Standard Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters[1]

This standard is issued under the fixed designation D5289; 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 a method for the measurement of selected vulcanization characteristics of rubber compounds using unsealed and sealed torsion shear cure meters. The two types of instruments may not give the same results.

NOTE 1—An alternative method for the measurement of vulcanization characteristics is given in Test Method D2084.

1.2 The values stated in SI units are to be regarded as the 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]

D1349 Practice for Rubber—Standard Conditions for Testing
D1566 Terminology Relating to Rubber
D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter
D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *rotorless cure meter, n*—a name for a class of cure meters that uses one of the two specimen shaping members or dies to sense the torque or stress during strain application. Rotorless cure meters do not have a third member in the form of a rotor (see definitions of cure meter in Terminology D1566 and Test Method D2084).

3.1.2 *S' torque, n—for an oscillating shear rotorless cure meter*, the value measured by a torque transducer at the peak strain amplitude of the oscillating cycle; represents the elastic response of the test material.

3.1.3 The following measurements may be taken from the recorded curve of torque as a function of time (see Fig. 1):

3.1.3.1 *minimum S' torque*—measure of the elastic stiffness of the unvulcanized test specimen at the specified vulcanizing temperature, taken at the lowest point in the vulcanization curve.

3.1.3.2 *maximum, plateau,* or *highest S' torque*—measure of the elastic stiffness of the vulcanized test specimen at the specified vulcanizing temperature, measured within a specified period of time.

3.1.3.3 *time to a percentage of full cure*—measure of cure based on the time to develop some percentage of the difference in S' torque from the minimum to the maximum.

3.1.3.4 *time to incipient cure (scorch time)*—measure of the time at which a specified small increase in S' torque has occurred; it indicates the beginning of vulcanization.

3.1.4 *S" torque, n—for an oscillating shear rotorless cure meter*, the value measured by a torque transducer at zero strain amplitude of the oscillating cycle; represents the viscous response of the test material (see Fig. 2).

3.1.5 *S* torque, n—for an oscillating shear rotorless cure meter*, the maximum value measured by a torque transducer during the oscillating cycle (see Fig. 2).

3.1.6 The relationship between S*, S', and S" for any oscillating cycle is:

$$S^* = \sqrt{(S')^2 + (S")^2} \quad (1)$$

3.1.7 *phase angle δ, n—for an oscillating shear cure meter*, the angle of shift between the sinusoidal strain and the sinusoidal S* torque during the oscillating cycle (see Fig. 2).

3.1.8 *Tan δ, n—for an oscillating shear rotorless cure meter*, the tangent of the phase angle δ.

3.1.8.1 *Discussion*—The relationship between Tan δ, S', and S" is:

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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.

