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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

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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 document 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 seventh edition cancels and replaces the sixth edition (ISO 1431-1:2022), which has been technically revised.

The main changes are as follows:

— sealing edges of a test piece has been added in 7.1;

— Annex D has been added.

A list of all parts in the ISO 1431 series can be found on the ISO website.

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.

Introduction

Ozone is generally present in small amounts in the atmosphere. However, even very small amounts of ozone can cause cracking in susceptible rubbers under tensile strain, resulting in loss of strength. Hence, it is necessary to test the resistance of rubbers to exposure to ozone.

Because of the uncertainties of natural exposure, testing for ozone resistance of rubbers is normally done in the laboratory using specially designed ozone cabinets.

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, temperature and relative humidity.[5] 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.

Rubber, vulcanized or thermoplastic — Resistance to ozone cracking —

Part 1:
Static and dynamic strain testing

WARNING 1 — 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.

WARNING 2 — Certain procedures specified in this document can involve the use or generation of substances, or the generation of waste, that can constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies the 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, at a definite temperature and, if required, at a definite relative humidity in circumstances that exclude the effects of direct light.

Either visual observation or image analysis, or both, are used to evaluate the formation and growth of cracks. The changes in physical or chemical properties resulting from exposure can also be determined.

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

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 1382, *Rubber — Vocabulary*

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, *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 terms and definitions given in ISO 1382 and the following apply.

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/

3.1
threshold strain
highest tensile strain at which 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 1 to entry: It is important to distinguish threshold strain from *limiting threshold strain* (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 a selected repetition rate or frequency

Note 1 to entry: 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 specified temperature and, at high or unspecified humidity, to an atmosphere containing a fixed concentration of ozone. The test pieces are examined periodically for cracking.

Three alternative procedures are described for exposure and evaluation of cracking:

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. The presence or absence of cracks is determined by either visual observation or image analysis, or both. If required, an estimate of the degree of cracking is made.

   If required, after the exposure, physical or chemical properties are measured to determine the deterioration of the sample materials by comparing with those of the original pieces.

b) The time until the first appearance of cracks is determined at any given static strain, dynamic strain or combination of dynamic and static strains.

c) The threshold and limiting threshold strain are determined for any given exposure period by either visual observation or image analysis (valid only for static tensile strain tests), or both.

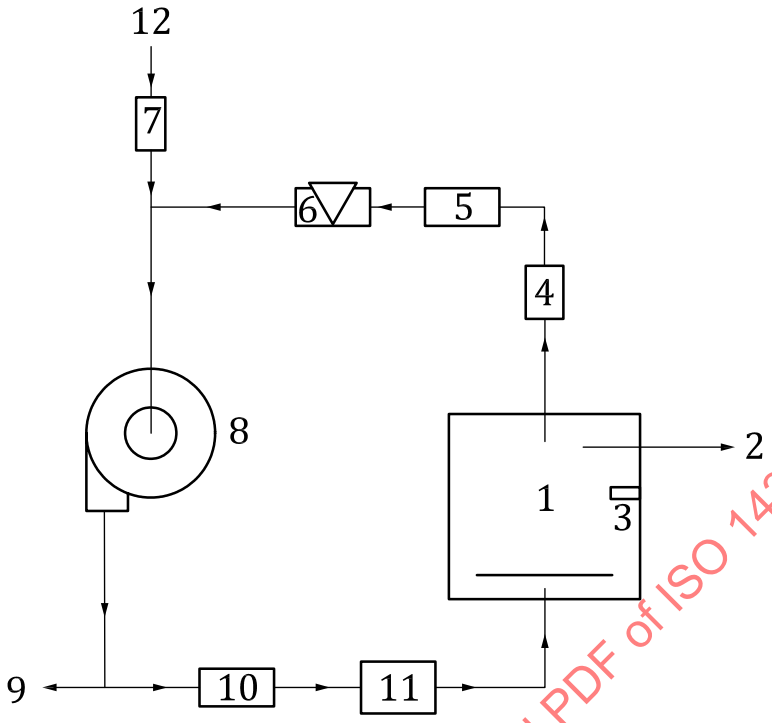
5 Apparatus

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, while 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.

The usual laboratory apparatus and, in particular, the following shall be used.

