

BS 903 : Part A29 : 1973

Contents

	Page		Page
Co-operating organizations	Inside front cover	Figures	
Foreword	2	1. Retraction apparatus	5
Method		2. Test piece	6
1. Scope	3		
2. Principle of test	3		
3. Apparatus	3		
4. Test piece	4		
5. Procedure	4		
6. Expression of results	4		
7. Test report	5		

Foreword

This British Standard has been prepared under the authority of the Rubber Industry Standards Committee.

This Part is based on a document which is being considered by Technical Committee 45, Rubber and Rubber Products, of the International Organization for Standardization (ISO).

This method of test is useful when employed in conjunction with other low-temperature tests for selection of materials suitable for low-temperature service, and is also of value in connection with research and development. The method is not at this stage recommended for use in specifications because there is insufficient evidence as to the repeatability and reproducibility of the test results.

British Standard

Methods of testing vulcanized rubber

Part A29. Determination of low-temperature retraction (TR test)

Method

1. Scope

This Part of this British Standard describes a temperature-retraction (TR) procedure for rapid evaluation of crystallization effects and for comparing viscoelastic properties of rubber and rubber-like materials at low temperatures.

2. Principle of test

The test is carried out by

- (1) elongating the specimen;
- (2) locking it in the elongated condition;
- (3) freezing it to practically a rigid state;
- (4) releasing the frozen specimen and allowing it to retract freely while raising the temperature at a uniform rate;
- (5) reading the length of the specimen at regular temperature intervals while it is retracting;
- (6) calculating the percentage retraction values at these temperatures from the data obtained.

In practice, the temperatures corresponding to 10 % and 70 % retraction are of particular importance, and are designated as TR 10 and TR 70, respectively.

The difference between the temperature at which a vulcanizate retracts 10 % (TR 10) and the temperature at which it retracts 70 % (TR 70) increases as the tendency to crystallize increases. Further, TR 70 has been found to correlate with low-temperature compression set, while TR 10 has been found to correlate with brittle points in vulcanizates based on polymers of similar type.

3. Apparatus

A suitable apparatus is illustrated in Fig. 1 and shall consist of:

- (1) *Container for coolant.* A container for coolant, insulated and equipped with an agitator, a temperature measuring device reading to 1 °C and a device for heating the coolant.
- (2) *Coolant.* A coolant which does not affect the rubber material to be tested. Among liquids that have been found suitable for use at low temperature are acetone, methanol, ethanol, butanol, silicone fluid and n-hexane with crushed dry ice added. Care should be taken to avoid the use of polar liquids in combination with polar rubbers or hydrocarbon liquids in combination with hydrocarbon rubbers. Gaseous media may be employed as coolants when the design of the apparatus is such that temperature control comparable with liquid media can be obtained.

- (3) *Rack with test piece holders.* Rack with loading device, holders for one or more test pieces and locking device for the upper (movable) test piece holders (see Fig. 1).

The rack shall be designed to maintain a slight tension (10–20 kPa*) on the test piece and permit it to be stretched up to a maximum of 350 %; the design shall permit the rack to be locked into position at the chosen elongation and subsequently released. Means shall be provided to enable the length of the test piece to be read, at any time during the test, with an accuracy of ± 1 mm.

Alternatively a series of removable scales graduated to enable the retraction to be read directly as a percentage of the elongation of the frozen rubber with an accuracy of ± 1 %, may be used.

The movable parts of the apparatus shall be constructed so that the lowest possible friction occurs.

* 1 kPa = 1 kN/m²

BS 903 : Part A29 : 1973

4. Test piece

4.1 Type and preparation of test piece

4.1.1 Standard test piece. The standard test piece shall have a reference length of either 100 mm or 50 mm (see Fig. 2). The test piece with reference length 100 mm is preferred for tests with small elongations, the test piece with reference length 50 mm for tests with larger elongations. Test pieces shall be cut with a sharp die from a flat sheet 2.0 ± 0.2 mm thick. The sheets may be prepared by moulding or from finished articles by cutting and buffing.

4.1.2 Test piece cut from products. Alternatively, other types of test piece cut from finished rubber products may be used (e.g. an O-ring with a cross section diameter between 1.5 mm and 4 mm).

