Designation: D3194 – 17

Standard Test Method for Rubber From Natural Sources—Plasticity Retention Index (PRI)[1]

This standard is issued under the fixed designation D3194; 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.

1. Scope

1.1 This test method gives an indication of the oxidation resistance of raw natural rubber at a specified temperature. This resistance is indicated by the Plasticity Retention Index (PRI), a ratio expressed as a percentage of the aged plasticity to the original plasticity determined by means of a parallel plate plastometer. A low PRI value indicates a poor resistance to oxidation.

1.2 The values stated in SI units are to be regarded as the standard.

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:*[2]

D1485 Practice for Rubber from Natural Sources—Sampling and Sample Preparation

D3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing (Withdrawn 1987)[3]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Summary of Test Method

3.1 A homogenized sample of raw rubber is prepared by a specified procedure to create a sheet from which six cylindrical pellets of specified dimensions are cut, three to be tested in original condition and three to be aged under specified conditions.

3.2 Plasticity on both the original and aged samples is measured as the median final thickness of each set of samples after a compressive force of 100 N ± 1 N is applied for a specified time. The PRI is the ratio expressed as a percentage of the aged to the original plasticity.

4. Significance and Use

4.1 This test method may be used to evaluate the heat and/or oxidation stability (aging) of raw rubber under controlled conditions. High original and aged plasticity values usually correspond to good aging properties. The ratio of the aged to original plasticity values expressed as a percentage, the PRI, is used as an indication of aging.

5. Apparatus

5.1 *Parallel Plate Plastometer*[4], with a 10 mm diameter platen. The platen temperature shall be 100 ± 1°C and the plastometer shall have a timer unit capable of giving a 15 ± 1 s initial conditioning period during which the sample shall be compressed to 1 ± 0.01 mm and a test period of 15 ± 1 s under a compression force of 100 N ±1 N. The plastometer shall also have a measuring device gauge reading to 0.01 mm. Calibration procedures recommended by the manufacturer of the plastometer should be followed.

5.2 *Punch,* that will die out cylindrical specimens approximately 13 mm in diameter from a prepared sheet of rubber.

5.3 *Circulating Air Oven,* specified in Specification E145, Type IIA. The oven must be capable of maintaining 140 ± 0.5°C.

5.4 *Dishes or Tray,* aluminum, for test specimens, lightweight aluminum dishes approximately 38 mm (1.5 in.) in diameter or an aluminum tray approximately 150 by 150 mm (7 by 7 in.) for aging specimens in the oven.

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.22 on Natural Rubber.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] The sole source of supply of the Wallace Rapid Plastimeter known to the committee at this time is Leverett A. Anderson Co., P.O. Box 5400, Akron, OH 44313. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

5.5 *Tissue paper or cigarette paper,* approximately 40 by 40 mm (1.5 by 1.5 in.). This paper shall be bleached, unglazed acid-free tissue paper, of approximately 1.7 g/m^2.

6. Precautions

6.1 The initial thickness of the prepared sheet can affect the final thickness after testing. Ascertain the required mill opening by preliminary trial; it will vary with the rubber and the mill.

6.2 The temperature and speed of the testing operations can affect the results. An increase in temperature or the time to preheat or time to read the dial micrometer can result in low, final plasticity readings.

7. Sampling

7.1 The natural rubber sample shall be obtained in accordance with Practice D1485.

8. Test Specimen Preparation

8.1 *Test Specimen Preparation Method A (original protocol):*

8.1.1 Homogenize the piece to be tested (mass approximately 360 g) 10 times through a mill as described in Practice D3182. The mill rolls should be at 70 ± 5°C (158 ± 9°F) with an opening of approximately 1.65 mm (0.065 in.). After each of the first six passes, roll the rubber into a cylinder and pass endwise through the mill rolls. After the sixth pass, the sample shall not be rolled, but is folded.

8.1.2 Take a test portion of about 30 g from the homogenized sample and pass it three times (doubling the sheet between passes) between mill rolls at room temperature with the opening adjusted so that the final sheet thickness is approximately 1.7 mm (0.07 in.). After the third pass, double the sheet, which should be uniform in texture and free of holes, and press the two halves lightly together.

8.1.3 Die out six or more test specimens from the doubled sheet with the punch and measure their thickness until six test pellets are obtained with a thickness of 3.4 ± 0.2 mm (0.134 ± 0.008 in.) having a volume of 0.40 ± 0.04 cm^3 (0.2441 ± 0.002441 in.^{3}). Randomly divide these into two sets of three, one for test before aging and the other for test after aging.

8.2 *Test Specimen Preparation Method B (alternative protocol):*

8.2.1 Homogenize the piece to be tested (mass approximately 360 g) 10 times through a mill as described in Practice D3182.

8.2.2 The mill rolls should be at room temperature with an opening of approximately 1.65 mm (0.065 in.). After each of the first six passes, roll the rubber into a cylinder and pass endwise through the mill rolls. After the sixth pass, the sample shall not be rolled, but is folded.

8.2.3 Take a test portion of about 30 g from the homogenized sample and pass it three times (doubling the sheet between passes) between mill rolls at room temperature with the opening adjusted so that the final sheet thickness is approximately 1.7 mm (0.07 in.). After the third pass, double the sheet, which should be uniform in texture and free of holes, and press the two halves lightly together.

8.2.4 Die out six or more test specimens from the doubled sheet with the punch and measure their thickness until six test pellets are obtained with a thickness of 3.4 ± 0.2 mm (0.134 ± 0.008 in.) having a volume of 0.40 ± 0.04 cm^3 (0.2441 ± 0.002441 in.^{3}). Randomly divide these into two sets of three, one for test before aging and the other for test after aging.

