



Designation: D8491 – 23

Standard Test Method for Recovered Carbon Black—Rheological Non-Linearity of a Rubber Compound by Fourier Transform Rheology¹

This standard is issued under the fixed designation D8491; 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 provides a measure of rheological non-linearity of a rubber compound filled with rCB to assess its reinforcement capabilities. This test method requires the use of a sealed cavity rotorless oscillating shear rheometer for the measurement of the torque with increasing sinusoidal strain applied to an uncured rubber compound containing significant amounts of colloidal fillers, such as recovered carbon black, alone or as blend with virgin carbon black.

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

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

D3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures

D3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)

¹ This test method is under the jurisdiction of ASTM Committee D36 on Recovered Carbon Black (rCB) and is the direct responsibility of Subcommittee D36.70 on rCB Testing in Rubber.

Current edition approved June 15, 2023. Published July 2023. Originally approved in 2022. Last previous edition approved in 2022 as D8491 – 22. DOI: 10.1520/D8491-23.

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.

D3896 Practice for Rubber From Synthetic Sources—Sampling

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) Using Sealed Cavity Rotorless Shear Rheometers

3. Significance and Use

3.1 This test method provides a measure of rheological non-linearity for filled rubber compounds under oscillatory shear conditions: the normalized 3rd harmonic of the torque $I_{3/1}$.

3.2 Rheological linearity means that the modulus is a function of frequency only. The shear modulus dependency on both frequency and amplitude of a dynamic deformation is a non-linear, rheological effect. Filled rubbers show a strain dependency of the modulus known as Payne effect. A test method for evaluating the Payne effect can be found in Test Method **D8059**.

3.3 One of the main contributions to the Payne effect is the so-called polymer-filler interaction in the range of mid amplitude oscillation shear, MAOS. The MAOS amplitude range is defined as the range where $I_{3/1}$ is already measurable and increases according to a scaling law, for example, $I_{3/1} \sim \gamma^2$. It has been shown that FT rheological measurements are very sensitive to changes in this interaction, and that it is possible to quantify the influence of the filler type and content on the nonlinearity. Interactions between the polymer and particle surface and between the filler particles create a network structure in the compound that not only increases the elasticity of the system but also introduces nonlinear contributions to the stress response.³

4. Apparatus

4.1 Torsion Strain Rotorless Oscillating Rheometer with a Sealed Cavity in accordance with Test Method **D6204**, operated in the test mode called strain sweep, in which the strain

³ Schwab, L. et al., "Fourier-Transform Rheology of Unvulcanized, Carbon Black Filled Styrene Butadiene Rubber," *Macromol. Mater. Eng.*, 2016, 301, pp. 457–468.