

JIS

JAPANESE INDUSTRIAL STANDARD

**Physical testing methods
for unvulcanized rubber**

JIS K 6300—1994

Translated and Published

by

Japanese Standards Association

Printed in Japan

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

1. Scope This Japanese Industrial Standard specifies the physical testing methods for unvulcanized rubber⁽¹⁾.

Note ⁽¹⁾ Unvulcanized rubber referred to here is material rubber and compounded rubber.

Remarks 1. The standards cited in this Standard are as follows.

JIS B 2401 O-rings

JIS G 3522 Piano wires

JIS K 6200 Glossary of terms used in rubber industry

JIS K 6253 Hardness testing methods for vulcanized rubber

JIS K 6299 Rubber test mixes - Preparation, mixing and vulcanization - Equipment and procedures

JIS Z 8401 Rules for rounding off of numerical values

2. The International standards corresponding to this Standard are as follows.

ISO 289: 1985 Rubber, unvulcanized - Determination of Mooney viscosity

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

ISO 667: 1981 Rubber, compounded - Determination of cure rate - Shearing disk method

ISO 1795: 1992 Rubber, raw, natural and synthetic - Sampling and further preparative procedures

ISO 2007: 1991 Rubber, unvulcanized - Determination of plasticity - Rapid-plastimeter method

ISO 2930: 1981 Rubber, raw natural - Determination of plasticity retention index (PRI)

ISO 3417: 1991 Rubber - Measurement of vulcanization characteristics with the oscillating disc curemeter

ISO 6502: 1991 Rubber - Measurement of vulcanization characteristics with rotorless curemeters

3. The units and numerical values in { } in this Standard are based on the traditional units and are standard values. The traditional values shall be only informative reference values on and after April 1st, 1995.

2
K 6300-1994

2. Definitions For the main terms used in this Standard the definitions in JIS K 6200 apply, and the rest of the terms shall be as follows.

- (1) Mooney unit The unit indicated or recorded on the torque indicator which is used in Mooney viscosity test or Mooney scorch test. The value shown when the torque on a shaft of rotor is $8.30 \text{ N}\cdot\text{m}$ { $84.6 \text{ kgf}\cdot\text{cm}$ }, is defined as 100 Mooney units.

The relation between torque and Mooney unit is linear.

3. Test items The test items specified in this Standard shall be as follows.

- (1) Mooney viscosity test
- (2) Mooney scorch test
- (3) Vulcanization test with oscillating curemeter
- (4) Plasticity test with rapid plastimeter
- (5) Test of plasticity retention index for natural rubber

4. General condition for test

4.1 Standard condition of laboratory The standard condition of laboratory shall be $23 \pm 2^\circ\text{C}$, and relative humidity preferably be $(50 \pm 5) \%$.

4.2 Standard condition of sample Principally, sample shall be the one in the period from 1 h to 72 h after kneading or procuring, and be set under the standard condition of 4.1 for not less than 30 min before test.

4.3 Record In a test record, the condition adopted in the test shall be recorded.

5. Preparing method of sample The preparing method of sample for test shall follow the description in JIS K 6299 except the method specified especially in each test item.

6. Mooney viscosity test

6.1 Purpose This test shall be carried out to measure the Mooney viscosity of unvulcanized rubber.

6.2 Test piece

6.2.1 Shape and dimensions of test piece One set of test pieces shall consist of two discs of about 50 mm in diameter and about 6 mm in thickness, sufficient to fill completely the die cavity. One of 2 test pieces shall be pierced with a hole, of about 11 mm in diameter in its center to permit the insertion of rotor shaft.

The test pieces shall be prepared so as not to contain air inside as far as possible, and shall have no pocket by which air might be left on the surface of a die or a rotor.

Remarks: The same sample may give different value on Mooney viscosity depending on its preparation method, therefore, when either Mooney viscosity is compared between the same types of rubber or the Mooney viscosity value of a certain rubber is standardized, the preparing method of rubber sample shall be specified.

6.2.2 Preparing method of sample The kind of sample shall be material rubber and compounded rubber. It is prepared according to the following method or the method which has been agreed between the parties concerned.

