BRITISH STANDARD

BS 903 :
Part A59 : 1990

Physical testing of rubber

Part A59. Methods using plastimeters

Essais physiques du caoutchouc
Partie A59. Méthodes avec plastimètre

Prüfung der mechanischen Eigenschaften von Kautschuk
Teil A59. Prüfungen mit dem Rheometer

STANDARDS

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Rubber Standards Policy Committee (RUM/-) to Technical Committee RUM/36, upon which the following bodies were represented:

British Railways Board
British Rubber Manufacturers' Association
ERA Technology Ltd.
GAMBICA (BEAMA Ltd.)
Institution of Mechanical Engineers
Institution of Water Engineers and Scientists
Malaysian Rubber Producers' Research Association
Ministry of Defence
National College of Rubber Technology
Rubber and Plastics Research Association of Great Britain
SATRA Footwear Technology Centre

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Contents

Foreword

This Part of BS 903 was prepared under the direction of the Rubber Standards Policy Committee and supersedes the methods in BS 1673 : Part 3 : 1969 using plastimeters. With the publication of this Part and BS 903 : Part A58, which will provide methods for the determination of Mooney viscosity, Delta Mooney values and pre-vulcanization characteristics using the Mooney viscometer, BS 1673 : Part 3 : 1969 is superseded entirely and withdrawn.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Method

1 Scope

This Part of BS 903 describes a method for the determination of the rapid plasticity number of raw rubber and unvulcanized compounded rubber and a method for the determination of the plasticity retention index of raw natural rubber, both methods using a parallel plate plastimeter.

NOTE. The titles of the publications referred to in this standard are listed on the inside back cover.

2 Method 1. Determination of the rapid plasticity number

2.1 Principle

A disc-shaped test piece is rapidly compressed between small parallel platens to a fixed thickness of 1 mm and held thus for 15 s to reach approximate temperature equilibrium with the platens. It is then subjected to a constant compressive force of 100 N for 15 s. Its thickness expressed in units of hundredths of a millimetre ($mm \times 10^2$) at the end of this period is taken as the measure of plasticity (the rapid plasticity number).

2.2 Apparatus

2.2.1 *A parallel plate plastimeter*, having two parallel circular platens with smooth flat surfaces, movable in relation to each other, both provided with a suitable means of heating, and a jacket so that the material being tested and the space surrounding it is maintained at the specified test temperature.

One of the two platens shall be a right cylinder of stainless steel and have one of the following diameters: 7.3 mm, 10.0 mm or 14.0 mm (tolerance ± 0.02 mm); its effective length shall be 4.5 ± 0.15 mm, and care shall be taken to ensure that the edge of the working face is neither worn nor damaged. The diameter shall be selected so that the measured plasticity lies between 20 and 85. The other of the two platens shall be of either stainless steel or of chromium-plated brass and shall be of a greater diameter than the first plate; its effective depth of inclusion within any heating jacket shall be 3.5 ± 0.25 mm. (Suitable forms for the two platens with prescribed dimensions are shown in figure 1). The platens are mounted coaxially one above the other.

2.2.2 *A mechanism for moving one or other of the two platens normal to its surface*, to compress the test piece to a thickness of 1.00 ± 0.01 mm. The mode of movement of the platen and the forces applied in this operation shall be such that, with or without the test piece in place, the movement is always completed within a period of 2 s. A force of at least 300 N is required and may be conveniently provided by springs.

2.2.3 *A mechanism for applying to one or other platen a test force of* 100 ± 1 N *normal to its surface*, to compress the test piece.

2.2.4 *A mechanism for measuring the thickness of the test piece*, to the nearest 0.005 mm when it is between the platens.

2.2.5 *A timing device*, so that the test may be timed in seconds to an accuracy of 0.2 s.

2.2.6 *A laboratory mixing mill*, operating at even speed and 24 ± 1 r/min but otherwise conforming to BS 1674. The roll surface temperature shall be in the range of 15 °C to 35 °C.

NOTE. The application of friction up to a ratio of 1.34 and the use of roll speeds in the range 18 r/min to 32 r/min have not been found to give significantly different results.

2.2.7 *Punch*, to produce test pieces of approximately constant volume quickly and without difficulty, consisting of a flat-ended cylindrical anvil and a coaxial tubular knife moving independently of one another; a single action of the handle shall compress a portion of the material to a thickness of approximately 3 mm and shall cut out a disk of approximately 13 mm diameter.

2.2.8 *Tissue paper*, unglazed, acid-free of approximately 17 g/m^2.

2.3 Calibration of plastimeter

Check the settings of the rapid plastimeter against the manufacturer's instructions. Recalibrate the loading mechanism (to be accurate to ± 1 N) every 6 weeks, and the timing unit (to be accurate to ± 0.2 s) every 4 weeks. Check the position of the top platen before each series of tests.

