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.

Designation: D6204 – 19a

Standard Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers[1]

This standard is issued under the fixed designation D6204; 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 covers the use of a rotorless oscillating shear rheometer for the measurement of the flow properties of raw rubber and unvulcanized rubber compounds. These flow properties are related to factory processing.

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

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

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D3896 Practice for Rubber From Synthetic Sources—Sampling

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

D5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

D6601 Test Method for Rubber Properties—Measurement of Cure and After-Cure Dynamic Properties Using a Rotorless Shear Rheometer

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *complex shear modulus, G*, n*—the ratio of peak amplitude shear stress to peak amplitude shear strain; mathematically, $G^* = [(S^*/Area)/Strain] = (G'^2 + G''^2)^{1/2}$.

3.1.2 *complex torque, S*, n*—the peak amplitude torque response measured by a reaction torque transducer for a sinusoidally applied strain; mathematically, S^* is computed by $S^* = (S'^2 + S''^2)^{1/2}$.

3.1.3 *dynamic complex viscosity η*, n*—the ratio of the complex shear modulus, G^* to the oscillation frequency, ω, in radians per second.

3.1.4 *elastic torque, S', n*—the peak amplitude torque component that is in phase with a sinusoidally applied strain.

3.1.5 *loss angle, δ, n*—the phase angle by which the complex torque (S^*) leads a sinusoidally applied strain.

3.1.6 *loss factor,* tan *δ, n*—the ratio of loss modulus to storage modulus, or the ratio of viscous torque to elastic torque; mathematically, $\tan \delta = G''/G' = S''/S'$.

3.1.7 *loss shear modulus G'', n*—the ratio of (viscous) peak amplitude shear stress to peak amplitude shear strain for the torque component 90° out of phase with a sinusoidally applied strain; mathematically, $G'' = [(S''/Area)/Peak\ Strain]$.

3.1.8 *real dynamic viscosity, η, n*—the ratio of the loss shear modulus, G'' to the oscillation frequency, ω, in radians per second.

3.1.9 *storage shear modulus, G', n*—the ratio of (elastic) peak amplitude shear stress to peak amplitude shear strain for the torque component in phase with a sinusoidally applied strain; mathematically, $G' = [(S'/Area)/Peak\ Strain]$.

3.1.10 *viscous torque, S'', n*—the peak amplitude torque component, which is 90° out of phase with a sinusoidally applied strain.

[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 D11.12 on Processability Tests.

Current edition approved Dec. 1, 2019. Published December 2019. Originally approved in 1997. Last previous edition approved in 2019 as D6204 – 19. DOI: 10.1520/D6204-19A.

[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.

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4. Summary of Test Method

4.1 A rubber test specimen is contained in a die cavity that is closed and maintained at an elevated temperature. The cavity is formed by two dies, one of which is oscillated through a rotary amplitude. This action produces a sinusoidal torsional strain in the test specimen, resulting in a sinusoidal torque, which measures a viscoelastic quality of the test specimen. The test specimen can be either a raw natural or synthetic rubber or an uncured rubber compound.

4.2 These viscoelastic measurements can be made based on *(1)* a *frequency sweep*, in which the frequency is programmed to change in steps under constant strain amplitude and temperature conditions, *(2)* a *strain sweep*, in which the strain amplitude is programmed to change in steps under constant frequency and temperature conditions, or *(3)* a *temperature sweep*, in which the temperature is programmed to either increase or decrease under constant strain amplitude and frequency conditions. A *timed test* may also be performed in which a sinusoidal strain is applied for a given time period under constant strain amplitude, frequency, and temperature conditions.

4.2.1 For a frequency sweep test, the instrument is typically programmed to increase the frequency with each subsequent step change. For a strain sweep test, the instrument is usually programmed to increase the strain amplitude with each subsequent step change. This is done to minimize the influence of prior test conditions on subsequent test steps. For temperature sweeps, the temperature may be programmed either to increase or decrease with each subsequent step change, depending on the effects to be studied. The results from increasing frequency, strain amplitude, or temperature may not be the same as results from decreasing these test parameters.

4.3 Rheological properties are measured for each set of frequency, strain, and temperature conditions. These properties can be measured as combinations of elastic torque *S'*, viscous torque *S"*, storage shear modulus *G'*, loss shear modulus *G"*, tan δ, complex dynamic viscosity η*, and real dynamic viscosity η'.

4.4 This standard is organized in three different parts (A, B, and C), which can be run in the following combinations:

A
B
A, B
A, B, C
A, C
B, C
C

4.5 These three parts are described below:

4.5.1 Part A is a rapid three-point frequency sweep performed at a low strain of 7 % to relate to differences in average molecular weight, molecular weight distribution, and long chain branching for raw elastomers and to relate to differences in flowability, shear thinning, and die swell for mixed batches.

4.5.2 Part B is a rapid two-point frequency sweep performed at a moderate strain of 100 % (or higher) to relate to gel differences with raw elastomers and to relate to differences in higher shear rate viscosity and die swell for mixed batches. The higher applied strain is commonly needed to help break up gel structure in some raw elastomers and break up filler aggregate networks for mixed batches. Although 100 % strain is the more common test condition, significantly higher test sensitivity is possible by performing this frequency sweep at 200 % strain or higher.

4.5.3 Part C is a linear ramped temperature rise from processing temperature (typically 100°C) to cure temperature (usually 140, 160, or 180°C) in a predetermined time period. This ramped temperature cure is performed to enhance statistical test sensitivity to real differences in scorch times providing better scorch control than traditional isothermal cure tests (Test Method D5289), and to provide a controlled transition from Part A or Part B tests, or both, in this method to a cure test.