- (1) Material rubber Principally, the quantity of material rubber to be sampled shall be 600 g to 1500 g in case of natural rubber and 250 ± 5 g in case of synthetic rubber, and respectively they shall be passed through rollers under the condition shown in Table 1.

The condition to pass through rollers, except the first trial, shall be as by multi-folding method for natural rubber and double layers method for synthetic rubber. Try 9 times of passings respectively, and shape it into sheet type at the last tenth trial. In case of CR synthetic rubber. However, shape it into sheet type with only 2 trials for CR. In this case, dimensions of a roller shall follow JIS K 6299.

Table 1. Sample preparing condition of various rubbers

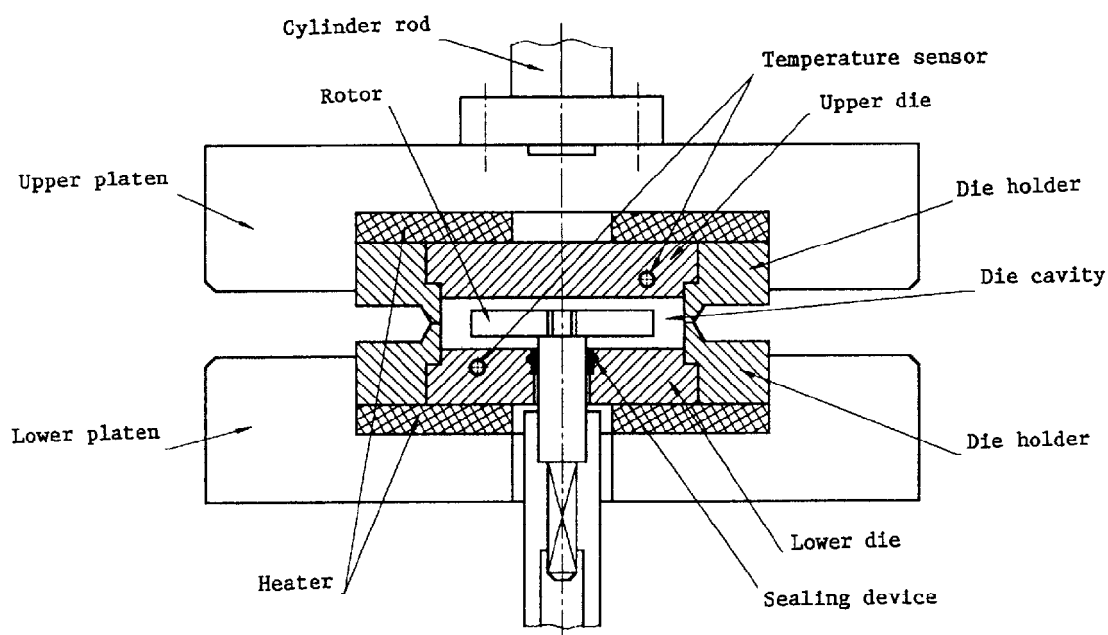
Type of rubber	Surface temperature of roller °C	Gap of rollers mm	Frequency of passing Time
Natural rubber (NR)	70 ± 5	1.3 ± 0.15	10
Synthetic rubber (Other than the below)	50 ± 5	1.4 ± 0.15	10
BR	35 ± 5	1.4 ± 0.15	10
CR	20 ± 5	0.4 ± 0.05	2
EPDM, EPM	35 ± 5	1.4 ± 0.15	10
11R, B11R and C11R ⁽²⁾	To be sampled directly from a bale.	-	-
Black masterbatch	50 ± 5	1.4 ± 0.15	10
Reclaimed rubber ⁽²⁾	To be sampled directly from a bale.	-	-

Note ⁽²⁾ When sampling directly from a bale, cut off sample measuring about 50 mm in diameter and about 6 mm in thickness. In case of crumb rubber, however, follow the condition given in the case of synthetic rubber.

- (2) Compounded rubber In case of compounded rubber, follow JIS K 6299.

