ASTM

Designation: D 2084 – 01

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

This standard is issued under the fixed designation D 2084; 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 Department of Defense.

1. Scope

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

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

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:*

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

D 3185 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Including Mixtures With Oil[2]

D 3186 Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil[2]

D 3187 Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Rubber)[2]

D 3190 Test Methods for Rubber—Evaluation of Chloroprene Rubber (CR)[2]

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

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

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

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

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

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

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 first 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
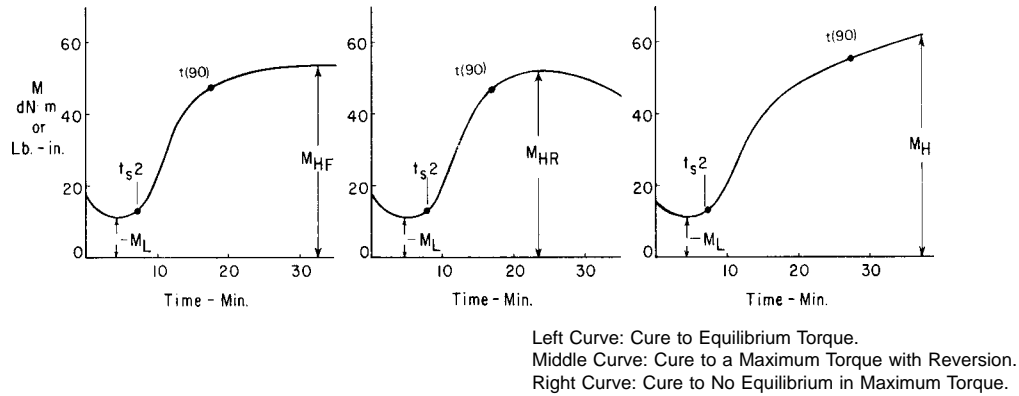
Left Curve: Cure to Equilibrium Torque.
Middle Curve: Cure to a Maximum Torque with Reversion.
Right Curve: Cure to No Equilibrium in Maximum Torque.
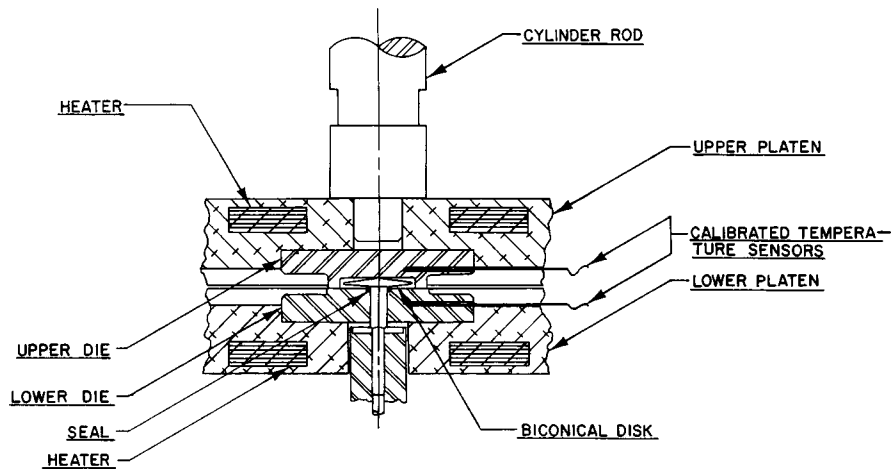
FIG. 1 Types of Cure Curve

FIG. 2 Cure Meter Assembly

characteristics of the rubber compound and of the test temperature (see Fig. 1 for typical cure curves).

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 Fig. 3 and Fig. 4 with dimensions and tolerances (see Table 1). The top and bottom surfaces of the die cavity shall contain rectangularly 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 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.

6.2.3 *Disk*—The biconical disk (see Fig. 5) 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. 5 (see Table 2).

6.2.3.1 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.

6.2.3.2 The standard frequency of the rotary oscillation of the disk shall be constant at 1.7 Hz (100 cpm). Other frequencies may be used, if required.

