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Standard Test Method for Rubber Property—Measurement of the Viscous and Elastic Behavior of Unvulcanized Raw Rubbers and Rubber Compounds by Compression Between Parallel Plates[1]

This standard is issued under the fixed designation D6049; 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 is an adaptation of the German Standard DIN 53514, a further development of the former "Defo Test" (see Appendix X1).

1.2 This test method is capable of measuring and characterizing the rheological behavior (viscosity and elasticity) of unvulcanized raw rubbers and rubber compounds, relating to the macro structure of rubber polymers (average molecular weight, molecular weight distribution, long chain branching, and micro- and macro-gel).

1.3 The viscosity and elasticity of unvulcanized rubbers and rubber compounds are determined by subjecting cylindrical test pieces to a compression/recovery cycle. The dependency on shear rate at constant shear stress is evaluated and the material fatigue behavior is determined in repeat cycle testing.

1.4 The non-Newtonian viscous and elastic behavior of rubbers and rubber compounds can also be evaluated.

1.5 Statistical evaluation of the test data provides an indication of data variation, which may be employed as an estimate of the homogeneity of the material tested.

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

1.7 *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]

D297 Test Methods for Rubber Products—Chemical Analysis

D926 Test Method for Rubber Property—Plasticity and Recovery (Parallel Plate Method)

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D6048 Practice for Stress Relaxation Testing of Raw Rubber, Unvulcanized Rubber Compounds, and Thermoplastic Elastomers

2.2 *DIN Standards:*[3]

DIN 53514 Testing of Rubber—Determination of Viscosity and Elasticity Related Numbers of Raw Rubber and Rubber Mixes in a Compression Test between Parallel Plates

DIN 53523, Part 1 Testing of Rubber and Elastomers—Testing with the Mooney Shearing Disk Viscometer; Preparation of Test Pieces

2.3 *ISO Standards:*[4]

ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests

ISO 7323 Rubber—Raw and Unvulcanized Compounded—Determination of Plasticity Number and Recovery Number; Parallel Plate Method

3. Terminology

3.1 *Definitions*—The following terms appear in logical order for the sake of clarity.

3.1.1 *Multiple Compression Force Test*—refer to Section 10 for more details.

3.1.1.1 *viscosity number,* V_{10} (Ns)—the product of the force F in N required to compress a test piece the final 0.5 mm (0.02 in.) in a 6.0 mm (0.24 in.) total compression cycle (from 13.0 to 7.0 mm (0.51 to 0.28 in.)) and the compression time dt_1 equaling 10 s.

[1] This test method is under the jurisdiction of Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.12 on Processability Tests.

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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] Available from Deutsches Institut für Normung, Burggrafenstr 6, D10787 Berlin 30, Germany.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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3.1.1.2 *total compression time,* t_T—the time in s required to compress a test piece the full 6.0 mm (0.24 in.), that is, from 13.0 to 7.0 mm (0.51 to 0.28 in.).

3.1.1.3 *elasticity number,* DE_{30}—the elastic recovery expressed in units of 0.1 mm (0.004 in.) calculated from the height h_2 of the test piece after compression from 13.0 to 7.0 mm (0.51 in. to 0.28 in.) within 30 s followed by a recovery period of 30 s.

3.1.1.4 *non-Newtonian viscosity exponent,* n_1—the slope of the line in a double log plot of the viscosity versus the shear rate; n_1 is dimensionless and always negative.

3.1.1.5 *elasticity coefficient, m*—the slope of the line in a plot of the elasticity number *DE* (see 3.1.2.3) versus the log of the shear rate; the dimension of *m* is mm (in units of 0.1 mm per decade).

3.1.1.6 *test data variation number,* s_v—the average standard deviation of the data points of the individual test pieces, from the regression line of the viscosity number in accordance with 3.1.1.4.

3.1.1.7 *test data variation number,* s_e—the average standard deviation of the data points of the individual test pieces from the regression line of the elasticity number in accordance with 3.1.1.5.

(*a*) *Discussion*—Both numbers, s_v and s_e, can be used to (*1*) characterize the homogeneity of the test pieces or (*2*) provide an estimate of the test precision where test pieces are known to be homogeneous. In addition, s_e can indicate rare cases of nonlinearity.

3.1.1.8 *viscous material fatigue,* ΔV_{21}—the decrease in percent of V_{10} for the first compression compared to V_{10} for the second compression.

(*a*) *Discussion*—When the first compression/recovery cycle (compression time = recovery time) is repeated, the viscosity number of the second cycle will be lower than that of the first cycle.

