



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 6257 : 2017

(JRMA/JSA)

**Rubber, vulcanized or
thermoplastic—Determination of
heat ageing properties**

ICS 83.060

Reference number : JIS K 6257 : 2017 (E)

PROTECTED BY COPYRIGHT



Documents

Document Sharing Hub

24 S

Date of Establishment: 1993-02-01

Date of Revision: 2017-10-20

Date of Public Notice in Official Gazette: 2017-10-20

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

JIS K 6257:2017, First English edition published in 2018-02

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 2018

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

PROTECTED BY COPYRIGHT

Contents

	Page
Introduction	1
1 Scope	1
2 Normative references	1
2A Terms and definitions	2
3 Principle	2
3.1 General	2
3.2 Test type	3
3.3 Test methods	3
4 Apparatus	4
4.1 General	4
4.2 Forced circulation type heat ageing test machine (lateral wind type)	5
4.3 Forced circulation type heat ageing test machine (vertical wind type)	6
4.4 Cell type heat ageing test machine	7
4.5 Natural ventilation type heat ageing test machine	8
5 Calibration of apparatus	9
6 Test pieces	9
7 Storage of samples and test pieces	10
7A Notes on heat ageing test	10
8 Test conditions (test duration, test temperature and air speed)	11
8.1 General	11
8.2 Accelerated ageing test	11
8.3 Heat resistance test	11
9 Procedure	11
10 Expression of test results	12
11 Precision	12
12 Test report	12
Annex A (normative) Measuring method for air exchange rate, air speed and temperature in the forced circulation type heat ageing test machine	14
Annex B (informative) Precision of forced circulation type heat ageing test machine of vertical wind type and lateral wind type	20
Annex C (informative) Precision of Method A and Method B	26

Annex D (normative)	Calibration of apparatus	30
Annex JA (informative)	General requirements for accelerated ageing test and heat resistance test	33
Annex JB (informative)	Determination of heat ageing properties using test tube type heat ageing test machine	35
Annex JC (informative)	Comparison table between JIS and corresponding International Standard	38

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision 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 applicable to the case of revision by the provision of Article 14. Consequently **JIS K 6257**:2010 is replaced with this Standard.

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.

Rubber, vulcanized or thermoplastic— Determination of heat ageing properties

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 188:2011**, Edition 5, with some modifications of the technical contents.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JC.

1 Scope

This Standard specifies the method for determining the heat ageing properties of vulcanized rubber and thermoplastic rubber according to the accelerated ageing test and/or the heat resistance test. The test methods include the following Method A and Method B.

- a) **Method A**, using the forced circulation type heat ageing test machine provided with an air blower
- b) **Method B**, using the cell type heat ageing test machine or the natural ventilation type heat ageing test machine in which the air in test oven is ventilated slowly by convection

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

ISO 188:2011 *Rubber, vulcanized or thermoplastic—Accelerated ageing and heat resistance tests* (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 to establish appropriate safety and health practices.

2 Normative references

The following standard contains provisions which, through reference in this text, constitute provisions of this Standard. The most recent edition of the standard (including amendments) indicated below shall be applied.

JIS C 1602 *Thermocouples*

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 thermoplastic—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 6253-2 *Rubber, vulcanized or thermoplastic—Determination of hardness—Part 2: IRHD method (hardness between 10 IRHD and 100 IRHD)*

NOTE : Corresponding International Standard: ISO 48 *Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)* (MOD)

JIS K 6253-3 *Rubber, vulcanized or thermoplastic—Determination of hardness—Part 3: Durometer method*

JIS T 8202 *Anemometer for general use*

JIS Z 8401 *Guide to the rounding of numbers*

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

2A Terms and definitions

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

3 Principle

3.1 General

The vulcanized or thermoplastic rubber is subjected to controlled deteriorating influences under atmospheric pressure at a specified temperature for a definite period, after which appropriate physical properties are measured and compared with the corresponding properties of the unaged rubber to determine the heat ageing properties of the rubber.

The physical properties to be used in the assessment of heat ageing properties should be determined according to the application of the vulcanized rubber and the thermoplastic rubber of concern. If no specification is made, the tensile strength, elongation at break and tensile stress measured in accordance with **JIS K 6251**, and the hardness, etc. measured in accordance with **JIS K 6253-2** or **JIS K 6253-3** shall be used as a basis for evaluation.

Tests applied for this assessment are classified into the accelerated ageing test and the heat resistance test. Applicable test methods include Method A which uses the forced circulation type heat ageing test machine and Method B which uses the cell type heat ageing test machine or the natural ventilation type heat ageing test machine.

