Designation: ~~D4065 – 12~~ D4065 – 20

Standard Practice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures [1]

This standard is issued under the fixed designation D4065; 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 practice is for general use in gathering and reporting dynamic mechanical data. It incorporates laboratory practice for determining dynamic mechanical properties of plastic specimens subjected to various oscillatory deformations on a variety of instruments of the type commonly called dynamic mechanical analyzers or dynamic thermomechanical analyzers.

1.2 This practice is intended to provide means of determining the transition temperatures, elastic, and loss moduli of plastics over a range of temperatures, frequencies, or time, by free vibration and resonant or nonresonant forced vibration techniques. Plots of elastic and loss moduli are indicative of the viscoelastic characteristics of a plastic. These moduli are functions of temperature or frequency in plastics, and change rapidly at particular temperatures or frequencies. The regions of rapid moduli change are normally referred to as transition regions.

1.3 The practice is primarily useful when conducted over a range of temperatures ~~from −160°C~~from −140°C to polymer ~~degradation~~softening and is valid for frequencies from 0.01 to 1000 Hz.

1.4 This practice is intended for materials that have an elastic modulus in the range from 0.5 MPa to 100 GPa (73 psi to 1.5×10^7 psi).

1.5 Discrepancies in results are known to arise when obtained under differing experimental conditions. Without changing the observed data, reporting in full (as described in this practice) the conditions under which the data were obtained will enable apparent differences observed in another study to be reconciled. An assumption of this technique is that testing is conducted in the region of linear viscoelastic behavior.

1.6 Different modes of deformation, such as tensile, bending and shear, are used, as listed in the referenced test methods.

1.7 Test data obtained by this practice are relevant and appropriate for use in engineering design.

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

1.9 *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 ~~practice~~standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 8.

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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***A Summary of Changes section appears at the end of this standard**

NOTE 1—This practice is equivalent to ISO 6721–1.

1.10 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D4000 Classification System for Specifying Plastic Materials
D4092 Terminology for Plastics: Dynamic Mechanical Properties
D4440 Test Method for Plastics: Dynamic Mechanical Properties Melt Rheology
D5023 Test Method for Plastics: Dynamic Mechanical Properties: In Flexure (Three-Point Bending)
D5024 Test Method for Plastics: Dynamic Mechanical Properties: In Compression
D5026 Test Method for Plastics: Dynamic Mechanical Properties: In Tension
D5279 Test Method for Plastics: Dynamic Mechanical Properties: In Torsion
D5418 Test Method for Plastics: Dynamic Mechanical Properties: In Flexure (Dual Cantilever Beam)
E1867 Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers
E2254 Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers
E2425 Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers
E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

2.2 *ISO Standard:*

ISO 6721–1 Plastics— Determination of Dynamic Mechanical Properties, Part 1, General Principles[3]

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this practice, see Terminology D4092.

4. Summary of Practice

4.1 A specimen of known geometry is placed in mechanical oscillation either at fixed or natural resonant frequencies. Elastic or loss moduli, or both of the specimen are measured while varying time, temperature of the specimen or frequency of the oscillation, or both the latter. Plots of the elastic or loss moduli, or both, are indicative of viscoelastic characteristics of the specimen. Rapid changes in viscoelastic properties at particular temperatures, times, or frequencies are normally referred to as transition regions.

NOTE 2—The particular method for measurement of elastic and loss moduli depends upon the operating principle of the instrument used.

4.2 D5023, D5024, D5026, D5279, and D5418 describe specific methods for determining dynamic mechanical properties.

5. Significance and Use

5.1 Dynamic mechanical testing provides a method for determining elastic and loss moduli as a function of temperature, frequency or time, or both. A plot of the elastic modulus and loss modulus of material versus temperature provides a graphical representation of elasticity and damping as a function of temperature or ~~frequency.~~frequency, respectively.

5.2 This procedure can be used to locate transition temperatures of plastics, that is, changes in the molecular motions of a polymer. In the temperature ranges where significant changes occur, elastic modulus decreases rapidly with increasing temperature (at constant or near constant frequency) or increases with increasing frequency (at constant temperature). A maximum is observed for the loss modulus, as well as for the tan delta curve, in the transition region.

