Designation: D6204 – 15

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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.

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

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[1] This test method is under the jurisdiction of ASTM 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.

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).

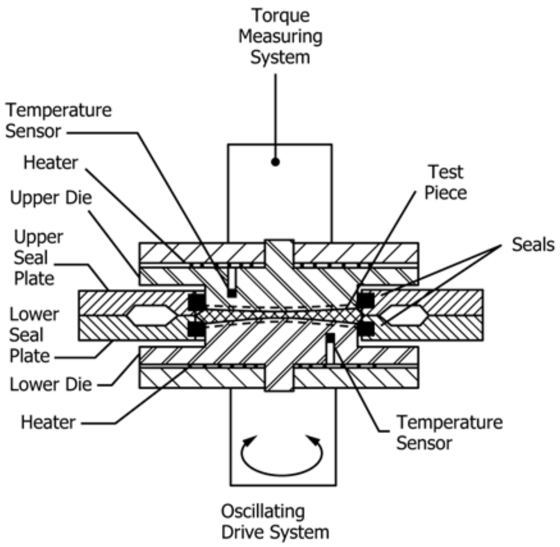
6.5 *Die Oscillating System*—The die oscillating system consists of a direct drive motor that imparts a torsional oscillating movement to the lower die in the cavity plane.

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.

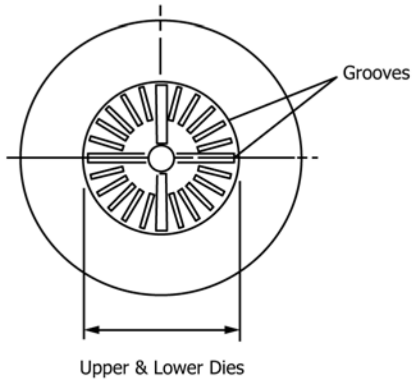
6.6 *Torque Measuring System*—The torque measuring system shall measure the resultant shear torque.

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 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.



A) Measuring Principle



B) Reaction Dies

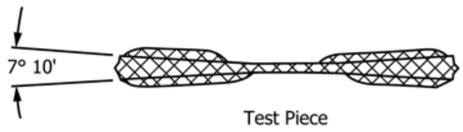


FIG. 1 Typical Sealed Torsion Shear Rotorless Curemeter

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 at least 30 s. When the set temperature is set for a ramped

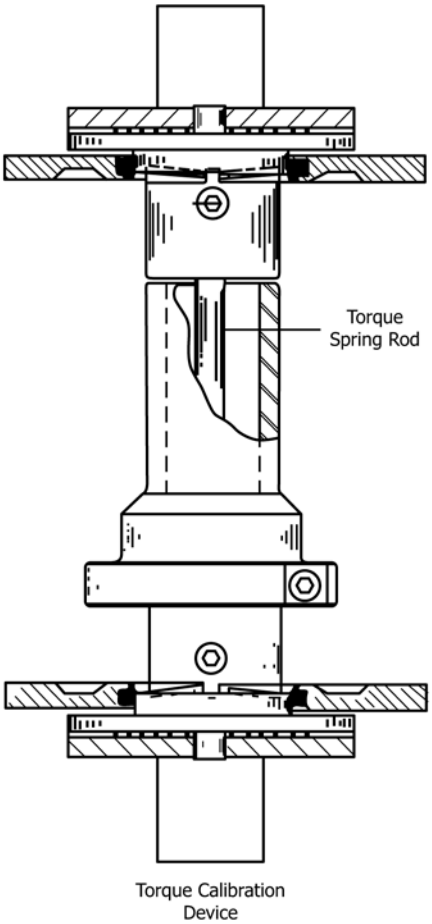


FIG. 2 Typical Torque Standard Calibration Devices for Torsion Shear Curemeters

increase as described in Part C of this test method, the temperature shall not overshoot by more than 3°C with recovery to within ±0.3°C within 1 min of control at the plateau temperature at the end of the temperature ramp.