2.4 Preparation of test piece

Prepare test pieces consisting of a disk of rubber approximately 13 mm in diameter and approximately 3 mm thick having a volume of 0.40 ± 0.04 cm^3.

NOTE 1. The test piece need only be approximately constant in volume because the final shaping to exact dimensions is carried out in the instrument during the preheating period.

For raw rubbers cut the test piece using the punch (**2.2.7**) from a test portion prepared in accordance with BS 6315. For compounded rubbers which are to be tested for referee purposes cut the test piece from a test portion taken from a compound prepared in accordance with the material standard relevant to the rubber. For compounded rubber in general cut the test piece from a sheet of maximum thickness 4 mm.

NOTE 2. The rapid plasticity number is affected by the manner in which the rubber is prepared and the conditions of storage prior to test. Accordingly, the prescribed procedure for preparing the particular rubber should be followed rigorously.

2.5 Temperature of test

The preferred temperature of test is 100 ± 1 °C.

2.6 Procedure

Place two pieces of the tissue paper (**2.2.8**) between the heated platens and set the thickness measuring device to zero when the platens are closed. Then insert the test piece between the two pieces of tissue paper and place immediately between the heated platens. Then compress the test piece to a thickness of 1.00 ± 0.01 mm in not more than 2 s and maintain it in the compressed state for a preheating period of 15 +1, −0 s.

On completion of the preheating period, apply a test force of 100 ± 1 N to the movable platen for a period of 15 ± 0.2 s. Take the reading of the thickness at the moment the 15 s test period is complete.

NOTE. On models with electronic digital read-out the measurement is held unless the instrument is re-set. On instruments with dial gauge read-out, the reading is taken before any drop back on to the locking mechanism occurs.

2.7 Expression of results

Determine the median value of the three results from three test pieces at the end of the 15 s test period. Express the result in units of hundredths of a millimetre as the rapid plasticity number.

2.8 Test report

The test report shall include the following information:

(a) identification of the rubber tested;

(b) a reference to this British Standard method of test, i.e. BS 903 : Part A59, method 1;

(c) the rapid plasticity number expressed as specified in 2.7;

(d) the size of platen used;

(e) the test temperature;

(f) date of test.

3 Method 2. Determination of the plasticity retention index of raw natural rubber

3.1 Principle

The rapid plasticity of raw natural rubber test pieces is measured before and after heat ageing in an oven for 30 min at 140 °C. The percentage ratio of the rapid plasticity numbers after and before heat ageing is taken as the plasticity retention index (PRI).

3.2 Apparatus

3.2.1 *Parallel plate plastimeter*, with a 10 mm diameter platen and otherwise as described in **2.2.1** to **2.2.5**.

3.2.2 *Punch*, as described in **2.2.7**.

3.2.3 *Thickness gauge*, having a scale graduated in unit divisions of 0.01 mm, fitted with a flat contact of diameter about 4 mm and operating with a pressure of 22 ± 5 kPa.

3.2.4 *Air circulation oven*, operating at 140 °C with control of the temperature in the vicinity of the test pieces to within ± 0.2 °C over a 30 min period; temperature uniformity to within ± 0.2 °C.

NOTE. A larger variation will impair the accuracy of test.

The oven shall allow temperature recovery of the oven and the inserted tray and dishes to within 1 °C of the set temperature in a time not exceeding 2 min after insertion of the tray in the oven. The air within the oven shall be replaced not less than 10 times per hour.

3.2.5 *Dishes*, of 40 mm to 50 mm diameter.

3.2.6 *Tray*, to carry the dishes (**3.2.5**).

3.3 Preparation of test pieces

NOTE 1. It is important to prepare the test pieces with care, as the PRI is affected by the sheet thickness.

Take 30 g from a test portion prepared in accordance with BS 6315 and pass three times (doubling the sheet between passes) between the mill rolls, with the nip adjusted so that the final sheet thickness is approximately 1.7 mm. Immediately double the sheet, which shall be uniform in texture and free from holes, and lightly press the two halves together by hand, ensuring that no air bubbles are formed.

NOTE 2. It is necessary to ascertain the required nip setting by preliminary trial, as it will vary with the rubber and with the mill.

Cut six test pieces from the doubled sheet using the punch (**3.2.2**). Using the thickness gauge (**3.2.3**), ensure that the thickness of each test piece is not less than 3.2 mm and not greater than 3.6 mm. If the six test pieces are not within these limits, prepare a new doubled sheet and prepare six new test pieces that are within these limits. Divide the six test pieces into two random sets each containing three test pieces. Use one random set of three test pieces for testing before ageing and the other random set of three test pieces for testing after ageing (see **3.4.1**).

3.4 Procedure

3.4.1 *Ageing of test pieces*

Before ageing is started, check the oven temperature to ensure that it has been stable at 140 ± 0.2 °C for at least 5 min.