3.2 Test type

3.2.1 Accelerated ageing test, At method

In this method, the test pieces are subjected to a higher temperatures than the rubber would experience in service in order to produce the effects of natural ageing in a shorter time.

3.2.2 Heat resistance test, Hr method

In this test method, the test pieces are subjected to the same temperature as they would experience in service.

3.3 Test methods

Test methods used are classified into Method A and Method B according to the test machine to be used.

Ageing tests of vulcanized rubber and thermoplastic rubber are largely divided into the following two methods based on the expected service condition.

- a) Method where ageing is achieved through forced air ventilation at a high air speed (Method A)
- b) Method where ageing is achieved through air ventilation at a low air speed (Method B)

The influence of heat is commonly relevant to Method A and Method B; the difference between them lies basically in how likely the vulcanized rubber or the thermoplastic rubber is to be in contact with air, and this is controlled through changing the moving speed of the air inside the test chamber.

For selection of the applicable test type and test method, Table 1 shall be referred to.

Table 1 Test types and test methods

Test type	Test method		
	Code designating the classification of test method		Test machine used
Accelerated ageing test (At method) (higher temperature than experienced in service)	Method A	AtA-1	Forced circulation type heat ageing test machine (lateral wind type) (also called Geer type ageing test machine)
		AtA-2	Forced circulation type heat ageing test machine (vertical wind type)
	Method B	AtB-3	Cell type heat ageing test machine
		AtB-4	Natural ventilation type heat ageing test machine
Heat resistance test (Hr method) (same temperatures as experienced in service)	Method A	HrA-1	Forced circulation type heat ageing test machine (lateral wind type) (also called Geer type ageing test machine)
		HrA-2	Forced circulation type heat ageing test machine (vertical wind type)
	Method B	HrB-3	Cell type heat ageing test machine
		HrB-4	Natural ventilation type heat ageing test machine
NOTE 1 In the accelerated ageing test or the heat resistance test, the results differ between Method A and Method B.			
NOTE 2 For the notes on the accelerated ageing test and the heat resistance test, see Annex JA.			
NOTE 3 For the test machine of Method B, the test tube type heat ageing test machine is also applicable (see Annex JB).			
NOTE 4 In the test method codes given above, At or Hr represents test type, A or B, test method, and the numeral at the end, the type of test machine used.			

4 Apparatus

4.1 General

The testing apparatus shall consist of a test oven, heating element to heat the air in oven, automatic temperature controller, etc. The oven shall be of such a size that the total volume of the test pieces does not exceed 10 % of the internal volume of the oven.

Test oven shall be provided with a test piece carrier at its upper part which is capable of suspending the test pieces so that they are at least 10 mm from each other, and in forced circulation type heat ageing test machine or natural ventilation type heat ageing test machine, at least 50 mm from the sides of the test oven.

The temperature of the test oven shall be controlled automatically so as to be kept within the specified tolerance for the specified ageing temperature in 8.1. A temperature sensor shall be placed inside the test oven to record the actual ageing temperature.

Care shall be taken to ensure that the incoming air into the test oven is heated to within ± 1 °C of the specified temperature before coming in contact with the test pieces.

No copper or copper alloys shall be used for the test oven.

The air in the oven shall be replaced with the air outside by the air exchange rate of 3 changes/h to 10 changes/h. The air exchange rate shall be determined by measuring the volume of the test oven and the flow rate of air inside the test oven.

The measuring method of air exchange rate and the temperature in the forced circulation type heat ageing test machine shall be in accordance with Annex A.

NOTE : To ensure good precision when carrying out the accelerated ageing test and the heat resistance test, it is important to keep the temperature constant and stable during the test and to verify that the test oven used is within the temperature permissible limits with regard to time fluctuation. Increasing the air speed in the oven improves temperature homogeneity; however, air circulation in the test oven and ventilation influence the ageing results. With a low air speed, such as that of natural ventilation, accumulation of degradation products and evaporated ingredients may cause the oxygen depletion. A high air speed increases the rate of deterioration, due to an increased oxidation and volatilization of plasticizers and antioxidants.

4.2 Forced circulation type heat ageing test machine (lateral wind type)

In the forced circulation type heat ageing test machine (lateral wind type), a test piece carrier which rotates around the vertical axis shall be provided. The rotating speed of the carrier should be five to ten rotations per minute. The air in the test oven shall be forcibly circulated, at an average air speed of $0.5 \text{ m/s} \pm 0.1 \text{ m/s}$. The measuring method of air speed shall be in accordance with Annex A.