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. Sampling

7.1 The sample shall be taken from a raw rubber or rubber compound as required by the mixing method or other sampling instructions. Condition a raw rubber sample in accordance with Practice D1485 or Practice D3896 until it has reached room temperature (23 ± 3°C (73 ± 5°F)) throughout.

7.2 The sample shall be homogeneous, at room temperature, and as free of air as possible. Raw rubber samples should be tested as received, that is unmassed (not milled). If the material cannot be tested unmassed, then follow the sample preparation section of Test Methods D1646. Rubber compound samples shall be in the form of a sheet. Rubber compounds with or without curatives may be tested using Parts A or B of this method. A vulcanizable rubber compound is required for Part C testing.

7.3 The temperature of the sample and its heat history can significantly affect test results. For part C testing, the sample shall be conditioned at 23 ± 1°C (73 ± 2°F) for at least 1 h before testing.

7.4 In production control testing, samples may be tested without the conditioning period, but care should be taken to minimize temperature and heat history variations prior to testing.

8. Test Specimen

8.1 The test specimen should be circular, with a diameter smaller than the test chamber of the instrument to be used.

8.2 The test specimen is considered to be of proper size (116 to 160 % of the test cavity volume) when a small bead of rubber compound is extruded uniformly around the periphery of the dies as they are closed. For example, an instrument with a test cavity volume of 3.44 cm^3 requires a test specimen volume between 4.0 and 5.5 cm^3 (4.6 to 6.3 g of rubber compound with a specific gravity of 1.15). Materials of either low or high viscosity may require a narrower range of sample volume for repeatable results. Undersized test specimens can cause low cavity pressure and low torque readings. Oversized test specimens cool the dies excessively during the early part of the test period, affecting the vulcanization characteristics measured in Part C.

8.3 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 preconditioning 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.

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

9. Procedure

9.1 *Part A—Measuring Rheological Properties At Low Strain:*

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

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

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

9.1.4 Load the test configuration to run the test.

9.1.5 Enter specimen identification.

9.1.6 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.

9.2 *Part B—Measuring Rheological Properties at High Strain:*

9.2.1 This procedure is similar to Part A, except that a two-point frequency sweep at 0.1 Hz and 1.0 Hz and 100 % strain is performed instead of the three-point frequency sweep at 0.1, 2, and 20 Hz and 7 % strain. Where more test sensitivity is needed, a strain of 200 % or higher may be selected.

9.2.2 Select the frequency, strain, temperature, and time for the conditioning step as listed in Table 2.

9.2.3 Select the frequency steps and the strain and temperature conditions for the frequency step as listed in Table 2.

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

9.2.5 Load the test configuration to run the test.

9.2.6 Enter specimen identification.

9.2.7 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.

9.3 *Part C—Measuring Cure Behavior Using a Linear Temperature Increase/Plateau:*

9.3.1 After completion of Part A or Part B, or both, a preprogrammed linear temperature increase and plateau may be applied to the specimen in order to measure cure behavior in the same test.

9.3.2 This linear temperature increase/plateau cure (or linear ramped transition to an isothermal cure) is shown in Fig. 3. This should be performed at 1.67 Hz.

9.3.2.1 In some cases, higher frequencies may be used to achieve higher statistical test sensitivity to the onset of scorch.

9.3.3 The recommended strain conditions are as follows.

TABLE 1 Standard Test Conditions for Oscillating Rheometer with Closed Die Cavity (Conditioning and Part A)

	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	5.0	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,	100	2.8	0.5	5.0	100	7.0	0.1	2.0	20
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	5.0	100 or 125	7.0	0.1	2.0	20
EPDM, EPM	125	2.8	0.5	5.0	125	7.0	0.1	2.0	20
Synthetic RubberBlack 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

TABLE 2 Standard Test Conditions for Oscillating Rheometer with Closed Die Cavity (Conditioning and Part B)

