BRITISH STANDARD

BS 903 :
Part A43 : 1990
ISO 1431 - 1 :
1989

Physical testing of rubber

Part A43. Method for determination of resistance to ozone cracking (static strain test)

Essais physique du caoutchouc
Partie A43. Méthode de détermination de la résistance au craquelage par l'ozone (essai sous allongement statique)

Prüfung der mechanischen Eigenschaften von Kautschuk
Teil A43. Prüfung der Ozonrissbeständigkeit (unter statischer Belastung)

BSI STANDARDS

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Rubber Standards Policy Committee (RUM/-) to Technical Committee RUM/36, upon which the following bodies were represented:

BP Chemicals Ltd.
British Railways Board
British Rubber Manufacturers' Association
GAMBICA (BEAMA Ltd.)
Institution of Mechanical Engineers
Institution of Water and Environmental Management
Malaysian Rubber Producers' Research Association
Ministry of Defence
RAPRA Technology Ltd.
SATRA Footwear Technology Centre

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Amendments issued since publication

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National foreword

This British Standard has been prepared under the direction of the Rubber Standards Policy Committee. This revision of Part A43 is identical with ISO 1431-1 : 1989 'Rubber, vulcanized or thermoplastic - Resistance to ozone cracking - Part 1 : Static strain test,' published by the International Organization for Standardization (ISO). This Part of BS 903 supersedes BS 903 : 1982 which is withdrawn.

The generic title of BS 903 has been changed to 'Physical testing of rubber' as many of the methods in the BS 903 : Part A series are suitable for testing thermoplastic as well as vulcanized rubber. The change of title also anticipates the transfer of the methods in the Part B series to a separate standard for the chemical testing of rubber and the omission of the words 'Methods for' anticipates the publication of a Part 0 'Introduction' and a 'Guide to the selection and use of methods of test.' Amendments are not being issued to the other Parts of BS 903, but their titles are being changed when these Parts are revised.

The major changes incorporated in this revision are as follows.

(a) Provision has been made for conducting tests, if required, at a velocity of flow of ozonized air of 600 ± 100 mm/s (see **5.5**).

(b) An alternative dumb-bell test piece has been added (see **6.3**).

(c) An ozone concentration of 100 ± 10 parts of ozone per hundred million parts of air by volume (pphm) has been included as one of the alternative test conditions (see **8.1**).

Warning note on toxicity

Attention is drawn to the warning in clause **5**. The recommendation that 10 pphm be regarded as an absolute maximum concentration in the surrounding atmosphere is in accordance with the value given for long term exposure (8 h TWA, Time Weighted Average, reference period) in Guidance Note EH 40/90 'Occupational exposure limits' published by the Health and Safety Executive, i.e. 0.1 ppm.

NOTE. Guidance Note EH 40/90 expresses occupational exposure limits in parts of ozone per million parts of air by volume (ppm). Units of pphm are used throughout this standard since this is normal practice in the rubber industry.

Attention is also drawn to Guidance Note EH 38/83, 'Ozone health hazards and precautionary measures.'

Cross-references

International standard	Corresponding British Standard
	BS 903 Methods of testing vulcanized rubber
ISO 471 : 1983	Part A35 : 1985 Temperatures, humidities and times for conditioning and testing of test pieces (Identical)
ISO 1431-2 : 1982	Part A44 : 1983 Determination of resistance to ozone cracking (dynamic strain test) (Identical)
ISO 4661-1 : 1986	Part A36 : 1988 Preparation of samples and test pieces (Identical)

It is intended that the international standard for the reference method for estimating the ozone concentration (ISO 1431-3, as referred to in clause **1** and in **5.4**), at present in preparation, will be implemented as a British Standard as BS 903 : Part A45. Until the reference method for estimating the ozone concentration is published, any suitable titrimetric or instrumental method may be used, although it will not be possible to calibrate such methods against the reference method. It is emphasized that details of the method of estimation of ozone concentration should be included in the test report (see item c) 1) of clause **11**).

Additional information. In UK practice, the standard temperature is 23 °C (see clause **7.1**), and is commonly referred to as the 'standard laboratory temperature'.