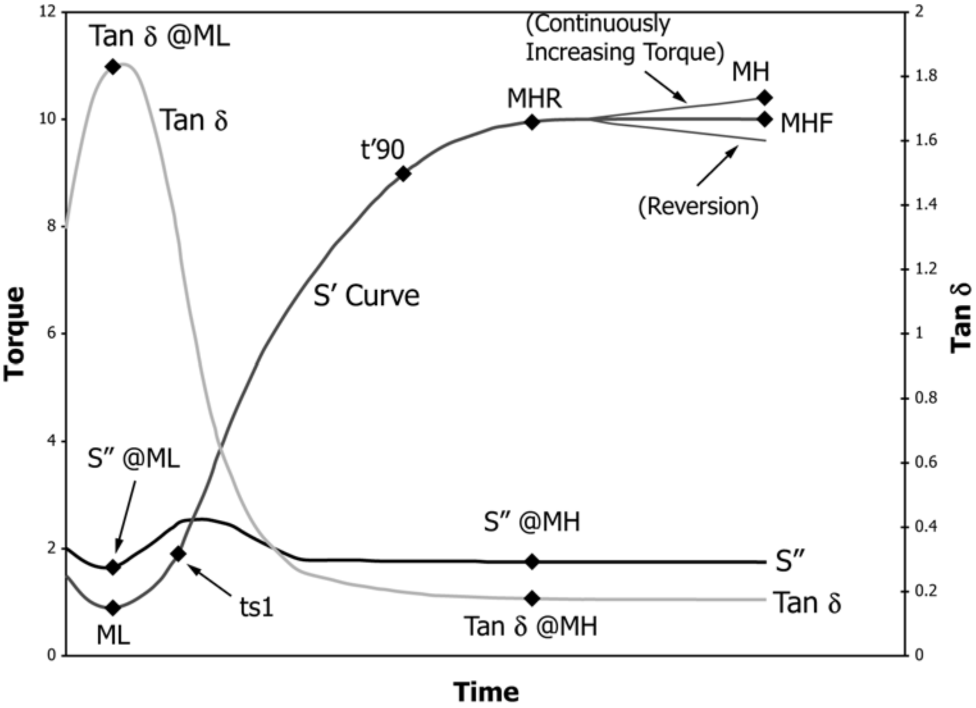


FIG. 1 Typical Vulcanization Curves

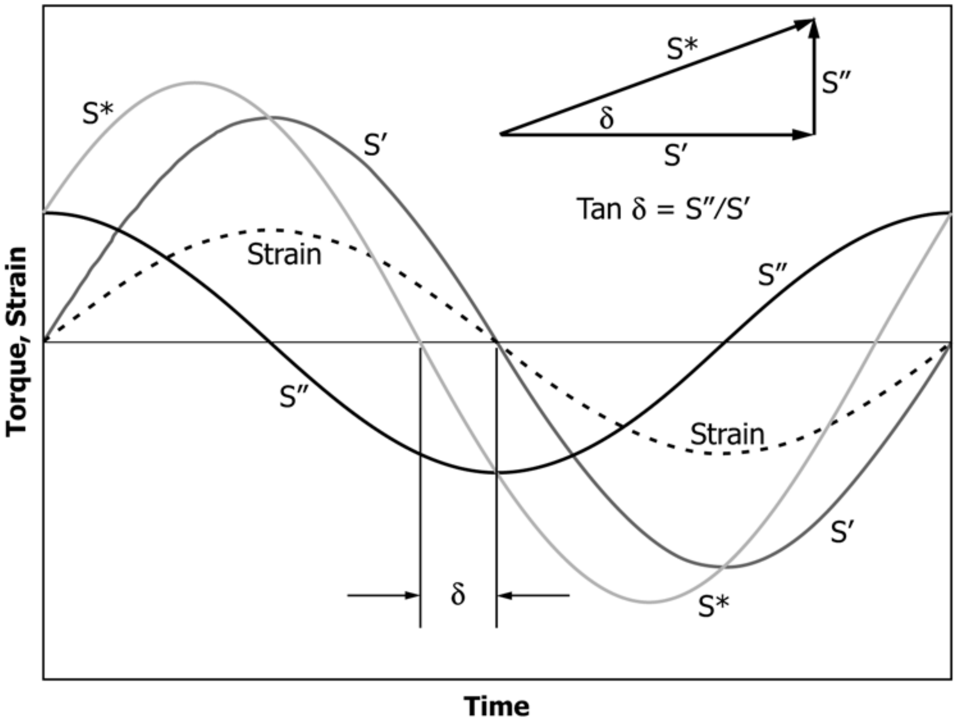


FIG. 2 Example of Torque Responses for an Oscillation Cycle

$$Tan\,\delta = \frac{S''}{S'} \qquad (2)$$

3.1.9 The following measurements may be taken from the recorded S" and Tan δ curves as a function of time (see Fig. 1):

3.1.9.1 *S" @ML*—value of S" torque when the minimum S' torque is taken.

3.1.9.2 *Tan* δ *@ML*—value of Tan δ when the minimum S' torque is measured.

3.1.9.3 *S" @MH*—value of S" torque when the maximum S' torque is taken.

3.1.9.4 *Tan* δ *@MH*—value of Tan δ when the maximum S' torque is measured.

4. Summary of Test Method

4.1 A rubber test piece is contained in a die cavity which may be closed or almost closed and maintained at an elevated temperature. The cavity is formed by two dies, one of which is oscillated through a small rotary amplitude. This action produces a sinusoidal alternating torsional strain in the test piece and a sinusoidal shear torque which depends on the stiffness (shear modulus) of the rubber compound.

4.2 The stiffness of the rubber test piece increases as vulcanization proceeds. The test is completed when the recorded torque rises to either an equilibrium or maximum value, or when a predetermined time has elapsed (see Fig. 1). A curve representing the torque at peak strain in one direction of the oscillation cycle is continuously recorded as a function of time.

5. Significance and Use

5.1 This test method is used to determine the vulcanization characteristics of (vulcanizable) rubber compounds.

5.2 This test method may be used for quality control in rubber manufacturing processes, for research and development testing of raw-rubber compounded in an evaluation formulation, and for evaluating various raw materials used in preparing (vulcanizable) rubber compounds.

5.3 The test specimen in a rotorless cure meter approaches the test temperature in a shorter time and there is a better temperature distribution in the test specimen due to the elimination of the unheated rotor found in oscillating disk cure meters.