5.3 This procedure can be used, for example, to evaluate by comparison to known reference materials or control materials:

[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,~~ Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY ~~10036.~~10036, http://www.ansi.org.

5.3.1 Degree of phase separation in multicomponent systems,

5.3.2 Filler type, amount, pretreatment, and dispersion, and

5.3.3 Effects of certain processing treatment.

5.4 This procedure can be used to determine the following:

5.4.1 Stiffness of polymer composites, especially as a function of temperature,

5.4.2 Degree of polymer crystallinity, and

5.4.3 Magnitude of triaxial stress state in the rubber phase of rubber modified polymers.

5.5 This procedure is useful for quality control, specification acceptance, and research.

5.6 Procedural modifications in material specifications take precedence to this practice. Therefore, consult the appropriate material specification before using this practice. Table 1 of Classification System D4000 lists the ASTM materials standards that currently exist.

6. Interferences

6.1 Since small quantities of specimen are used, it is essential that the specimens be homogeneous or representative, or both.

7. Apparatus

7.1 The function of the apparatus is to hold a plastic specimen of uniform cross section, so that the specimen acts as the elastic and dissipative element in a mechanically oscillated system. Instruments of this type are commonly called dynamic mechanical or dynamic thermomechanical analyzers. They typically operate in one of seven oscillatory modes: (*1*) freely decaying torsional oscillation, (*2*) forced constant amplitude, resonant, flexural oscillation, (*3*) forced constant amplitude, fixed frequency, compressive oscillation, (*4*) forced constant amplitude, fixed frequency, flexural oscillation, (*5*) forced, constant amplitude, fixed frequency, tensile oscillation, (*6*) forced constant amplitude, fixed frequency, torsional oscillation and (*7*) forced constant amplitude, fixed frequency, or variable frequency dual cantilever.

7.2 The apparatus shall consist of the following:

7.2.1 *Clamps*—A clamping arrangement that permits gripping of the sample.

7.2.2 *Oscillatory Deformation (Strain)*—A device for applying an oscillatory deformation (strain) to the specimen. The deformation (strain) shall be applied and then released, as in free-vibration devices, or continuously applied, as in forced-vibration ~~devices (see~~ devices.~~Table 1~~~~).~~

7.2.3 *Detectors*—A device or devices for determining dependent and independent experimental parameters, such as force (stress or strain), frequency, and temperature. Temperature shall be ~~measurable with an accuracy of ±1°C,~~ readable to ±1 °C, frequency to ±1 %, and force to ±1 %.

7.2.4 *Temperature Controller and Oven*—A device for controlling the specimen temperature, either by heating (in steps or ramps), cooling (in steps or ramps), or maintaining a constant specimen environment. Any temperature programmer should be sufficiently stable to permit measurement of sample temperature to ~~±0.5°C.~~ ±0.5 °C.

7.3 Nitrogen or other gas supply for purging purposes.

7.4 Calipers or other length-measuring device capable of measuring to an accuracy of ±0.01 mm.

8. Hazards

8.1 *Precautions:*

8.1.1 Certain materials, when heated near their decomposition point, can release potentially toxic, or corrosive effluents, or both that can be harmful to personnel or to the apparatus.

8.1.2 Buckling of the clamped specimen due to thermal expansion during the test.

9. Test Specimens

9.1 Specimens are of any uniform size or shape but are ordinarily analyzed in rectangular form. If some heat treatment is applied to the specimen to obtain this preferred analytical form, this treatment shall be noted in the report.

9.2 Due to the numerous types of dynamic mechanical instruments, specimen size is not fixed by this practice. ~~In many cases, a specimen of 0.75 by 9.4 by 50 mm (0.03 by 0.38 by 2.0 in.) is found to be usable and convenient.~~The selection of sample size depends on material modulus, and DMA geometry used. A general recommendation is that length divided by thickness is greater than or equal to 10.

NOTE 3—It is important to select a specimen size consistent with the modulus of the material under test and capabilities of the measuring apparatus. ~~For example, while thick specimens of low modulus materials are suitable for measurement, thin specimens of high modulus materials are required.~~Instrument manufacturers often provide sample size recommendations based on material modulus.

