Designation: D2084 – 11 (Reapproved 2016)

Standard Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter[1]

This standard is issued under the fixed designation D2084; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the use of the oscillating disk cure meter for determining selected vulcanization characteristics of vulcanizable rubber compounds.

1.2 ISO 3417 is very similar to this test method. It has minor technical differences that are not considered to be significant.

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

1.4 *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

D3185 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Including Mixtures With Oil

D3186 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil

D3187 Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Rubber)

D3190 Test Method for Rubber—Evaluation of Chloroprene Rubber (CR)

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

2.2 *ISO Standard:*

ISO 3417 Rubber—Measurement of Vulcanization Characteristics With the Oscillating Disk Rheometer[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 The following measurements may be taken from the torque versus time curve (see Fig. 1).

3.1.2 *cure rate index*—measure of rate of vulcanization based on the difference between optimum vulcanization and incipient scorch time.

3.1.3 *peak cure rate*—measure of rate of vulcanization expressed as the maximum slope of the torque versus time curve.

3.1.4 *maximum, plateau, or highest torque*—measure of stiffness or shear modulus of the fully vulcanized test specimen at the vulcanization temperature.

3.1.5 *minimum torque*—measure of the stiffness of the unvulcanized test specimen taken at the lowest point of the curve.

3.1.6 *time to incipient cure (scorch time)*—measure of the time at which vulcanization begins.

3.1.7 *time to a percentage of full cure*—measure of cure based on the time to develop some percentage of the highest torque or difference in torque from the minimum.

3.1.8 *torque—for an oscillating shear cure meter*, the value measured by a torque transducer at the peak strain amplitude of the oscillating cycle.

3.1.9 *optimum cure time*—measure of the time required to reach a percentage of full cure that corresponds to a desired level of a property of the cured compound.

3.1.9.1 *Discussion*—The time to reach 90 % cure corresponds to a maximum in tensile strength for some rubber compounds. This does not apply in all cases.

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[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.

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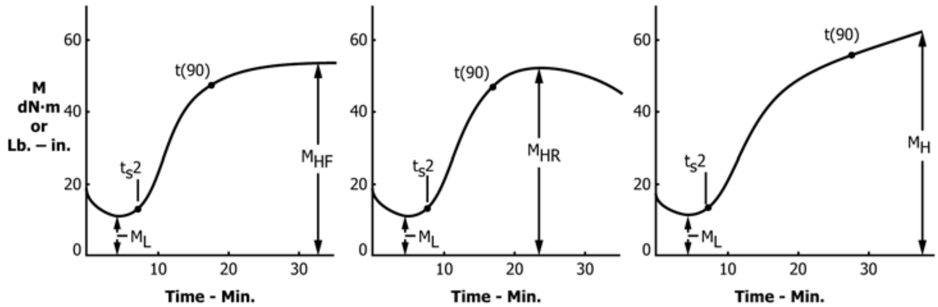
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.



Left Curve: Cure to Equilibrium Torque.
Middle Curve: Cure to a Maximum Torque with Reversion.
Right Curve: Cure to No Equilibrium in Maximum Torque.

FIG. 1 Types of Cure Curve

4. Summary of Test Method

4.1 A test specimen of vulcanizable rubber compound is inserted into the cure meter test cavity and after a closure action is contained in a sealed cavity under positive pressure. The cavity is maintained at some elevated vulcanization temperature. The rubber totally surrounds a biconical disk after the dies are closed (see Fig. 2). The disk is oscillated through a small rotational amplitude (1° or 3°) and this action exerts a shear strain on the test specimen. The force required to oscillate or rotate the disk to maximum amplitude is continuously recorded as a function of time, with the force being proportional to the shear modulus (stiffness) of the test specimen at the test temperature. This stiffness initially decreases as it warms up; then it increases due to vulcanization. The test is completed when the recorded torque either rises to an equilibrium or maximum value, or when a predetermined time has elapsed. The time required to obtain a cure curve is a function of the characteristics of the rubber compound and of the test temperature (see Fig. 1 for typical cure curves).

4.2 Several configurations of the oscillating disk cure meter are currently in use. Fig. 3 illustrates example shifts of the cure curves associated with the configuration differences included in this standard. Results between tests using rapid and slow temperature recovery, or between heated and unheated disks cannot be compared without taking the heating differences into account. The differences between test curves will vary with the compound being tested. Configurations included in this test method are listed in this section.