4.2 Number of test pieces. For each test at least three test pieces shall be used.

4.3 Conditioning of test pieces. Unless otherwise specified for technical reasons the following procedures shall be used:

(1) For all test purposes the minimum time between vulcanization and testing shall be 16 hours. For non-product tests the maximum time between vulcanization and testing shall be 28 days 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, whenever possible, the time between vulcanization and testing shall not exceed 90 days. In other cases tests shall be made within 60 days of the date of receipt by the customer of the product.

(2) Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

(3) Test pieces shall be conditioned for not less than three hours at $23 \pm 2^\circ\text{C}$ immediately before testing.

5. Procedure

The bath shall be filled with coolant so that the test pieces are covered during testing by liquid to a depth of at least 25 mm. Start the stirrer, then add dry ice to the coolant until the temperature is below -70°C . Care should be exercised at the start to avoid excessive frothing.

Insert the test piece in the rack and, at room temperature, stretch the reference length to the chosen elongation (see Fig. 2). The elongation should be chosen in the light of the following criteria.

(1) To avoid the effect of crystallization use an elongation of 50 %.

(2) To study the combined effect of crystallization and low temperature use a value of:

- a. 250 % if the elongation at break is less than 600 %
- b. half the elongation at break if 250 % is unobtainable
- c. 350 % if the elongation at break is greater than 600 %.

(3) For O-rings use an elongation of 50 %.

Lock the upper test piece holder with the locking device.

For normal tests immerse the stretched test piece into the cold bath as soon as possible after stretching.

Where studies of crystallization rates are required, the stretched test pieces should first be subjected to a specified time and temperature conditioning period before immersion into the cold bath.

After immersion in the bath at a temperature of -70°C to -73°C for 10 min to 12 min, release the locking device of the upper holder and allow the specimen to retract freely. If the elongated test piece retracts by more than 75 % of the applied elongation, use another cooling medium and cool to a lower initial temperature.

Raise the temperature of the liquid at a rate of $1 \pm 0.5^\circ\text{C}$ per min.

Take the first reading of specimen length or percentage retraction at the initial temperature, or at -70°C , and continue to read the length or percentage retraction at every 2°C rise until retraction has reached 75 % of the applied elongation.

6. Expression of results

Calculate retraction values at any specific temperature as follows:

$$\text{Retraction \%} = \left(\frac{L_e - L_t}{L_e - L_o} \right) \times 100$$

where L_o is the length of specimen in the unstretched condition,

L_e is the length of specimen in the stretched condition,

L_t is the length of specimen at the observed temperature.

Plot a curve of the percentage retraction values against the actual temperature for each test piece.

Read the temperatures from the curve which show retractions of 10 %, 30 %, 50 % and 70 %. These temperatures are designated as TR 10, TR 30, TR 50 and TR 70.

Calculate the median value of the three determinations of the temperature for TR 10, TR 30, TR 50 and TR 70.

7. Test report

The test report shall include at least the following information.

- (1) identification of the sample;
- (2) type and dimensions of the test pieces;
- (3) coolant used;
- (4) elongation at freezing;
- (5) time and temperature of low temperature conditioning;
- (6) temperature TR 10, TR 30, TR 50 and TR 70 calculated according to Clause 6;
- (7) method used, i.e. BS 903:Part A29;
- (8) date of test.