6.2.3.3 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 $\pm 1.00°$ with a tolerance of $\pm 0.03°$ about the center position, that is, a total amplitude of 2°. Other amplitudes may be used, if specified.

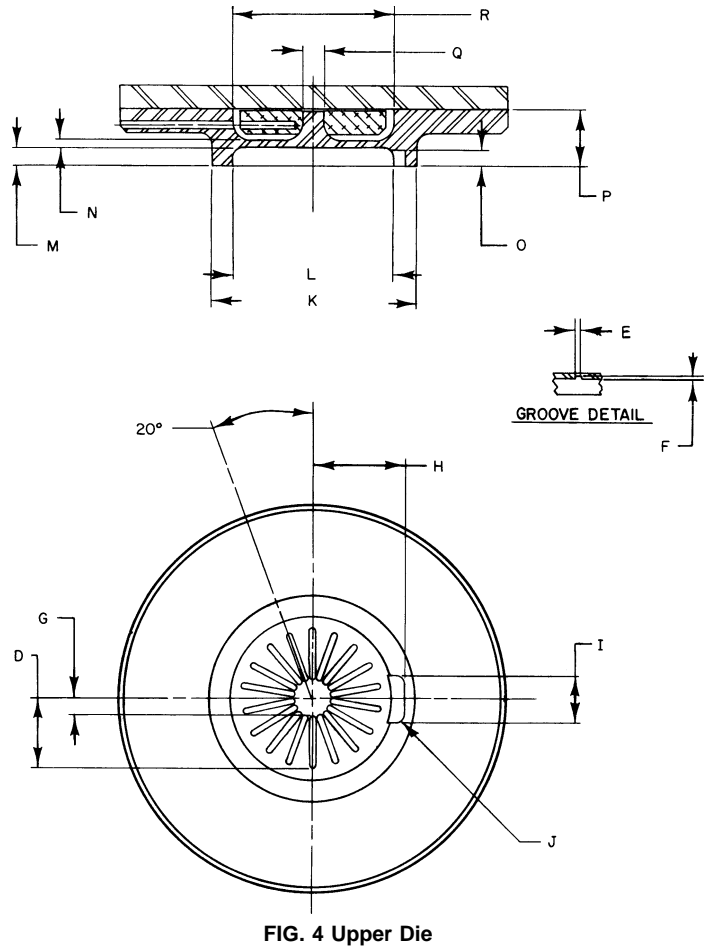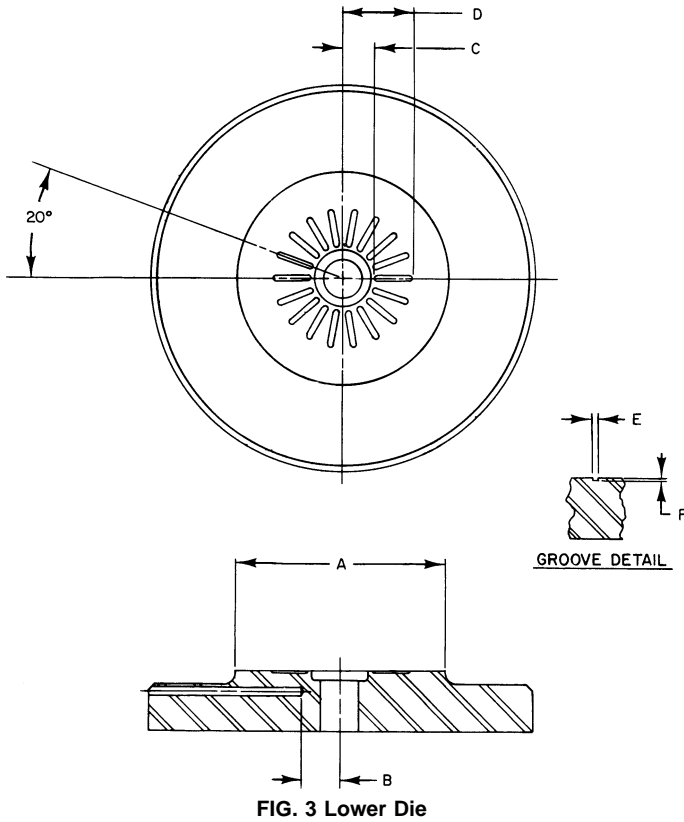NOTE 1—Where the effect of surface contamination on the disk is not