6.3 Test apparatus The viscometer for this test (generally called Mooney viscometer) shall consist of; the rotor to measure rubber viscosity which is driven by a motor, the hollow cylinder-type die in which test piece and rotor are put, the device to indicate the torque necessary for rotating rotor, and heating and temperature controlling device to keep the temperature of the die constant (see Fig. 1). Each of them shall satisfy the following conditions.

Fig. 1. Example of main parts of Mooney viscometer



- (1) **Rotor** There are two types of rotor, that is, a large rotor (hereafter referred to as "L type") and a small rotor (hereafter referred to as "S type"), both of them are made of anticorrosive and antiabrasive steel with HRC 60 or harder by Rockwell hardness, and the rotor surface shall be grooved for preventing slippage between it and test piece.

Dimensions of a rotor shall be as shown in Table 2.

The dimensions shall be measured from the top of grooves.

Table 2. Dimensions of rotor

Unit: mm

Type of rotor	Diameter of rotor	Thickness of rotor
L-type rotor	38.10 ± 0.03	5.54 ± 0.03
S-type rotor	30.48 ± 0.03	5.54 ± 0.03

Shape and dimensions of grooves to prevent slippage on the surface of rotor are as follows (see Fig. 2).

- (a) Shape: Square type

- AVA** Documents
Document Sharing Hub

- (2) Dies Dies are made of anticorrosive and antiabrasive steel with HRC 60 or harder by Rockwell hardness, and one set of dies and die holder make a cylinder-type cavity (see Fig. 3). The size of the die cavity shall be, when being measured from its top surface, 50.9 ± 0.1 mm in diameter and 10.59 ± 0.03 mm in depth.

At central position of lower die, provide a hole for inserting the shaft of a rotor. Make the inside diameter of the hole about 0.4 mm larger than the shaft diameter of the rotor.

Use a sealing device for preventing test piece from overflowing into the hole (see Fig. 3).

On the surface which is facing to the die cavity, provide square-shaped grooves or V-shaped grooves for preventing slippage of test piece. The dimensions of grooves and the way for notching them shall be as follows (see Fig. 4).

(2.1) Square-shaped grooves

- (a) Shape: Square shape
- (b) Dimensions: 0.80 ± 0.02 mm in width, 0.30 ± 0.05 mm in depth, and 1.60 ± 0.04 mm spaced for notching.
- (c) Way for notching: Upper and lower surfates shall be notched in parallel crosses intersecting at a right angle, and the outside periphery shall be notched to internal gear shape.

(2.2) V-shaped grooves

- (a) Shape: V-shape
- (b) Dimensions: 1.0 ± 0.1 mm in width, 90° in angle, and 20 mm, from the point at 3.5 mm apart its center toward its circumference, in length.
- (c) Way for notching: 18 V-shape grooves shall be notched radiately at 20° angle intervals directing towards circumference.

