INTERNATIONAL STANDARD

ISO 1431-2

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Rubber, vulcanized or thermoplastic — Resistance to ozone cracking —

Part 2:
Dynamic strain test

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

Partie 2: Essai de déformation dynamique

Reference number
ISO 1431-2:1994(E)

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.

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.

International Standard ISO 1431-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This second edition cancels and replaces the first edition (ISO 1431-2:1982), of which it constitutes a technical revision.

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

— *Part 1: Static strain test*

— *Part 2: Dynamic strain test*

— *Part 3: Reference method for determining the ozone concentration in laboratory test chambers*

Annex A of this part of ISO 1431 is for information only.

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Rubber, vulcanized or thermoplastic — Resistance to ozone cracking —

Part 2:
Dynamic strain test

1 Scope

This part of ISO 1431 specifies a method intended for use in estimating the resistance of vulcanized or thermoplastic rubbers to cracking when exposed, under 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 may be quite different owing to the effects of size and of the type and magnitude of deformation. Explanatory notes on the nature of ozone cracking are given in informative annex A.

Methods for determining resistance to ozone cracking under static strain conditions are specified in ISO 1431-1. A reference method for estimating the ozone concentration will form the subject of ISO 1431-3.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 1431. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 1431 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 471:1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1431-1:1989, *Rubber, vulcanized or thermoplastic — Resistance to ozone cracking — Part 1: Static strain test.*

ISO 4661-1:1993, *Rubber, vulcanized or thermoplastic — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Definition

For the purposes of this part of ISO 1431, the following definition applies.

3.1 dynamic strain: A strain (normally a tensile strain) with a sinusoidal nature varying with time at some selected repetition rate or frequency.

NOTE 1 The maximum strain and the repetition rate are used to describe the dynamic strain conditions.

4 Principle

Test pieces are exposed, either 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.

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

a) Determination of the presence or absence of cracks after exposure for a fixed period of time at a given dynamic strain or combination of dynamic and static strains.

b) Determination of the time to the first appearance of cracks at any given dynamic strain or combination of dynamic and static strains.

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 member body countries, it is recommended that 10 parts of ozone per hundred 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 ± 2 °C of the test temperature, lined with, or constructed of, a material (for example aluminium) that does not readily decompose ozone. 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.

5.2 Source of ozonized air.

Either of the following items of apparatus may be used:

a) an ultra-violet lamp;

b) a silent-discharge tube.

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

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

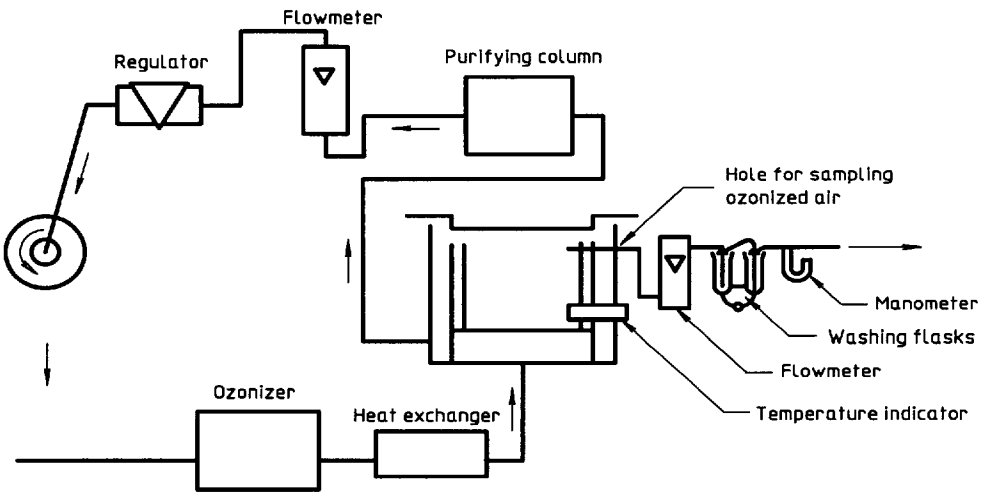

Figure 1 — Schematic diagram of the apparatus

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

5.3 Means for adjusting the concentration of ozone.

This may be, but does not have to be, automatic.

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

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 means of estimating the ozone content shall be provided.

Methods of estimating the ozone concentration will form the subject of ISO 1431-3.

5.5 Means of adjusting the gas flow.

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

If high velocities are desired, a fan may be installed in the chamber to raise the velocity of ozonized air to 600 mm/s $\pm$ 100 mm/s.

NOTES

2 The ratio expressed in s/m is derived from surface area in m^2 and volumetric flow rate in m^3/s.

3 Different results may be obtained if different velocities of ozonized air are used.

5.6 Dynamic testing apparatus.

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

Its essential features are its 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 shall be such that initially the 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 Hz $\pm$ 0,025 Hz. If necessary, a timing device may be provided which stops the apparatus after a period of dynamic exposure and starts it again after the rest period.

The grips shall hold the test pieces firmly, without any slipping or tearing, and shall enable adjustment 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 its length is in the direction of the air flow.

6 Test piece

6.1 General

Standard test pieces shall be strips or dumb-bells as specified in 6.2 or 6.3.

Test pieces shall be cut from freshly moulded sheet or, if required, from a finished product in accordance with ISO 4666-1. 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.

6.2 Strip test piece

The test piece shall consist of a strip of not less than 10 mm width, thickness 2,0 mm ± 0,2 mm, and 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 in selecting a 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 breakage at the grips during ozone exposure.

6.3 Dumb-bell test piece

The test piece shall consist of a strip of 5 mm width, 2,0 mm ± 0,2 mm thickness and 50 mm length, between enlarged tab ends 12 mm square (see figure 2). This test piece shall not be used for procedure A.

