Standard Test Method for Rubber Property—Plasticity and Recovery (Parallel Plate Method)\(^1\)

This standard is issued under the fixed designation D 926; 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 (\(\epsilon\)) 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 This test method describes the determination of plasticity and recovery of unvulcanized rubber by means of the parallel plate plastometer. Uncompounded, compounded, and reclaim rubber may be tested.

Note 1—ISO 2007 and D 3194 use a principle similar to this test method although the apparatus, test conditions, and procedure are different.

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 problems, 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:
D 3194 Test Method for Rubber from Natural Sources—Plasticity Retention Index (PRI)\(^2\)
D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries\(^3\)
E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens\(^3\)

2.2 ISO Standard:
2007 Rubber Unvulcanized—Determination of Plasticity—Rapid Plastometer Method\(^4\)

3. Summary of Test Method

3.1 Plasticity:

3.1.1 A specimen of specific volume and height is tested at room temperature or heated to a specified test temperature for 15 min and then subjected to a compressive force of 49 N for a specified time.

3.1.2 At the end of this time, the height of the specimen is measured and the plasticity number is calculated.

5. Apparatus

5.1 The essential features of the parallel plate plastometer, illustrated in Fig. 1, are as follows:

5.1.1 Two metal plates, not less than 10 mm in thickness and 40 mm in diameter shall be mounted on a suitable frame so that one plate moves with respect to the other and so that the two plates are parallel within 0.017 rad (\(1^\circ\)) at all times.

5.1.2 The apparatus shall be so designed that a specimen between the parallel plates is subjected to a force of 49 ± 0.5 N including the effect of the spring in the dial gage during test.
5.1.3 A digital or dial indicator gage calibrated in hundredths of a millimetre (0.01 mm) shall be mounted on the apparatus in such a manner that the distance between the plates can be determined at all times.

5.1.4 When required, the apparatus shall be operated in any oven as described in Specification E 145 that is capable of containing the plastometer.

5.1.5 A separate digital or dial micrometer gage, calibrated in hundredths of a millimetre and having a presser foot about 25 ± 2 mm in diameter and actuated by a force of 800 ± 30 mN, is required for recovery measurements.

5.2 Test Surfaces—Films or material with low coefficients of friction to prevent sticking of the test specimen to the plates. The most commonly used test surface is polyester film.\(^5\)

Note 2—The test surface used can affect the plasticity number. Even different grades of the same type of polyester film can cause different results. When comparing results, the same test surface must be used.

6. Sampling

6.1 Samples may be taken from raw rubber or unvulcanized rubber compounds undergoing processing operations.

7. Test Specimens

7.1 The specimen shall be a right cylinder 2.00 ± 0.02 cm\(^3\) in volume and free from air pockets. (A cylinder 16 mm in diameter and 10 mm in height is appropriate.) The specimen may be prepared from either a solid piece or plied sheets of unvulcanized rubber approximately 1.5 mm in thickness, care being taken to exclude air pockets in the sheet. The specimens may be cut from the sheet either by means of two mating dies conforming to Fig. 2, which produce cylinders of the correct volume, or a rotating die having an internal diameter of approximately 16 mm. If a rotating die is used, the height of the cylinder shall be adjusted until the volume is 2.00 ± 0.02 cm\(^3\). If the density is known, weighing may be used to adjust to the correct volume. If necessary the specimen shall be placed in a suitable holder so that it is not appreciably deformed before testing.

8. Test Conditions

8.1 The plasticity and recovery values obtained for a given sample will depend largely upon the manner in which the test specimen is prepared. It is therefore imperative that, when comparisons are made between similar materials, the test specimen preparation should be in accordance with some rigidly specified method. This is necessary because the plasticity and recovery may be affected by the amount of mechanical working and the conditions under which such work is done. This effect is not constant for all materials. The time interval between test specimen preparation and testing is important and should be the same for test specimens between which comparisons are to be made.

8.2 The test temperature shall be 23 (room temperature), 40, 70, 85, or 100°C unless another temperature is agreed upon. The most frequently used temperature is 23°C.

8.3 Readings may be taken at any desired interval following the application of the load. The most frequently used intervals are between 3 and 10 min.

9. Plasticity Procedure

9.1 Set the indicator to read zero with two thicknesses of the test surface between the plates. Condition the plastometer at the test temperature until temperature equilibrium is achieved.