4.2.1 Diaphragm dies, unheated rotor, temperature recovery within 4.5 min.

4.2.2 Solid dies, unheated rotor, temperature recovery within 4.5 min.

4.2.3 Solid dies, unheated rotor, temperature recovery in less than 2 min.

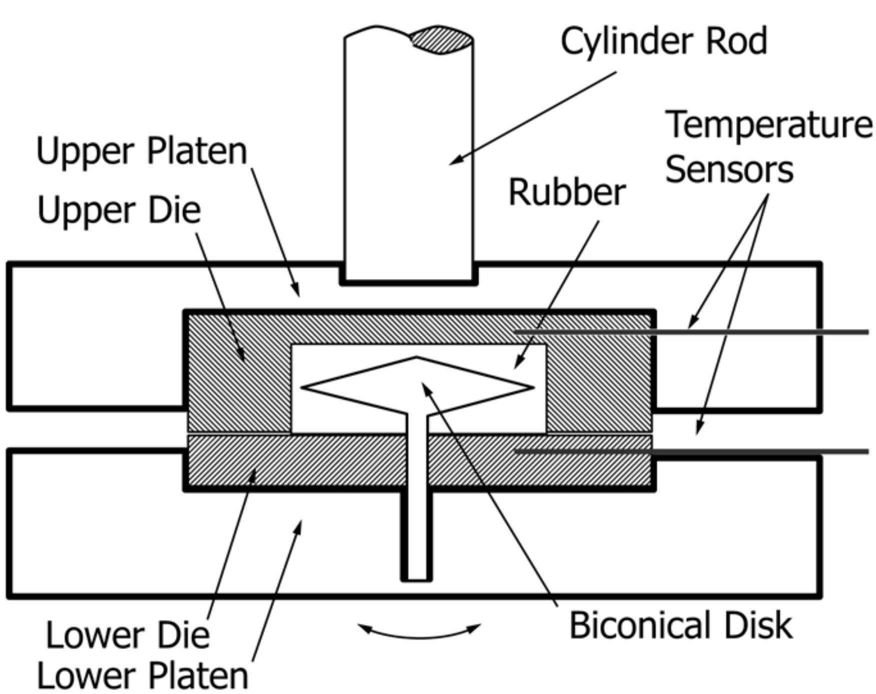


FIG. 2 Cure Meter Assembly

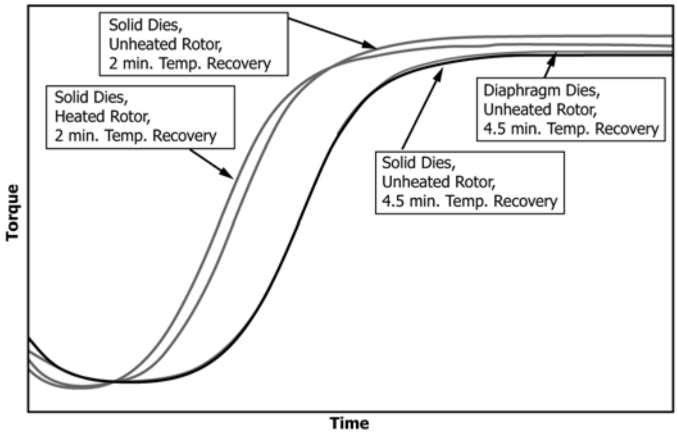


FIG. 3 Example Cure Curves from ODR Configurations

4.2.4 Solid dies, heated rotor, temperature recovery in less than 2 min.

NOTE 1—Diaphragm dies are unique to cure meters developed before rapid temperature recovery and heated rotors were introduced. Diaphragm dies in combination with rapid temperature recovery or heated rotors are not a normal configuration for Oscillating Disk Cure Meters.

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.

6. Apparatus

6.1 *Cure meter,* consists of the following major components: specimen chamber and closure mechanism, temperature control system, rotor drive and torque measuring system (see Fig. 2 for a detailed drawing of cure meter assembly).

6.2 *Specimen Chamber*—Consists of platens, dies, and a biconical disk.

6.2.1 *Platens*—Two platens made of aluminum alloy, each containing an electric heater, and each having in the center, a cavity to accommodate a die and from the side, a well for inserting a temperature sensor.

6.2.2 *Dies*—Two which form a cavity when closed and which shall be fabricated from tool steel having a minimum Rockwell Hardness HRC 50. The geometry of the standard dies is shown in Figs. 4-6 with dimensions and tolerances (see Table 1). The top and bottom surfaces of the die cavity shall contain rectangular-shaped grooves arranged radially about the center and spaced at 20° intervals. Each die shall have a well or hole drilled from the side to accommodate a temperature sensor inserted through the platen. The upper die may be either solid or diaphragm type. The lower dies shall have a hole in the center to allow for the insertion of the disk shaft. A suitable low-friction seal shall be provided in this hole to prevent material leaking from the cavity.

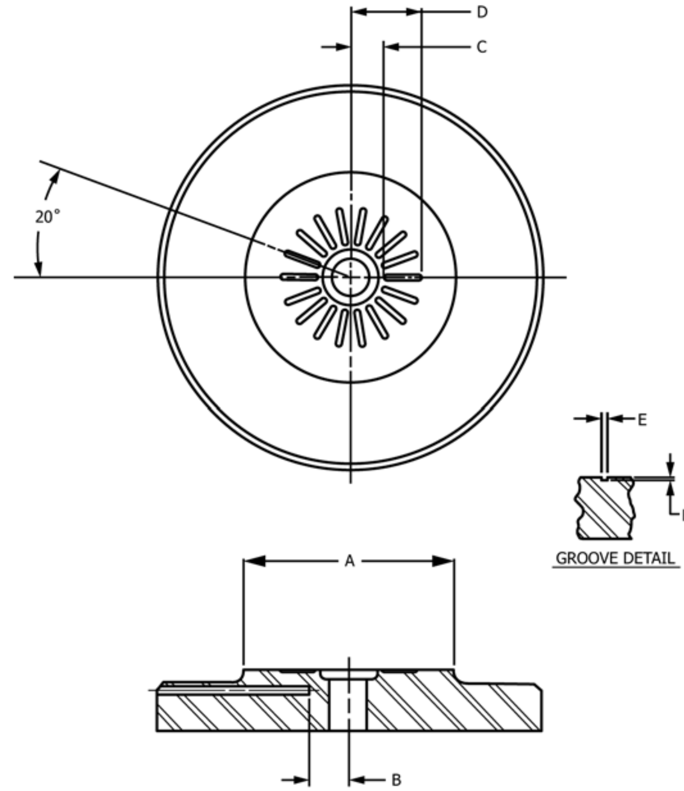


FIG. 4 Lower Die

6.2.2.1 *Diaphragm Upper Die*—Upper die manufactured so that the grooved die face is allowed to flex when closed on a specimen and then to maintain essentially constant pressure on the specimen as it shrinks slightly in volume during vulcanization. To provide thermal conduction to the metal body of the diaphragm die, an aluminum or stainless steel insert is placed in the diaphragm space with a hole designed to accommodate the temperature sensor. Fig. 5 describes the diaphragm type upper die.

6.2.2.2 *Solid Upper Die*—Upper die formed from one piece of steel, as described in Fig. 6.

6.2.3 *Disk*—The biconical disk shall be fabricated from tool steel having a minimum Rockwell Hardness of HRC 50. The disk shall be fitted with a stem that fits into the torque shaft. The disk is shown in Fig. 7 (see Table 2).

