Designation: D 1917 – 023

Standard Test Methods for Rubber Property—Shrinkage of Raw and Compounded Hot-Polymerized Styrene-Butadiene Rubber (SBR)[1]

This standard is issued under the fixed designation D 1917; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of mill shrinkage of hot-polymerized styrene-butadiene rubbers (SBR) and their compounds.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.12 on Processability Tests .

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D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]
D 3896 Practice for Rubber from Synthetic Sources—Sampling[2]
D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]
E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[3]

3. Summary of Test Methods

3.1 A specimen of raw or compounded rubber is prepared on a standard laboratory mill, its dimensions are measured or controlled by a cutting technique, it is heat aged in a circulating oven at 100°C, cooled at room temperature, and the dimensions are again measured. The linear percent shrinkage of the specimen is calculated.

3.2 The following test methods are given:

3.2.1 *Test Method A*— For raw SBR which is particularly applicable to rubbers having shrinkage below 25 %, such as cross-linked types.

3.2.2 *Test Method B*— For compounded SBR.

4. Significance and Use

4.1 The shrinkage of SBR rubbers and their compounds after processing is of importance in many applications. This property can also be used for control, specifications, and fabrication purposes.

5. Apparatus

5.1 *Mill*—A standard laboratory mill as described in Practice D 3182.

5.2 *Laboratory Oven*, having internal horizontal dimensions in excess of 300 by 480 mm (12 by 19 in.) and conforming to the requirements for Type II, Grade B ovens given in Specification E 145.

5.3 *Mill Knife*.

5.4 *Cutting Tools*:

5.4.1 A tool having two parallel blades spaced about 20 mm (0.8 in.) apart. Two standard mill knives bolted together, properly spaced, may be used.

5.4.2 A 150 mm long by 100 mm wide (6.0 by 4.0 in.) metal template approximately 1 mm (0.04 in.) thick, equipped with a handle, and having its longitudinal dimension curved to fit the contour of the mill roll, may also be used. It is especially suitable for raw rubbers that are disjunctive or form a poor sheet. A mill knife is used for cutting the rubber.

5.5 *Smooth Sheet*, of TFE-fluorocarbon, metal, or glass, having dimensions to accommodate the specimen.

5.6 *Linear Scale*, 500 mm (20 in.) long, graduated in 1.0 mm (0.05 in.).

5.7 *Dusting powder*, for example talc or soapstone.

6. Sampling

6.1 Take samples in accordance with Practice D 3896.

NOTE 1—Make sure the sample is talc-free.

7. Test Specimens

7.1 *Standard Specimens*—The standard specimen is a strip cut with a double-bladed knife. Three specimens are cut, one in the center, and two, each halfway between the center and the edge of the banded material.

7.2 *Template Specimens*—An alternative method is the use of a template. When the template is used, only one specimen is cut exactly around the edges of the template from the center of the material banded on the mill.

NOTE 2—These two types of specimens will not necessarily give identical results. Results obtained on one type of specimen should not be compared with those obtained using the other.

TEST METHOD A—RAW RUBBER

8. Procedure

8.1 With the mill rolls maintained at 50 ± 5°C (122 ± 9°F) and roll separation of 0.20 ± 0.05 mm (0.008 ± 0.002 in.), pass 200 g of rubber twice without banding.

8.2 Adjust the roll separation to 0.6 ± 0.1 mm (0.024 ± 0.004 in.), band the rubber on the slow (front) roll, and mill exactly 10 min. Make three-quarter cuts from alternate sides every ½ min throughout the milling.

8.3 Open the mill rolls slowly and evenly until the bank disappears and the rubber receives no pressure from the opposite roll. This is done by opening the rolls in increments of no more than 0.25 mm (0.01 in.) on alternate ends of the mill rolls.

[2] *Annual Book of ASTM Standards*, Vol 09.01.
[3] *Annual Book of ASTM Standards*, Vol 14.04.

