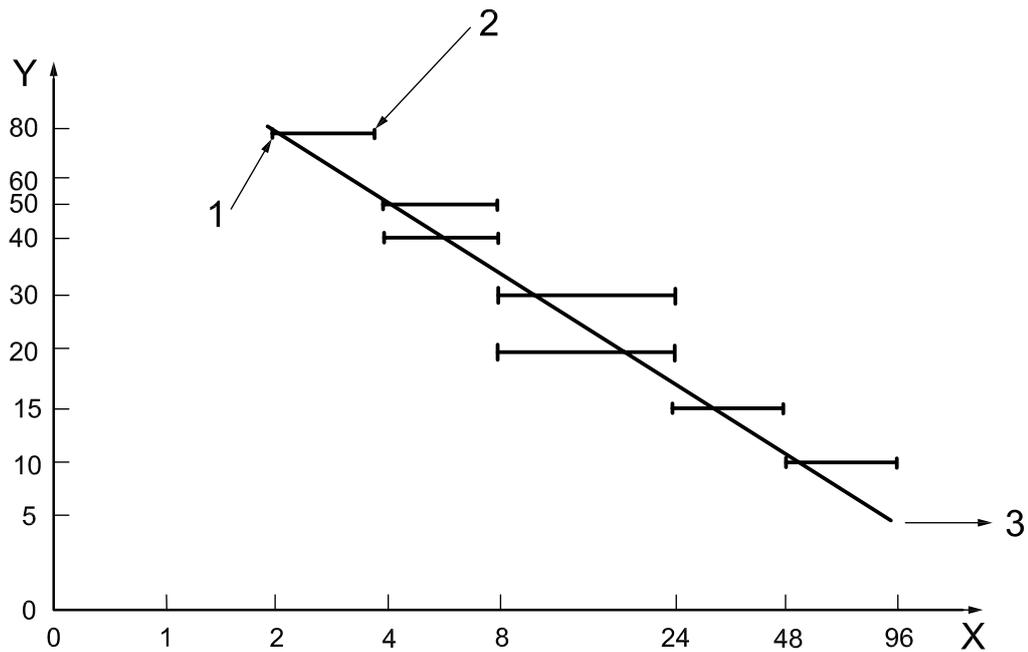
If required, the results of a continuous dynamic exposure test may also be expressed in terms of the number of cycles to the first appearance of cracks.

### 12.3 Procedure C (for static tests only)

Indicate the range within which the threshold strain is found to lie by reporting the highest strain at which cracking was not detected and the lowest strain at which cracking was observed after the specified exposure period. If replicate tests give different results, quote the extreme range observed. For example, if three test pieces were used at each of 10 %, 15 % and 20 % elongation and cracking observed on only one test piece at 10 %, on only one test piece at 15 % but on all three at 20 %, the quoted range shall be 10 % to 20 %. Graphical presentation may be used to assist in interpretation of the results.

A method that has been found useful is to plot the logarithm of the strain against the logarithm of the time to first cracking — both the longest time for which no cracks are seen and the earliest time when cracks are observed may be plotted. Where possible, a smooth curve may be drawn taking into account the gap between the longest time with no cracks and the earliest time with cracks at each strain to assist in the estimation of the threshold strain for any time within the test period (see Figure 4). For some rubbers, the curve can approximate to a straight line but this shall not be assumed since it can lead to large errors in estimating the threshold strain. Unless otherwise specified, report the threshold strain for the longest test period.

NOTE With some rubbers, a linear plot of strain against time to first cracking will enable the existence of a limiting threshold strain to be observed.

**Key**

X time, h (log scale)  
 Y strain, % (log scale)

- 1 last observation with no cracking
- 2 first observation with cracking
- 3 no cracking

NOTE For the example shown, the threshold strain at 48 h = 10 %.