6.2.3.1 *Heated Disk*—Some manufacturers of oscillating disk cure meters offer a heated rotor as an option. If the disk is heated, both torque values and cure times may be significantly altered. The heated disk is a modification of the biconical disk shown in Fig. 7. This modification has provisions for directly controlling the disk temperature, as shown in Fig. 8. In this example, an electrical heater and temperature sensor are located in a metal tube, which is inserted in the disk through a vertical well in the disk shaft. The well is typically 0.325 cm (0.128 in.) in diameter and extends to within approximately 0.25 cm (0.100 in.) of the disk apex. The insertion tube is typically 0.0125 cm (0.005 in.) less than the well diameter to allow for easy tube removal for cleaning.

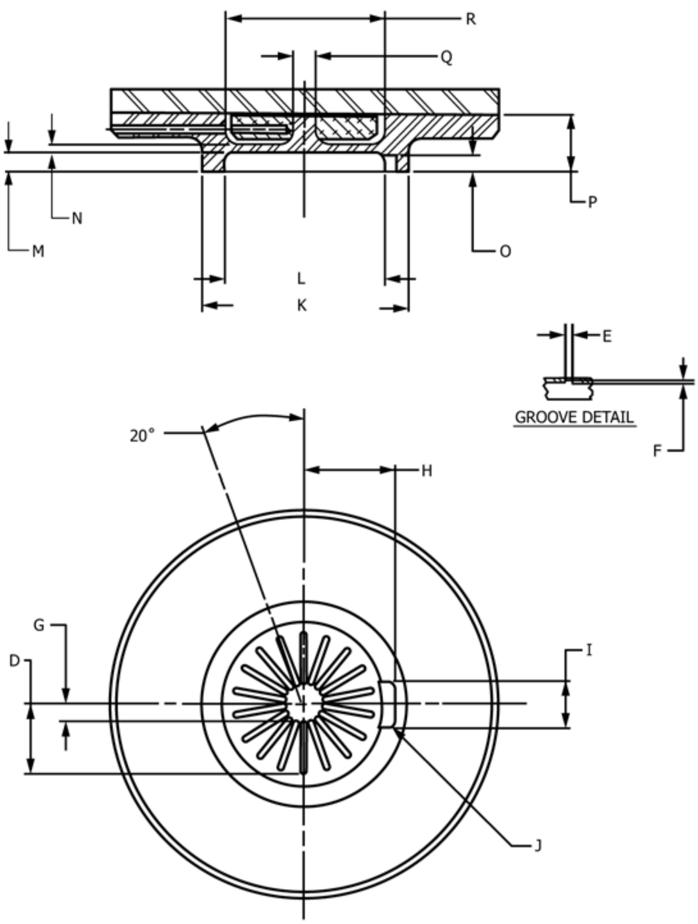


FIG. 5 Diaphragm Type Upper Die

6.2.3.2 Disk wear will affect test results. A disk worn to such an extent that the disk diameter is less than the minimum diameter shown in this procedure shall not be used.

6.2.3.3 The standard frequency of the rotary oscillation of the disk shall be constant at 1.67 Hz (100 cpm) ±1 %. Other frequencies may be used, if required.

6.2.3.4 A rotary drive system shall be provided for oscillatory rotation of the disk. The amplitude of oscillation of the unloaded disk shall be constant at ±1.00° with a tolerance of ±0.03° about the center position, that is, a total amplitude of 2°. Other amplitudes may be used, if specified.

NOTE 2—Disk and die surface contamination may contribute to slippage. Typically, torque values over 40 dNm may be subject to slipping, thus reducing torque values. Torque values approaching 100 dNm are also typically compromised by a significant torsion deformation of the disk shaft. Where slipping or torsion deformation is not a concern, greater sensitivity may be possible using ±3° arc of oscillation.

6.2.4 *Die Closing Mechanism*—A pneumatic cylinder or other device shall close the dies and hold them closed during the test with a force of 11.0 ± 0.5 kN (2500 ± 100 lbf).

NOTE 3—One manufacturer recommends the source air pressure be adjusted to 345 kPa (50 psi) for a 203-mm (8-in.) diameter air cylinder. Provisions are made for this adjustment to the instrument. This pressure acting on a 203-mm (8-in.) diameter air cylinder will produce a force of 11 kN (2500 lbf) on the die per the following equation:

$$F = P\left(\frac{\pi D^2}{4}\right) \quad (1)$$

where:

F = closure force on die,
P = source air pressure, and
D = diameter of piston in pneumatic cylinder.

To calculate maximum cavity pressure, the effect of this force acting on the surface area of the upper die may be calculated per the following equation:

$$P_c = \frac{4F}{\pi d^2} \quad (2)$$

where:

P_c = pressure on sample in upper die cavity, and
d = diameter of upper die cavity (55.9 mm (2.2 in.)).

$$\left(\text{For example},\quad P_c = \left(\frac{(4)(11)}{\pi\,(55.9)^2}\right) = 4485 \quad kPa = 650 \quad psi\right) \quad (3)$$

6.3 *Temperature Controlling System*—A temperature controller shall be provided for maintaining the dies within ±0.5°C (±1°F) of the specified test temperature.

6.3.1 *Heated Disk Temperature Control*—When the disk is heated, a temperature controller shall be provided for maintaining the disk temperature within ±0.5°C (±1°F) of the specified test temperature.

6.4 *Torque Measuring System*—The torque measuring system shall consist of a device, such as a torque transducer, producing a signal that is directly proportional to the torque required to oscillate the disk. A recording system, as used in this test method, may consist of any suitable data collection device, including computers, printers, plotters, and chart recorders. The recording system shall have a full-scale deflection response on the torque scale of 1 s or less and be capable of recording the torque with accuracy of ±0.5 % of the torque range. A minimum of four torque ranges shall be provided; 0 to 25, 0 to 50, 0 to 100, and 0 to 200 dN·m (or 0 to 25, 0 to 50, 0 to 100, and 0 to 200 lbf· in.)

NOTE 4—Direct proportionality between torque and stiffness cannot be expected under all test conditions, particularly in higher torque ranges, because elastic deformation of the disk shaft and driving device must be taken into account. However, for routine quality control test purposes corrections are not necessary.

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 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 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.