9.3 Unless otherwise specified in the appropriate material specification, condition the specimen at a set temperature of ~~23°C (73°F)~~23 °C (73 °F) that is maintained ~~±2°C (±4°F)~~±2 °C (±4 °F) and at a set relative humidity of 50 % that is maintained ~~±10 %~~ ±10 % for not less than 40 h prior to test in accordance to Procedure A of Practice D618, for those tests where conditioning is required. If other specimen conditioning is used, it should be noted in the report.

10. Calibration

10.1 Using the same heating rate or schedule to be used for specimens, calibrate the instrument temperature axis, using the instrument manufacturer's procedures ~~with either or both of the following substances. Refer to~~ and recommended materials (refer to standards E1867, E2254, and E2425 ~~for additional details on calibration.~~).

~~Standard~~	~~Transition Temperature, °C~~	~~Type of Transition~~
~~Water~~	~~0.0~~	~~fusion~~
~~Indium~~	~~156.6~~	~~fusion~~

11. Procedure

11.1 Measure the length, width, and thickness of the specimen to an accuracy of ±1 %.

11.2 Maximum strain amplitude shall be within the linear viscoelastic range of the material. Strains of less than 1 % are recommended.

11.3 If temperature is to be the independent variable:

11.3.1 The test frequency shall be from ~~0.01~~0.01 Hz to ~~500 Hz,~~ 500 Hz, fixed or changing as the dependent variable.

~~TABLE 1 Summary of Techniques and Calculations Used to Determine Dynamic Mechanical Properties~~

~~Technique~~	~~Input Excitation~~	~~Mode of Oscillation~~	~~Frequency Range, Hz~~	~~Specimen Size, mm~~	~~Calculations: Oscillating Strain~~	~~Calculations: Elastic Component~~	~~Calculations: Damping Component~~
~~Dynamic mechanical analyzer~~	~~Sinusoidal/ fixed or resonance frequency~~	~~Forced constant amplitude fixed or resonance frequency flexural oscillation~~	~~0.001 to 60 Hz~~	~~t = 0.01–1.6~~ ~~b = 0.02–13~~ ~~L = 18, 25, or 33~~	~~± 3tA (2 D + L)/L^2 R~~	~~Rectangular:~~	~~Tan δ = JV/f^2~~

TABLE 1 *Continued*

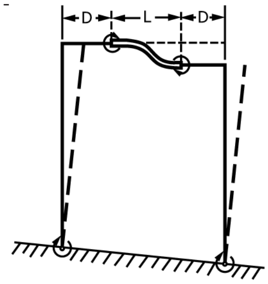
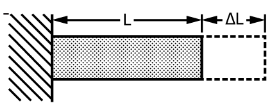
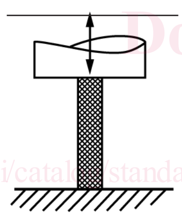
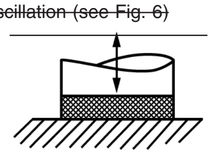
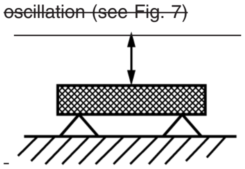
Technique	Input Excitation	Mode of Oscillation	Frequency Range, Hz	Specimen Size, mm	Calculations: Oscillating Strain	Calculations: Elastic Component	Calculations: Damping Component
						$E' = \frac{4\pi^2 f^2 I - H}{2b(L/2+D)^2}[L/t]^3$ Circular: $E' = 4\pi^2 f^2 I H/3r^4$ $(2D + L)^2 [2L^3]$	
Visco-elastometer[A]	Sinusoidal fixed frequency	Forced constant amplitude fixed frequency tensile oscillation (see Fig. 4)	3.5, 11, 35, 110	L = 7 cm T = 0.05 cm B = 0.4 cm	$\Delta L/L$	Rectangular cross section: $E' = NL/bt\Delta L$ $\cos\delta$ Circular cross section:	$E'' = NL/tb\Delta L \sin\delta$ Tan δ directly read
					$\Delta L/L$	$E' = NL\cos\delta/\pi r^2$ ΔL	$E' = NL\sin\delta/\pi r^2$ ΔL Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed or variable frequency	Forced constant amplitude; fixed or variable frequency tensile oscillation (see Fig. 5)	0.0016 to 80	t = 0.025–1.0 b = 12.7 L = 63.5	$\Delta L/L$	Rectangular cross section: $E' = NL\cos\delta/bt$ Δ/L Circular cross section:	$E'' = NL\sin\delta/$ $tb\Delta L$ Tan δ directly read
				r = 1.6, 2.35, 3.15 L = 63.5	$\Delta L/L$	$E' = NL\cos\delta/\pi r^2$ Δ/L	$E' = NL\sin\delta/\pi r^2$ Δ/L Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed or variable frequency	Forced constant amplitude; fixed or variable frequency compressive oscillation (see Fig. 6) 	0.0016–80	Up to 38 × 38: t = 38 b = 38 L = 1–10 r = 8–50 t = 1–10	$\Delta L/L$	Rectangular cross section: $E' = NL\cos\delta/tb$ ΔL Circular cross section: $E' = NL\cos\delta/\pi r^2$ ΔL	$E'' = NL\sin\delta/$ $tb\Delta L$ Tan δ directly read $E'' = NL\sin\delta/$ $\pi r^2 \Delta L$ Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed or variable frequency	Forced constant amplitude; fixed or variable frequency flexural oscillation (see Fig. 7) 	0.0016–80	t = 0.5–6.4 b = 12.7 L = 63.5	$3\,ta/L^2$	Rectangular cross section: $E'\,NL^3\cos\delta/$ $2\,bt^3\,a$	$E'' = NL^3\sin\delta/$ $2bt^3\,a$ Tan δ directly read
				r = 0.25–3.2 L = 63.5	$3\,ra/L^2$	Circular cross section: $E' = 4NL^3$ $\cos\delta/3r^4\,a$	$E'' = 4NL^3\sin\delta/$ $3\,r^4\,a$ Tan δ directly read