Fig. 3. Dies, die holder, and die cavity

Unit: mm

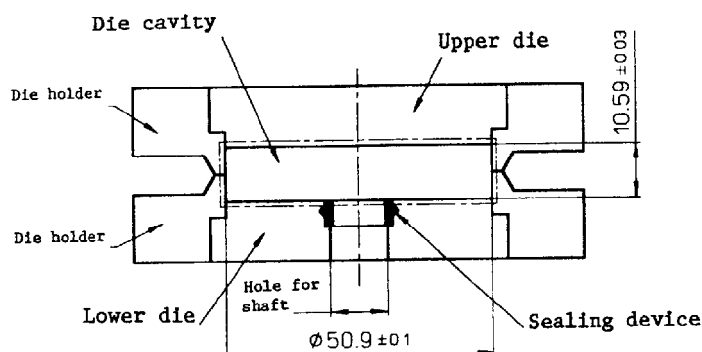
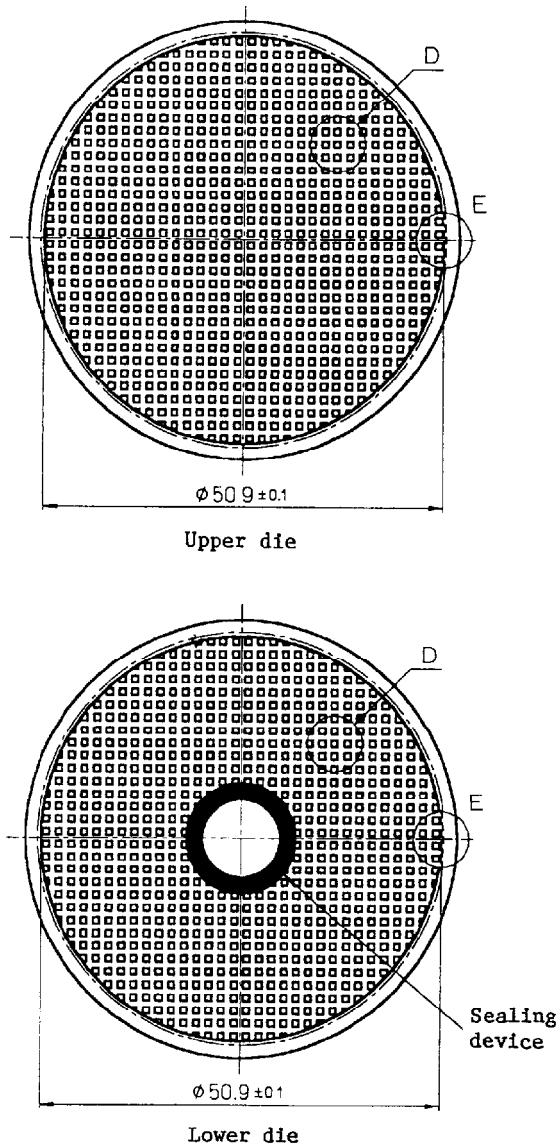