5.1 Test chamber without humidity control. This shall be a closed, non-illuminated chamber, thermostatically controlled to within ±2 °C of the test temperature, lined with, or constructed of, a material (e.g. aluminium) that does not readily decompose ozone. The dimensions shall be such that the requirements of 5.7 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. See Figure 1.

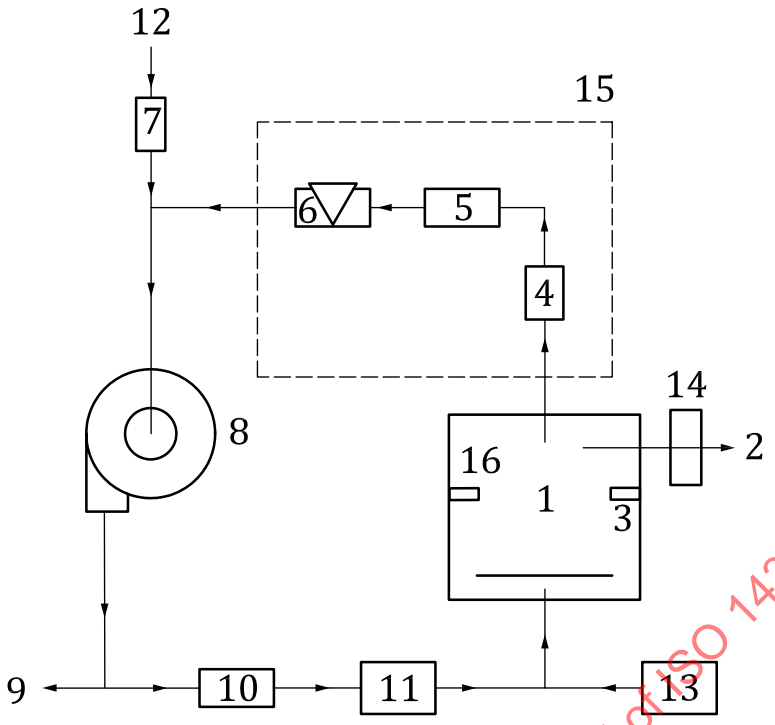


Key

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 without humidity control

5.2 Test chamber with humidity control. This shall be a chamber conforming to the requirements of 5.1, with the addition of being capable of controlling the relative humidity to within ±5 % of test relative humidity. See Figure 2.



Key

1	test chamber	7	air filter	13	humidity controller
2	to ozone concentration measurement device	8	circulation fan	14	dehumidifier for ozone concentration measurement device
3	temperature indicator	9	air outlet	15	arrangement to prevent dew condensation of flowmeter and filter
4	purifying column	10	heat exchanger	16	humidity indicator
5	flowmeter	11	ozonizer		
6	regulator	12	air inlet		

Figure 2 — Example of a test apparatus with humidity control

5.3 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) ultraviolet lamp;

b) silent-discharge tube.

Air used for the generation of ozone or for the dilution of ozonized air shall first be purified by passing it over activated charcoal. The air 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 ± 2 °C via a heat exchanger.

The source via a heat exchanger shall be ozonized and fed into the chamber at the temperature and specified relative humidity (see 9.3) required for the test.

5.4 Means of adjusting the ozone concentration. When an ultraviolet 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 time 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 can be manual or automatic.

5.5 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.

In the case of humidity control, a device that dehumidifies the gas sample and prevents condensation of moisture in the sampling line shall be used in order to accurately measure the ozone concentration.

Referencing and alternative methods of determining the ozone concentration shall be in accordance with ISO 1431-3.

5.6 Means of adjusting the humidity. For apparatus with humidity control (5.2), a humidity indicator for measuring the relative humidity in the test chamber and a humidifier for humidifying the ozone gas introduced into the test chamber shall be used. The humidifier shall be capable of maintaining the specified relative humidity. The gas flow rate measurement device (flowmeter) shall not be influenced by high humidity. Dew condensation at the flowmeter and the purifying column from high humidity shall be prevented.

5.7 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 ±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 from 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 does 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, an 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 ± 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^2 and volumetric flow rate in m^3/s.

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

5.8 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 which does not readily decompose ozone (e.g. aluminium).

