Designation: D 6204 – 99

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

This standard is issued under the fixed designation D 6204; 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 the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1349 Practice for Rubber—Standard Temperatures for Testing[2]

D 1485 Test Methods for Rubber from Natural Sources—Sampling and Sample Preparation[2]

D 3896 Practice for Rubber from Synthetic Sources—Sampling[2]

D 4483 Practice for Preparing Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

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.

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, a *frequency sweep* in which the frequency is programmed to change in steps under constant strain amplitude and temperature conditions, a *strain sweep* in which the strain amplitude is programmed to change in steps under constant frequency and temperature conditions, or, 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

[1] This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.12 on Processability Tests.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

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'.

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 should 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 which is proportional to the torque. The total error resulting from 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 may 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 if required. Temperatures should be selected in accordance with Practice D 1349.

6.8 *Temperature Control System*—This system shall permit the reference 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, rheological measurements should not be recorded until the die temperatures are within ± 0.3°C of the new set temperature for at least 30 s.

6.8.2 Temperature distribution within the test piece shall be as uniform as possible. Within the deformation zone, a tolerance of ± 1°C of the average test piece temperature shall not be exceeded.

6.8.3 Die temperature is determined by a temperature sensor used for control. The difference between the die temperature and the average test piece temperature shall not be more than 2°C. Temperature measurement accuracy shall be ± 0.3°C for the die temperature sensor.

7. Test Specimen

7.1 A test specimen taken from a sample shall be between 5 and 6 cm^3 for the sealed cavity oscillating rheometer. The specimen volume should exceed the test cavity volume by a small amount, to be determined by preliminary tests. Typically, specimen volume should be 130 to 150 % of the test cavity volume. Once a target weight for a desired volume has been established, specimen weights should be controlled to within ± 0.5 g for best repeatability. The initial test specimen shape should fit well within the perimeter of the test cavity.

7.2 *Raw Rubber Specimens*—Condition the specimen obtained in accordance with Methods D 1485 or Practice D 3896 until it has reached room temperature (23 ± 3°C (73 ± 5°F)) throughout. The raw rubber test specimen should be tested as received, that is unmassed (not milled).

7.2.1 Raw rubber test specimens in a sealed cavity oscillating rheometer must be pre-conditioned in the instrument before rheological measurements are made to improve test precision. A programmed pre-conditioning step shall consist of oscillating the specimen at 0.5 Hz, ± 2.8 % strain, 100°C (or 125°C) for the time interval specified in Table 1.

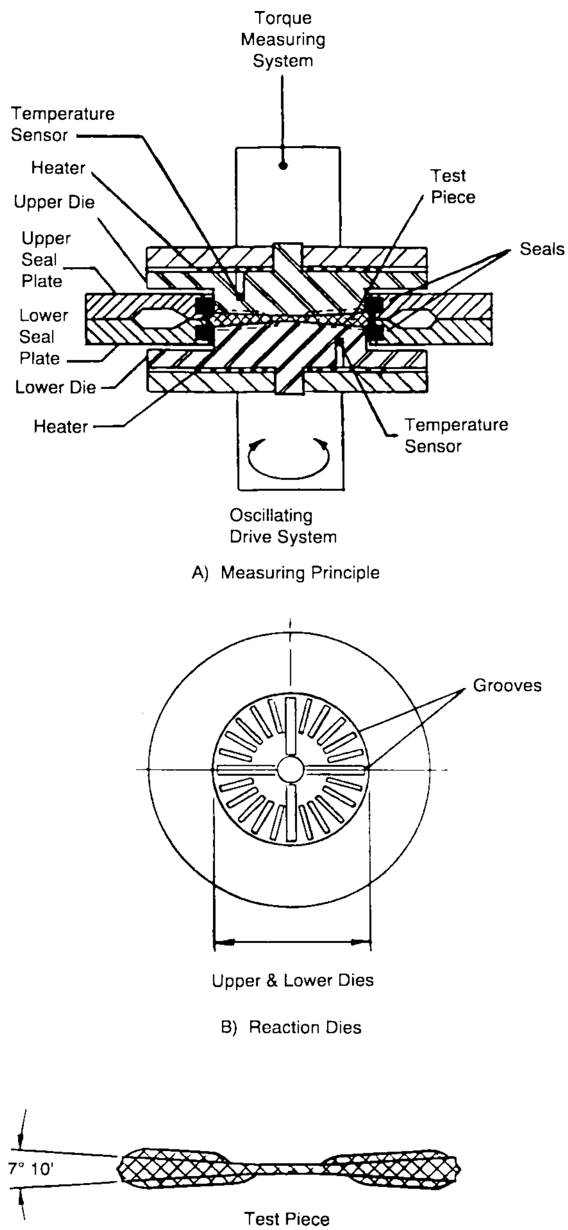

FIG. 1 Typical Sealed Torsion Shear Rotorless Curemeter

7.3 *Compounded Rubber Specimens*—Test specimens shall be taken from a rubber compound as required by the mixing method or other sampling instructions. Rubber compounds with or without curatives may be tested.