FIG. 3 Lower Die

a problem, greater sensitivity in determining curing characteristics may be obtained using ±3° angle 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 2—The manufacturer recommends the source air pressure be adjusted to 345 kPa (50 psi). Provisions are made for this adjustment in the instrument. Therefore, 345 kPa acting on the instrument's 203-mm (8-in.) diameter air cylinder will produce a force of 11 kN (2500 lbf) on the die per the following equation:

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

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:

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

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

$$\left(\text{For example, } P_c = \left(P_c\right.\right.$$
$$= \frac{(4)(2500)}{(3.14)(2.2)^2}$$
$$= \frac{10\ 000}{(3.14)(4.84)}$$

FIG. 4 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

$$= \frac{10\ 000}{12.06}$$
$$\left. = 829 \text{ psi.}\right) \qquad (3)$$

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.
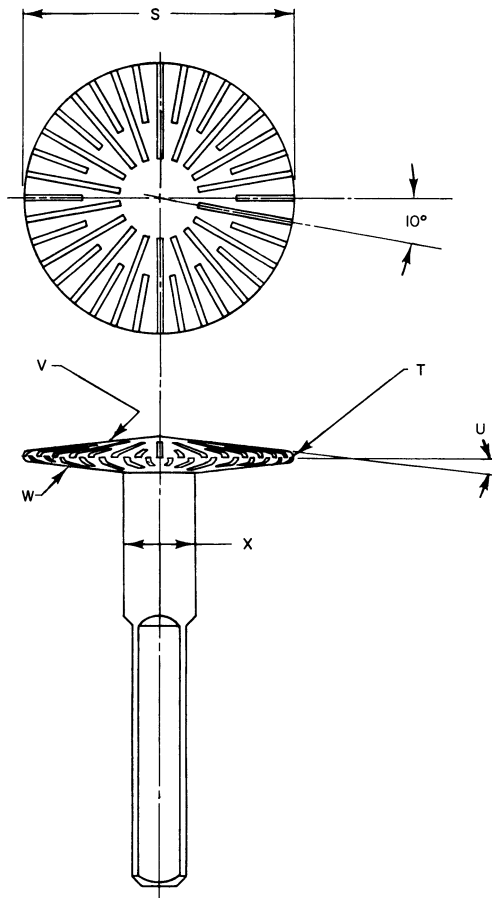
FIG. 5 Biconical Disk

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°.

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 recorder to record the maximum amplitude signal from the torque transducer shall be provided. The recorder 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. 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 3—The term "recorder" as used in this test method implies the use of any suitable data collection device, including printers, plotters, and computers.

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.

8. Test Specimen

8.1 A circular test specimen taken from a sample shall be 30 ± 2 mm (1.2 ± 0.1 in.) in diameter and 11.5 ± 1.5 mm (0.45 ± 0.05 in.) in thickness or equivalent to a volume of 9 cm^3 (0.5 in.^{3}).

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. This is achieved when the specimen volume is between 8 and 11 cm^3(9 to 13 g of rubber compound with a specific gravity of 1.15). Undersized specimens can cause low cavity pressure and low torque readings. Oversized specimens cool the dies excessively during the early part of the test cycle affecting the vulcanization characteristics.

9. Test Temperatures

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

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 D 1349.

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 recorder 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 the 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 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 temperature of test with the disk in place and the dies in the closed position. 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.

11.1.2 "Running Zero" may be checked at this point and should be off no more than 0.5 dN·m (or 0.5 lbf·in.). If in excess of this, check the cure meter for frictional drag that could be caused either by bad bearings, by improper friction seal (6.2.2), by rotor misalignment, or by sample "build-up" around the rotor shaft. If the error persists, consult the manufacturers 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. This operation must be completed within 20 s.

11.2.2 Start the recorder at the instant the dies are closed. 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 as required in 12.1.8.