Fig. 4. Shape and dimensions of die and die grooves

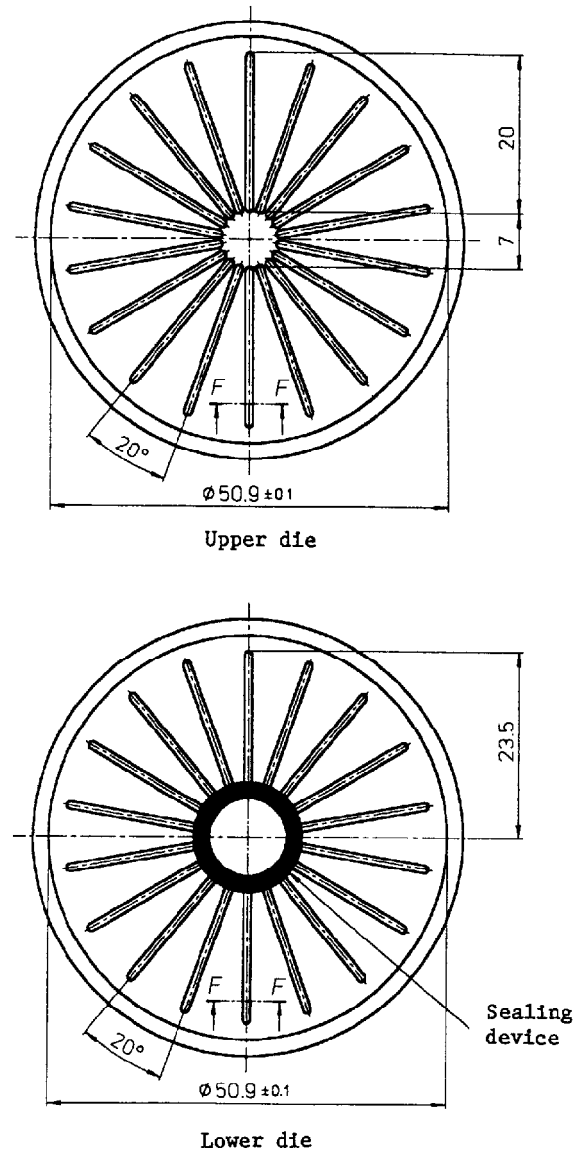
Unit: mm

Square-shaped grooves



Enlarged drawing of D part

V-shaped grooves



Enlarged drawing of E part

Enlarged drawing of F-F part

- (3) Driving device of rotor The driving device of rotor shall be capable of rotating at the rate of 2.00 ± 0.02 revolutions per minute.
- (4) Device of heating and temperature controlling The device for heating and temperature controlling capable of holding the temperature of dies at test temperature⁽³⁾ with $\pm 0.5^\circ\text{C}$ tolerance, and of recovering the test temperature of dies within 5 min with $\pm 0.5^\circ\text{C}$ tolerance after a test piece is inserted, shall be provided.

Note ⁽³⁾ See the items of test temperature in 6.5 (3).

- (5) Closing device for dies The tester shall be equipped with a suitable device by which, while testing, the die cavity is kept under closed condition⁽⁴⁾ with the force of 11.5 ± 0.5 kN { 1173 ± 51 kgf}.

Note ⁽⁴⁾ Closed condition means that the paper or cellophane with 0.04 mm or less in thickness, will leave uniform pattern made by grooves on it when it is inserted between two mating surfaces of die periphery or die holder.

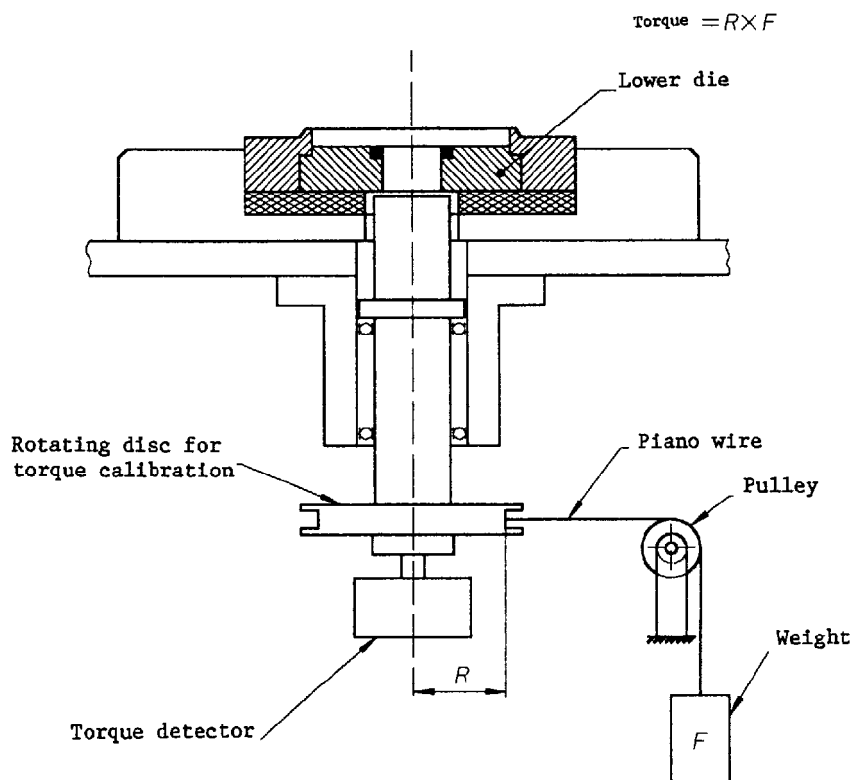
- (6) Torque indicating device Torque indicating device can indicate or record the value of torque working on the shaft of a rotor with Mooney unit (hereafter referred to as "M"), and its maximum measurement shall be 200 M and its minimum graduation be 1.0 M or less.

6.4 Calibration of torque indicating device Torque indicating device shall be calibrated at every suitable interval.

Calibration shall be carried out as follows; prepare a rotor for torque calibration or a rotating disc for torque calibration by which torque of 8.30 ± 0.02 N·m { 84.6 ± 0.2 kgf·cm} applies on the shaft of the rotor, connect it with piano wire class A, with 0.45 mm in diameter, specified in JIS G 3522 via a pulley, and suspend a weight for torque calibration at its other end, to make calibration (see Fig. 5).