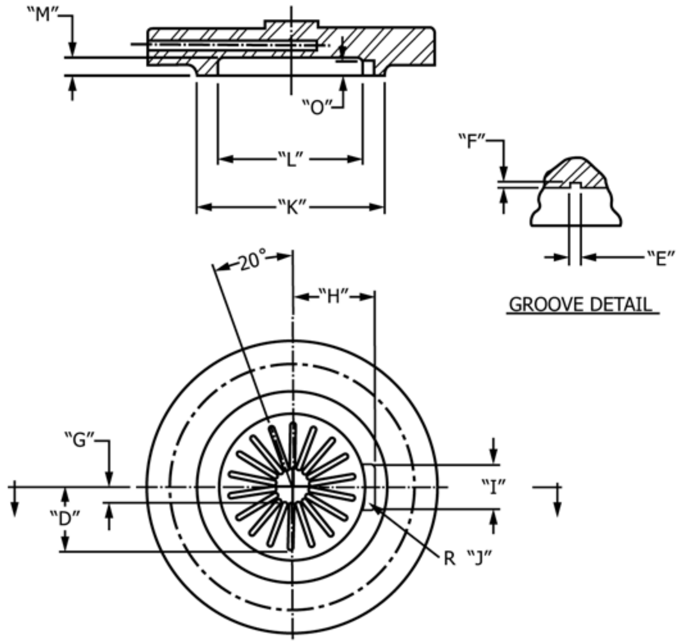


FIG. 6 Solid Type Upper Die

TABLE 1 Die Dimensions

Code	Dimension, mm	Tolerance, mm
A	55.88	±0.13
B	10.64	±0.25
C	7.94	±0.13
D	18.26	±0.13
E	1.57	±0.13
F	0.8	±0.1
G	4.76	±0.13
H	24.07	±0.06
I	12.70	±0.40
J	2.38	±0.40
K	54.61	±0.05
L	41.91	±0.03
M	5.35	±0.01
N	1.65	±0.03
O	4.57	±0.13
P	15.38	±0.03
Q	5.08	±0.05
R	41.91	±0.03

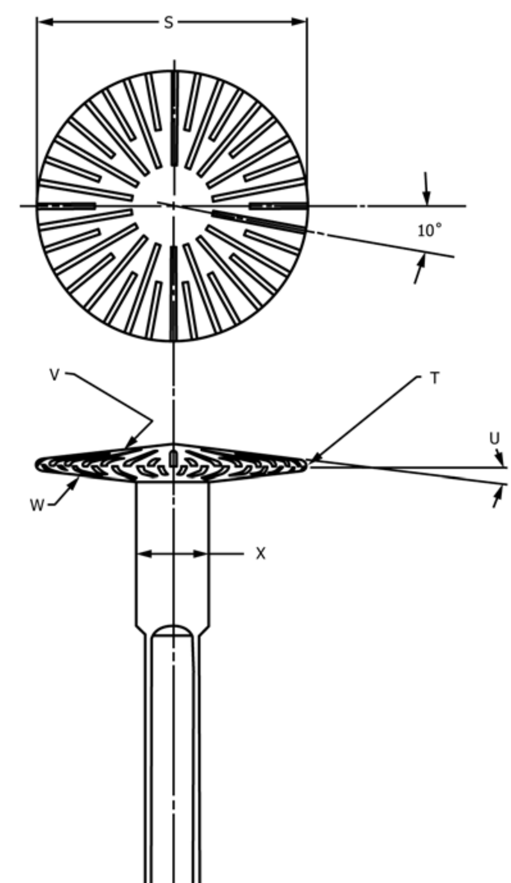


FIG. 7 Biconical Disk

8. Test Specimen

8.1 A nearly circular test specimen taken from a sample shall have a volume of 9.5 ± 1.5 cm^3 (0.58 ± 0.09 $in.^3$) (for example, approximately 30 mm (1.2 in.) in diameter and 13.5 mm (0.53 in.) in thickness).

8.2 The test specimen is considered to be of proper size when a small bead of compound is extruded uniformly around the periphery of the die as it is closed (116 to 160 % of the test cavity volume). This is achieved when the test specimen volume is between 8 and 11 cm^3 (9 to 13 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. Test Temperatures

9.1 The standard test temperature shall be 160°C (320°F).

TABLE 2 Disk Dimensions

	Code	Dimension, mm	Tolerance, mm
S	Diameter	35.55	±0.01
T	Radius	0.80	±0.03
U		6.28°	±0.08°
V[A]	Groove width	0.80	±0.05
	Groove depth	0.8	±0.1
	Groove lengths, min	7.5	
		12.5	
W[A]	Groove width	0.80	±0.05
	Groove depth	0.8	±0.1
	Groove lengths, min	7.5	
		9.5	
X	Diameter	9.50	±0.02
			−0.00

[A] Grooves on top and bottom surfaces should be displaced 5°.

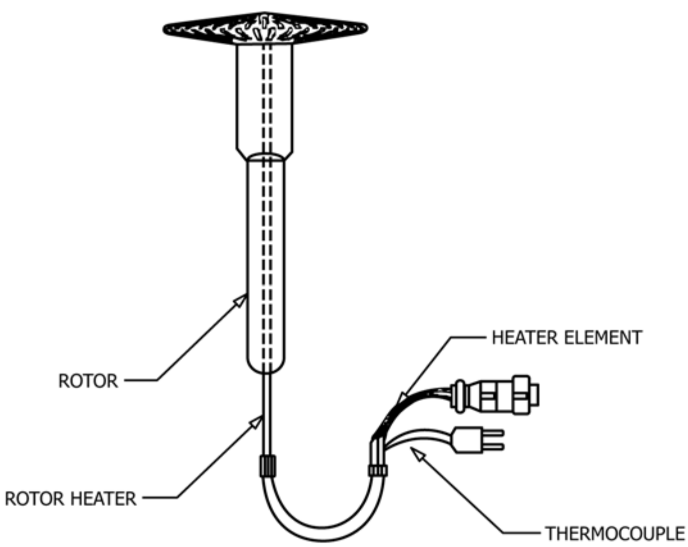


FIG. 8 Example of an ODC Rotor With Provision for Heating

9.2 The test temperature tolerance shall be ±0.5°C (±1.0°F).

9.3 Tests may be carried out at other temperatures, if required. They should be selected in accordance with Practice D1349.