NOTE : This test apparatus makes turbulent air flow in the horizontal direction.

An example of the test oven of the forced circulation type heat ageing test machine (lateral wind type) is shown in Figure 1.

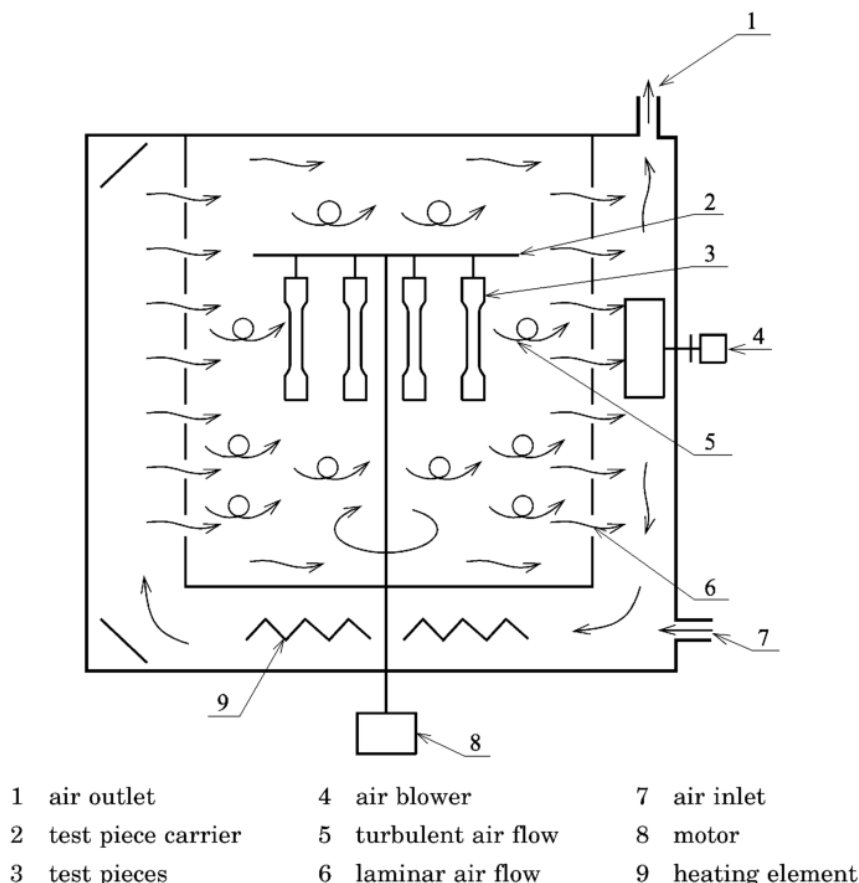


Figure 1 Example of test oven of forced circulation type heat ageing test machine (lateral wind type) with conceptual airflow diagram

4.3 Forced circulation type heat ageing test machine (vertical wind type)

In the forced circulation type heat ageing test machine (vertical wind type), the air in the test oven is forcibly circulated at an average air speed of 0.5 m/s to 1.5 m/s. The air speed shall be measured by means of an anemometer close to the test piece. The measuring method of air speed shall be in accordance with Annex A.

NOTE : This test apparatus makes laminar air flow in the vertical direction.

An example of the test oven of the forced circulation type heat ageing test machine (vertical wind type) is shown in Figure 2.