**Figure 4 — Presentation of results in graphical form**

### 13 Test report

The test report shall include the following information:

a) sample details:

- 1) a full description of the sample and its origin,
- 2) compound identification,
- 3) the method of preparation of the test pieces, for example whether moulded or cut;

b) test method:

- 1) a reference to this part of ISO 1431 (ISO 1431-1),
- 2) whether testing was carried out in the static or dynamic test mode,
- 3) if dynamic, the type of exposure (continuous or intermittent),
- 4) the procedure used (A, B or C),
- 5) the type of test piece and its dimensions,

- 6) whether a rotating carrier was used;
- c) test details:
- 1) the ozone concentration and the method of estimation,
  - 2) the temperature of testing,
  - 3) the temperature of conditioning,
  - 4) the humidity, if other than that specified,
  - 5) the ozonized-air flow rate, in cubic metres per second, and the ozonized-air velocity, in metres per second,
  - 6) the maximum strain(s) on the test pieces,
  - 7) the number of test pieces tested at each strain,
  - 8) the duration of the test,
  - 9) for intermittent dynamic exposure only, the duration of the alternate dynamic and static exposure periods,
  - 10) details of any non-standard procedures;
- d) test results:
- 1) for procedure A, whether cracking occurred (if required, the nature of any cracking may also be given),
  - 2) for procedure B, the time to the first appearance of cracks for each elongation or, for continuous dynamic exposure tests, either the time or the number of cycles to the first appearance of cracks,
  - 3) for procedure C (static strain test), the observed range of threshold strains for a suitable exposure period or periods, or the limiting threshold strain;
- e) the date of the test.

## Annex A (informative)

### Ozone cracking — Explanatory notes

#### A.1 Introduction

Cracks develop in rubber only on surfaces subjected to tensile strain. The patterns of cracks, and the severity of cracking, vary depending on the magnitude and nature of the applied strain. The strain on an article in service will vary from a minimum, which need not necessarily be zero, at one point to a maximum at some other point. The pattern of cracks at all elongations in this range should be considered when ozone resistance is being measured.

The first criterion for describing a material as ozone-resistant is total freedom from cracking. Thus, the higher the strain to which the rubber can be subjected for a given exposure period without cracking, or the longer the time before cracks appear on a test piece at a given elongation, the better is the ozone resistance.

However, an alternative criterion might be necessary when cracks below a certain limit of size are permitted on the rubber over a given range of strains. This criterion is based on the concept that one rubber can be described as more ozone-resistant than another if the cracks on it are less severe over the range of elongations encountered in service, which should be specified. The visual nature of the cracks which develop in the test piece should then be reported so that the whole relationship between strain and severity of cracking is determined.

NOTE Crack description schemes are available from various sources, such as DIN 53509-1 [1] and JIS K 6259 [2].

#### A.2 Static strain exposure

The way in which ozone cracking depends on strain is not a simple relationship. The number of cracks on a test piece is related to their size and this relationship depends on the threshold strain for a given exposure period and the elongation of the test piece, for any given material.

Thus no ozone cracking will occur for a given exposure period at strains between zero and the threshold (by definition). A few cracks, which will be large, will be found at strains slightly above the threshold, and the cracks will become more numerous and smaller at progressively higher strains. At very high strains, the cracks can sometimes be so small as to be invisible to the naked eye.

Cracks will coalesce as the exposure increases, particularly when they are very numerous on the surface of the test piece. This will result in the length of some cracks being increased, but without a proportionate increase in depth. Coalescence is probably due to a tearing process as well as ozone attack, and will sometimes result in a number of larger cracks being scattered among the general mass of small, dense cracks which often cover the test piece surface at high strains.

#### A.3 Dynamic strain exposure

Under dynamic strain conditions, a distinction should be made between ozone cracking and the cracking resulting from fatigue failure. Ozone attack is the sole cause of crack initiation at cyclic strains below a characteristic strain known as the mechanical fatigue limit. Once this limit is exceeded, the rate of crack growth increases rapidly and is mainly the result of mechanical fatigue, assisted in many rubbers by the presence of atmospheric oxygen. In this region, the effect of ozone is small and becomes negligible at higher strains. Mechanical fatigue can also occur at low strains once cracks reach a certain size. For these reasons, the ranking order of different rubbers can vary according to the magnitude of the strain, so that the test conditions used should, as far as possible, match those anticipated in service.