10. Calibration

10.1 The cure meter shall be calibrated mechanically in accordance with the manufacturer's instructions.

10.2 Provisions shall be made for electronic verification of the recording system and for torque transducer calibration by means of a resistor incorporated in the torque measuring circuit that simulates an applied torque of specified value.

10.3 The cure meter shall be calibrated with a mechanical torque standard supplied by the manufacturer any time the results are suspected of being inaccurate, after any repairs, any change in arc, or frequently enough to ensure the maintenance of proper calibration. The cure meter shall read zero when running empty with no disk seal in place and read the certified value with the torque standard inserted.

11. Procedure

11.1 *Preparation for Test:*

11.1.1 Bring the temperature of both dies to the test temperature with the disk in place and the dies in the closed position. When a chart recorder is used, set recorder range to zero and adjust the recorder pen to zero torque and zero time position on the chart. Select the correct running time and choose the torque range to give maximum torque in the upper half of the recorder chart. Computer data acquisition systems may require none or different adjustments to properly record data, but may still require setting of the test time.

11.1.2 "Running Zero" with the disk seal in place may be checked at this point and should be off no more than 0.5 dN·m (or 0.5 lbf·in.). If the torque is higher, check the cure meter for frictional drag that could be caused by bad bearings, excessive seal friction (6.2.2), rotor misalignment, or by sample "build-up" around the rotor shaft. If the error persists, consult the manufacturer's manual.

11.2 *Loading the Cure Meter:*

11.2.1 Open the dies, place the test specimen (Note 5 and Note 6) on top of the disk and close the dies. Placement of the test specimen and activation of die closure shall be completed within 20 s. When running a test where the cure meter die cavity is empty prior to testing, the rotor shall be in place a minimum of 1 min before opening the dies to place the test specimen. When loading a sample immediately following a previous test, the process of removing the tested specimen and placing the new test specimen shall be completed within 20 s. If test specimen removal takes more than 20 s, replace the rotor and close the dies on the empty cavity for 1 min before loading the next test specimen.

11.2.2 The recording system shall start at the instant the dies are closed. In some instruments, the operator must start the recorder. In others, the recorder starts automatically. The disk may be oscillating at zero time or oscillation may be started not later than 1 min after the dies are closed. In the latter case, report preheat time as required in 12.1.8.

NOTE 5—When testing sticky rubber compounds, thin film that will not melt at the test temperature may be inserted below and above the test specimen, but not against the rotor, to prevent the rubber from sticking to the dies.

NOTE 6—A material deposit from the rubber compounds under test may build up on the disk and dies. This may affect the final torque values. It is suggested that stable vulcanizable rubber compound be tested daily to detect this occurrence. If such contamination develops, it may be removed by cleaning with a noncorrosive compound or solution that does not degrade the aluminum insert contained in some diaphragm dies. After solvent cleaning one or two runs on a nonessential rubber compound are required to eliminate solvent or residue completely. Abrasive cleaning may be used with caution. The recommended abrasive cleaning agent is 220 grit aluminum oxide.

12. Report

12.1 Report the following information on the sample and instrument used:

12.1.1 Sample identification,

12.1.2 Method of specimen preparation (for example, amount of milling),

12.1.3 Make and model of the cure meter,

12.1.4 Type of dies, unheated or heated rotor, and temperature recovery classification,

12.1.5 Die Temperature,

12.1.6 Oscillation amplitude used, reported as half of total amplitude (for example, ±1° arc is reported as 1° arc),

12.1.7 Oscillation frequency, Hz (or cpm),

12.1.8 Test time, and preheat time, if not zero, and

12.1.9 Recording scales for time and torque.

12.2 Test results reported are normally chosen from the following parameters (refer to Fig. 1 for guidance). The conversion from dN·m to lbf·in. is: 1.13 (dN·m) = 1.00 (lbf·in.).

12.2.1 M_L—Minimum torque, dN·m (lbf·in.), also referred to as M_L.

12.2.2 *Maximum torque*—All in dN·m (or lbf·in.).

12.2.2.1 M_{HF}—Maximum torque where curve plateaus.

12.2.2.2 M_{HR}—Maximum torque of reverting curve.

12.2.2.3 M_H—Highest torque attained during specified period of time when no plateau or maximum torque is obtained, also referred to as M_H.

12.2.3 Scorch time, min.

12.2.3.1 t_S1 is equal to the time to 1 dN·m (or lbf·in.) rise above M_L; is used with 1° oscillation amplitude.

12.2.3.2 t_S2 is equal to the time to 2 dN·m (or 2 lbf·in.) rise above M_L; is used with 3° (and 5°) oscillation amplitudes.

12.2.4 Cure time, min.

12.2.4.1 t' x (also TCx) is equal to the time to x % of torque increase or t'x = minutes to $M_L + x(M_H - M_L)/100$ torque.

NOTE 7—This test method of determining the cure times is considered the standard. The most commonly used values of x are 10, 50, and 90.

12.2.4.2 $t$xis equal to the time to x % of maximum torque, or t x = minutes to x $M_H/100$ torque.

NOTE 8—This is an alternative test method for cure time determination.

12.2.5 Cure Rate Index = 100/(cure time – scorch time).

12.2.6 Peak or Maximum Cure Rate (also PCR or MCR) is the highest slope obtained for the torque versus time curve after minimum torque has been plotted, usually in dNm/min (or lbf-in./min).

12.2.6.1 Time to Peak or Maximum Cure Rate (also TPCR or TMCR) is the test time at the point where maximum cure rate is reached.

13. Precision and Bias

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

13.2 Two precision studies were conducted, one using the diaphragm upper die shown in Fig. 5, and one using a solid upper die as shown in Fig. 6.

13.3 Both Type 1 and Type 2 precision results of Oscillating Disc Cure Meters (ODC) with diaphragm upper dies are given in Table 3. For both types of precision repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the test value, as specified by this test method, obtained on one determination or measurement of the property or parameter in question.

