

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 6259-1 : 2015

(JRMA/JSA)

**Rubber, vulcanized or
thermoplastic—Determination of
ozone resistance—Part 1: Static
and dynamic strain testing**

ICS 83.060

Reference number : **JIS K 6259-1 : 2015 (E)**

PROTECTED BY COPYRIGHT

Date of Establishment: 2015-09-24

Date of Public Notice in Official Gazette: 2015-09-24

Investigated by: Japanese Industrial Standards Committee
Standards Board for ISO area
Technical Committee on Chemical Products and
Analytical Methods

JIS K 6259-1:2015, First English edition published in 2016-08

Translated and published by: Japanese Standards Association
Mita MT Building, 3-13-12, Mita, Minato-ku, Tokyo, 108-0073 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 2016

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

NH/AT

PROTECTED BY COPYRIGHT

Contents

	Page
Introduction	1
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	2
5 Apparatus	4
6 Calibration of test apparatus	8
7 Test pieces	8
7.1 General	8
7.2 Wide test piece	9
7.3 Narrow test piece	9
7.3A Preparation of the test pieces	9
7.3B Measuring the thickness and width of the test pieces	9
7.3C Making method of bench mark for measuring tensile strain	9
8 Conditioning	10
8.1 Conditioning in the unstrained state	10
8.1A Standard laboratory temperature	10
8.1B Storage of test specimens and test pieces	10
8.1C Conditioning of test pieces	10
8.2 Conditioning in the strained state (for static strain testing only)	10
9 Test conditions	10
9.1 Ozone concentration	10
9.2 Temperature of test	11
9.3 Relative humidity	11
9.4 Maximum elongation	11
10 Static strain testing	11
10.1 General	11
10.2 Choice of procedure	12
11 Dynamic strain testing	13
11.1 General	13
11.2 Continuous dynamic exposure	14
11.3 Intermittent dynamic exposure	15
12 Expression of results	16
12.1 Procedure A (Crack condition observation procedure)	16

12.2	Procedure B (Crack appearance time measuring procedure)	16
12.3	Procedure C (Threshold strain and limiting threshold strain measuring procedure)	16
13	Test report	17
Annex A (informative)	Ozone cracking—Explanatory notes	19
Annex B (normative)	Calibration schedule	21
Annex C (informative)	Ozone cracking—Rating scales	23
Annex JA (normative)	Estimate of the degree of cracking	24
Annex JB (informative)	Comparison table between JIS and corresponding International Standard	26

Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment of Japanese Industrial Standard submitted by The Japan Rubber Manufacturers Association (JRMA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

This **JIS** document is protected by the Copyright Law.

Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

JIS K 6259 series consists of the following 2 parts under the general title “*Rubber, vulcanized or thermoplastic—Determination of ozone resistance*”:

Part 1: Static and dynamic strain testing

Part 2: Determination of the ozone concentration

Rubber, vulcanized or thermoplastic— Determination of ozone resistance— Part 1: Static and dynamic strain testing

Introduction

This Japanese Industrial Standard has been prepared based on the fifth edition of **ISO 1431-1** published in 2012 with some modifications of the technical contents.

The portions given sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with the explanations is given in Annex JB.

1 Scope

This Standard specifies the test method for the determination of the ozone resistance of vulcanized or thermoplastic rubber under static and dynamic strain testing.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 1431-1:2012 *Rubber, vulcanized or thermoplastic—Resistance to ozone cracking—Part 1: Static and dynamic strain testing* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

WARNING Persons using this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this Standard to establish appropriate safety and health practices.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. For standards with the year indication, only the editions of the indicated year shall be applied and the revisions (including amendments) made thereafter shall not be applied. For those without the indication of the year, the most recent edition (including amendments) shall be applied.

JIS K 6200 *Rubber—Vocabulary*

JIS K 6250 *Rubber—General procedures for preparing and conditioning test pieces for physical test methods*

NOTE : Corresponding international Standard: ISO 23529 *Rubber—General procedures for preparing and conditioning test pieces for physical test methods* (MOD)

JIS K 6251 *Rubber, vulcanized or thermoplastics—Determination of tensile stress-strain properties*

NOTE : Corresponding international Standard: ISO 37 *Rubber, vulcanized or thermoplastic—Determination of tensile stress-strain properties* (MOD)

JIS K 6259-2 *Rubber, vulcanized or thermoplastic—Determination of ozone resistance—Part 2: Determination of the ozone concentration*

NOTE : Corresponding international Standard: 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* (MOD)

ISO 18899:2004 *Rubber—Guide to the calibration of test equipment*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 6200**, and the following apply.

3.1 threshold strain

the maximum tensile strain at which a vulcanized or thermoplastic rubber can be exposed to an atmosphere containing a fixed concentration of ozone without ozone cracks developing on it after a given exposure period

3.2 limiting threshold strain

tensile strain below which the time required for the development of ozone cracks increases very markedly and can become virtually infinite

3.3 dynamic strain

strain (normally a tensile strain) varying sinusoidally with time at some selected repetition rate or frequency

NOTE : The maximum strain and the frequency are used to describe the dynamic strain.