	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
IRM 241	100 or 125	2.8	0.5	5.0	100 or 125	100	0.1	1.0
NR	100	2.8	0.5	8.0	100	100	0.1	1.0
BR, CR, IR, NBR	100	2.8	0.5	5.0	100	100	0.1	1.0
SBR	100	2.8	0.5	4.0	100	100	0.1	1.0
BIIR, CIIR, IIR	100 or 125	2.8	0.5	5.0	100 or 125	100	0.1	1.0
EPDM, EPM	125	2.8	0.5	5.0	125	100	0.1	1.0
Synthetic RubberBlack Masterbatch	100	2.8	0.5	4.0	100	100	0.1	1.0
Rubber Compound, Reclaimed Material	100	2.8	0.5	2.0	100	100	0.1	1.0

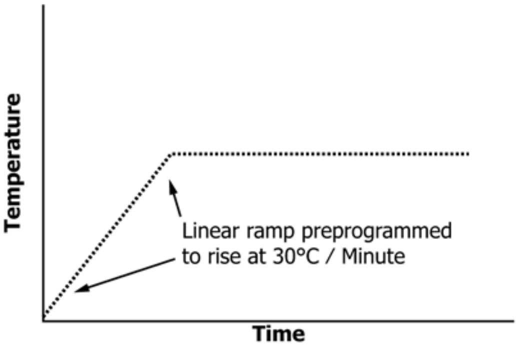


FIG. 3 Linear Ramped Transition/Plateau Profile

9.3.3.1 Apply 0.2° arc when one wishes to perform an after-cure strain sweep (reference Test Method D6601) and minimize the suppression of the after-cure modulus.

9.3.3.2 Apply 1.0° arc when one wishes to increase test sensitivity to scorch differences even though this higher strain may suppress the after-cure modulus (if an after-cure strain test is performed).

9.3.3.3 Apply 0.5° arc as a compromise between test sensitivity to scorch and suppression of the after-cure modulus.

9.3.4 The ramped transition between the processability temperature (100 or 125°C) and the cure temperature (140, 160, or 180°C) shall increase at 30 ± 0.5°C/min. This means that ramped transitions will require the time periods to reach the stated cure temperature plateaus as shown in Table 3.

10. Report

10.1 Report the following information:

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

10.1.2 Type and model of oscillating rheometer,

TABLE 3 Time Table for Transition Time

Processability Temperature	Cure Temperature	Transition Time (min.)
100	140	1.33
100	160	2.00
100	180	2.67
125	140	0.50
125	160	1.17
125	180	1.83

10.1.3 The frequency, strain, temperature, and time for the conditioning step,

10.1.4 The strain amplitude in ± degrees of arc or ± percent strain for the frequency sweep,

10.1.5 The temperature of the frequency sweep,

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

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

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

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

10.2 Use Test Method D5289, Section 10 on Report for parameters from Part C—Measuring Cure Behavior Using a Linear Temperature Increase/Plateau.

11. Precision and Bias[3]

11.1 This precision statement was prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

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[3] A research report is forthcoming.

11.2 The results in this precision and bias section give an estimate of the precision of this test method with the materials used in the interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method.

11.3 A type I interlaboratory precision program was conducted. Both repeatability and reproducibility represent short term (daily) testing conditions. The testing was performed using one operator in each laboratory performing the test twice on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured.

11.4 The results of the precision calculations are given in Tables 4-6. The materials are arranged in ascending "mean level" order. The relative repeatability and reproducibility are more independent of the mean level so the relative repeatability, (r), and reproducibility, (R), are the preferred parameters.

11.5 *Repeatability*—The pooled relative repeatability, (r), of this test has been established and may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value must be suspected of being from different populations and some appropriate action taken.

NOTE 1—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, and so forth, which generated the two test results.

11.6 *Reproducibility*—The pooled relative reproducibility, (R), of this test has been established. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

11.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Test method bias, therefore, cannot be determined.

11.8 *Precision Type*—All precision statements given in this section are Type I Precision in accordance with Practice D4483. These data were all calculated from the parameters of eight (8) separate laboratories independently performing Test Method D6204, Parts A and B.

