



Designation: D8362 – 21

Standard Test Method for Rubber—Determination of the Degree of Silanization by Dynamic Strain Softening¹

This standard is issued under the fixed designation D8362; 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 used to rheologically determine the degree of silanization in a silica filled compound. Silanization is the chemical reaction related to covering the silica surface with organosilanes for a better incorporation of silica into rubber. Bifunctional silanes can provide coupling between silica and rubber for enhanced rubber reinforcement.

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

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

D6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

D8059 Test Method for Rubber Compounds—Measurement of Unvulcanized Dynamic Strain Softening (Payne Effect)

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 D11.20 on Compounding Materials and Procedures.

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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.

Using Sealed Cavity Rotorless Shear Rheometers

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *complex shear modulus, G^* , n* —ratio of shear stress to shear strain, where both may include elastic and viscous components.

3.1.2 *elastic (storage) shear modulus, G' , n* —ratio of the shear stress component in phase with the shear strain, to the shear strain.

3.1.3 *Payne Effect, n* —in the low amplitude dynamic testing of filled rubbers, the decrease in modulus (G' or E') as the amplitude of deformation is increased.

3.1.4 *silanization, n* —chemical reaction of organosilane molecules with the silanol groups of synthetic amorphous silica covering its surface and improving its compatibility with rubber leading to an enhanced dispersion.

3.1.5 *viscous (loss) shear modulus, G'' , n* —ratio of the shear stress component 90° out of phase with the shear strain, to the shear strain.

4. Summary of Test Method

4.1 This test method covers the determination of the degree of silanization in a silica-filled rubber using a rotorless oscillating shear rheometer for the measurement of the strain softening (Payne Effect) before and after thermal annealing at 160°C.

5. Significance and Use

5.1 This test method is based on the Payne Effect. It is a reasonable approach to evaluate the compound's filler-network formation to quantify the degree of silanization in a silica-filled rubber using a rotorless oscillating shear rheometer for the measurement of the strain softening.

5.2 This test is based on silane's capability to suppress silica filler network formation (flocculation) and correlates this behavior to the resultant degree of silanization.

5.3 The optimal mixing conditions can be selected based on the silanization/property correlations to give desired compound properties. The degree of silanization quantified by this test

method is used for controlling and predicting compound properties regardless of the mixing scheme or equipment, or both.

6. Apparatus

6.1 *Rotorless Oscillating Shear Rheometer*, according to Test Method [D6204](#).

7. Test Specimen

7.1 A test specimen taken from a sample shall be between 5 and 6 cm³ for the sealed cavity oscillating rheometer according to Test Method [D8059](#).

7.2 The test specimen is an uncured silica-filled compound containing silane (that is, the last non-productive stage, a master batch containing no ZnO and no curatives). Two additional reference compounds are required, which are comparable, silica-filled compounds without silane and with an octyl silane, respectively.

7.3 Since the test specimens are uncured rubber specimens, a thermal conditioning of the mixed stock specimen is needed until it has reached room temperature (23 ± 3) °C throughout. It is recommended to condition the compound after mixing at room temperature for 16 h.

7.4 The uncured rubber test specimen should be tested as received, that is unmassed (not milled). The rubber compound shall be in the form of a sheet, at room temperature, and as free of air as possible.

8. Procedure

8.1 The method measures the difference in the strain softening (Payne Effect) before and after the uncured rubber compound is annealed at 160°C for 20 min.

8.2 Place the test specimen into the test chamber of the rotorless shear rheometer.

8.3 Precondition the test specimen under mechanical and thermal histories in analogy to a productive mix. The preconditioning scheme is listed below and is to be included in the test program of the rheometer.

8.3.1 75°C for 1 min at 1Hz and 200 % strain.

8.3.2 93°C for 1 min at 1Hz and 200 % strain.

8.3.3 102°C for 0.3 min at 1Hz and 200 % strain.

8.4 Precondition the sample at 45°C for 4 min, and subsequently perform a strain sweep at 45°C and 0.1 Hz in the range of 0.25 to 500 %. Report the value for Δ' between 0.25 and 500 % according to [Eq 1](#):

$$\Delta G' = G'_{0.25\%} - G'_{500\%} \quad (1)$$

8.5 Replace the test specimen by a new sample of the same compound.

8.6 Precondition the new test specimen according to [8.3](#).

8.7 Increase the temperature to 160°C and hold it for 20 min as the annealing step.

8.8 Perform a strain sweep with the new test specimen according [8.4](#).

8.9 Repeat the steps according [8.2 – 8.8](#) using the two reference compounds without silane and with an octyl silane, respectively.

9. Calculation or Interpretation of Results

9.1 The capability of suppressing the filler flocculation by a silane is calculated by measuring the difference in the Payne Effect values $\delta(\Delta G')$ of the last non-productive stage stock from the strain sweep taken before and after thermal annealing at 160°C where:

$$\delta(\Delta G') = \Delta G'_{\text{before annealing}} - \Delta G'_{\text{after annealing}} \quad (2)$$

9.2 The degree of silanization (expressed as Silanization%) of a silane containing compound can be calculated using the following equation:

$$\text{Silanization}\% = \frac{\delta(\Delta G')_{\text{no coverage}} - \delta(\Delta G')_{\text{tested coverage}}}{\delta(\Delta G')_{\text{no coverage}} - \delta(\Delta G')_{\text{reference coverage}}} \quad (3)$$

where:

$\delta(\Delta G')_{\text{tested coverage}}$ is obtained from the test compound,

$\delta(\Delta G')_{\text{no coverage}}$ is obtained from the reference compound without silane, and

$\delta(\Delta G')_{\text{reference coverage}}$ is obtained from the reference compound containing an octyl silane.

10. Report

10.1 Identification of the test compound, and the reference compounds without silane and with an octyl silane, respectively.

10.2 Type and model of oscillating rheometer.

10.3 $\Delta G'$ values for the compounds in [8.1](#).

10.4 $\delta(\Delta G')$ values for the compounds in [8.1](#).

10.5 Degree of Silanization in %.

11. Precision and Bias

11.1 Precision and bias statements according to Practice [D4483](#) have not been generated but will be available within five years of publication of the test method.

12. Keywords

12.1 Payne Effect; rotorless oscillating shear rheometer; silane; silanization; silica; strain softening