4 Principle

It is said that the degradation of rubbers is produced by the combination of heat, light, mechanical operation and chemical influence due to oxygen, ozone, chemicals. This Standard specifies the following three evaluation procedures, two test methods combining the presence or absence of the degradation by ozone and mechanical operation, and one method to evaluate the test results.

a) Test methods

- 1) **Static strain testing** The test piece is examined periodically for cracking after exposure for a fixed period of time at a given static tensile strain in a closed chamber at a constant temperature, to an atmosphere containing a fixed concentration of ozone, and the ozone resistance is determined.
- 2) **Dynamic strain testing** The dynamic strain testing includes two methods as follows.

- 2.1) The test piece is examined periodically for cracking after exposure for a fixed period of time at a dynamic tensile strain to give the reciprocating motion to the test piece at a given tensile strain, in a closed chamber at a constant temperature, to an atmosphere containing a fixed concentration of ozone, and the ozone resistance is determined.
- 2.2) The test piece is examined periodically for cracking after exposure for an alternate periods of combination of dynamic and static tensile strain, and the ozone resistance is determined.

b) **Evaluation procedures**

- 1) **Procedure A (Crack condition observation procedure)** The ozone resistance is evaluated by the presence or absence of cracks determined after exposure under a condition of static or dynamic strain testing. If required, an estimate of the degree of cracking is made.
- 2) **Procedure B (Crack appearance time measuring procedure)** The ozone resistance is evaluated by the time to the first appearance of cracks determined after exposure under a condition of static or dynamic strain testing.
- 3) **Procedure C (Threshold strain and limiting threshold strain measuring procedure)** The ozone resistance is evaluated by the threshold strain or the exposure period determined by the limiting threshold strain for static strain testing.

Moreover, the classification of tests and the evaluation procedures are given in Table 1.

Table 1 Classification of test methods and evaluation procedures

Classification of test methods and evaluation procedures			Strain at the exposure	Exposure period
Static strain testing	Procedure A (Crack condition observation procedure)		20 % or arbitrary	72 h or arbitrary
	Procedure B (Crack appearance time measuring procedure)		20 % or arbitrary	Examine periodically (2, 4, 8, 24, 48, 72 and 96 h. Arbitrary after 96 h.)
	Procedure C (Threshold strain and limiting threshold strain measuring procedure)		Select not less than four elongations from 9.4.	Examine periodically at each elongation (2, 4, 8, 24, 48, 72 and 96 h. Arbitrary after 96 h.)
Dynamic strain testing	Continuous method (The dynamic strain is repeated)	Procedure A (Crack condition observation procedure)	0.5 Hz, 10 %	72 h
		Procedure B (Crack appearance time measuring procedure)	0.5 Hz, 10 % or arbitrary	Examine periodically (2, 4, 8, 24, 48, 72 and 96 h. Arbitrary after 96 h.)
	Continuous method (The dynamic and static strains are alternately repeated)	Procedure A (Crack condition observation procedure)	0.5 Hz, 10 % or arbitrary	Arbitrary (Agreed between the parties concerned)
		Procedure B (Crack appearance time measuring procedure)	0.5 Hz, 10 % or arbitrary	Arbitrary (Agreed between the parties concerned)

Great caution is necessary in attempting to relate standard test results to service performance since the relative ozone resistance of different rubbers can vary markedly depending on the conditions, especially ozone concentration and temperature. In addition, tests are carried out on thin test pieces deformed in tension and the significance of attack for articles in service can be quite different owing to the effects of size and of the type and magnitude of the deformation. Explanatory notes on the nature of ozone cracking are given in Annex A.

Reference and alternative methods for determining the ozone concentration are described in **JIS K 6259-2**.

5 Apparatus

The test apparatus consists of a test chamber, heat exchanger, ozone concentration controlling device, regulator, test piece mounting device (for the static strain testing) and dynamic testing apparatus (for the dynamic strain testing), and those shall be as follows respectively.

The example of a test apparatus is shown in Figure 1.

WARNING Attention is drawn to the highly toxic nature of ozone. Efforts shall be made to minimize the ozone exposure of operators of this test. To ensure the safety of human body against the flue gas from the test chamber, the maximum ozone concentration shall be not more than 0.1 ppm. Unless a totally enclosed system is being used, an exhaust vent to remove ozone-laden air is advised.