Note 5—When testing sticky rubber compounds, cellophane, or other thin film that will not melt at the test temperature, may be inserted below and above the test specimen, but not against the rotor. This prevents the rubber from sticking to the dies. Suitable film materials are Dupont 600 PD/001,[4] Avisco 300P1,[5] or Polyester Film Mylar Type A-100.[6]

Note 6—A deposit of material 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 the upper die of most cure meters. 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 cleaner is 220 grit aluminum oxide.

12. Report

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

12.1.1 Sample or specimen identification, or both,

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 Temperature of the dies,

12.1.5 Amplitude of oscillation used, reported as half of total amplitude,

12.1.6 Frequency of oscillation, Hz (or cpm),

12.1.7 Recorder range,

12.1.8 Full-scale recorder time, and

12.1.9 Preheat time, if not zero.

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.).

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.

12.2.3 Scorch time, min.

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

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

12.2.4 Cure time, min.

12.2.4.1 t'x 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 test method.

12.2.4.2 tx is equal to the time to x % of maximum torque, or tx = minutes to x M_H/100 torque.

Note 8—This is an alternative test method for cure time determination. The most commonly used values of x are 50 and 90.

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

13. Precision and Bias

13.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

13.2 All precision data were obtained using the diaphragm upper die shown in Fig. 4. The use of the alternative solid die shown in Fig. A1.1 may cause different results.

13.3 Both Type 1 and Type 2 precision results 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 D 3185 (SBR, OE-SBR) and Methods D 3186 (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 The precision of this test method may be 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, to be used in decisions about test results (obtained with the test method).

13.7 *Repeatability*—The repeatability, r, of this test method has been established as the appropriate value for any parameter as tabulated in Table 3. 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.

13.8 *Reproducibility*—The reproducibility, R, of this test method has been established as the appropriate value for any parameter as tabulated in Table 3. 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.

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[4] Dupont 600 PD/001 is suitable for this purpose.
[5] Avisco 300P1 is suitable for this purpose.
[6] Polyester Film Mylar Type A-100 is suitable for this purpose.

TABLE 3 Precision

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_sl (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.

13.9 Repeatability and reproducibility expressed as a percentage of the mean level, (r) and (R), have equivalent application statements as 13.7 and 13.8 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.

13.10 *Precision (International Interlaboratory Testing)*—The Appendix gives precision results obtained in an international interlaboratory program conducted in ISO Technical Committee 45 on Rubber/Rubber Products in 1984/1985. These results are given for additional background on a broad-based comprehensive interlaboratory program. Please refer to the appendix for details and a full report on the precision results.

13.11 *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

ANNEX

(Mandatory Information)

A1. ALTERNATIVE UPPER DIE

A1.1 This annex describes an alternative upper die for use in the oscillating disk cure meter (ODC). This die does not contain a diaphram as shown in the die in Fig. 4.

A1.2 Some manufacturers of ODC instruments furnish upper dies with a diaphram while others do not.

A1.3 The stated purpose of the diaphram die is to allow the diaphram to be flexed when the dies are closed on a specimen and then to maintain essentially constant pressure on the specimen as it shrinks slightly in volume during vulcanization.

A1.4 The precision and bias observed with the alternative solid upper die as well as any differences in observed results have not been determined.

A1.5 The solid upper die is shown in Fig. A1.1. Dimensions are listed in Table 1.
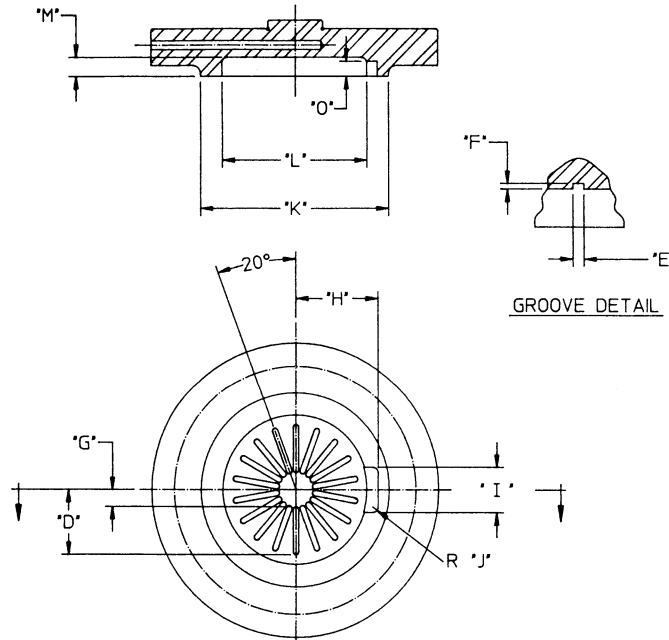
FIG. A1.1 Alternative Upper Die