8.4 Within 15 s after the mill rolls are opened, trim the band (if necessary) by cutting off no more than 15 mm (0.6 in.) from each of the outside edges (see Note 3).

8.5 Cut three standard specimens, one from the center and one halfway between the center and each edge. Make the cuts while the mill is running by holding the cutting tool (see 5.4.1) firmly against the roll at the indicated locations for the duration of one revolution.

8.6 Stop the mill and make a cut straight across the banded rubber. Start the mill and remove the specimens, taking care not to stretch or otherwise distort them. Place the three specimens on a smooth, well-dusted metal, glass, or TFE-fluorocarbon sheet, with the inside or smooth surface down (see Note 4).

NOTE 3—Steps 8.4-8.6 should take no more than 2 min.

8.7 If the template specimen is used, stop the mill after the procedure in 8.4, place the template (see 5.4.2) in the middle of the banded rubber and cut one specimen as fast as possible, making sure that the cuts are made exactly along the edges. Remove the specimen, place it on a smooth well-dusted surface with the inside or smooth surface down, and mark the longitudinal direction.

NOTE 4—Steps 8.4 and 8.7 should take no more than 2 min.
NOTE 5—The dusted surface is used to prevent any adhesion or sticking.

8.8 Place the sheet holding the specimens in the oven controlled at 100 ± 2°C (212 ± 3.8°F) for 1 h.

8.9 Remove the sheet with the specimens from the oven and without removing the specimens, allow them to cool at ~~25~~ 23 ± 2°C ~~(77~~ (73.4 ± 3.8~~6~~°F) for 1 h.

8.10 Measure the length of each of three standard specimens to the nearest 1 mm (0.05 in.). Measure the shrunken length of the template specimen on three places. Record all three values.

8.11 Calculate the percentage of shrinkage using the equation shown in 10.1.

TEST METHOD B—COMPOUNDED RUBBER

9. Procedure

9.1 With the mill rolls maintained at 50 ± 5°C (122 ± 9°F) and roll separation of 0.20 ± 0.05 mm (0.008 ± 0.002 in.) pass twice without banding 300 g of the compound that had at least 1-h rest period after mixing.

9.2 Adjust the roll separation to 1.30 ± 0.15 mm (0.050 ± 0.006 in.), band the compound on the fast (back) roll, and mill for 4 min, making three-quarter cuts from alternate sides every ½ min. The fast roll is used at this point to improve the banding of tacky or baggy compounds.

9.3 Continue milling of the compound on the fast roll for 1 min without cutting.

9.4 Open the mill rolls slowly and evenly until the bank disappears and the compound receives no pressure from the opposite roll. Increase the opening by adjusting in increments of no more than 0.25 mm (0.01 in.) on alternate ends of the mill roll.

9.5 Within 15 s after the mill rolls are opened, trim the band by cutting off no more than 15 mm (0.6 in.) from each of the outside edges (see Note 6).

9.6 Cut three standard specimens about 20 mm (0.8 in.) in width, one from the center and one halfway between the center and each edge. Make the cuts while the mill is running by holding the cutting tool firmly against the roll at the indicated locations for the duration of one revolution.

9.7 Stop the mill and make a cut straight across the banded compound. Start the mill and remove the specimens, taking care not to stretch or otherwise distort them. Place the three specimens on a smooth, well-dusted metal, glass, or TFE-fluorocarbon surface with the inside or smooth surface down.

NOTE 6—Steps 9.5, 9.6, and 9.7 should take no more than 2 min.
NOTE 7—Dusted surface is used to prevent any adhesion or sticking.

9.8 If the template specimen is used, stop the mill after 9.5, place the template in the middle of the banded compound and cut the specimen as fast as possible, making sure that the cuts are made exactly along the edges. Remove the specimen, place it on a smooth well-dusted metal, glass, or TFE-fluorocarbon surface, with the inside or smooth surface down, and mark the longitudinal direction (see Note 8).