TABLE 1 *Continued*

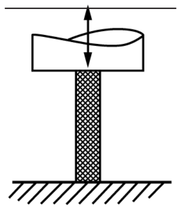
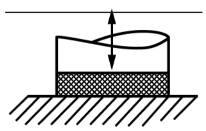
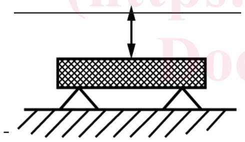
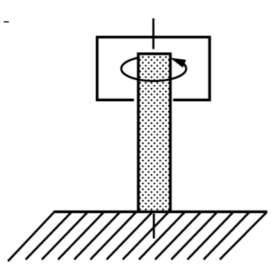
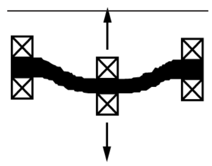
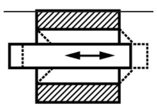
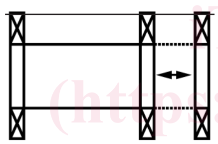
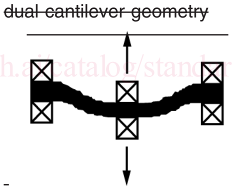
Technique	Input Excitation	Mode of Oscillation	Frequency Range, Hz	Specimen Size, mm	Calculations		
					Oscillating Strain	Elastic Component	Damping Component
Dynamic Mechanical Analyzer[B,D]	Sinusoidal fixed or variable frequency	Constant force amplitude; fixed or variable frequency tensile oscillation (see Fig. 5)	0.01–50	t = up to 2.0 b = up to 10 L = up to 24	$\Delta L/L$	Rectangular cross section: $E' = NL \cos\delta / bt \Delta/L$	$E'' = NL \sin\delta / tb\Delta L$ Tan δ directly read
				r = up to 2.0 L = up to 24	$\Delta L/L$	Circular cross section: $E' = NL \cos\delta/\pi r^2 \Delta/L$	$E'' = NL \sin\delta/\pi r^2 \Delta/L$ Tan δ directly read
Dynamic Mechanical Analyzer[B,D]	Sinusoidal fixed or variable frequency	Constant force amplitude; fixed or variable frequency compression oscillation (see Fig. 6)	0.01–50	Up to 3 × 20 t = up to 20 b = up to 20 L = 0.001-24 r = 1–20 t = up to 20	$\Delta L/L$	Rectangular cross section: $E' = NL \cos\delta / tb \Delta L$	$E'' = NL \sin\delta / tb\Delta L$ Tan δ directly read
					$\Delta L/L$	Circular cross section: $E' = NL \cos\delta/\pi r^2 \Delta L$	$E'' = NL \sin\delta / \pi r^2 \Delta L$ Tan δ directly read
Dynamic Mechanical Analyzer[B,D]	Sinusoidal fixed or variable frequency	Constant force amplitude; fixed or variable frequency flexural oscillation (see Fig. 7)	0.01–50	t = up to 24 b = up to 10 L = up to 20	$3\ ta/L^2$	Rectangular cross section: $E' = NL^3 \cos\delta / 2\ bt^3 a$	$E'' = NL^3 \sin\delta / 2bt^3 a$ Tan δ directly read
				r = up to 5 L = up to 20	$3\ ra/L^2$	Circular cross section: $E' = 4NL^3 \cos\delta/3\ r^4 a$	$E'' = 4NL^3 \sin\delta / 3r^4 a$ Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed or variable frequency	Forced constant amplitude; fixed or variable frequency torsional oscillation (see Fig. 8)	0.0016–80	0.5 to 6.4t 12.7b 63.5L	Rectangular cross section: $K\theta (3a + 1.8b) / 8b^2 L t^2$	Rectangular cross section: $G' = \frac{TL\cos\delta}{K\Theta}$ where: $K = bt^3 \left[16/3 - 3.36\frac{t}{b}(1 - t^4/12b^4)\right]$	$G'' = TL \sin\delta/\theta K$ Tan δ directly read
				3.2, 4.7, 6.4 dia, 63.5L	Circular cross section: $r\theta/L$	Circular cross section: $G' = 2\ TL \cos\delta/\pi r^4 \theta$	$G'' = 2\ TL \sin\delta/\pi r^4 \theta$ Tan δ directly read