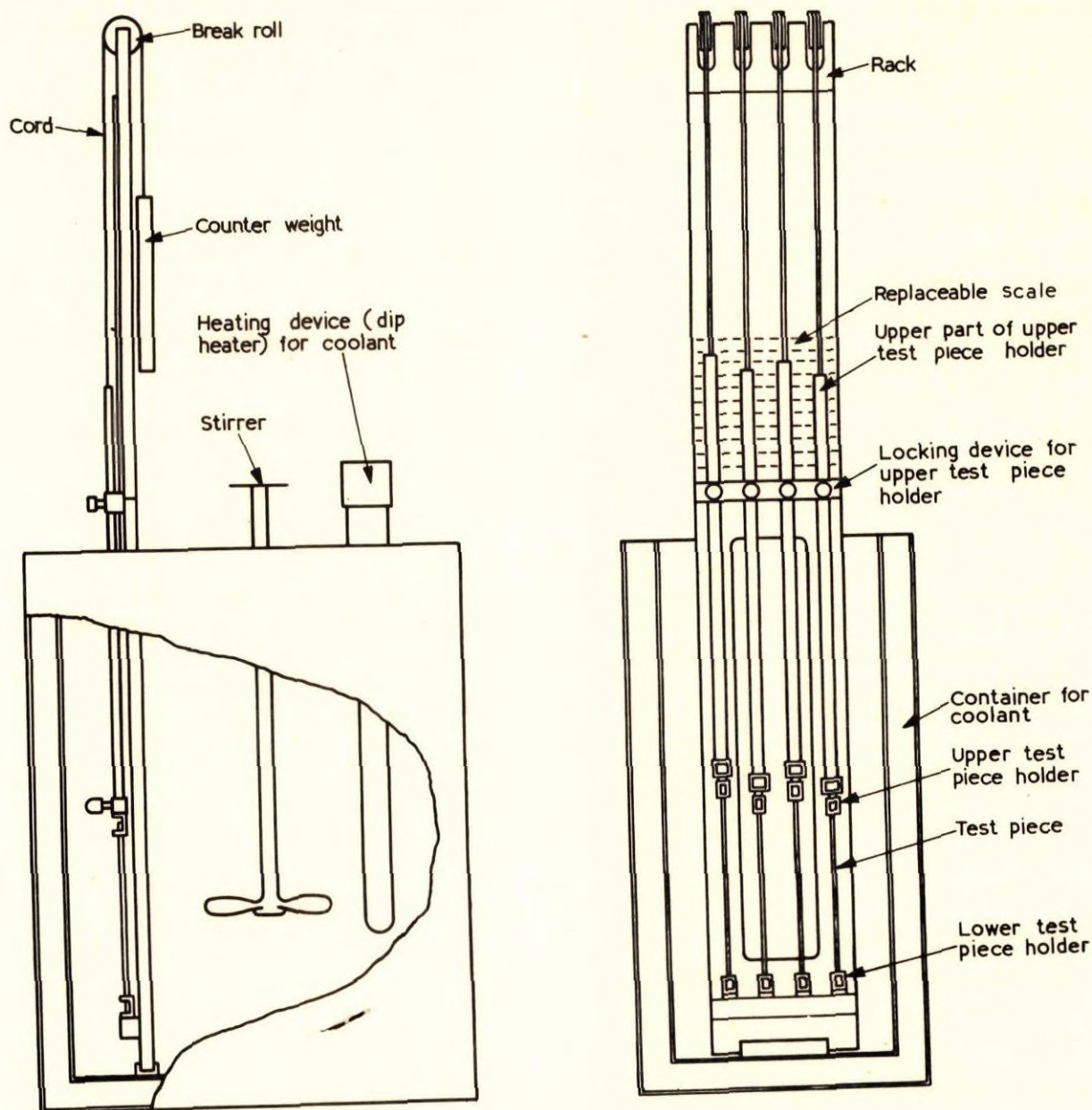
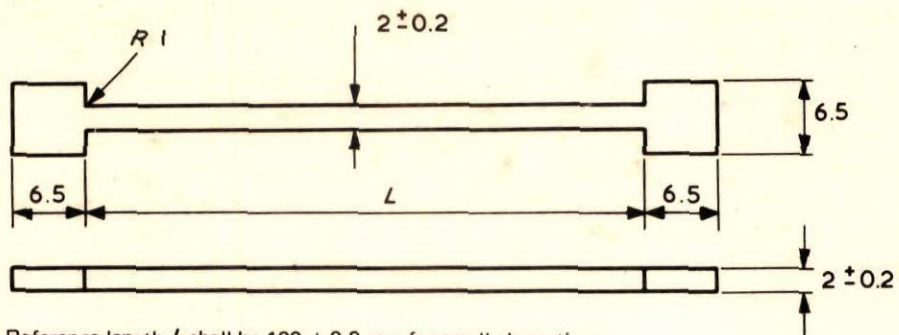


Fig. 1. Retraction apparatus

BS 903 : Part A29 : 1973



Reference length L shall be 100 ± 0.2 mm for small elongations
and 50 ± 0.2 mm for large elongations

All dimensions in millimetres

Fig. 2. Test piece