NOTE 8—Steps 9.5 and 9.8 should take no more than 2 min.

9.9 Place the sheet with the specimens in the oven controlled at 100 ± 2°C (212 ± 3.8°F) for 1 h.

9.10 Remove the sheet with the specimens from the oven and without removing the specimens allow them to cool at 23 ± 2°C (73.4 ± 3.6°F) for 1 h.

9.11 Measure the length of each standard specimen to the nearest 1 mm (0.05 in.). Measure the shrunken length of the template specimen on three places. Record all three values.

9.12 Calculate the percentage of shrinkage using the equation shown in 10.1.

10. Calculation

10.1 Calculate the percentage of shrinkage, *S*, as follows:

$$S = [(L_0 - L)/L_0] \times 100 \quad (1)$$

where:
L_0 = original length of the standard specimen which is equal to the circumference of the mill roll (see Note 9), and
L = shrunken length or the average value of three length measurements after oven aging and cooling.

NOTE 9—The circumference of a standard mill is between 471.2 and 486.9 mm (18.54 and 19.16 in.). The average value of 479.0 mm (18.85 in.) may be used except in referee or precision testing, when the actual circumference should be determined. If the template is used, the original length shall be 150 mm.

11. Report

11.1 Report the following information:
11.1.1 Sample identification,
11.1.2 Method used,
11.1.3 Type of specimen used,
11.1.4 Aging conditions,
11.1.5 Average value of three measurements, and
11.1.6 Date of test.

PRECISION AND BIAS

12. Precision and Bias

12.1 This precision and bias section has been prepared in accordance with Practice D 4483. Please refer to this practice for terminology and other statistical calculation details.

12.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, etc.) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocols of the test method.

12.3 A Type 1 interlaboratory test program (ITP) was conducted in 1995. Three materials (polymers) with different levels of shrinkage were supplied to four laboratories; in each laboratory two replicate tests were conducted on each of two successive test days. A test result is defined as a single measurement of percent shrinkage using Method A.

12.4 The precision results are given in Table 1; see notes at the bottom of the table. Precision may be expressed in the format of the following statements that use an "appropriate value" of *r* and *R* or (*r*) and (*R*) associated with a mean level or material in the table closest to the mean level under consideration for any material in routine testing operations.

12.5 For two of the materials the within-laboratory and the between-laboratory standard deviations are equal. This occurs mainly because the within-laboratory variation is large and the "average of two" between-laboratory values have decreased variation. This situation frequently occurs in ITP that have only a minimal number of laboratories.

12.6 *Repeatability*— The repeatability, *r* (in percent actual shrinkage) of this test method has been established as the appropriate value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or non-identical sample populations.

12.7 *Reproducibility*— The reproducibility, *R* (in percent actual shrinkage) of this test method has been established as the appropriate value tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or non-identical sample populations.

12.8 *Relative Precision*—The relative repeatability (*r*) and the relative reproducibility (*R*) are defined for this standard as a percent of the measured percent shrinkage. These values are also found in Table 1.

12.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by

TABLE 1 Precision for Percent Shrinkage: Method A

		Within Lab			Between Lab		
Material	Mean	*Sr*	*r*	(*r*)	*SR*	*R*	(*R*)
B	10.4	0.654	1.83	17.6	0.654	1.83	17.6
A	14.4	0.742	2.08	14.4	0.808	2.26	15.7
C	25.5	1.755	4.91	19.3	1.755	4.91	19.3

Note: Only four laboratories participated in the ITP
Sr = repeatability standard deviation
SR = reproducibility standard deviation
r = repeatability, in measurement units
R = reproducibility, in measurement units
(r) = repeatability (relative) in percent
(R) = reproducibility (relative) in percent

the test method. Bias cannot therefore be determined.

13. Keywords

13.1 compounded rubber; hot-polymerized SBR; mill shrinkage; raw rubber