A mechanically rotating carrier mounted in the test chamber and upon which the clamps or frames holding the test pieces are mounted should be used 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 (see Figure 3) is at least 40 % of the available cross-sectional area of the chamber.

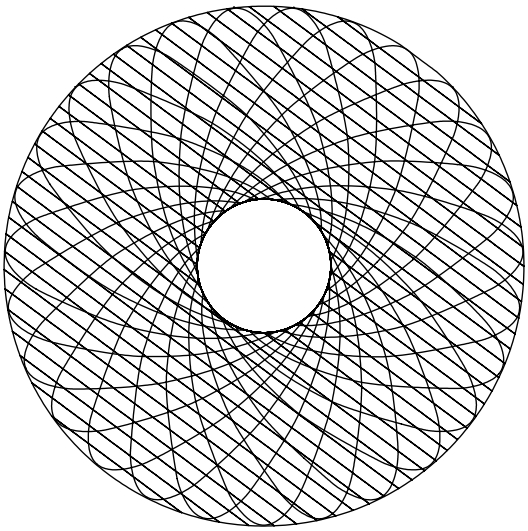


Figure 3 — Path of test pieces and swept area

5.9 Mounting test pieces for dynamic strain testing. The apparatus shall be constructed of a material that does not readily decompose ozone (e.g. aluminium).

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 arranged such 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 ± 0,025) 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.

5.10 Purifying column and filter (key items 7 and 4 in Figure 1 and Figure 2).

5.10.1 Purifying column, for removing ozone gas.

5.10.2 Air filter, for removing undesirable gas in the air introduced into the test chamber.

5.11 Image analysis. If required, a means of analysing the degree of cracking by image analysis.

5.12 Apparatus for measuring properties of the material. If required, apparatus for measuring change in tensile properties in accordance with ISO 37.

6 Calibration

The requirements for calibration of the test apparatus given in Annex B shall be followed.

7 Test pieces

7.1 General

Standard test pieces shall be as specified in 7.2, 7.3 or 7.4.

Test pieces shall be cut from moulded sheet or, if required, from a finished product, in accordance with ISO 23529. Test pieces shall, wherever possible, be cut parallel to the grain of the material unless otherwise specified. Test pieces shall have an undamaged test surface; ozone resistance shall not be assessed on surfaces that have been cut or buffed. Any pattern or flaws on the test piece surface will also tend to act as stress raisers and show preferential cracking. The surfaces of the mould which form the test surfaces should be highly polished.

Test sheets should be moulded between 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.

Evaluation of ozone resistance greatly depends on the surface condition of the test piece, in particular the bloom of the antiozonants or waxes. Where comparisons are to be made, the surfaces should be brought to the same condition as received or cleaned. Do not clean the test pieces with organic materials that will attack or swell the rubber. When the test surfaces are cleaned, the test pieces should be conditioned for a period sufficient to allow the antiozonants or waxes to bloom out again.

To minimize the variation of cracks, especially at the edges of a test piece, it is recommended that the edges of the test piece are sealed with chemical adhesives or paints. The details are described in Annex D.

This kind of pre-treatment shall be agreed between interested parties.

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.

To avoid undesired contamination on the test surface, the operator should not touch the surface of the test pieces.

Avoid simultaneous exposure of different types of composition in the same chamber to prevent the migration of ingredients.

To make an accurate elongation, reference marks shall be marked on the test pieces using a suitable marker and an ink which does not affect the material.

7.2 Wide strip test piece

This test piece shall consist of a strip of not less than 10 mm in width, of thickness (2,0 ± 0,2) 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. Polyacrylate emulsion (see D.3.1) can also be used. 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 strip test piece

This test piece shall consist of a strip of width (2,0 ± 0,2) mm, thickness (2,0 ± 0,2) mm and length 50 mm, between enlarged tab ends 6,5 mm^2 (see Figure 4). This test piece shall not be used for procedure A (10.2).