APPENDIXES

(Nonmandatory Information)

X1. INTERNATIONAL INTERLABORATORY TESTING

X1.1 This appendix gives the precision results of an international interlaboratory Oscillating Disc Cure Meter (ODC) test program conducted in ISO TC-45 in 1984 and 1985. It is added as an appendix to this test method to give additional information on the precision of ODC testing. The ISO/TC45 standard that is analogous to Test Method D 2084 is ISO 3417.

X1.2 The practice for analysis and expression of precision results for ISO TC45 is equivalent in its basic fundamentals and format to Practice D 4483.

X1.3 *Test Details:*

X1.3.1 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 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:

X1.3.1.1 *Part I of 160C*—One test (determination) on each of two days, one week apart, for all four compounds.

X1.3.1.2 *Part II of 150C*—One test on each of two days, one week apart, for all four compounds.

X1.3.2 The formulations for the four compounds are listed in Table X1.1. Compound A has a moderate black level with a non-free sulfur (TMTD) cure system. Compounds B and C are relatively high black with conventional cure systems. Com-

TABLE X1.1 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 D-24 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.

pound D is a gum compound with a conventional cure system.

X1.3.3 A *Type 1* precision was measured in the ITP (no processing operations required on the circulated materials in any given laboratory). The time period for repeatability and reproducibility is on a scale of days.

X1.3.4 A test result is (the test value) obtained from one

measurement or determination with the ODC at any temperature.

X1.3.5 A total of 50 laboratories participated in Part I, and 45 laboratories participated in Part II in addition to their participation in Part I.

X1.4 *Precision Results:*

X1.4.1 The precision results for both Parts I and II are given for five ODC cure parameters. These five parameters are as follows:

X1.4.1.1 M_L—Minimum torque (N·m).

X1.4.1.2 M_{HF}—Maximum torque (N·m).

X1.4.1.3 t_s—Scorch time, (minutes).

X1.4.1.4 t^1_c *(50)*—50 % cure time, (minutes).

X1.4.1.5 t^1_c *(90)*—90 % cure time, (minutes).

X1.4.2 For Part I of 160C, the precision results are given in Table X1.2.

X1.4.3 For Part II of 150C, the precision results are given in Table X1.3.

X1.5 *Use of Precision Results:*

X1.5.1 The general procedure for using precision results is with the symbol $|x_1 - x_2|$ designating a positive difference, that is, without regard to sign. The symbol x refers to any parameter value.

X1.5.1.1 Select the ODC parameter on which decisions are to be made. Find in Table X1.2 (160C) or Table X1.3 (150C) the Summary section for this parameter. If the tests and data under consideration are not at 150 or 160C, select the summary

results for the temperature nearest to the actual test temperature under consideration.

X1.5.1.2 Enter the Summary Precision Result section of either Table X1.2 or X1.3 at an average material value nearest to the test data average under consideration. This will give the applicable r, (r), R, and (R) for use in the decision process.

X1.5.1.3 With these r and (r) values the following general repeatability statements may be used to make decisions. For an absolute difference, the difference, $|x_1 - x_2|$, between two measured ODC parameters values, found on (presumed) identical material samples under normal and correct ODC operation conditions, will exceed the tabulated r, on average not more than once in 20 cases. For a percentage difference, the percent difference, $|x_1 - x_2|/(x_1 + x_2)/2]100$, between two measured ODC parameter values, found on (presumed) identical material samples under normal and correct ODC operation conditions, will exceed the tabulated (r), on average not more than once in 20 cases.