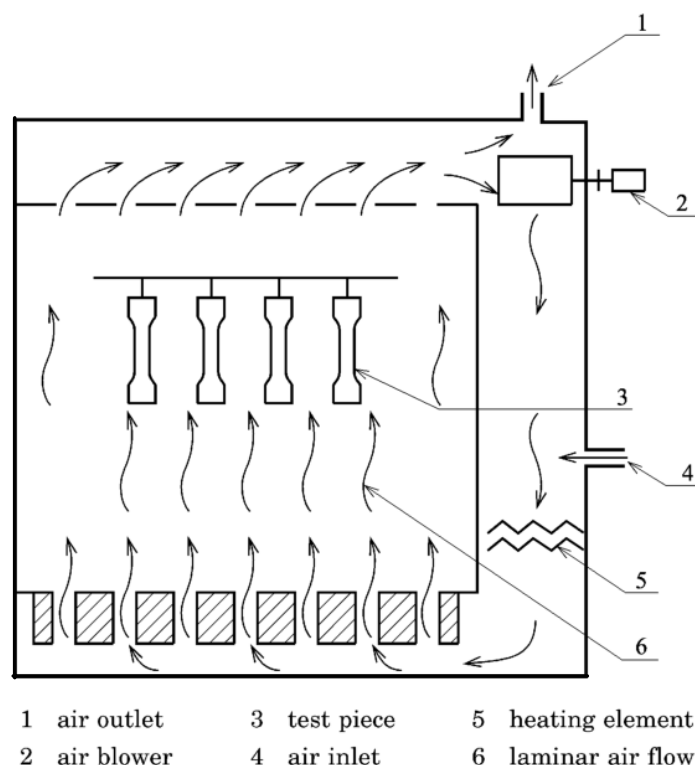


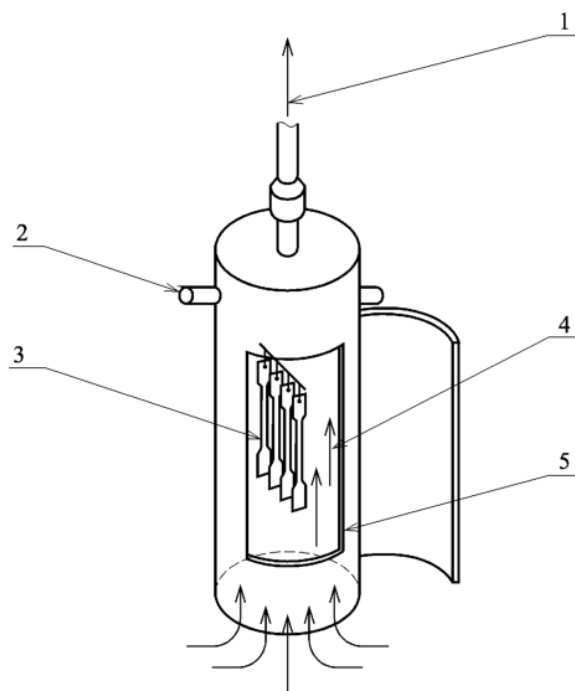
Figure 2 Example of test oven of forced circulation type heat ageing test machine (vertical wind type) with conceptual airflow diagram

4.4 Cell type heat ageing test machine

The cell type heat ageing test machine shall consist of one or more vertical cylindrical cells having a minimum height of 300 mm and the heating element.

The cells shall be surrounded by good heat transfer medium (aluminium block, liquid bath or saturated vapour), and shall be thermostatically controlled to the temperature range specified in 8.1 by the heating element. Air passing through one cell shall not enter other cells. Provision shall be made for a slow flow of air at an air speed under 0.4 m/s through the cell. The air speed shall be determined so that the air exchange rate becomes 3 changes/h to 10 changes/h.

An example of cell is shown in Figure 3.



- | | | |
|--------------------|--------------------|-----------------------------------|
| 1 air outlet | 3 test piece | 5 opening for test piece mounting |
| 2 fixture for cell | 4 laminar air flow | |

Figure 3 Example of cell of cell type heat ageing test machine with conceptual airflow diagram

4.5 Natural ventilation type heat ageing test machine

The natural ventilation type heat ageing test machine shall comprise a test oven without separating walls so that the heated air is taken from the bottom part of the test oven and it is exhausted from the above. No fans are allowed inside the oven. The construction shall allow a slow exchange with outside air. The air speed shall be determined so that the air exchange rate becomes 3 changes/h to 10 changes/h.

An example of natural ventilation type heat ageing test machine is shown in Figure 4.