3.1.1.9 *elastic material fatigue,* Q_{21}—the quotient of the recovery times, $(t_{RV})_2$ and $(t_{RV})_1$, after the second and first compression/recovery cycle, allowing the test piece to reach equivalent recovery heights in both cycles; Q_{21} is dimensionless.

(*a*) *Discussion*—In the second compression/recovery cycle the test piece requires a longer recovery time to reach the same height as in the first cycle.

3.1.2 *Single Compression Force Test*—refer to Section 10 for more details.

3.1.2.1 *viscosity*—specific to this test method, the ratio of compression force to compression time, where compression force and compression time are proportional to shear stress and shear rate, respectively.

(*a*) *Discussion*—The compression force *F* specified for a particular material determines the deformation stress, and the compression time dt_1 required to compress a test piece the final 0.5 mm (0.02 in.) in a 6.0 mm (0.24 in.) total compression cycle (from 13.0 to 7.0 mm (0.51 to 0.28 in.)) determines the deformation rate.

3.1.2.2 *total compression time,* t_T—refer to 3.1.1.2.

3.1.2.3 *elastic recovery, DE*—calculated from the height h_2 of the test piece after compression from 13.0 to 7.0 mm (0.51 in. to 0.28 in.) followed by a recovery period, equal in length to the compression time, expressed in units of 0.1 mm (0.004 in.).

3.1.2.4 *non-Newtonian viscosity number, q*—the quotient of the two compression time values dt_1 and t_T (see 3.1.2.1 and 3.1.1.2); *q* is dimensionless.

3.1.2.5 *test data variation*—the standard deviations s_r for the compression time dt_1 (3.1.2.1) and s_r for the elastic recovery *DE* (3.1.2.3).

(*a*) *Discussion*—see 3.1.1.7.

3.1.2.6 *viscous material fatigue,* $\Delta\ dt_{21}$—the decrease in percent of the compression time dt_2 versus the compression time dt_1.

(*a*) *Discussion*—When the first compression/recovery cycle (compression time = recovery time) is repeated with the same force, the compression time dt_2 of the second cycle will be lower than the compression time dt_1 of the first cycle.

4. Summary of Test Method

4.1 This test method provides procedures for preparing cylindrical test pieces of specified diameter and height from unvulcanized raw rubbers and rubber compounds and for testing their viscous and elastic behavior at a specified temperature in a compression/recovery operation between parallel plates.

4.2 The compression device is mounted in an environmental chamber. The preferred test temperature is 105°C (221°F).

4.3 The change in test piece height is measured under a constant compression force and in the recovery phase after releasing the force.

4.4 Viscosity is characterized by compression force and compression time, elasticity by the recovery height of the test piece after release of the compression force (recovery phase). Material fatigue is measured through repeat compression/recovery cycles.

4.5 The test can be performed with multiple compression forces for a more comprehensive evaluation of the viscous and elastic properties, including non-Newtonian behavior, or with a time saving single compression force, preferred in quality control to test primarily uniformity of viscosity and elasticity.

4.6 Statistical evaluation of the test results gives an indication of data scatter, and permits also an assessment of the homogeneity of the material under test.

5. Significance and Use

5.1 The viscous and elastic behavior of unvulcanized rubbers and rubber compounds is of paramount importance in rubber manufacturing, since it affects processing, such as mixing, calendering, extrusion, and molding. The uniformity of these properties is equally important, as fluctuations will cause upsets in manufacturing processes.

5.2 A test capable of measuring viscosity and elasticity of unvulcanized rubbers and rubber compounds, including their uniformity and prediction of processing behavior, is therefore highly desirable (see Practice D6048 for further interpretation).

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5.3 Compared to many other rheological tests, this test method measures viscosity and elasticity related parameters under conditions of low shear and has a high discriminating power. It can detect small rheological differences. A full discussion of the principles behind stress relaxation testing is given in Practice D6048.

5.4 Test results of this test method may be useful in predicting processability, but correlation with actual manufacturing processes must be established in each individual case, since conditions vary too widely.

5.5 This test method is suitable for specification compliance testing, quality control, referee purposes, and research and development work.

6. Interferences

6.1 For reliable test results it is important that test pieces are of accurate dimensions, are free of air inclusions and blisters, and contain negligible residual stresses.

6.2 Although this test method is practically unlimited in testing unvulcanized rubbers, it may be necessary to consider smaller test pieces in the case of very hard and rigid substances.

7. Apparatus[5]

7.1 *Compression Device:*

7.1.1 Two circular platens with a diameter of 30.0 ± 0.1 mm (1.181 ± 0.004 in.) and a thickness of at least 2 mm (0.08 in.) mounted in an angle iron frame, are facing each other horizontally. The upper platen can be vertically moved and raised at least 14 mm (0.55 in.) above the lower platen to permit insertion of the 13-mm (0.51-in.) high test piece.