Rubber, vulcanized or thermoplastic — Resistance to ozone cracking —

Part 1:

Static strain test

1 Scope

This part of ISO 1431 specifies a method for the determination of resistance of vulcanized or thermoplastic rubbers to cracking when exposed, under static 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 according to 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 the type and magnitude of deformation. Explanatory notes on the nature of ozone cracking are given in annex A.

Methods for determining resistance to ozone cracking under dynamic strain conditions and combined dynamic and static strain conditions are specified in ISO 1431-2. 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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 1431-2 : 1982, *Rubber, vulcanized — Resistance to ozone cracking — Part 2: Dynamic strain test.*

ISO 4661-1 : 1986, *Rubber, vulcanized — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Definitions

For the purpose of this part of ISO 1431, the following definitions apply.

3.1 threshold strain: The 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.

Threshold strain must be distinguished from limiting threshold strain, defined in 3.2.

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

4 Principle

Test pieces are exposed under static tensile 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 Determination of the presence or absence of cracks, and if required, an estimate of the degree of cracking, after exposure for a fixed period of time at a given strain.

B Determination of time to the first appearance of cracks at any given strain.

C Determination of the threshold strain for any given exposure period.

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.

NOTE — 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.

5.2 Source of ozonized air

Either of the following 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.

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 light source is used the amount of ozone produced can be controlled by adjusting 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.

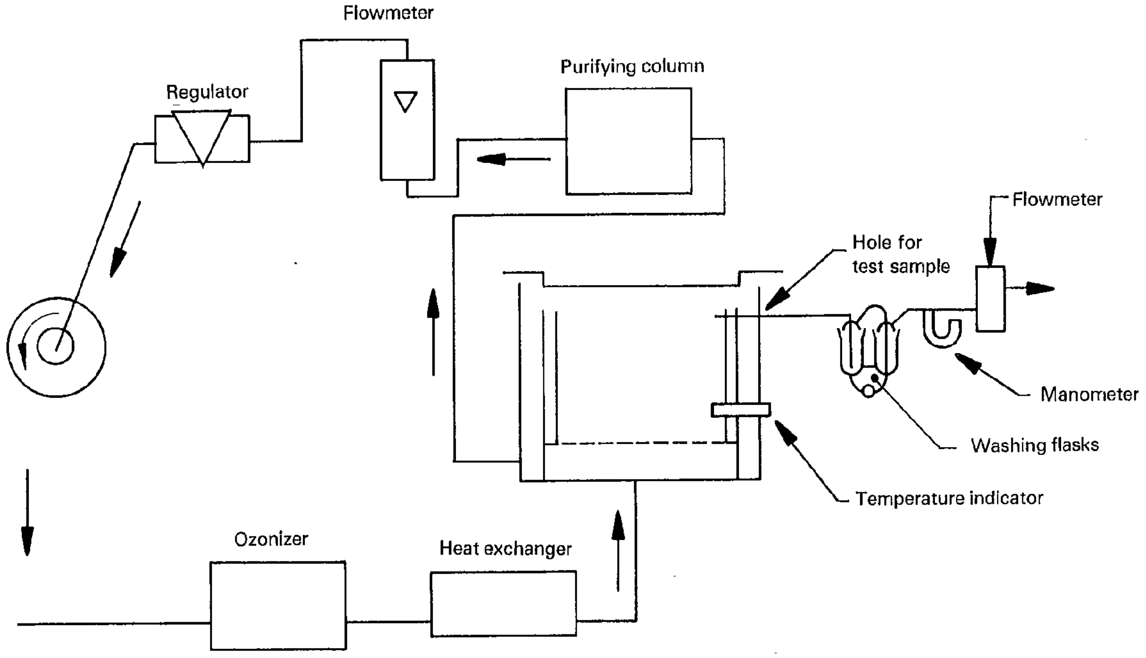

Figure 1 — Schematic diagram of the apparatus

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 its ozone content shall be provided. The methods for estimating the ozone concentration will form the subject of ISO 1431-3.

5.5 Means of adjusting gas flow

A mechanism shall be provided which is capable of adjusting the average velocity of 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 being significantly reduced owing to ozone destruction by the test pieces. The rates of destruction will vary according to 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 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 flow of ozonized air to 600 mm/s ± 100 mm/s.