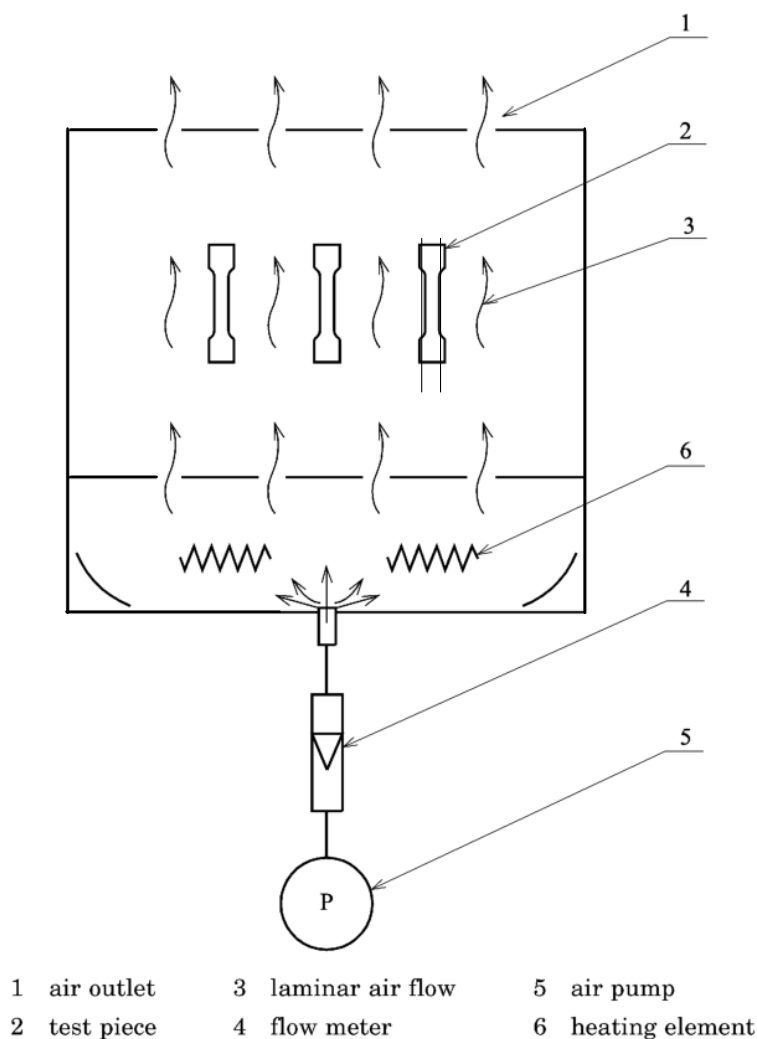


Figure 4 Example of natural ventilation type heat ageing test machine with conceptual airflow diagram

5 Calibration of apparatus

The calibration of testing apparatus shall be in accordance with Annex D.

6 Test pieces

It is recommended that the accelerated ageing test or the heat resistance test be carried out on test pieces prepared and conditioned as required for the relevant physical properties tests, and not on the products or the samples before punching. Also, the test pieces should not be subjected to any mechanical, chemical or heat treatment after ageing. If the preparation method and treatment are specified for the test pieces for the related physical properties tests or the product tests, they shall be observed.

Only test pieces of same dimensions and having been aged under approximately the same conditions shall be compared with each other.

The number of test pieces shall be in accordance with the related standards for the physical property tests. The dimensions of test pieces shall be measured before ageing. Whenever possible, marking shall be carried out after heating as some marking inks can affect the ageing of the rubber.

Care shall be taken to ensure that the markings used to identify the test pieces are not applied in any critical area of the test piece and will not damage the rubber or disappear during testing.

NOTE : For sampling, preparation and dimensional measurement of test pieces, see **JIS K 6250**.

7 Storage of samples and test pieces

Unless otherwise specified for technical reasons, the following requirements shall be observed for the storage of samples and test pieces.

- The storage temperature shall be the standard temperature of the laboratory specified in **JIS K 6250**.
- For all normal test purposes, the minimum duration between vulcanization or moulding and testing shall be 16 h. In cases of arbitration, the minimum duration shall be 72 h.
- For the tests of samples and test pieces, the maximum period between vulcanization or moulding and testing shall be four weeks and, for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.
- For product tests, the period between the production and testing shall not exceed three months. In other cases, tests shall be carried out within two months of the date of receipt of the product.

7A Notes on heat ageing test

Simultaneous ageing of rubber of different types of compound in the same ageing test oven shall be avoided to prevent the migration of accelerator, crosslinking agent, antioxidant, peroxide, plasticizer and the like that may change the heat ageing properties of other samples. To fulfil this purpose, one cell for each type of sample is required. For testing multiple types of samples simultaneously and efficiently, the use of heat ageing test machine with individual cells is highly recommended. In this case, either the types of materials as agreed between the interested parties, or the following types of material should be heated together:

- a) vulcanized rubber and thermoplastic rubber containing popular rubber material of same type;
- b) vulcanized rubber containing the same type of accelerator and approximately the same ratio of sulphur to accelerator;
- c) vulcanized rubber and thermoplastic rubber containing the same type of antioxidant;
- d) vulcanized rubber and thermoplastic rubber containing the same type of plasticizer.

NOTE : When multiple testing apparatuses are used at the same place, care shall be taken so that the air exhausted from one apparatus is not introduced to another apparatus.