5. Significance and Use

5.1 This test method is used to measure viscoelastic properties of raw rubber as well as unvulcanized rubber compounds. These viscoelastic properties may relate to factory processing behavior.

5.2 This test method may be used for quality control in rubber manufacturing processes and for research and development testing of raw rubber and rubber compounds. This test method may also be used for evaluating compound differences resulting from the use of different compounding materials.

6. Apparatus

6.1 *Torsion Strain Rotorless Oscillating Rheometer With a Sealed Cavity*—This type of rheometer measures the elastic torque *S'* and viscous torque *S"* produced by oscillating angular strain of set amplitude and frequency in a completely closed and sealed test cavity.

6.2 *Sealed Die Cavity*—The sealed die cavity is formed by two conical surface dies. In the measuring position, the two dies are fixed a specified distance apart so that the cavity is closed and sealed (see Fig. 1).

6.3 *Die Gap*—For the sealed cavity, no gap shall exist at the edges of the dies. At the center of the dies, the die gap shall be set at 0.45 ± 0.05 mm.

6.4 *Die Closing Mechanism*—For the sealed cavity, a pneumatic cylinder or other device shall close the dies and hold them closed during the test with a force not less than 11 kN (2500 lbf).

6.5 *Die Oscillating System*—The die oscillating system consists of a direct drive motor that imparts a torsional oscillating movement to the lower die in the cavity plane.

6.5.1 The oscillation amplitude can be varied, but a selection of ±0.5° arc (7.0 % shear strain) is preferred for frequency sweep tests. The oscillation frequency can be varied between 0.03 and 30 Hz.

6.6 *Torque Measuring System*—The torque measuring system shall measure the resultant shear torque.

6.6.1 The torque measuring device shall be rigidly coupled to one of the dies, any deformation between the die and device shall be negligibly small, and the device shall generate a signal that is proportional to the torque. The total error resulting from

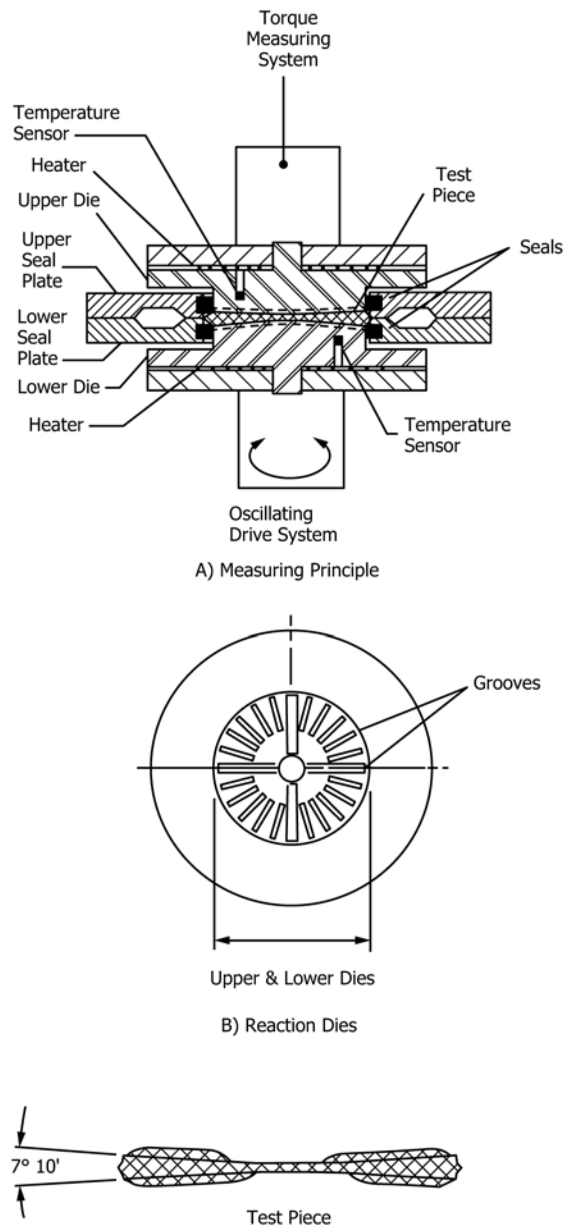

FIG. 1 Typical Sealed Torsion Shear Rotorless Curemeter

zero point error, sensitivity error, linearity, and repeatability errors shall not exceed 1 % of the selected measuring range.

6.6.2 The torque recording device shall be used to record the signal from the torque measuring device and shall have a response time for full scale deflection of the torque scale of 1 s or less. The torque shall be recorded with an accuracy of ±0.5 % of the range. Torque recording devices may include analog chart recorders, printers, plotters, or computers.

6.6.3 A reference torque device is required to calibrate the torque measurement system. A torque standard shall be used to calibrate the torque measuring system at the selected angular displacement by clamping a steel torsion rod to the oscillating and the torque measuring dies of the torsion shear rheometer (see Fig. 2). The reference values for angular displacement and corresponding torque shall be established by the manufacturer for each torque standard.

6.7 *Reference Test Temperature*—The standard reference test temperature shall be 100°C (212°F) or 125°C (257°F) for processability measurements. Tests may be carried out at other temperatures when appropriate.

6.8 *Temperature Control System*—This system shall permit the set point temperature to be varied between 40°C and 220°C with an accuracy of ±0.3°C or better.

6.8.1 The dies shall heat to the set point temperature in 1.0 min or less from closure of the test cavity. Once the initial heating-up time has been completed, die temperature shall not vary by more than ±0.3°C for the remainder of a test at a set temperature. When the set temperature is changed in a programmed temperature sweep (step changes in temperature), rheological measurements shall not be recorded until the die temperatures are within ±0.3°C of the new set temperature for