9. Procedure

9.1 *Aging:*

9.1.1 Before aging is started, the oven must be stabilized at 140 ± 0.5°C for at least ½ h.

9.1.2 The dishes or tray containing the three pellets (8.3) to be aged must be arranged within the calibrated region of the oven. Insert the tray and close the oven door quickly to prevent excessive oven heat loss. Start timing as soon as the oven door is closed.

9.1.3 After 30 ± 0.25 min remove the tray from the oven and remove the dishes from the tray. Allow them to cool to room temperature for a minimum of 30 min and a maximum of 2 h before testing.

9.2 *Measurement of Plasticity:*

9.2.1 The platen of the plastometer shall have come to equilibrium at 100 ± 1°C for 15 min before making measurements.

9.2.2 Place two pieces of tissue paper (5.5) between the heated platens and set the thickness measuring device to zero when the platens are closed.

9.2.3 Insert a pellet at room temperature between two pieces of tissue paper and place the whole assembly centrally between the heated platens. Put the machine lever into operation. After a 15-s conditioning period, the timing device automatically releases the force of 100 N ±1 N to compress the specimen. This load period is automatically adjusted, exactly 15-s duration. The final thickness expressed in units equivalent to 0.01 mm remains locked after the 15–s load period on the dial micrometer until the operating handle is moved to open the instrument. Record the measured thickness from the dial micrometer. Repeat the above for each of the pellets, both aged and in original condition.

9.2.4 The measured thickness for each pellet within either of the groups should not vary by more than ± 0.01 mm from the median value of that group. Run additional pellets, if needed, to get three of each group within this tolerance.

10. Calculation

10.1 The plasticity is 100 times the final thickness as expressed in units equivalent to 0.01 mm.

10.2 Using the median values of these plasticity results of both original (Po) and aged test specimens, calculate the PRI as follows:

$$\text{PRI} = (\text{plasticity aged}/\text{plasticity original}) \times 100 \quad (1)$$

11. Report

11.1 The report shall include the following:

11.1.1 Three values of the plasticity for the original and the aged test pieces,

11.1.2 Type of oven used, and

11.1.3 Plasticity Retention Index (PRI) using Method A or Method B.

12. Precision and Bias[5]

12.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

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

12.3 A Type 1, Class II interlaboratory precision program was conducted (except for sample No. 6, which was Type 1, Class I). Both repeatability and reproducibility are short term. A period of a few days separate test results, which were repeated on three separate days. Participation included 13 laboratories, and six materials were used. Therefore, $p = 13$, $q = 6$, and $n = 3$. A test result is the median value obtained from three determinations as specified in Section 10.

12.4 Other than sample No. 1, which was IIR, the materials used were natural rubber. They were prepared through step 8.2 of this test method. Participants were required to die out and test the cut pellets. Sample identification: No. 1 = Butyl rubber, No. 2 = PA80 coagulated latex, No. 3 = SIR20, No. 4 = No. 1RSS, No. 5 = CV50, and No. 6 = SIR20 cut pellets.

12.5 The results of the precision calculations are given in Table 1, with the materials arranged in increasing mean PRI value.

12.6 The precision for these tests on a relative basis may be expressed as follows (sample No. 1 IIR excluded):

12.6.1 *Repeatability*—The repeatability (r) of this test has been established as 9.8 %. Two single measurements (determinations) that differ by more than the tabulated (r) (expressed as percentage of their mean value) must be considered suspect, that is, having arisen from different sample populations. Such a decision dictates that appropriate action be taken.

12.6.2 *Reproducibility*—The reproducibility (R) of this test has been established as 34.5 %. Two single measurements (determinations) that differ by more than the tabulated (R) (expressed as percentage of their mean value) must be considered suspect, that is, having arisen from different sample populations. Such a decision dictates that appropriate action be taken.

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

TABLE 1 ASTM Test Method D3194 Type 1 Precision—Wallace Plasticity (PRI)[AB]

Sample	Mean Value	Within Laboratories			Between Laboratories		
		s_r	r	(r)	S_R	R	(R)
6	58.95	2.58	7.30	12.4	9.33	26.41	44.8
4	63.22	1.70	4.82	7.6	4.91	13.88	22.0
3	63.44	2.22	6.28	9.9	9.40	26.57	41.9
5	79.56	2.10	5.94	7.5	7.31	20.68	26.0
2	87.23	3.34	9.45	10.8	10.79	30.55	35.0
1[C]	95.15	1.95	5.52	5.8	2.52	7.13	7.5
Mean	70.48						
Pooled		2.45	6.94	9.8	8.6	24.32	34.5

[A] This is short-term precision with $p = 13$, $q = 6$, and $n = 3$.
s_r = Within laboratory standard deviation,
r = Repeatability in measured units (s_r x 2.83),
(r) = Repeatability in percent ((r/mean) x 100),
S_R = Between laboratory standard deviation,
R = Reproducibility in measured units (S_R x 2.83), and
(R) = Reproducibility in percent ((R/mean) x 100).
Outliers have been rejected from the tabled data.
[B] Units: PRI is the ratio expressed as a percentage of the aged to the original plasticity. The basic plasticity units represent the thickness of the samples in 0.01 mm times 100.
[C] Sample No. 1 excluded from mean and pooled values.

13. Keywords

13.1 natural rubber; plasticity; P.R.I.; retention index

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1066.