NOTES

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

5 It is sometimes impracticable to cut the standard test pieces. In such cases, one form of test piece which may be used is the T 50 dumb-bell with a length of 50 mm and width of 2 mm. When used to detect the onset of cracking, these test pieces have been shown to give approximately equivalent results to the standard test pieces at the same percentage elongations. Dumb-bell test pieces in accordance with ISO 37 may also be used.

7 Conditioning

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

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

For product tests, wherever possible, the time between vulcanization and straining 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 cabinet, be allowed to come into contact with rubbers of a different composition. This is necessary to prevent additives, which may affect the development of ozone cracks, such as antiozonants, 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 any other method which prevents migration of additives may be used.

Dimensions in millimetres

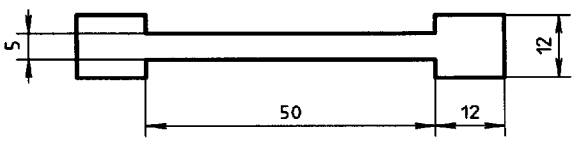

Figure 2 — Dumb-bell test piece

Samples and test pieces shall be stored in the dark, in an essentially ozone-free atmosphere, during the period between vulcanization and testing; the normal storage temperature should be a standard temperature (see ISO 471) but other, controlled, temperatures may be used, if appropriate for particular applications. These storage conditions should also be used, as far as possible, for products. For evaluations intended to be comparable, the storage time and conditions shall be the same.

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

8 Test conditions

8.1 Ozone concentration

The test shall be carried out at one of the following ozone concentrations, expressed in parts of ozone per hundred million of air by volume (pphm):

25 pphm ± 5 pphm

50 pphm ± 5 pphm

100 pphm ± 10 pphm

200 pphm ± 20 pphm

Unless otherwise specified, the test shall be carried out at an ozone concentration of 50 pphm ± 5 pphm. If a lower concentration is required for testing rubbers known to be used under low ambient ozone concentrations, an ozone concentration of 25 pphm ± 5 pphm is recommended. If highly resistant polymers are being tested, a test concentration of 100 pphm ± 10 pphm or 200 pphm ± 20 pphm is recommended.

NOTE 6 It has been found that differences in atmospheric pressure can influence ozone cracking when test pieces are exposed to constant ozone concentrations expressed in parts per hundred million by volume. This effect may be taken into account by expressing the ozone content in the ozonized air in terms of the partial pressure of ozone, i.e. in millipascals, and making comparisons at constant partial pressures of ozone. At standard conditions of atmospheric pressure and temperature (101 kPa, 273 K), a concentration of 1 pphm is equivalent to an ozone partial pressure of 1,01 mPa. Further guidance will be given in ISO 1431-3.

8.2 Temperature

The preferred temperature of test is 40 °C ± 2 °C. Other temperatures, such as 30 °C ± 2 °C or 23 °C ± 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 ± 2 °C.

NOTE 7 For applications where markedly varying temperatures may be encountered, it is recommended that two or more temperatures, covering the service range, be used.

8.3 Relative humidity

The relative humidity of the ozonized air should 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.

8.4 Maximum elongation

Tests shall normally be carried out with the maximum elongation of the dynamic straining cycle at one or more of the following levels:

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

NOTE 8 The elongation(s) used should be similar to those anticipated in service.

9 Test procedure

9.1 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 about × 7, the test pieces being illuminated at the time of examination by a suitably arranged light

source. The lens may either 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.

NOTE 9 Cracking on edges and surfaces which have been cut or buffed should 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.

9.2 Continuous dynamic exposure

Two alternative procedures for exposure of test pieces are permissible.

9.2.1 Procedure A

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 elongations and alternative exposure periods may be given in the material specification).

9.2.2 Procedure B

Cycle the test pieces between zero and one or more of the maximum elongations given in 8.4 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, 16 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 maximum elongation.

NOTE 10 It is sometimes satisfactory to omit examination at 16 h.

9.3 Intermittent dynamic exposure

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 exposure periods as necessary.

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

Two alternative procedures for evaluation are permissible.

9.3.1 Procedure A

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

9.3.2 Procedure B

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

10 Expression of results

10.1 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 appearance of single 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.

10.2 Procedure B

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

11 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) method of preparation of test pieces, for example whether moulded or cut;

b) test method:

1) a reference to this part of ISO 1431,

2) the type of exposure (continuous or intermittent),

3) the procedure used (A or B),

4) the type of test piece and its dimensions;

c) test details:

1) the ozone concentration and the method of estimation,

2) the temperature of test,

3) the temperature of conditioning,

4) the humidity, if other than that specified,

5) the air flow rate, in cubic metres per second, and the air velocity, in metres per second,

6) the maximum strain(s) on the test pieces,

7) the duration of the test,

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

9) any non-standardized procedures;

d) test results:

1) the number of test pieces tested at each strain,

2) for procedure A only, whether cracking occurred (if required, the nature of any cracking may also be given),

3) for procedure B, the times or the number of cycles to the first appearance of cracks;

e) the date of the test.

Annex A
(informative)

Explanatory notes

Cracks develop in rubber only on surfaces subjected to tensile strain. The pattern of cracks, and the severity of cracking, vary according to the magnitude and nature of the applied strain. The strain on an article in service will vary from a minimum at one point, which need not necessarily be zero, to a maximum at some other point. The pattern of cracks at all extensions 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 exposed 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 may be necessary when ozone 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 ozone cracks on it are less severe over the range of extensions encountered in service, which should be specified. The visual nature of the ozone cracks which develop in the test piece should then be reported so that the whole relationship between strain and severity of cracking is determined.

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.

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 increasingly negligible at higher strains. Mechanical fatigue can also occur at low strains once ozone 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.

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