Dimensions in millimetres

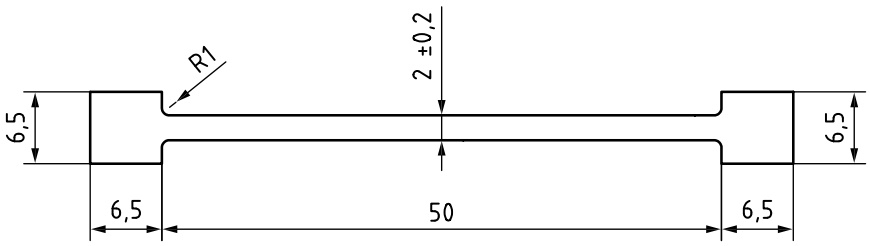


Figure 4 — Narrow strip test piece

7.4 Dumb-bell test piece

Dumb-bell test pieces should be according to ISO 37, but other dumb-bells may be used. Dumb-bells with a wide centre section are preferable for observation of cracking.

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 antiozonants, from migrating by diffusion from one rubber into adjacent rubbers.

Aluminium foil should be placed between test pieces and sheets of different compositions, but other methods which prevent migration of additives can also be used.

During the period between vulcanization and testing, samples and test pieces shall be stored in the dark, in an essentially ozone-free atmosphere. The storage temperature shall normally be a standard laboratory temperature (see ISO 23529), but other environmental conditions (temperature and relative humidity) may be used if appropriate for particular applications. As far as possible the same storage conditions shall also be used 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.

NOTE Some equipment, such as mercury vapour lamps or high-voltage electrical equipment giving rise to electric sparks or silent electrical discharges, is capable of generating ozone.

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

After extending to the required elongation, 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 storage temperature shall normally be a standard laboratory temperature (see ISO 23529), but other environmental conditions (temperature and relative humidity) may be used if appropriate for particular applications. The test piece surface shall not be touched or otherwise disturbed in any way during the conditioning period and subsequent handling. For tests intended to be comparative, the conditions shall be as far as possible 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) or parts per hundred million (pphm) (see NOTE 1):

— (250 ± 50) ppb or (25 ± 5) pphm;

— (500 ± 50) ppb or (50 ± 5) pphm;

— (1 000 ± 100) ppb or (100 ± 10) pphm;

— (2 000 ± 200) ppb or (200 ± 20) pphm.

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

NOTE 1 ppb is used in environmental science for atmospheric pollutants, while pphm is 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 (e.g. 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 ± 2) °C. Other temperatures, such as (30 ± 2) °C or (23 ± 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 ± 2) °C.

For applications where markedly varying temperatures can 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 such that results of resistance to ozone cracking at high relative humidity can differ from those obtained at low humidity. When applicable, for products intended for use in damp climates, the test shall be carried out at a relative humidity above 65 %. For testing at high humidity, unless otherwise specified, the test shall be carried out at one of the following relative humidities:

— (80 ± 5) %;

— (90 ± 5) %.

Other high-humidity conditions may be used by agreement between the interested parties. In this case, it shall be stated in the test report.

The test relative humidity shall be chosen as appropriate for the material being tested and its application. For applications where markedly varying humidity can be encountered, it is recommended that two or more humidity conditions, covering the service range, be used.

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.

9.5 Exposure period

The resistance to ozone cracking will depend upon the type of rubber and the formulation. Also, the test conditions, such as ozone concentration, temperature, humidity and strain, markedly affect formation and growth of cracks. The exposure period shall be selected to obtain a given degree of deterioration of the test pieces.

Trial runs should be made to establish suitable exposure periods.

10 Static strain testing

10.1 General

Adjust the ozone concentration, rate of flow, temperature and, if applicable, relative humidity to the required levels 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 x5 and x10, 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 and evaluation 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). Unless otherwise specified, the procedures for expression of results are in accordance with 12.1.

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. Examination after 16 h can also be desirable, even though it is not convenient in practice. Unless otherwise specified, the procedure for expression of results is in accordance with 12.2.

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. Examination after 16 h can also be desirable, even though it is not convenient in practice. Unless otherwise specified, the procedures for expression of results are in accordance with 12.3.

11 Dynamic strain testing

11.1 General

Adjust the ozone concentration, rate of flow, temperature and, if applicable, relative humidity to the required levels. 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 x5 and x10, 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, while 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 0 % 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 can be given in the material specification). Unless otherwise specified, the procedures for expression of results are in accordance with 12.1.

11.2.3 Procedure B

Cycle the test pieces between 0 % and 10 % 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. Examination after 16 h can also be desirable, even though it is not convenient in practice. Unless otherwise specified, the procedure for expression of results is in accordance with 12.2.