11.9 The materials used in this interlab test program included: SBR 1502, IRM241 Lot E Butyl, and an SBR Reference Compound containing carbon black. Eight laboratories reported results for all of the test conditions listed in Tables 4-6.

11.10 Part A test conditions were 100°C, 7 % strain, at consecutive frequencies of 0.1 Hz, 2 Hz, and 20 Hz. Part A test results are reported as Tan delta (unitless, 3 decimal places), G' (elastic Modulus), and G" (viscous modulus) in kPa, reported to 1 decimal place (Table 4A–I). Part B test conditions were 100°C, 100 % strain, at consecutive frequencies of 0.1 Hz and 1 Hz (Table 5A–F). A second Part B test was then run at 100°C, 200 % strain, at consecutive frequencies of 0.1 Hz and 1 Hz (Table 6A–F). Part B test results are also reported as Tan delta, G' (elastic Modulus) in kPa, and G" (viscous modulus) in kPa.

12. Keywords

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

TABLE 4 Precision Parameters

Table 4A Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, Tan delta (unitless) at 100°C, 7 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	0.582	0.010	0.028	**4.9**	0.018	0.052	**9.0**
SBR 1502	8	0.710	0.016	0.045	**6.3**	0.026	0.073	**10.2**
SBR Reference Compound	8	0.915	0.012	0.035	**3.8**	0.028	0.078	**8.6**
Average		0.735						
Pooled Values			0.013	0.037	**5.0**	0.024	0.069	**9.3**

Table 4B Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, Tan delta (unitless) at 100°C, 7 % strain at 2 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	0.255	0.004	0.010	**4.0**	0.007	0.021	**8.1**
SBR 1502	8	0.613	0.007	0.020	**3.3**	0.011	0.030	**4.9**
SBR Reference Compound	8	0.779	0.005	0.015	**1.9**	0.011	0.030	**3.9**
Average		0.549						
Pooled Values			0.006	0.016	**2.9**	0.010	0.027	**5.0**

Table 4C Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, Tan delta (unitless) at 100°C, 7 % strain at 20 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	0.161	0.004	0.012	**7.4**	0.008	0.022	**13.8**
SBR 1502	8	0.438	0.007	0.019	**4.3**	0.006	0.018	**4.0**
SBR Reference Compound	8	0.593	0.004	0.012	**1.9**	0.009	0.025	**4.2**
Average		0.397						
Pooled Values			0.005	0.015	**3.7**	0.008	0.022	**5.5**

Table 4D Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, G' (elastic modulus, kPa) at 100°C, 7 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	35.4	1.04	2.94	**8.3**	1.18	3.33	**9.4**
IRM241 Lot E	8	86.1	1.36	3.84	**4.5**	1.49	4.21	**4.9**
SBR Reference Compound	8	96.8	1.17	3.30	**3.4**	3.82	10.82	**11.2**
Average		72.76						
Pooled Values			1.195	3.381	**4.6**	2.464	6.973	**9.6**

Table 4E Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, G' (elastic modulus, kPa) at 100°C, 7 % strain at 2 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	120.7	1.29	3.65	**3.0**	2.21	6.26	**5.2**
IRM241 Lot E	8	189.4	1.27	3.59	**1.9**	4.06	11.49	**6.1**
SBR Reference Compound	8	225.2	2.04	5.78	**2.6**	4.04	11.43	**5.1**
Average		178.39						
Pooled Values			1.575	4.458	**2.5**	3.544	10.029	**5.6**

Table 4F Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, G' (elastic modulus, kPa) at 100°C, 7 % strain at 20 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	269.1	2.74	7.76	**2.9**	6.04	17.09	**6.3**
IRM241 Lot E	8	252.2	2.30	6.50	**2.6**	6.16	17.44	**6.9**
SBR Reference Compound	8	452.4	3.04	8.61	**1.9**	6.73	19.05	**4.2**
Average		324.58						
Pooled Values			2.711	7.671	**2.4**	6.317	17.877	**5.5**