To ensure that all test pieces are aged at the correct temperature do not overload the oven; this may cause a severe prolonged temperature drop and may upset temperature uniformity (see **3.2.4**).

Quickly insert the tray taking care that it is within the calibrated region of the oven. Close the oven door and start timing.

Check that the temperature is regained to 140 ± 1 °C within 2 min and retained at the correct level.

After 30 ± 0.25 min remove the tray from the oven and the dishes from the tray. Allow them to cool at standard laboratory temperature as defined in BS 903 : Part A35.

3.4.2 ***Measurement of plasticity***

Determine the rapid plasticity number in accordance with clause **2** not less than 30 min and not more than 2 h after ageing. Take readings of the rapid plasticity number to the nearest 0.5 unit, i.e. 0.005 mm.

Make plasticity determinations on unaged and aged test pieces at the same time.

3.5 Expression of results

Calculate the plasticity retention index (PRI) according to the following:

$$PRI = \frac{\text{Median value of rapid plasticity number of aged test pieces}}{\text{Median value of rapid plasticity number of unaged test pieces}} \times 100$$

NOTE. See appendix A.

3.6 Test report

The test report shall include the following information:

(a) identification of the rubber tested;

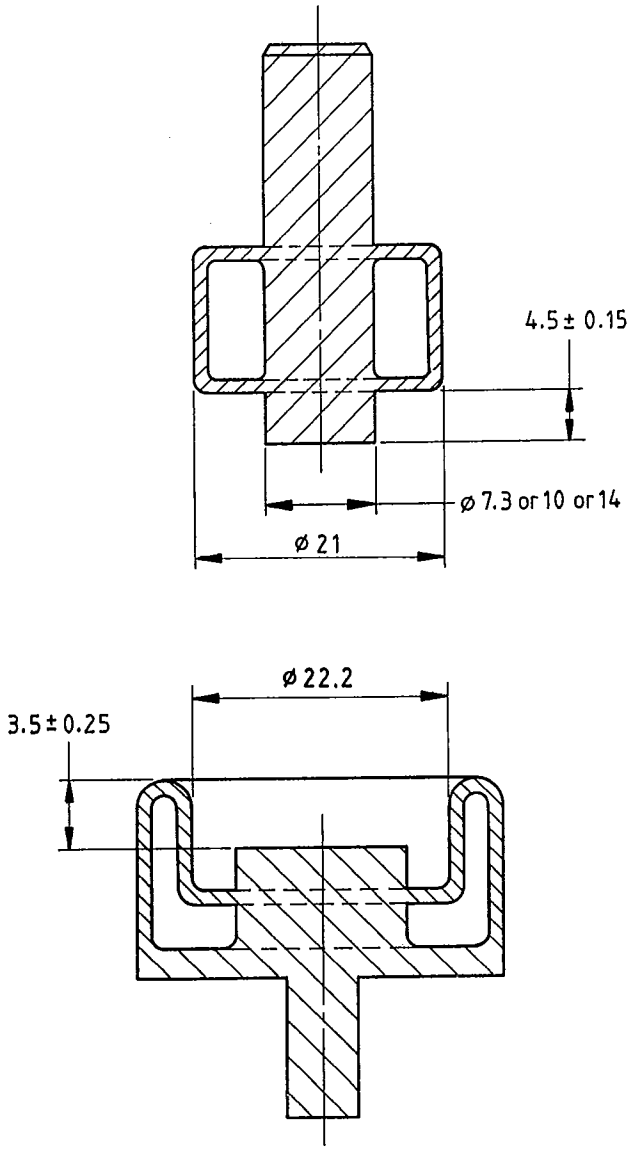
(b) a reference to this British Standard method of test, i.e. BS 903 : Part A59, method 2;

(c) median rapid plasticity number for unaged and aged test pieces;

(d) the PRI;

(e) the type of oven used;

(f) date of test.



All dimensions are in millimetres

Figure 1. Parallel plate plastimeter

Appendix

Appendix A. Repeatability

It has been found that the coefficient of variation (V) is dependent upon the accuracy of the ageing temperature. For PRI calculated as in **3.5** from the median plasticity values, V is 3 % when ageing is at 140 ± 0.2 °C and 6 % at 140 ± 1 °C. Both values of V are consistent with an accuracy of 3 % for single determinations of the rapid plasticity number.

Publication(s) referred to

BS 903	Physical testing of rubber Part A35 Temperatures, humidities and times for conditioning and testing of test pieces Part A58 Methods of using the Mooney viscometer[1)]
BS 1674	Specification for equipment and general procedure for mixing and vulcanizing rubber test mixes
BS 6315	Methods for sampling and sample preparation of raw rubber

[1)]Referred to in the foreword only.

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