13.4 For the Type 1 precision, four compounds (or materials) were used; these were tested in eleven laboratories on two different days (see Table 3).

13.5 For the Type 2 precision, the precision results reported in Table 3 represent pooled average values obtained from four (other) rubber evaluation standards: Test Methods D3185 (SBR, OE-SBR), D3186 (SBR-BMB), D3187 (NBR), and D3190 (CR). These precision values are derived from interlaboratory programs with two different types of materials (for each rubber as listed above), in seven laboratories with the mixing and testing both conducted on two different days essentially one week apart.

13.6 ISO TC 45 conducted a Type 1 precision study of Oscillating Disk Cure Meters with solid upper dies in 1984 and 1985 using ISO 3417. ISO 3417 is analogous to Test Method D2084. The practice for analysis and expression of results for ISO TC 45 is equivalent in its basic fundamentals and format to Practice D4483.

13.7 *ISO TC 45 Test Details:*

13.7.1 An interlaboratory test program (ITP) was organized in late 1984 to obtain precision results. Four compounds with a range of cure properties were mixed and prepared in one laboratory, sealed in metal foil packets, and distributed to

TABLE 3 Precision (Diaphragm Upper Die)

NOTE 1—S_r = within laboratory standard deviation. r = repeatability (in measurement units). (r) = repeatability (in percent). S_r = between laboratory standard deviation. R = reproducibility (in measurement units). (R) = reproducibility (in percent).

Test Parameter	Range of Values	Mean Value	Within Laboratory			Between Laboratory		
			Sr	*r*	$(r)^A$	*SR*	*R*	$(R)^A$
Type 1 Precision:								
M_L (dN·m)	7.1 to 9.7	8.4	0.158	0.45	5.4	1.36	3.85	45.8
M_H (dN·m)	28.4 to 38.9	33.7	0.181	0.51	1.5	1.98	5.60	16.6
t_sl (min)	2.3 to 5.3	3.8	0.12	0.34	9.0	0.25	0.71	18.7
t' 50 (min)	3.9 to 8.8	6.4	0.15	0.43	6.7	0.34	0.96	15.0
t' 90 (min)	6.4 to 14.0	10.2	0.20	0.57	5.6	0.60	1.70	16.7
Type 2 Precision:								
M_L (dN·m)	7.6 to 9.8	8.7	0.215	0.61	7.0	0.735	2.08	23.9
M_H (dN·m)	32.0 to 47.7	39.9	0.35	0.99	2.5	2.60	7.36	18.4
t_s l (min)	4.4 to 5.8	5.1	0.12	0.34	6.7	0.37	1.05	20.6
t' 50 (min)	7.7 to 9.9	8.8	0.18	0.51	5.8	0.90	2.55	29.0
t' 90 (min)	15.9 to 18.0	17.0	0.19	0.54	3.2	1.50	4.25	25.0

[A] These are estimated values, using the mid-point of the range for the parameter mean value.

Type 1 precision is obtained from fully prepared test specimens (compounds mixed in one laboratory); these are circulated to all participating laboratories.

Type 2 precision is obtained by circulating all compounding materials (drawn from a common source) to each participating laboratory. The mixing to prepare the compound is done in each laboratory and therefore mixing variation is part of the "total test" variation or test precision.

laboratories located in 19 countries in Europe, Asia, and North and South America. Tests were conducted in late January and early February 1985 according to the following schedule:

13.7.1.1 Part I at 160°C—One test (determination) on each of two days, one week apart, for all four compounds.

13.7.1.2 Part II at 150°C—One test (determination) on each of two days, one week apart, for all four compounds.

13.7.2 Formulations for the four compounds are listed in Table 4. Compound A has a moderate carbon black level with a non-free sulfur (TMTD) cure system. Compounds B and C are relatively high black with conventional cure systems. Compound D is a gum compound with a conventional cure system.

13.7.3 Type 1 precision was measured in the ITP (no processing operations required on the circulated materials in any participating laboratory). The time period for repeatability and reproducibility is on a scale of days.

TABLE 4 Compound Formulations (ISO 3417-ITP)

Material	Formulations Used			
	A	B	C	D
SBR 1502	100.0	...	...	100.0
SBR 1712[A]	...	68.8	137.5	...
BR (CB441)[B]	...	68.8	...	...
Zinc oxide	5.0	5.0	5.0	5.0
Stearic acid	1.0	1.5	1.5	1.5
IRB Number 5[C]	...	80.0	60.0	...
N330	45.0	...	...	...
Process Oil[D]	...	8.8	5.0	5.0
DPPD[E]	...	1.5	1.5	1.5
Antiozonant[F]	...	1.5	1.5	1.5
TBBS[G]	...	1.2	1.0	1.0
TMTD[H]	3.0	...	...	...
Sulfur	...	2.0	2.0	2.0
Specific Gravity	1.13	1.16	1.16	0.98

[A] 37.5 (phr) oil extended SBR.
[B] 37.5 (phr) oil extended, BR rubber.
[C] ASTM Committee D24 Industry Reference Carbon Black Number 5.
[D] Sundex 7260T or equivalent.
[E] Dimethyl-butylphenyl-phenylene diamine.
[F] Trimethyl-dihydroquinoline.
[G] N-tert-butyl-2-benzothiazole-sulfenamide.
[H] Tetramethylthiuram disulfide.

13.7.4 A total of 50 laboratories participated in Part I, and 45 laboratories participated in Part II, in addition to their participation in Part I.

13.7.5 Part I (160°C) precision results are given in Table 5.

13.7.6 Part II (150°C) precision results are given in Table 6.

13.8 Precision of this test method is expressed in the format of the following statements that use what is called an "appropriate value" of *r*, *R*, (*r*), or (*R*), that is, that value obtained from Table 3, Table 5, and Table 6 to be used in decisions about test results (obtained with the test method).

13.9 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value for any parameter as tabulated in Table 3, Table 5, and Table 6. 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 nonidentical sample populations.

13.10 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value for any parameter as tabulated in Table 3, Table 5, and Table 6. 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 nonidentical sample populations.