When making calibration, raise temperature of dies up to testing temperature, set the device for torque calibration, and rotate the rotor at the rate of 2.00 ± 0.02 revolutions per minute. In this case, while no torque the indicating device shall read 0 ± 0.5 M, and while torque is 8.30 N·m { 84.6 kgf·cm} it shall read 100 ± 0.5 M.

Fig. 5. Example of torque calibration device



6.5 Test method

- (1) Mounting of test piece Attach a rotor, heat the die cavity up to test temperature, after getting temperature stable, open the dies, take swiftly out the rotor, insert the shaft of the rotor through a hole in one test piece of 1 set, and again attach the rotor. Then, place another test piece on the rotor, and close the die rapidly.

Remarks: When starting up the apparatus from ordinary temperature, raise temperature of a rotor before test sufficiently. Usually, the first test result shall not be adopted, and the results of the second trial and after that shall be adopted.

- (2) Rotor Principally, the rotor shall be L-type, but when viscosity exceeds 100 M, S-type may be used.

When testing rubber with either low viscosity or sticky property, for the purpose of easy detachment of test piece after test, the thermally stable film (for instance, cellophane) with about 0.03 mm in thickness may be placed between surfaces of test piece and metal. In this case, employing film may give some influence on test results, so it is preferable to record whether film is used or not.

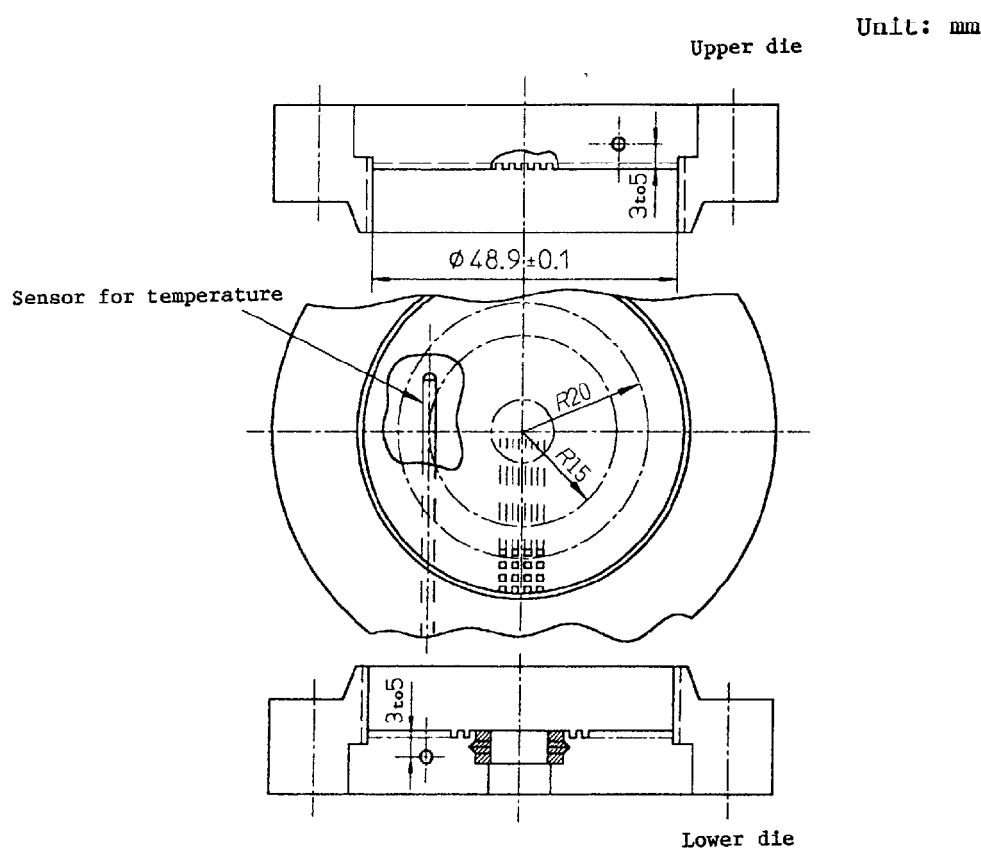
10
K 6300-1994

(3) Test temperature Principally, test temperature shall be $100.0 \pm 0.5^{\circ}\text{C}$. The test temperature is the one of dies measured under the following condition.