5.4 Several manufacturers produce rotorless cure meters with design differences that may result in different torque responses and cure times for each design. Correlations of test results between cure meters of different designs should be established for each compound tested, and for each set of test conditions.

6. Apparatus

6.1 Rotorless cure meters of two types can be used. In each case, an oscillation of small amplitude is applied to one die.

6.1.1 *Unsealed Torsion Strain Rotorless Cure Meter*—This type of cure meter measures the torque produced by an angular strain of constant amplitude in a cavity that is not completely closed (see Fig. 3(a)).

6.1.2 *Sealed Torsion Strain Rotorless Cure Meter*—This type of cure meter measures the torque produced by an angular strain of constant amplitude in a cavity that is completely closed and sealed (see Fig. 4(a)).

6.2 *Die Cavity*—The die cavity is formed by two dies. In the measuring position, the two dies are fixed a specified distance apart so that the cavity is almost closed (see Fig. 3(b)), or closed and sealed (see Fig. 4(a)).

6.2.1 The dimensions for typical torsional shear curemeters include biconical-shaped dies having a diameter of 40 ± 2 mm (1.57 ± 0.08 in.), and an angle of separation ranging from 7 to 18°, depending on the manufacturer's design. In the center of the dies, a separation equal to 0.5 mm (0.02 in.) plus the die gap should be maintained (see Fig. 3(b) or Fig. 4(b)). Manufacturer's guidelines should be followed to determine if the dies have been excessively worn and should be replaced.

6.2.2 *Die Gap*—The gap between the edges of the dies in the closed position shall be between 0.05 and 0.20 mm (0.002 to 0.008 in.), preferably 0.1 mm (0.004 in.) for unsealed cavities. For sealed cavities, no gap should exist at the edges of the dies.

6.2.3 *Die Closing Mechanism*—A pneumatic cylinder or other device shall close the dies and hold them closed during the test with a force of not less than 8.0 kN (1820 lbf).

6.3 *Die Oscillating System*—The die oscillating system imparts a torsional oscillating movement to one of the dies, in the plane of the cavity.

6.3.1 The amplitude of the oscillation should be ±0.1 to ±3.0°, preferably ±0.5° of arc for torsional shear cure meters.

6.3.2 The frequency of oscillation should be between 0.5 and 2 Hz, preferably 1.7 ± 0.1 Hz.

6.4 *Torque Measuring System*—A torque measuring system shall measure the resultant torque.

6.4.1 The torque measuring device shall be rigidly coupled to one of the dies and any deformation shall be negligibly small and shall generate a signal which is proportional to the torque. The total error resulting from zero point error, sensitivity error, linearity, and reproducibility errors shall not exceed 1 % of the measuring range selected.

NOTE 2—The elastic deformation of the oscillating and measuring system should not be more than 1 % of the oscillating amplitude; otherwise, the curemeter curves must be corrected.

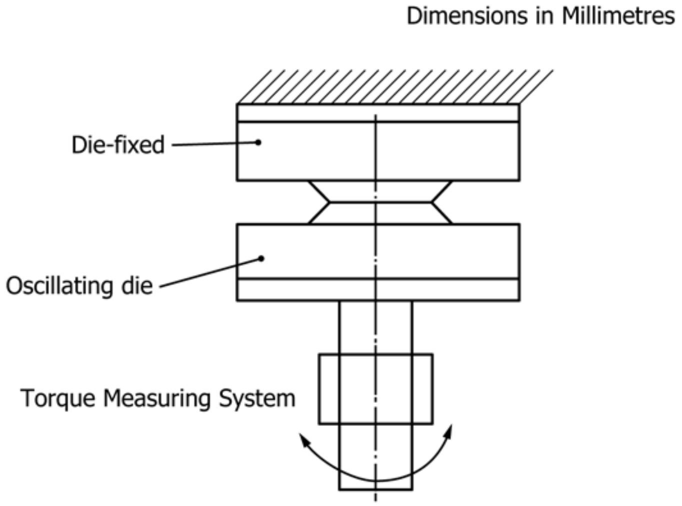
6.4.2 The torque recorder device shall be used to record the signal from the torque measuring device. It shall record the S' torque at maximum oscillation as a function of time.

6.4.3 The torque recorder device shall be used to record the signal from the torque measuring device. It shall record the torque at maximum oscillation continuously as a function of time (see Fig. 1) and shall have a response time for full-scale deflection on 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.