13.11 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as 13.9 and 13.10 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.

13.12 *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.

14. Keywords

14.1 compounds; ODR oscillating disk cure meter; vulcanization characteristics

TABLE 5 ISO 3417: Type 1—Precision of 160°C, Solid Upper Dies

Parameter 1—Min torque, ML (N-M) 160°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
4. Compound D	0.39	0.0315	0.0892	23.040	0.1404	0.3973	102.649
3. Compound C	0.70	0.0316	0.0893	12.700	0.1997	0.5652	80.380
2. Compound B	0.99	0.0339	0.0960	9.652	0.4399	1.2448	125.147
1. Compound A	1.65	0.0656	0.1857	11.249	0.1442	0.4081	24.715
Pooled values	0.92	0.0432	0.1223	13.298	0.2645	0.7486	81.422

Parameter 2—Max torque, MHF (N-M) 160°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
4. Compound D	2.21	0.0582	0.1646	7.447	0.1996	0.5648	25.550
3. Compound C	2.71	0.0511	0.1446	5.328	0.2207	0.6246	23.007
2. Compound B	3.26	0.0750	0.2123	6.521	0.2625	0.7430	22.820
1. Compound A	4.26	0.0546	0.1544	3.621	0.3628	1.0266	24.073
Pooled Values	3.11	0.0605	0.1713	5.506	0.2686	0.7602	24.432

Parameter 3—Scorch time, (min) 160°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
1. Compound A	1.41	0.1028	0.2910	20.580	0.1547	0.4377	30.957
4. Compound D	5.04	0.2017	0.5708	11.324	0.7011	1.9842	39.364
2. Compound B	5.08	0.1836	0.5197	10.222	0.7278	2.0598	40.512
3. Compound C	5.57	0.2302	0.6515	11.689	0.6752	1.9109	34.284
Pooled Values	4.32	0.1854	0.5247	12.140	0.6234	1.7642	40.822

TABLE 6 ISO 3417: Type 1—Precision of 150°C, Solid Upper Dies

NOTE 1—*Sr* = repeatability standard deviation. *r* = repeatability = 2.83 (square root of the repeatability variance). (*r*) = repeatability (as percentage of material average). *SR* = reproducibility standard deviation. *R* = reproducibility = 2.83 (square root of the reproducibility variance). (*R*) = reproducibility (as percentage of material average).

Parameter 1—Min torque, ML (N-M) 150°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
4. Compound D	0.40	0.0300	0.0848	21.000	0.1286	0.3639	90.135
3. Compound C	0.69	0.0403	0.1141	16.448	0.1025	0.2901	41.798
2. Compound B	0.77	0.0258	0.0730	9.511	0.0743	0.2101	27.376
1. Compound A	1.74	0.0517	0.1463	8.399	0.1616	0.4574	26.250
Pooled Values	0.90	0.0384	0.1086	12.098	0.1221	0.3457	38.505

Parameter 2—Max torque, MHF (N-M) 150°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
4. Compound D	2.31	0.0324	0.0916	3.960	0.1942	0.5495	23.768
3. Compound C	2.82	0.0684	0.1937	6.861	0.2431	0.6878	24.364
2. Compound B	3.39	0.0642	0.1818	5.354	0.2654	0.7511	22.124
1. Compound A	4.34	0.0787	0.2228	5.134	0.2464	0.6972	16.066
Pooled Values	3.20	0.0633	0.1793	5.594	0.2397	0.6783	21.169

Parameter 3—Scorch time, (min) 150°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
1. Compound A	1.99	0.1141	0.3230	16.265	0.2471	0.6992	35.214
2. Compound B	8.61	0.4932	1.3958	16.213	1.2572	3.5577	41.325
4. Compound D	8.77	0.2524	0.7143	8.149	0.6697	1.8951	21.621
3. Compound C	9.73	0.3233	0.9150	9.406	1.3838	3.9161	40.257
Pooled Values	7.31	0.3266	0.9243	12.650	0.9867	2.7924	38.217

Parameter 4—50 % cure time, (min) 150°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
1. Compound A	5.56	0.2100	0.5943	10.691	0.4654	1.3172	23.696
2. Compound B	13.34	0.5602	1.5852	11.882	0.9905	2.8032	21.011
3. Compound C	17.20	0.3975	1.1250	6.539	1.1619	3.2883	19.113
4. Compound D	17.56	0.3393	0.9603	5.470	1.1049	3.1269	17.812
Pooled Values	13.46	0.3983	1.1272	8.374	0.9726	2.7525	20.448

Parameter 5—90 % cure time, (min) 150°C
Final Summary Table: Precision Values
Averages given in increasing order

Material	Average	Within Laboratories			Between Laboratories		
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)
1. Compound A	11.91	0.6954	1.9680	16.521	1.2683	3.5894	30.133
2. Compound B	19.90	0.7055	1.9964	10.031	1.4111	3.9934	20.065
3. Compound C	30.07	0.6387	1.8075	6.010	2.1003	5.9439	19.763
4. Compound D	33.52	0.9787	2.7699	8.264	2.5473	7.2088	21.507
Pooled Values	23.91	0.7671	2.1708	9.080	1.9092	5.4030	22.599

APPENDIX

X1. HISTORY OF THE OSCILLATING DISK CURE METER

X1.1 Oscillating disk cure meters were first made commercially available in 1963. The first units oscillated at a frequency of three cycles per minute, typically at ±3° of arc. The dies (SCD) for these early cure meters were commonly a 2-in. square cavity 0.4 in. high, with a biconical rotor centered in the cavity. A typical rubber sample of 1.15 specific gravity weighed 22 g, and was loaded in two pieces, above and below the rotor. A20 to 60-s preheat was required after closure before collecting data. The strain on the sample at 3° arc was 21 %.

X1.2 Frequencies of oscillation of 10, 100, and 900 cpm were made available over the next five years. These created different curve shapes due to the heat energy added to the cavity in working the rubber, breakdown of polymer structure when curing under dynamic conditions, and the shear rate dependence of the rubber flow resistance. Fig. X1.1 compares the cure meter curves for the various speeds of oscillation using the square dies and an arc of ±3°.