7.3.1 The rubber compound shall be in the form of a sheet, at room temperature, and as free of air as possible.

7.3.2 Compounded rubber test specimens in a sealed cavity oscillating rheometer must be pre-conditioned in the instrument before rheological measurements are made to improve test precision. A programmed pre-conditioning step shall consist of oscillating the specimen at 0.5 Hz, ± 2.8 % strain, 100°C for the time interval specified in Table 1.

8. Procedure

8.1 Select the frequency, strain, temperature and time for the conditioning step as listed in Table 1.

8.2 Select the frequency steps and the strain and temperature conditions for the frequency step as listed in Table 1.

8.3 Program a test configuration that incorporates these conditions and store on the instrument computer operating system.

8.4 Load the test configuration to run the test.

8.5 Enter specimen identification.

8.6 Wait until both dies are at the initial test temperature. Open the test cavity and visually check both upper and lower dies for cleanliness. Clean the dies if necessary. Place the test specimen on the center of the lower die and close the dies within 20 s.

9. Report

9.1 Report the following information:

9.1.1 A full description of the sample or test specimen(s), or both, including their origin.

9.1.2 Type and model of oscillating rheometer.

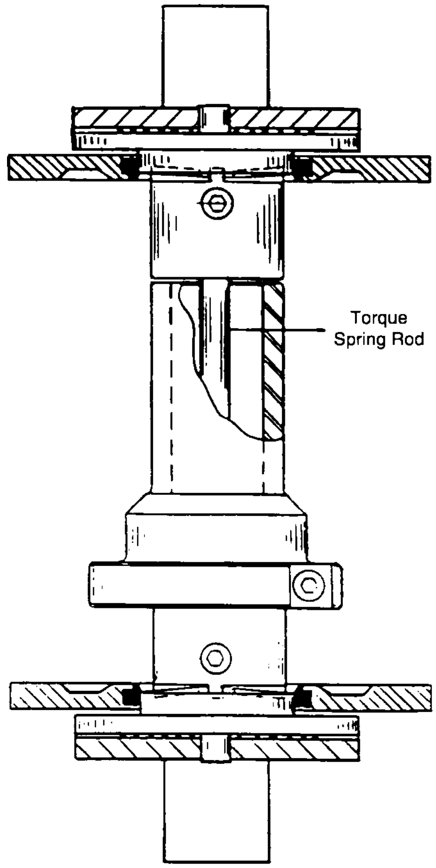

FIG. 2 Typical Torque Standard Calibration Devics for Torsion Shear Curemeters

TABLE 1 Standard Test Conditions for Oscillating Rheometer with Closed Die Cavity

	Conditioning Step				Frequency Sweep				
Rubber Type	Temperature, (± 0.3°C)	Strain, ± %	Frequency, Hz	Time, min	Temperature (± 0.3°C)	Strain, ± %	First Frequency, Hz	Second Frequency, Hz	Third Frequency, Hz
IRM 241	100 or 125	2.8	0.5	4.0	100 or 125	7.0	0.1	2.0	20
NR	100	2.8	0.5	8.0	100	7.0	0.1	2.0	20
BR, CR, IR, NBR, SBR	100	2.8	0.5	4.0	100	7.0	0.1	2.0	20
BIIR, CIIR, IIR	100 or 125	2.8	0.5	4.0	100 or 125	7.0	0.1	2.0	20
EPDM, EPM	100 or 125	2.8	0.5	4.0	100 or 125	7.0	0.1	2.0	20
Synthetic Rubber Black Masterbatch	100	2.8	0.5	4.0	100	7.0	0.1	2.0	20
Rubber Compound, Reclaimed Material	100	2.8	0.5	2.0	100	7.0	0.1	2.0	20

9.1.3 The frequency, strain, temperature and time for the conditioning step.

9.1.4 The strain amplitude in ± degrees of arc or ± % strain for the frequency sweep.

9.1.5 The temperature of the frequency sweep.

9.1.6 The storage shear modulus *G*' in kPa and the frequency in Hz for each step in the programmed frequency sweep.

9.1.7 The loss shear modulus *G"* in kPa and the frequency in Hz for each step in the programmed frequency sweep.

9.1.8 The dynamic complex viscosity η^* in kPa/s, and the frequency in radians per second for each step in the programmed frequency sweep.

9.1.9 The tangent delta (tan δ) and the frequency in Hz for each step in the programmed frequency sweep.

10. Precision and Bias

10.1 A precision and bias estimate has not been completed for this test method at this time.

11. Keywords

11.1 dynamic complex viscosity; loss modulus; processability; rheological properties; rotorless oscillating shear rheometer; storage modulus; viscosity