7.1.2 The upper platen shall be able to move nearly without friction, that is, it must smoothly descend under the contact force specified in 7.1.5.

7.1.3 The upper platen shall be capable of applying a compressive force between 1 and 800 N (0.22 and 180 lbf) ± 0.5 % to the test piece within 1 s. The force should be applied rapidly but not abruptly.

7.1.4 After the height of the test piece has been reduced from 13.0 to 7.0 mm (0.51 to 0.28 in.) the compression force shall be released in less than 0.5 s.

7.1.5 A residual compression force of 0.040 ± 0.005 N (0.009 ± 0.001 lbf) shall always be maintained by the upper platen to warrant an intimate contact with the test piece prior to compression and during the recovery cycle. The mass of the upper platen must be taken into account and compensated for.

7.1.6 The position of the upper platen shall be continuously recorded during the test to the nearest 0.01 mm (0.0004 in.) in at least 0.05 s intervals. The starting time of the compression cycle, the time of reaching compression heights of 7.5 and 7.0 mm (0.30 and 0.28 in.), and the recovery times shall be recorded to the nearest 0.05 s.

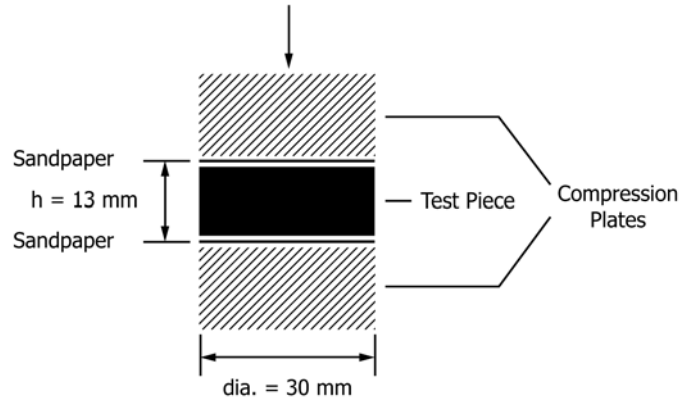

FIG. 1 Test Piece Arrangement

7.1.7 The compressed test pieces shall only be in contact with the two platens and the platens shall be kept free of contamination. The test piece arrangement is illustrated in Fig. 1.

7.2 *Test Chamber:*

7.2.1 The compression device shall be contained in a test chamber that can be temperature controlled between 20 and 170 ± 1°C (68 and 338 ± 2°F).

7.2.2 The test chamber shall have holding devices to accommodate test pieces of 13.0 ± 0.1 mm (0.51 ± 0.004 in.) plus twice the thickness of the sandpaper in height for conditioning.

7.3 *Recording and Evaluation of Test Results:*

7.3.1 The test shall be run and controlled automatically, including recording of the compression heights and times.

7.3.2 Evaluation of test results is best carried out immediately after the test. The use of a computer to control the test sequence and record data is recommended.

8. Preparation of Test Pieces

8.1 The cylindrical test piece shall have a height of 13.0 ± 0.1 mm (0.51 ± 0.004 in.) and a diameter of 30.0 ± 0.1 mm (1.18 ± 0.004 in.). It is typically produced by molding and should be free of blisters and internal residual stresses. Sandpaper disks[6] shall be molded to the top and bottom of the cylindrical test piece to stabilize the surfaces and ensure an even transmission of the compression forces without slippage. The sandpaper facing also prevents fouling of the platens of the compression device and can be used for identification purposes.

8.2 *Test Pieces from Rubber in Bale Form:*

8.2.1 Sheets of approximately 2 mm (0.08 in.) in thickness are sliced from the bale, and disks of approximately 30 mm (1.18 in.) in diameter are die cut from these sheets. The disks are loosely plied up to produce a specimen sufficiently large in volume for a test piece, including a mold flash of 0.05 to 0.35 g.

[5] A suitable instrument can be obtained under the name Defo-Elastometer (System Bayer) from Haake, Inc., West Century Road, Paramus, NJ 07652, or Haake G.m.b.H., Dieselstr., D76227 Karlsruhe, Germany.

[6] A suitable sandpaper is 3M 230N (120 grit, aluminum oxide) available from 3M Abrasive Systems Div., 3M Center, St. Paul, MN 55144, or 3M 204 (120 grit, aluminum oxide) available from Fa. Krueckemeyer, D57225 Wilmsdorf, Germany.

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