X1.5.1.4 With these R and (R) values the following general reproducibility statements may be used to make decisions. For an absolute difference, the difference $|x_1 - x_2|$, between two independently measured ODC parameter values, found in two laboratories using normal and correct ODC procedures on identical test material samples, will exceed the tabulated reproducibility R, not more than once in 20 cases. For a percentage difference, the percent difference, $|x_1 - x_2|/(x_1 + x_2)/2]100$, between two independently measured ODC parameter values, found in two laboratories using normal and

TABLE X1.2 ISO 3417: Type 1—Precision of 160 °C

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

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TABLE X1.3 ISO 3417: Type 1—Precision of 150 °C

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

correct ODC procedures on identical material samples, will exceed the tabulated reproducibility (R), not more than once in 20 cases.

X2. HISTORY OF THE OSCILLATING DISK CURE METER

X2.1   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. A 20 to 60-s preheat was required after closure before collecting data. The strain on the sample at 3° arc was 21 %.

X2.2   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. X2.1 compares the cure meter curves for the various speeds of oscillation using the square dies and an arc of ±3°.

X2.3   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.

X2.4   In 1971, a smaller version of the LPC die, called the MPC die, was adopted in conformance with Test Method D 2084. 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. X2.2. 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 D 2084 specifies a 1° arc of oscillation as standard, with a 16 % shear strain.

X2.5   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.

X2.6   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 X2.1 illustrates the potential operator effects.

X2.7   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. X2.3 compares typical cure curves for an SBR stock from the rotorless and oscillating disk cure meters.
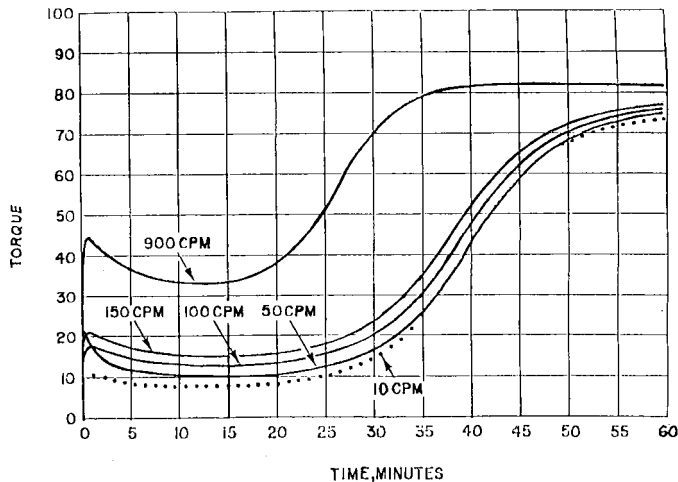
FIG. X2.1 Comparison of Oscillation Frequencies Using the
Square Die and ±3° Arc
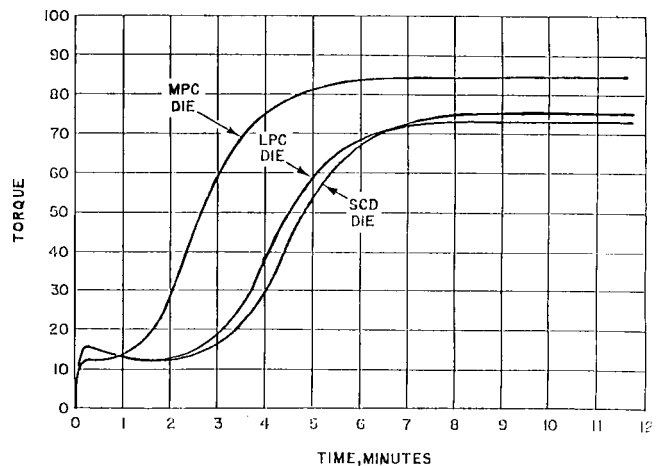
FIG. X2.2 Comparison of Cure Meter Dies