Table 4G Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, G″ (viscous modulus, kPa) at 100°C, 7 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	25.1	1.08	3.05	**12.2**	1.20	3.39	**13.5**
IRM241 Lot E	8	50.0	0.51	1.43	**2.9**	1.23	3.49	**7.0**
SBR Reference Compound	8	89.1	1.44	4.07	**4.6**	3.67	10.38	**11.7**
Average		54.72						
Pooled Values			1.078	3.050	**5.6**	2.339	6.619	**12.1**

Table 4H Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, G″ (viscous modulus, kPa) at 100°C, 7 % strain at 2 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	48.3	0.52	1.48	**3.1**	1.28	3.62	**7.5**
SBR 1502	8	73.8	0.66	1.86	**2.5**	1.40	3.96	**5.4**
SBR Reference Compound	8	177.6	1.72	4.86	**2.7**	5.85	16.55	**9.3**
Average		99.91						
Pooled Values			1.103	3.122	**3.1**	3.550	10.045	**10.1**

Table 4I Precision Parameters for Test Method D6204 (Type 1 Precision), Part A, G″ (viscous modulus, kPa) at 100°C, 7 % strain at 20 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	40.3	0.71	2.00	**5.0**	1.78	5.04	**12.5**
SBR 1502	8	117.5	1.46	4.13	**3.5**	2.87	8.11	**6.9**
SBR Reference Compound	8	267.6	1.53	4.33	**1.6**	5.57	15.76	**5.9**
Average		141.82						
Pooled Values			1.288	3.645	**2.6**	3.759	10.637	**7.5**

TABLE 5 Precision Parameters

Table 5A Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, Tan delta (unitless) at 100°C, 100 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	0.766	0.009	0.027	**3.5**	0.014	0.039	**5.1**
SBR 1502	8	0.898	0.012	0.033	**3.6**	0.016	0.045	**5.1**
SBR Reference Compound	8	1.364	0.010	0.029	**2.1**	0.016	0.045	**3.3**
Average		1.009						
Pooled Values			0.010	0.030	**2.9**	0.015	0.043	**4.3**

Table 5B Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, Tan delta (unitless) at 100°C, 100 % strain at 1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	0.473	0.005	0.015	**3.3**	0.007	0.020	**4.2**
SBR 1502	8	0.846	0.007	0.019	**2.2**	0.011	0.030	**3.6**
SBR Reference Compound	8	1.311	0.007	0.020	**1.5**	0.021	0.059	**4.5**
Average		0.877						
Pooled Values			0.006	0.018	**2.1**	0.014	0.040	**4.6**

Table 5C Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G’ (elastic modulus, kPa) at 100°C, 100 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR Reference Compound	8	21.0	0.17	0.48	**2.3**	0.53	1.50	**7.1**
SBR 1502	8	27.2	0.39	1.11	**4.1**	0.54	1.53	**5.6**
IRM241 Lot E	8	61.6	0.71	2.02	**3.3**	1.93	5.47	**8.9**
Average		36.60						
Pooled Values			0.480	1.360	**3.7**	1.198	3.389	**9.3**

Table 5D Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G’ (elastic modulus, kPa) at 100°C, 100 % strain at 1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR Reference Compound	8	46.4	0.41	1.16	**2.5**	0.76	2.14	**4.6**
SBR 1502	8	66.6	0.58	1.64	**2.5**	0.85	2.41	**3.6**
IRM241 Lot E	8	112.5	0.67	1.92	**1.7**	2.64	7.46	**6.6**
Average		75.15						
Pooled Values			0.565	1.599	**2.1**	1.657	4.691	**6.2**