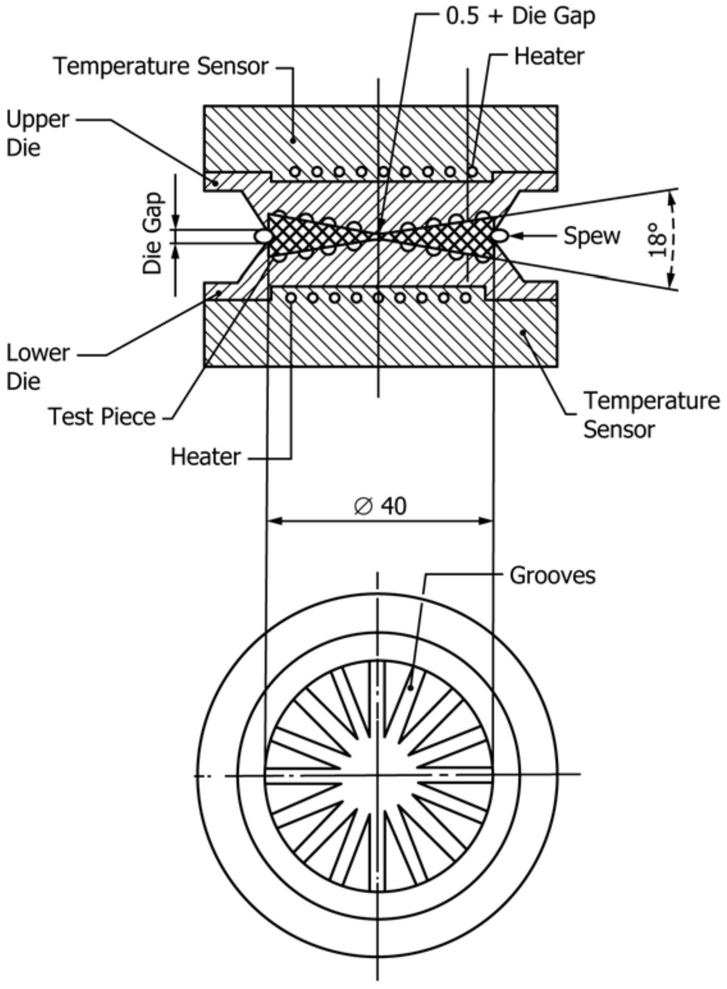
6.5 Torque calibration equipment is required to measure the angular strain amplitude and to calibrate the torque measuring device. Examples of calibration equipment are shown in Fig. 5 and Fig. 6. The amplitude of oscillation of the device shall be checked with no test specimen in it. A displacement transducer shall be used to measure the amplitude and torque measurements shall be checked against standard masses using a device as shown in Fig. 5. An alternate technique shall use a torque standard.

6.5.1 For calibrating torsion shear curemeters, either a displacement transducer and wire-mass calibration or a torque standard shall be used.

6.5.1.1 A displacement transducer for checking angular displacement shall be coupled by a knife-edge bearing in contact with a rod fixed to one of the dies (see Fig. 5). The force measuring system shall be checked by loading a wire, attached to the die or block by a pulley, with masses corresponding to the full-scale force being measured. The torque



a) Measuring Principle

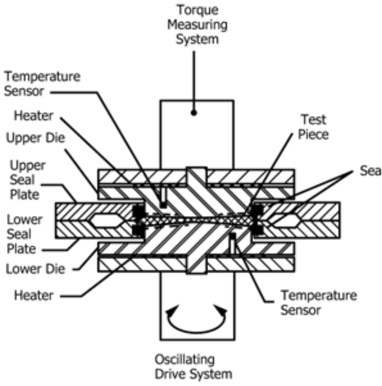


b) Reaction Die

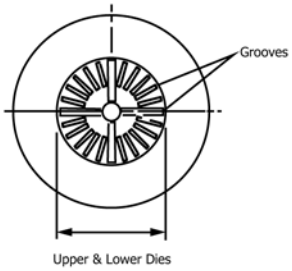
FIG. 3 (a) and (b)—Typical Unsealed Torsion Shear Rotorless Curemeter

shall be calculated in this case from the product of the applied force and the radius of the die block where the wire is attached.