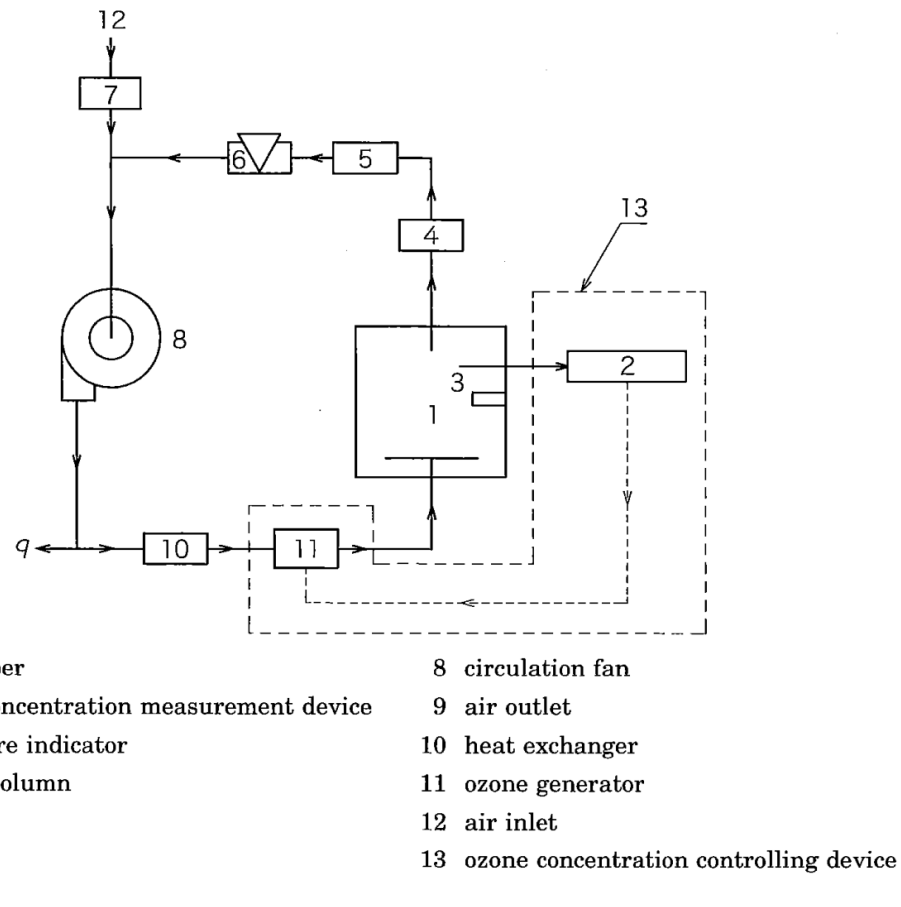


Figure 1 Example of a test apparatus

- a) **Test chamber** This shall be a closed, non-illuminated chamber, thermostatically controlled to within $\pm 2^\circ\text{C}$ of the test temperature, lined with, or constructed of, a material (for example stainless steel, aluminium) that does not readily decompose ozone. The dimensions shall be such that the requirements of **d)** 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.
- b) **Heat exchanger** The air introduced from the outside of chamber (12 in Figure 1) is passed through the cleaning filter (7 in Figure 1) removing contaminant by activated carbon or according to other appropriate method is adjusted to the test temperature by heat exchanger before it is supplied to an ozone generator (11 in Figure 1). The heat exchanger (10 in Figure 1) shall be capable of controlling to within $\pm 2^\circ\text{C}$ and become the relative humidity specified in **9.3**.
- c) **Ozone concentration controlling device**
- 1) **Summary** The ozone concentration controlling device (13 in Figure 1) shall consist of the ozone generator and ozone concentration measuring device and be capable of controlling automatically the ozone concentration. It shall be capable of maintaining the ozone concentration specified while the test piece is present in test chamber. And the air containing the ozone of higher concentration than the test concentration shall not be put in the test chamber.

- 2) **The ozone generator** The ozone generator includes the following two items of apparatus and one of them may be used.

- ultra-violet lamp to generate ozone
- silent-discharge tube

When an ultra-violet lamp is used, the ozone concentration can be controlled by adjusting either the voltage applied to the tube or the input-gas or diluent-air flow rate, or by shielding part of the tube from the UV light. When a silent-discharge tube is used, the ozone concentration can be controlled by adjusting the voltage applied to the generator, the dimensions of the electrodes, or the oxygen or diluent-air flow rate. Two-stage dilution of the ozonized air may also be used.

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 ozonized air shall be largely free of nitrogen oxides in order to avoid errors in the ozone concentration.

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.

- 3) **Ozone concentration measurement device** A means of sampling the ozonized air from the vicinity of the test pieces in the chamber and a means of estimating the ozone content shall be provided. Reference and alternative methods for determining the ozone concentration are described in **JIS K 6259-2**.

- d) **Regulator** A mechanism shall be provided that is capable of adjusting the average velocity of the flow of ozonized air in the test chamber by discharging a part of the gas from the chamber and circulating it, 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 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 ratio¹⁾ of the exposed surface area of the test pieces to the gas flow rate not exceed 12 s/m². 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 should be used to assist thorough mixing of incoming gas with that in the chamber.

If high velocities are desired³⁾, a fan may be installed in the chamber to raise the velocity of the ozonized air to 600 mm/s $\pm$ 100 mm/s.

Notes ¹⁾ The ratio, expressed in seconds per metre (s/m), is derived from surface area in (m²) and volumetric flow rate in (m³/s).