TABLE X2.1 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
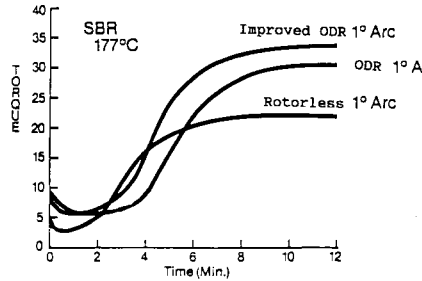
FIG. X2.3 Comparison of Cure Meters Using SBR Stock

X3. ALTERNATIVE BICONICAL DISK

X3.1 This appendix describes an alternative biconical disk for use in the oscillating disk cure meter (ODC). The alternative disk described in this appendix is directly heated for the purpose of maintaining the temperature of the disk at the specified test temperature during the total testing interval.

X3.2 Some manufactures of ODC instruments offer a heated rotor, while others do not. If the disk is heatd, both torque values and cure times may be signifcantly altered. Fig. X3.1 shows an example of the difference in cure curves. Test results between ODC tests using heated and unheated disks canot be compared without taking the differences in heating into account. When a heated rotor is used, it shall be reported with the test results.

X3.3 The alternative heated disk is a modification of the biconical disk show in Fig. 5. This modification has provisions for directly controlling the disk temperature, as shown in Fig. X3.2. In this example, an electrical heater and temerature sensor are located in a metal tube, which is inserted in the disk throught a vertical well in the disk shaft. The well is typically 33 cm (0.130 in.) in diameter and extends to within 0.25 cm (0.100 in.) of the disk apex. The insertion tube diameter is typically 0.1 cm (0.005 in.) less than the well diameter, allowing easy tube removal for cleaning.

X3.4 A temperature controller shall be provided for maintaining the disk temperature within ± 0.5°C (± 1.0°F) of the specified test temperature.
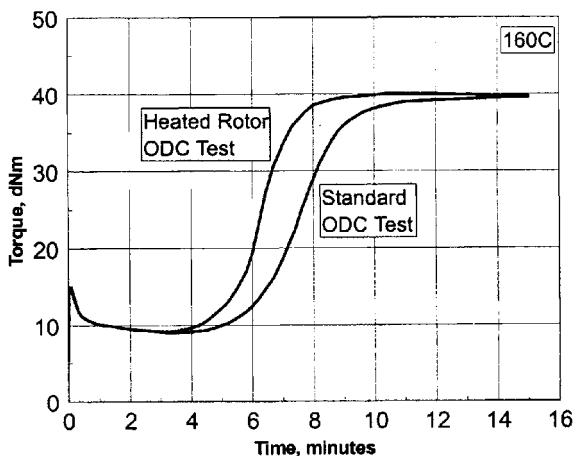

FIG. X3.1 Comparison of Cure Curves With Heated and Unheated Rotors
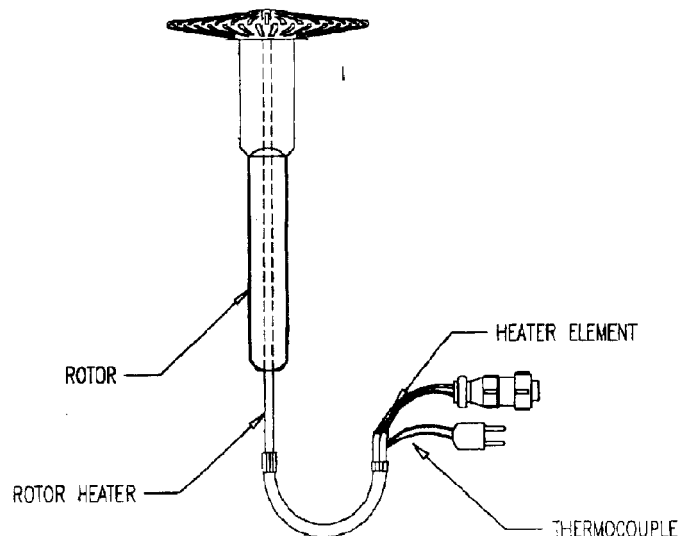

FIG. X3.2 Example of an ODC Rotor With Provision for Heating

X3.5 The precision and bias of ODC tests run with the alternative heated biconical disk have not been statistically determined.