X1.3 When the oscillating disk cure meter was first proposed as an ASTM standard in 1968, a smaller, production-sized table model of the cure meter was introduced along with a new die that was 2 in. in diameter (LPC dies). This circular die had the same height and used the same rotor as the square dies. The LPC die produced similar torque values for minimum and maximum torques as the SCD die. The sample could be loaded as one piece on top of the rotor for most stocks. The practice of adding a preheat, as commonly used with the SCD die, was eliminated. The elimination of the preheat time led to slightly faster cures for the LPC die. Another advantage of this die was the flat lower die surface which allowed easier removal of the cured sample for most stocks.

X1.4 In 1971, a smaller version of the LPC die, called the MPC die, was adopted in conformance with Test Method D2084. The MPC die used a rotor with a different conical angle. At 3° of arc, the strain is 48 % versus 21 % for the larger dies and rotor. Higher strain leads to higher torques, and the smaller specimen (10 to 12 g for a stock with a specific gravity of 1.15) gives a cure curve with a significantly different shape, as shown in Fig. X1.2. Higher torques have been shown to cause slippage at the surface of the rotor for many stocks, and that slippage is a potential source of variation in test results. Studies conducted with the smaller dies and rotor indicated slippage due to rotor contamination was common above 50 in.·lbs of torque. As a result, Test Method D2084 specifies a 1° arc of oscillation as standard, with a 16 % shear strain.

X1.5 The curves obtained with MPC dies at 1° arc were used as standards until 1987, when further reductions in the temperature recovery time became possible due to improved temperature controllers.

X1.6 Another improvement introduced at this time was the reduction of the mechanical compliance of the rotor drive system for improved reproducibility between instruments. The improved mechanical design increased maximum torques, but lowered minimum torques by reducing friction. Faster temperature recovery has two advantages: first, the cure is closer to the desired cure temperature of the test for more accurate results; second, the potential for variation in results due to cooling of the rotor during loading and unloading is greatly reduced. Table X1.1 illustrates the potential operator effects.

X1.7 As part of the continuing evolution in cure meter design, a number of manufacturers have introduced rotorless cure meters. These cure meters use a sample of 3 to 5 g, with a thinner cross section to obtain more rapid temperature recovery and more uniform temperature throughout the specimen. The dies are usually directly heated, and smaller in mass than for the oscillating disk cure meters, so that faster temperature recovery can be achieved. By eliminating the rotor, the surface area under load is reduced so that smaller torque values are achieved. Faster temperature recovery leads to faster cure times. Fig. X1.3 compares typical cure curves for an SBR stock from the rotorless and oscillating disk cure meters.

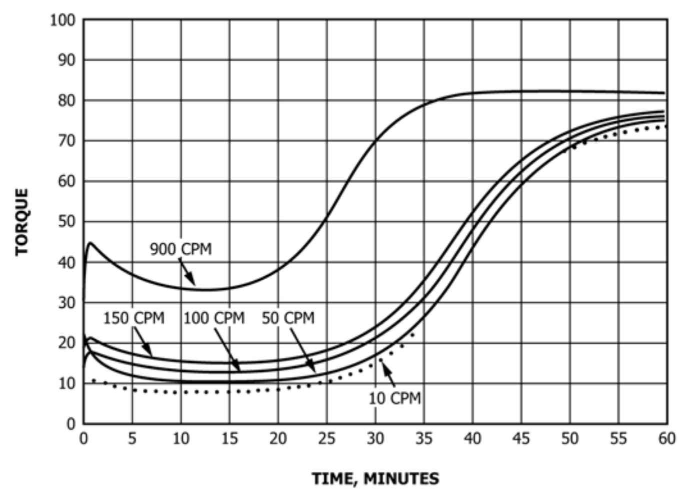


FIG. X1.1 Comparison of Oscillation Frequencies Using the Square Die and ±3° Arc

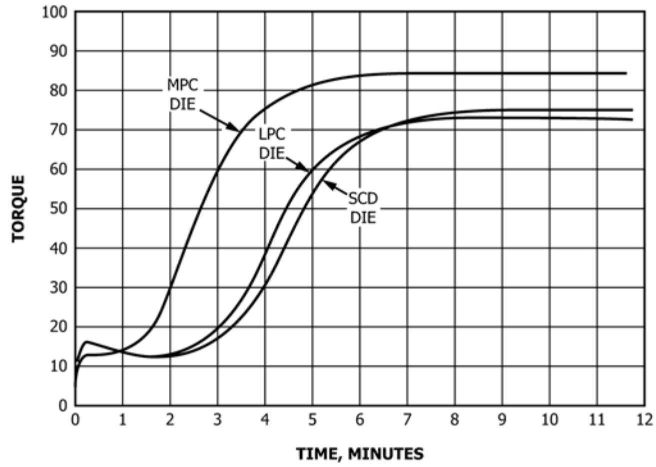


FIG. X1.2 Comparison of Cure Meter Dies

TABLE X1.1 Operator Effects on ODR Cure Meter Tests

Sample Loading Variable	Cure Times, min		
	TS2	T'50	T'90
Slow Temperature Recovery:			
Optimum loading time (20 s)	2.01	3.51	5.86
2-min loading time	2.33	3.90	6.33
% change with loading time	15.9	11.1	8.0
Rapid Temperature Recovery:			
Optimum loading time (20 s)	1.71	3.06	5.40
2-min loading time	1.86	3.23	5.60
% change with loading time	8.8	5.6	3.7

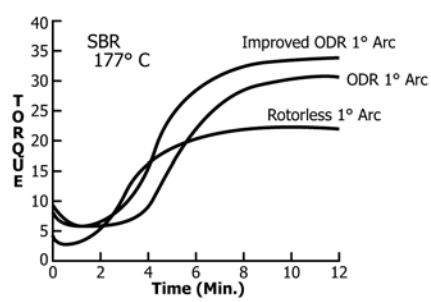


FIG. X1.3 Comparison of Cure Meters Using SBR Stock