11.3 Intermittent dynamic exposure

11.3.1 Exposure procedure

Cycle the test pieces between 0 % 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. Unless otherwise specified, the procedures for expression of results are in accordance with 12.1.

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. Unless otherwise specified, the procedure for expression of results is in accordance with 12.2.

12 Expression of results

12.1 Procedure A

12.1.1 Procedure A.1: evaluation with visual assessment

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 (e.g. 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 Annex A and Annex C.

12.1.2 Procedure A.2: evaluation with image analysing technique

Use image analysis software to create a binary image from the photograph. A binary image of a photograph taken from a cracked sample will result in cracks with black colour and the matrix with white colour and will give a numerical value to the degree of ozone cracking. The total black area divided by the total area represents the percentage area of cracks and is taken as the degree of cracking.

The number of cracks can also be used to add numerical value to the degree of cracking, but it will not take into account the size of cracks, which is an important aspect in grading ozone cracking. Therefore, the use of percentage area is recommended.

NOTE Software is used to convert a photograph of a cracked sample to a binary black and white image and gives a ratio of the black and white areas. Such software can be found free of charge on the internet.

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

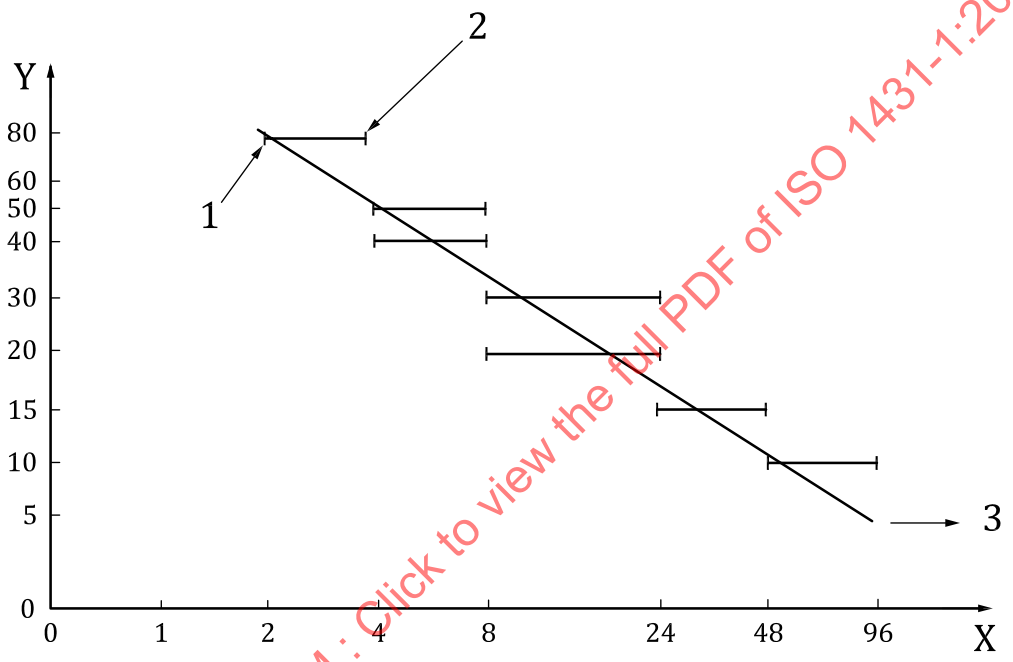
12.3.1 Procedure C.1: evaluation with visual assessment

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 can be plotted. Where possible, a smooth curve can 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 5). 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