## Annex B (normative)

### Calibration schedule

#### B.1 Inspection

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

#### B.2 Schedule

Verification/calibration of the test apparatus is a normative part of this part of ISO 1431. The frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table B.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 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:

- C requirement to be confirmed but no measurement;
- N initial verification only;
- S standard interval as advised in ISO 18899.

Table B.1 — Calibration frequency schedule

Parameter	Requirement	Subclause in ISO 18899: 2004	Verifi- cation frequency guide	Notes
Test chamber	Material does not readily decompose ozone	C	N	E.g. aluminium
	Non-illuminated	C	N	But window and light to view test pieces may be installed
Temperature of test chamber	Constant to $\pm 2$ °C	18	S	
Ozonized air	Obtained by UV lamp or silent-discharge tube	C	N	Largely free from oxides of nitrogen
	Air used passed over activated charcoal	C	N	To be free from contaminants
Temperature of ozone source	Constant to $\pm 2$ °C	18	S	
Relative humidity	Normally < 65 %	20	S	Or 80 % to 90 %
Ozone concentration	As specified in 9.1 Shall not exceed specified concentration and shall return in $\leq 30$ min to specified concentration after test chamber opened	ISO 1431-3	S	
Velocity of ozonized air	Not less than 8 mm/s, preferably 12 mm/s to 16 mm/s, shall not vary by more than $\pm 10$ % in comparative tests	16.2	S	Calculated as in 5.5 If high velocity is required, use 600 mm/s $\pm$ 100 mm/s
Diffusing screen	To assist mixing of gas	C	N	
Clamps to mount test piece parallel to gas flow	Material does not readily decompose ozone	C	N	
Dynamic-testing apparatus	Material does not readily decompose ozone	C	N	
Grips	Arranged such that test piece is reciprocated between zero and a maximum strain as specified in 5.7	C	N	
Frequency	0,5 Hz $\pm$ 0,025 Hz	23.3	S	
Lens	Magnification between $\times 5$ and $\times 10$	C	N	
Materials	Ozone-resistant lacquer may be used to protect ends of test pieces held in grips			

In addition to the items listed in Table B.1, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- timer;
- thermometer for monitoring the conditioning and test temperatures;
- hygrometer for monitoring the conditioning and test humidities;
- instruments for determining dimensions of the test pieces and the imposed strain.

## Annex C (informative)

### Ozone cracking — Rating scales

A rating scale to qualify the degree of cracking observed in an ozone exposure test is sometimes useful. Over the years, several rating scales have been developed, some being fully described in References [3] to [6] of the Bibliography. Generally, these rating scales are based on one or more parameters, such as crack length, crack depth, crack width, crack density, crack localization or crack distribution with respect to size. However, experience has shown that even the most elaborate methods can give poor results in terms of repeatability and reproducibility. Therefore, it is important to bear in mind that the rating operation is very subjective, and it is recommended that the result be considered as additional information, complementing the results of the test, and not as a result in itself. Also, the more complex scales involving measurement of crack dimensions have been found to be excessively time-consuming.

For the sake of simplicity, the following qualitative two-parameter rating scale is suggested:

Crack size:

- 0: no cracks
- 1: cracks only seen under magnification
- 2: cracks visible to the naked eye but very small (less than 0,5 mm)
- 3: anything worse

Crack density:

- S: cracks scarce
- F: relatively few cracks
- N: anything worse

Additional information on crack localization:

- E: cracks on the edge

## Bibliography

- [1] DIN 53509-1, *Prüfung von Kautschuk und Elastomeren — Bestimmung der Beständigkeit gegen Rissbildung unter Ozonwirkung — Teil 1: Statische Beanspruchung*
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- [6] KEMPERMANN, Th., CLAMROTH, R., *Kaut. u. Gummi*, 15, 5, 135 WT, 1962

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