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

Partie 1: Essais sous allongement statique et dynamique

thermoplastic — Resistance to

Rubber, vulcanized or

ozone cracking —

ISO/FDIS 1431-1

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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:

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- sealing edges of a test piece has been added in 7.1;
- <u>Annex D</u> 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 <u>www.iso.org/members.html</u>.

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 <u>Annex A</u>.

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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.

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2ht Normative references g/standards/iso/40a4f0f3-70a4-47e9-8387-20a4372083cc/iso-fdis-1431-1

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 <u>https://www.iso.org/obp</u>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

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.



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.



https://standards.it Figure 2 - Example of a test apparatus with humidity control cc/iso-fdis-1431-1

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 <u>9.3</u>) 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 <u>9.1</u>. 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