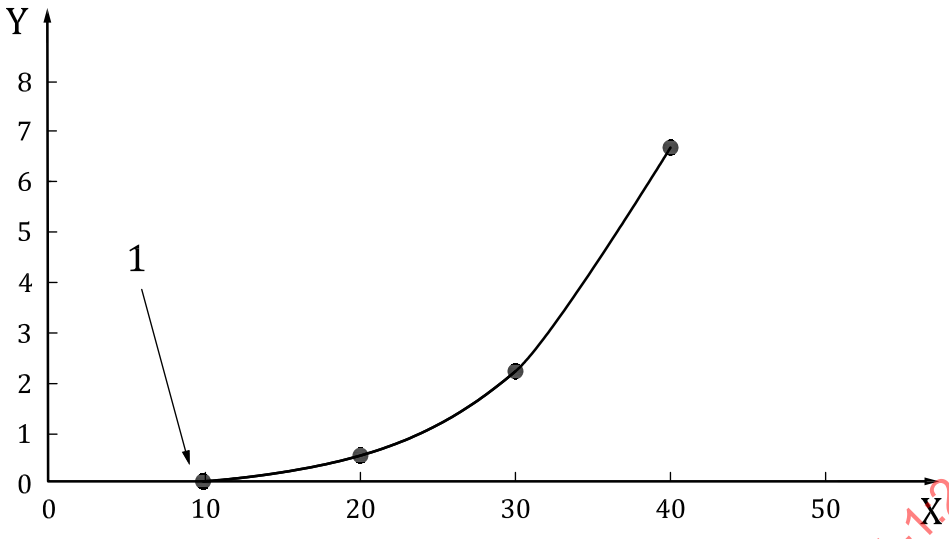
X time, h (log scale)
Y strain, % (log scale)
1 last observation with no cracking
2 first observation with cracking
3 no cracking

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

Figure 5 — Presentation of results in graphical form (visual assessment)

12.3.2 Procedure C.2: evaluation with image analysing technique

For exposures made in accordance with procedure C, plot the percentage crack area against the strain for each condition (temperature and relative humidity) and draw the best-fit curve through the points. The threshold strain is where the curve meets the abscissa (see Figure 6).



Key

X strain, % (linear scale)

Y percentage crack area, % (linear scale)

1 threshold strain

Figure 6 — Presentation of results in graphical form (image analysis)

12.4 Procedure D: evaluation with physical properties change

Express the measurement result as a percentage of the change in each property calculated by Formula (1).

$$R = \frac{T_m - T_0}{T_0} \times 100 \quad (1)$$

where

R is the percentage of the change;

T_0 is the original value (before exposure);

T_m is the value after exposure.

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.

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 document, i.e. ISO 1431-1:2024;

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 laboratory temperature;

2) the ozone concentration and the method of estimation;

3) the test temperature;

4) the temperature and the relative humidity of conditioning;

5) if measured, the test relative humidity;

6) the ozonized-air flow rate, in cubic metres per second, and the ozonized-air velocity, in metres per second;

7) the maximum strain(s) on the test pieces;

8) the number of test pieces tested at each strain;

9) the duration of the test;

10) for intermittent dynamic exposure only, the duration of the alternate dynamic and static exposure periods;

11) 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) and, if measured, the percentage crack area or number of cracks;

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;

4) for procedure D, percentage of the change in each property,

e) any deviations from the procedure;

f) any unusual features observed;

g) the date of the test.

Annex A
(informative)

Ozone cracking — Explanatory notes

A.1 General

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 is not necessarily zero) at one point to a maximum at another 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 the ozone resistance.

However, an alternative criterion can 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 and JIS K 6259-1.

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 several 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 or calibration of the test apparatus is a normative part of this document. 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 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.)

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 18899:2013	Verification frequency guide	Notes
Test chamber	Material does not readily decompose ozone Non-illuminated	C C	N N	e.g. aluminium But window and light to view test pieces can be installed
Temperature of test chamber	Constant to ± 2 °C	18	S	
Ozonized air	Obtained by UV lamp or silent-discharge tube Air used passed over activated charcoal	C C	N N	Largely free from oxides of nitrogen To be free from contaminants
Temperature of ozone source	Constant to ± 2 °C	18	S	
Relative humidity of test chamber	Normally < 65 % or constant to ± 5° %	20	S	
Ozone concentration	As specified in 9.1 Shall not exceed specified concentration and shall return in ≤ 30 min to specified concentration after test chamber opened		S	See ISO 1431-3
Velocity of ozonized air	Not less than 8 mm/s, preferably 12 mm/s to 16 mm/s, shall not vary by more than ± 10 % in comparative tests	16.2	S	Calculated as in 5.7 If high velocity is required, use 600 mm/s ± 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 5.9	C	N	
Frequency	(0,5 ± 0,025) Hz	23.3	S	
Lens	Magnification between x5 and x10	C	N	
Materials	Ozone-resistant lacquer can 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 shall be calibrated 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.