- (a) Measurement spot Temperature sensor shall be located at 3 mm to 5 mm apart from the inside wall of die cavity, and at 15 mm to 20 mm apart from the rotating shaft of a rotor (see Fig. 6).
- (b) Measuring method Shut the dies as the rotor was attached, run the device for heating and temperature control, and read the temperature given when the temperature comes to constant.

Remarks: Test temperature may be raised up to $125.0 \pm 0.5^{\circ}\text{C}$ depending on the type of sample.

Fig. 6. Measuring spot of test temperature



(4) Measurement Record the time when dies are shut according to (1), and thereafter preheat principally at test temperature for one min. Immediately after preheating, rotate the rotor, principally rotate it for 4 min, then finish the test. Find the minimum value of M shown while 30 s prior to finish of test, and make it Mooney viscosity of the rubber.

Remarks: The running time of a rotor may be for 8 min depending on the type of sample.

6.6 Roundign off of test result Principally, round off the value obtained by one test piece according to JIS Z 8401 to get a whole number, and make it test result.

6.7 Record Test result shall be recorded the following items.

- (1) Mooney viscosity
- (2) Shape of rotor
- (3) Period of preheating
- (4) Running time of a rotor
- (5) Test temperature
- (6) Type of die
- (7) Other items to be specially mentioned

Remarks: Test result is generally expressed as follows.

Example: 50 ML (1+4) 100°C

where, 50 M: Mooney viscosity, L: L-type of a rotor, (1+4): one min of preheating and 4 min of rotor running time, and 100°C: test temperature of 100°C.

7. Mooney scorch test

7.1 Purpose This test shall be carried out to measure the scorch characteristic of unvulcanized rubber.

7.2 Test piece Test piece shall follow 6.2.

7.3 Test apparatus Test apparatus shall follow 6.3.

7.4 Calibration of torque indicating device The calibration of a torque indicating device shall follow 6.4.

7.5 Test method

- (1) Mounting of test piece The mounting of test piece shall follow 6.5 (1).
- (2) Rotor Rotor shall follow 6.5 (2).
- (3) Test temperature Principally, test temperature shall be $125.0 \pm 0.5^\circ\text{C}$. The measuring method of test temperature shall follow 6.5 (3).

Remarks: Other test temperatures may be applied depending on the type of sample.

- (4) **Measurement** Record the time at which dies are shut according to 6.5 (1), thereafter preheat principally at test temperature for one min. Immediately after preheating, rotate a rotor, take readings of enough number of values of both Mooney viscosity and time to plot a smooth curve on a diagram which represents relation between them, and prepare Mooney viscosity - time curve (see Fig. 7)⁽⁵⁾. On this curve find the minimum value of Mooney viscosity, and make it V_m .

After shutting dies, find the duration until Mooney viscosity gets 5 M higher point than V_m , make it duration t_5 , and call it Mooney scorch time. Similarly, find the duration until Mooney viscosity gets 35 M higher point than V_m , and make it t_{35} . When t_5 or t_{35} is obtained on the Mooney viscosity - time curve, the test shall be judged to finish. The value Δt , defined in the following formula, is called vulcanization index. This index expresses the degree of vulcanization speed⁽⁶⁾.

$$\Delta t = (t_{35} - t_5)$$

Notes ⁽⁵⁾ It should be preferable to use apparatus which can automatically record Mooney viscosity - time curve.

- ⁽⁶⁾ When employing S-type rotor, the values of t_5 and t_{18} give nearly equal to t_5 and t_{35} given by L-type rotor. When employing S-type rotor, vulcanization index, Δt_s is expressed by the following formula.

$$\Delta t_s = (t_{18} - t_5)$$

Fig. 7. Mooney viscosity - time curve