NOTE — The ratio, expressed in seconds per metre, is derived from surface area in square metres and volumetric flow rate in cubic metres per second.

5.6 Test piece carrier

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

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

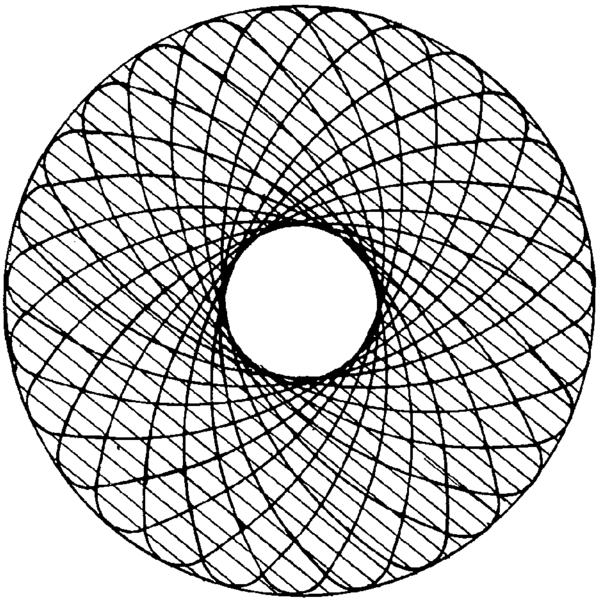

Figure 2 — Path of test piece and swept area (shaded)

6 Test piece

6.1 General

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

Test pieces shall be cut from freshly moulded sheet or, if required, from a finished product in accordance with ISO 4661-1. 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 test condition, 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 that 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 and 50 mm length, between enlarged tab ends 12 mm square (see figure 3). This test piece shall not be used for procedure A.

Dimensions in millimetres

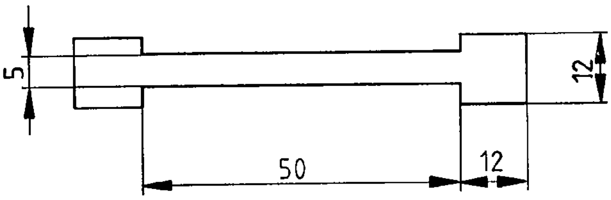

Figure 3 — Dumb-bell test piece

NOTES

1 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.

2 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 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.

3 As an alternative to the exposure of test pieces 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.

4 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 gradation of extensions within one test piece.

7 Conditioning

7.1 Conditioning in the unstrained state

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 can be used.

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

For thermoplastic rubbers, the storage period shall begin immediately after shaping.

7.2 Conditioning in the strained state

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 the standard temperature (see ISO 471), 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 comparable, the conditioning time and temperature shall be the same.

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 — 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. 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 ozone partial pressures. At standard conditions of atmospheric pressure and temperature (101 kPa, 273 K), a concentration of 1 pphm is equivalent to a partial pressure of 1,01 mPa. Further guidance will be given in ISO 1431-3.

8.2 Temperature

The preferred temperature of test shall be 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 — 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 not normally be 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 Elongation

Tests should normally be carried out with test pieces stretched to one or more of the following percentage elongations:

5 ± 1	10 ± 1	15 ± 2
20 ± 2	30 ± 2	40 ± 2
50 ± 2	60 ± 2	80 ± 2

NOTE — The elongation(s) used for procedures A and B 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 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 about X 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 from the chamber for a short period, in their clamps. The test pieces shall not be handled or bumped when carrying out the examination.

NOTE — Cracking on surfaces which have been cut or buffed should be ignored.

Three alternative procedures for exposure of test pieces are permissible.

9.2 Procedure A

Strain the test pieces at 20 % elongation, condition them in accordance with 7.2, 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.

9.3 Procedure B

Strain the test pieces at one or more of the elongations given in 8.4 and condition them in accordance with 7.2. If only one elongation is used, this shall be 20 % unless otherwise specified. Examine the test pieces after 2 h, 4 h, 8 h, 16 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.

9.4 Procedure C

Strain the test pieces at no fewer than four of the elongations given in 8.4 and condition them in accordance with 7.2. 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 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 — For procedures B and C it is sometimes satisfactory to omit examination after 16 h.