6.5.1.2 Torque standard calibration checks the torque measurement at the selected angular displacement by clamping a



A) Measuring Principle



B) Reaction Dies

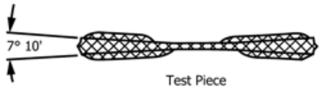


FIG. 4 Typical Sealed Torsion Shear Rotorless Curemeter

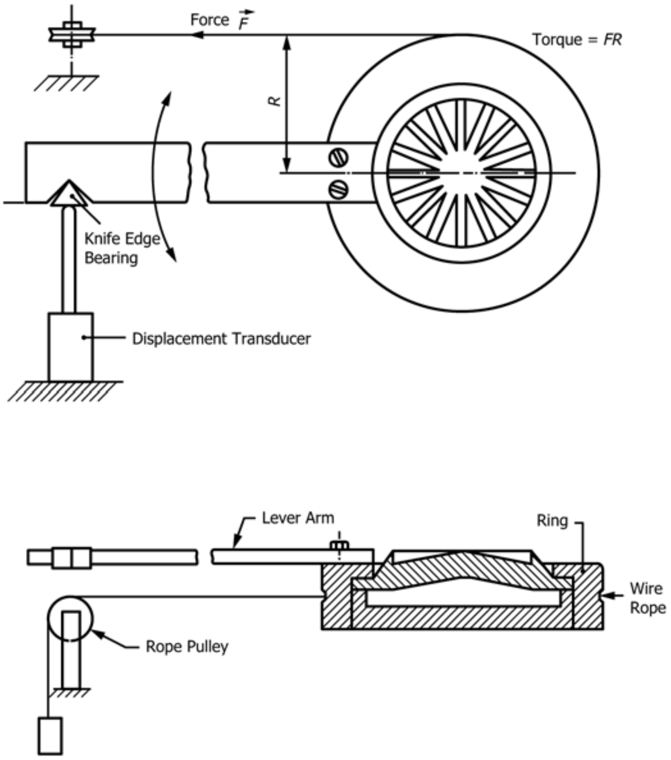


FIG. 5 Displacement Transducer and Wire-Mass Calibration Equipment for Torsion Shear Curemeters

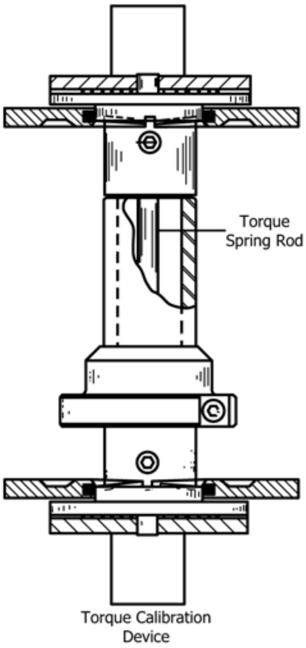


FIG. 6 Typical Torque Standard Calibration Device for Torsion Shear Curemeters

reference steel torsion rod to the oscillating die and the torque measuring die of the torsion shear cure meter (see Fig. 6). The reference values for angular displacement and corresponding torque have been established by the manufacturer for each torque standard.

6.6 *Temperature Controlling System*—The method of temperature control shall maintain the following process parameters: heating up time, curing temperature, temperature distribution, and reference temperature, which are necessary for reproducible measurement of the vulcanization curve. The temperature control system shall permit the reference temperature to be varied between 110°C and 200°C with an accuracy of ±0.3°C or better.

6.6.1 Die shall heat up in 1.5 min or less from closure of the die cavity.

6.6.2 Once heating up time hs been completed, die temperature shall not vary by more than ±0.3°C for the rest of the test.

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

6.6.4 The reference temperature is determined by a temperature sensor used for control. The difference between the reference temperature and the average test specimen temperature shall not be more than 2°C.

6.6.5 Temperature measurement accuracy shall be ±0.3°C for the reference temperature sensor.

6.7 *Reference Test Temperature*—The standard reference test temperature shall be 160°C (320°F). Tests may be carried out at other temperatures if required. Temperatures should be selected in accordance with Practice D1349.

7. Sampling

7.1 The sample shall be taken from a vulcanizable rubber compound as required by the mixing method or other sampling instructions.

7.2 The sample shall be homogeneous, in sheeted form, at room temperature, and as free of air as possible.

7.3 The temperature of the sample and its heat history can significantly affect test results. For referee testing and for testing under controlled circumstances, the sample shall be conditions 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 recommended test specimen volume is between 3 and 6 cm^3, depending on the instrument model being used.

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

8.3 The test specimen is considered to be of proper size when a small bead of rubber compound is extruded uniformly around the periphery of the dies as they are closed (116 to 160 % of the test cavity volume). For an instrument with a test cavity volume of 3.44 cm^3, this is achieved when the test specimen volume is between 4.0 and 5.5 cm^3 (4.6 to 6.3 g of rubber compound with a specific gravity of 1.15). 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.

9. Procedure

9.1 *Preparation for Test*—Bring the temperature of both dies to the reference temperature with the cavity closed. Adjust the zero of the force or torque measuring device, if necessary.