TABLE 1 *Continued*

Technique	Input Excitation	Mode of Oscillation	Frequency Range, Hz	Specimen Size, mm	Calculations: Oscillating Strain	Calculations: Elastic Component	Calculations: Damping Component
Mechanical spectrometer[B,C]	Sinusoidal fixed frequency	Forced constant amplitude fixed frequency flexural oscillation (single or dual cantilever) 	.01–200	L = 1–46 t = .1–5 b = .1–18	3 ta/L^2	$E' = \frac{S'10^D}{2b(t/L)^3}$	Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed frequency	Forced constant amplitude fixed frequency shear oscillation 	.01–200	t = .1–3	a/t	$G' = \frac{S'10^D\ t}{2A}$	Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal fixed frequency	Forced constant amplitude fixed frequency tensile oscillation 	.01–100	t = .005–1 b = .01–18 L = 1–20	a/L	$E' = \frac{S'10^D L}{wt}$	Tan δ directly read
Mechanical spectrometer[B,C]	Sinusoidal, fixed or variable frequency	Forced constant amplitude fixed or variable frequency in dual cantilever geometry 	0.00016–16.0	t = up to 1.59 b = up to 6.4 L = up to 44.5	12 tA/L	$E' = (NL^3/Bt^3\ A)\cos\delta$	$E'' = (NL^3/Bt^3\ A)\sin\delta$

[A] Instruments of this type are available from IMASS, Inc., Box 134, Accord, MA 02018.
[B] Instruments of this type are available from TA Instruments, 109 Lukens Drive, New Castle, DE 19720.
[C] Instruments of this type are available from Rheometric-Scientific, Inc., One Possumtown Road, Piscataway, NJ 08854.
[D] Instruments of this type are available from The Perkin-Elmer Corp., 761 Main Avenue, Norwalk, CT 06859-0256.

Symbols:

I = moment of inertia of the inertial member
f = frequency of oscillation
L = specimen length between clamp
b = specimen width
t = specimen depth
r = specimen radius

G' = elastic modulus in shear
Δ = logarithmic decrement
G'' = loss modulus in shear
ΔL = change in length
E' = elastic modulus
E'' = loss modulus
S' = in-phase stiffness (force/displacement)

D = clamping distance
H = instrument constant
J = instrument constant
V = instrument provided dependent variable
A = oscillation amplitude
θ = angular displacement
k = constant
a = parallel axes displacement

T = torque,
RR = relative rigidity
n = number of cycles for oscillation to decay a specific amount
P = period of oscillation
δ = phase angle
N = axial force
Z = elapsed time
R = instrument arm length