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 ozone resistance at the specified strain.

10.3 Procedure C

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 are used at each of 10 %, 15 % and 20 % strains and one cracks at 10 % only, one at 15 % and all three at 20 %, the quoted range should be 10 % to 20 %. Graphical presentation may be used to assist interpretation of the results.

NOTES

1 A method that has been found useful is to plot the logarithm of strain against the logarithm of the time to first cracking — both the longest time at 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 estimation of the threshold strain for any time within the test period

(see figure 4). For some rubbers the curve may approximate to a straight line but this should not be assumed since it can lead to large errors in estimating threshold strain. Unless otherwise specified, the threshold strain at the longest test period should be reported.

2 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.

11 Test report

The test report shall contain 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 procedure used (A, B or C);

   3) the type and dimensions of the test piece;

   4) whether a rotating carrier was used;

c) test details:

   1) the ozone concentration and the method of estimation;

   2) the temperature of the test;

   3) the temperature of conditioning, if other than the standard temperature;

   4) the humidity, if other than specified;

   5) the air flow rate;

   6) the strain(s) on the test pieces;

   7) the duration of the test;

   8) any non-standard procedures;

d) test results:

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

   2) for procedure A only, whether cracking occurred. If required, the nature of cracking may also be given;

   3) for procedure B only, the times to the first appearance of cracks;

   4) for procedure C only, the observed range of threshold strain for a suitable exposure period or periods, or the limiting threshold strain;

e) the date of the test.

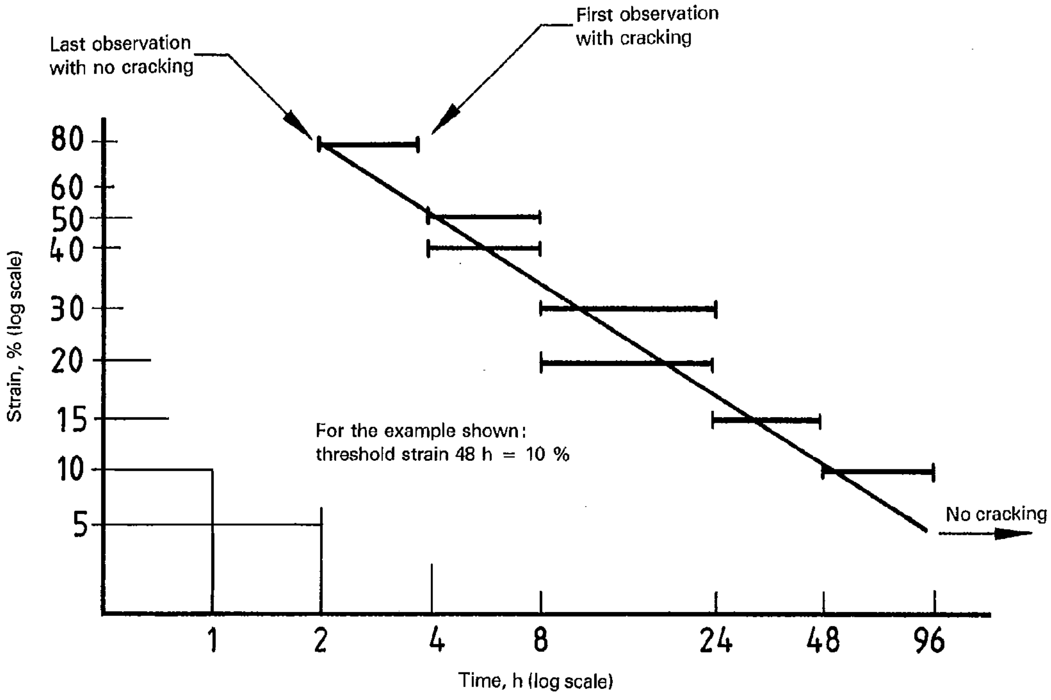

Figure 4 — Results in graphic form

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 threshold strain for a given exposure period or the higher the limiting threshold strain 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.

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 applied to 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 may 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.

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Publications referred to

See national foreword.

BS 903 :
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