9.2 *Loading the Curemeter:*

9.2.1 Open the dies, unload the previous sample (if necessary), place the test piece in the cavity, and close the dies within 20 s.

9.2.2 The test time shall be counted from the instant that the dies are closed. Oscillation of the movable die shall be started at zero time or before.

10. Report

10.1 Report the following information:

10.1.1 A full description of the sample, its origin and compound details,

10.1.2 Test method and test details,

10.1.2.1 Reference to this test method,

10.1.2.2 Type and model curemeter used (sealed or unsealed rotorless, manufacturer, die options, if any),

10.1.2.3 Amplitude of the die oscillation, in degrees,

10.1.2.4 Frequency of oscillation,

10.1.2.5 Torque range selected, in deci Newton metres (dN·m). (The equation for conversion from dN·m to lbf·in. is 1.13 (dN·m) = 1.00 (lbf·in.)),

10.1.2.6 Time scale of the recording device,

10.1.2.7 Curing temperature in degrees Celsius, and

10.1.2.8 Date of the test.

10.2 Test results reported are normally chosen from the following parameters (refer to Fig. 1 for guidance):

10.2.1 *ML*—Minimum S' torque, in dN·m (lbf·in.).

10.2.2 *Maximum S' Torque*—All in dN·m (lbf·in.).

10.2.2.1 *MHF*—Maximum S' torque where curve plateaus.

10.2.2.2 *MHR*—Maximum S' torque of reverting curve.

10.2.2.3 *MH*—Highest S' torque attained during a specified period of time when no plateau or maximum torque is obtained.

10.2.3 *tsx*—Scorch time, in minutes (time to an increase of *x* units of S' torque from the ML value). The preferred scorch time for tests at an oscillation amplitude of ±0.5° is *ts1* (1.0 S' torque unit rise).

NOTE 3—The value of *ts1* is different if the units of S' torque rise are in lbf·in instead of dN·m.

10.2.4 *Cure Time,* in minutes.

10.2.4.1 *t'x*—equal to the time to *x* % of S' torque increase or $t'x$ = minutes to $(ML + x(MH - ML)/100)$ S' torque. This test result may also be called *TCx.*

NOTE 4—This method of determining the cure times is considered the standard method. The most commonly used values of *x* are 50 and 90. A cure time of *t'*10 is sometimes used as a measure of scorch time.

10.2.4.2 *tx*—equal to the time to *x* % of S' torque increase or tx = minutes to $(x(MH)/100)$ S' torque.

NOTE 5—This is an alternate method for cure time determination.

10.2.5 *Cure Rate Index*—equal to 100/(cure time − scorch time).

10.2.6 *PCR (Peak Cure Rate)*—Maximum slope of the S' torque curve as a function of time, in dN·m/min. This value is usually measured with the aid of computer data recording.

10.2.7 *PCR Time*—test time at which the Peak Cure Rate is reached, in minutes.

10.2.8 "*t10 % Rise*" is the time for the torque to rise to 110 % of the minimum value. This may be used to measure scorch in some cases.

10.2.9 *S" @ML*—The value of S" torque when the ML value of S' torque is taken.

10.2.10 *Tan δ @ML*—The value of Tan δ when the ML value of S' torque is taken.

10.2.11 *S" @MH*—The value of S" torque when the MH value of S' torque is measured.

10.2.12 *Tan δ @MH*—The value of Tan δ when the MH value of S' torque is measured.

11. Precision and Bias

11.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical calculation details.

11.1.1 The precision results in this precision and bias section give an estimate of the precision of this test method

TABLE 1 Precision Program 1[A]

NOTE 1—
Sr = within laboratory standard deviation,
r = repeatability (in measurement units),
(r) = repeatability (in percent),
SR = between-laboratory standard deviation,
R = reproducibility (in measurement units), and
(R) = reproducibility (in percent).