9.2 Place the test specimen between the two test surfaces to prevent sticking of the test specimen to the plates.

9.3 If required, condition the test specimen at the test temperature for 15 ± 0.5 min.

9.4 Quickly place the test specimen between the two parallel plates, lower the mass and at the end of the required time

\(^5\) Mylar 300A polyester film has been found satisfactory for this purpose.
interval read the thickness gage to hundredths of a millimetre. Record the height of the specimen with the micrometer gage specified in 5 min at the test temperature.

9.5 Calculate plasticity, \( P \), as follows:

\[
P = 100h_1
\]

(1)

10. Recovery Procedures

10.1 Recovery values may be determined by the procedure described in either 10.2 or 10.3, but the results will differ.

10.2 Procedure A:

10.2.1 After determining the plasticity as described in Section 9 immediately remove the test specimen from the plastometer and allow it to cool exactly 1 min at 23 ± 2°C. Then measure the height of the specimen with the two test surfaces using the dial micrometer gage specified in 5.1.5. Record this reading as \( h_2 \).

10.2.2 Calculate recovery, \( R_A \), as follows:

\[
R_A = 100(h_2 - h_1)
\]

(2)

10.3 Procedure B:

10.3.1 Follow the procedure described in Section 9 for determining plasticity, except that the test specimen shall be compressed to a height of 5 mm for 30 s. This may be done by placing three spacers 5.00 ± 0.01 mm in thickness on two thicknesses of test surface at suitable positions between the plates to ensure parallelism and prevent misalignment of the plates.

10.3.2 At the end of the compression period, remove the specimen from between the plates and permit it to recover for 5 min at the test temperature.

10.3.3 At the end of the 5-min recovery period, measure the height of the specimen with the micrometer gage specified in 5.1.5. Record as \( h_3 \).

10.3.4 Calculate recovery, \( R_B \), as follows:

\[
R_B = 100(h_3 - 5)
\]

(3)

11. Report

11.1 Report the following information:

11.1.1 Sample Identification:

11.1.1.1 A full description of the sample and its origin,

11.1.1.2 Compound details, where appropriate,

11.1.1.3 Preparation method of the test piece (plied or solid), and

11.1.1.4 Time between preparation and test.

11.1.2 Test Conditions:

11.1.2.1 Test surface used,

11.1.2.2 Test temperature,

11.1.2.3 Time in minutes the force was applied to the test piece,

11.1.2.4 Recovery time in minutes, and

11.1.2.5 Date of the test.

11.1.3 Test Results:

11.1.3.1 Plasticity number, \( P \), and

11.1.3.2 Recovery number, \( R_A \) or \( R_B \).

12. Precision and Bias

12.1 These precision and bias statements have been prepared in accordance with Practice D 4483. Please refer to Practice D 4483 for terminology and other testing and statistical concept explanations.

12.2 The Type 1 precision was determined from an inter-laboratory study of three compounds that were mixed in one laboratory. Four laboratories prepared samples and tested each compound.

12.3 A “test result” for this plasticity and recovery testing represents one measurement with a parallel plate plastometer.

12.4 Table 1 lists the precision data. A Type 1 precision, as defined in Practice D 4483, includes as components of variation the specimen preparation and testing operation within each laboratory, but not the mixing or compound preparation components of variation.

12.5 The precision of this test method may be expressed in the format of the following statements which use an “appropriate value” of \( r \), \( R \), \( r \), or \( R \), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of \( r \) or \( R \) associated with a mean level in Table 1 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

12.6 Repeatability—The repeatability, \( r \), 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, \( R \), 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 Repeatability and reproducibility expressed as a percent of the mean level, \( r \) and \( R \), have equivalent application statements as above for \( r \) and \( R \). For the \( r \) and \( R \) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

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 values of plasticity and recovery are exclusively defined by the test method. Bias, therefore, cannot be determined.

13. Keywords

13.1 elastic properties; flow properties; parallel plate plastometer; plasticity; plasticity and recovery; plastometer; Williams plastometer
<table>
<thead>
<tr>
<th>Test</th>
<th>Test Temperature, °C</th>
<th>Range of Values[^a]</th>
<th>Within Laboratory[^b]</th>
<th>Between Laboratory[^b]</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>S_r</td>
<td>r</td>
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<tr>
<td>Plasticity</td>
<td>70</td>
<td>415–755</td>
<td>22.7</td>
<td>64.2</td>
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<td>15.8</td>
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</tbody>
</table>

[^a]: Units = mm × 100.
[^b]: S_r = repeatability standard deviation, in measurement units.
      r = repeatability in measurement units.
      (r) = repeatability (relative) percent.
      S_R = reproducibility standard deviation, in measurement units.
      R = reproducibility in measurement units.
      (R) = reproducibility (relative) percent.
[^c]: Midpoint of range used to calculate (r) and (R).