Table 5E Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G″ (viscous modulus, kPa) at 100°C, 100 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	24.4	0.20	0.55	**2.3**	0.26	0.74	**3.0**
SBR Reference Compound	8	28.8	0.20	0.56	**1.9**	0.51	1.44	**5.0**
IRM241 Lot E	8	47.4	0.31	0.89	**1.9**	0.73	2.07	**4.4**
Average		33.53						
Pooled Values			0.242	0.685	**2.0**	0.536	1.517	**4.5**

Table 5F Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G″ (viscous modulus, kPa) at 100°C, 100 % strain at 1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	53.5	0.48	1.36	**2.5**	1.01	2.86	**5.3**
SBR 1502	8	56.4	0.43	1.22	**2.2**	0.81	2.28	**4.0**
SBR Reference Compound	8	60.4	0.37	1.06	**1.8**	1.19	3.38	**5.6**
Average		56.79						
Pooled Values			0.431	1.219	**2.1**	1.015	2.872	**5.1**

TABLE 6 Precision Parameters

Table 6A Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, Tan delta (unitless) at 100°C, 200 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	1.038	0.008	0.021	**2.1**	0.009	0.025	**2.5**
SBR 1502	8	1.303	0.015	0.042	**3.2**	0.020	0.057	**4.4**
SBR Reference Compound	8	1.854	0.011	0.032	**1.7**	0.033	0.093	**5.0**
Average		1.398						
Pooled Values			0.012	0.033	**2.3**	0.023	0.065	**4.6**

Table 6B Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, Tan delta (unitless) at 100°C, 200 % strain at 1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
IRM241 Lot E	8	1.030	0.009	0.027	**2.6**	0.025	0.072	**7.0**
SBR 1502	8	1.249	0.009	0.025	**2.0**	0.011	0.031	**2.4**
SBR Reference Compound	8	1.769	0.007	0.021	**1.2**	0.016	0.045	**2.6**
Average		1.349						
Pooled Values			0.009	0.024	**1.8**	0.018	0.052	**3.9**

Table 6C Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G’ (elastic modulus, kPa) at 100°C, 200 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR Reference Compound	8	13.3	0.11	0.32	**2.4**	0.37	1.05	**7.9**
SBR 1502	8	16.3	0.22	0.63	**3.9**	0.30	0.86	**5.3**
IRM241 Lot E	8	37.9	0.35	1.00	**2.6**	0.90	2.55	**6.7**
Average		22.48						
Pooled Values			0.250	0.707	**3.1**	0.590	1.670	**7.4**

Table 6D Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G’ (elastic modulus, kPa) at 100°C, 200 % strain at 1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR Reference Compound	8	29.0	0.21	0.60	**2.1**	0.60	1.69	**5.8**
SBR 1502	8	39.1	0.39	1.09	**2.8**	0.60	1.69	**4.3**
IRM241 Lot E	8	52.8	0.45	1.27	**2.4**	0.91	2.57	**4.9**
Average		40.32						
Pooled Values			0.363	1.027	**2.5**	0.715	2.024	**5.0**

Table 6E Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G″ (viscous modulus, kPa) at 100°C, 200 % strain at 0.1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	21.1	0.12	0.33	**1.5**	0.19	0.53	**2.5**
SBR Reference Compound	8	24.7	0.12	0.34	**1.4**	0.29	0.83	**3.3**
IRM241 Lot E	8	39.7	0.24	0.67	**1.7**	0.56	1.60	**4.0**
Average		28.50						
Pooled Values			0.167	0.472	**1.7**	0.382	1.081	**3.8**

Table 6F Precision Parameters for Test Method D6204 (Type 1 Precision), Part B, G″ (viscous modulus, kPa) at 100°C, 200 % strain at 1 Hz

Material	Number of Laboratories	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SBR 1502	8	48.7	0.29	0.82	**1.7**	0.51	1.43	**2.9**
SBR Reference Compound	8	51.1	0.30	0.84	**1.6**	0.90	2.54	**5.0**
IRM241 Lot E	8	54.4	0.34	0.97	**1.8**	1.14	3.21	**5.9**
Average		51.36						
Pooled Values			0.310	0.88	**1.7**	0.885	2.504	**4.9**

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