Compound, Test Parameter	Mean Value	Within Labs			Between Labs		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
Compound A:							
M_L (dN·m)	1.35	0.007	0.02	1.4	0.071	0.20	14.8
M_H (dN·m)	13.60	0.025	0.07	0.5	0.608	1.72	12.7
t_s1 (min)	1.55	0.009	0.02	1.6	0.065	0.18	11.8
t'50 (min)	3.10	0.007	0.03	0.8	0.061	0.17	5.6
t'90 (min)	4.93	0.015	0.04	0.9	0.147	0.42	8.5
	Gr. Avg.	**0.013**	**0.036**	**1.04**	**0.190**	**0.538**	**10.68**
Compound B:							
M_L (dN·m)	1.00	0.008	0.02	2.2	0.048	0.14	13.6
M_H (dN·m)	10.17	0.024	0.07	0.7	0.478	1.35	13.3
t_s1 (min)	2.22	0.008	0.02	1.0	0.076	0.21	9.6
t'50 (min)	3.55	0.007	0.02	0.6	0.098	0.28	7.8
t'90 (min)	5.74	0.023	0.06	1.1	0.156	0.44	7.7
	Gr. Avg.	**0.014**	**0.036**	**1.12**	**0.171**	**0.484**	**10.40**
Compound C:							
M_L (dN·m)	1.50	0.011	0.03	2.1	0.076	0.22	14.3
M_H (dN·m)	12.30	0.045	0.13	1.0	0.571	1.62	13.1
t_s1 (min)	1.80	0.011	0.03	1.7	0.063	0.18	9.9
t'50 (min)	3.34	0.009	0.03	0.8	0.098	0.28	8.3
t'90 (min)	5.73	0.018	0.05	0.9	0.156	0.44	7.7
	Gr. Avg.	**0.019**	**0.054**	**1.30**	**0.193**	**0.548**	**10.66**

[A] These values are Type I precision values, obtained from fully prepared test specimens (compounds mixed in one laboratory) which were circulated to all participating laboratories.

with the materials (rubbers, etc.) used in the particular interlaboratory test programs (ITPs) as 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 that include this test method.

11.1.2 The precision has been evaluated in two separate interlaboratory test programs: Program 1 in 1989 and Program 2 in 2000. Each program is described below and then repeatability and reproducibility statements are given that apply to both programs. Both ITPs evaluated Type 1 precision, with fully prepared test specimens sent to all participants.

11.1.3 The data generated in Precision Programs I and II were collected from tests run on instruments from a single manufacturer. This is due to most participants in both interlaboratory studies having similar equipment. This test method is intended to cover use of a wide range of rotorless curemeter designs, and the fact that the data reported is from a single manufacturer's instrument is not to be considered an endorsement of that instrument over the use of other manufacturer's instruments. Each manufacturer's rotorless curemeter design has unique characteristics that may cause some shifts in test results. As noted in the report section of this test method, reports of test results should include notation of the instrument used to obtain the results.

11.2 *Precision Program 1*—In this ITP three compounds based on SBR and SBR/NR polymers with sulfenamide cure systems and carbon black reinforcement were carefully mixed and prepared for testing. Precut specimens were sent to 11 laboratories along with instructions to test duplicate samples of each compound in one day, and repeat the testing, after checking calibration, for day 2 one week later. The testing was conducted using a sealed torsion type rotorless curemeter,[3] with typical dies as illustrated in Fig. 4, at 175°C and ±0.5° arc. The duplicate test values for each day were averaged to get a single value for each day and the analysis was conducted on these day 1–day 2 test results. The precision output for Program 1 is listed in Table 1.

11.3 *Precision Program 2*—In this ITP four compounds were used for testing. Compounds A, B, and C were based on SBR with a sulfenamide cure system and carbon black reinforcement with increasing sulfur content for A, B, and C. Compound D was a Fluoroelastomer compound that required the use of film between the sample and the dies for testing. All compounds were carefully mixed and prepared for testing. Precut specimens were sent to 20 laboratories for compounds A, B, and C and 12 laboratories for compound D, along with instructions to test duplicate samples of each compound in one day, and repeat the testing, after checking calibration, for day 2 one week later. The testing was conducted using a sealed torsion type rotorless curemeter,[3] with typical dies as illustrated in Fig. 4, at 160°C and ±0.5° arc. The duplicate test

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[3] The MDR-2000 from Alpha Technologies, 3030 Gilchrist Road, Akron, OH 44305 was used by all participants in both Interlaboratory Test Programs. There are several other manufacturers of rotorless cure meters, but not enough participants using other instruments were available to generate meaningful statistics for this precision and bias statement. No endorsement of any instrument is implied in this study.

TABLE 2 Precision Program 2

NOTE 1—
Sr = within laboratory standard deviation,
r = repeatability (in measurement units),
(r) = repeatability (in percent of mean level),
SR = between-laboratory standard deviation (for total between-laboratory variation in measurement units),
R = reproducibility (in measurement units), and
(R) = reproducibility (in percent of mean level).

Compound, Test Parameter	Mean Value	Within Labs			Between Labs			No. Labs^{A,B}
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*	
Compound A:								
M_L (dN·m)	2.13	0.025	0.071	3.34	0.076	0.210	10.0	17
M_H (dN·m)	14.43	0.176	0.49	3.42	0.548	1.53	10.6	17
t_s1 (min)	3.14	0.055	0.15	4.90	0.095	0.270	8.47	16
t'10 (min)	3.26	0.048	0.14	4.14	0.085	0.240	7.29	16
t'50 (min)	5.37	0.091	0.25	4.73	0.204	0.572	10.7	19
t'90 (min)	10.34	0.172	0.48	4.66	0.586	1.64	15.9	19
	Gr. Avg.	**0.079**	**0.222**	**4.11**	**0.202**	**0.564**	**9.39**	
Compound B:								
M_L (dN·m)	2.00	0.021	0.059	2.93	0.067	0.19	9.3	18
M_H (dN·m)	16.32	0.122	0.341	2.09	0.635	1.78	10.9	17
t_s1 (min)	2.78	0.023	0.066	2.37	0.069	0.19	6.9	14
t'10 (min)	3.00	0.027	0.077	2.55	0.086	0.24	8.0	16
t'50 (min)	4.61	0.032	0.091	1.97	0.090	0.25	5.4	16
t'90 (min)	8.10	0.081	0.226	2.79	0.303	0.85	10.5	18
	Gr. Avg.	**0.045**	**0.127**	**2.38**	**0.189**	**0.530**	**8.1**	
Compound C:								
M_L (dN·m)	2.23	0.026	0.074	3.30	0.063	0.18	7.9	17
M_H (dN·m)	10.68	0.118	0.329	3.08	0.391	1.10	10.3	17
t_s1 (min)	6.25	0.126	0.359	5.75	0.228	0.64	10.2	17
t'10 (min)	5.81	0.106	0.297	5.12	0.157	0.44	7.56	18
t'50 (min)	5.37	0.091	0.254	4.73	0.204	0.57	10.7	19
t'90 (min)	10.31	0.156	0.438	4.25	0.583	1.63	15.8	19
	Gr. Avg.	**0.093**	**0.263**	**4.40**	**0.209**	**0.586**	**9.3**	
Compound D:								
M_L (dN·m)	2.19	0.0098	0.027	1.25	0.034	0.100	4.4	9
M_H (dN·m)	31.52	0.245	0.685	2.17	0.536	1.50	4.8	9
t_s1 (min)	2.56	0.027	0.076	2.97	0.107	0.300	11.7	9
t'10 (min)	3.76	0.065	0.183	4.87	0.082	0.230	6.1	10
t'50 (min)	4.98	0.061	0.170	3.42	0.119	0.333	6.7	11
t'90 (min)	7.32	0.117	0.327	4.47	0.322	0.901	12.3	8
	Gr. Avg.	**0.103**	**0.288**	**3.73**	**0.233**	**0.65**	**8.3**	

[A] The final number of laboratories remaining in the ITP after (Option 1) deletion of outliers. Compounds A, B, and C had a total of 20 labs participating in the ITP.
[B] Compound D had a total of 12 labs participating in the ITP.
ML = minimum torque; MH = maximum torque; ts1 = time to 1 unit torque rise; t'10, t'50, and t'90 = times to 10, 50, and 90 %, respectively, of full cure time.

values for each day were averaged to get a single value for each day and the analysis was conducted on these day 1–day 2 test results. The precision output for Program 2 is listed in Table 2.

11.3.1 The analysis of the ITP data for Program 2 was conducted after Practice D4483 was revised (see 2004 published version). This Practice D4483 revision has improved procedures for outlier identification and subsequent deletion. The 2004 version of Practice D4483 permits identifying the core group of laboratories in any ITP that have good control over their testing operations and thus represent the industry benchmark for high quality testing. Note the last column of Table 2 that indicates the number of laboratories used for precision calculation after deletion of outliers.

11.4 The precision may be expressed in the format of the following statements that use an appropriate value of *r*, *R*, *(r)*, or *(R)*, that is, that value obtained from Table 1 or Table 2 for any of the measured properties, to be used in decisions about results obtained for that property, with the test method.

11.4.1 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value for any parameter as tabulated in Table 1 or Table 2. Two single test results, obtained under normal test method procedures that differ by more than this tabulated *r* must be considered as derived from different or non-identical sample populations.

11.4.2 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value for any parameter as tabulated in Table 1 or Table 2. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* must be considered to have come from different or non-identical sample populations.

11.4.3 Repeatability and reproducibility expressed as a percentage of the mean level, *(r)* and *(R)*, have equivalent application statements as cited above for *r* and *R*. For the *(r)* and *(R)* statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

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

12. Keywords

12.1 compounds; rheometer; rotorless cure meter; vulcanization characteristics

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