Designation: C 1350M – 96

METRIC

Standard Test Method for Measurement of Viscosity of Glass Between Softening Point and Annealing Range (Approximately 10^8 Pa·s to Approximately 10^{13} Pa·s) by Beam Bending (Metric)[1]

This standard is issued under the fixed designation C 1350M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of glass viscosity from approximately 10^8 Pa·s to approximately 10^{13} Pa·s by measuring the rate of viscous bending of a simply loaded glass beam.[2] Due to the thermal history of the glass, the viscosity may not represent conditions of thermal equilibrium at the high end of the measured viscosity range. Measurements carried out over extended periods of time at any temperature or thermal preconditioning will minimize these effects by allowing the glass to approach equilibrium structural conditions. Conversely, the method also may be used in experimental programs that focus on nonequilibrium conditions.

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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 336 Test Method for Annealing and Strain Point of Glass by Fiber Elongation[3]

C 338 Test Method for Softening Point of Glass[3]

C 598 Test Method for Annealing Point and Strain Point of Glass by Beam Bending[3]

C 965 Practice for Measuring Viscosity of Glass Above the Softening Point[3]

C 1351 Test Method for Measurement of Viscosity of Glass between 10^4 Pa·s and 10^8 Pa·s by Viscous Compression of a Solid Right Cylinder[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *beam bending viscometer*—a device used to determine the viscosity of glass from approximately 10^8 Pa·s to approximately 10^{13} Pa·s by measuring the deflection rate of a simply supported beam. The equation for calculating viscosity by this method is:

$$\eta = \frac{gL^3}{1440\, I_c\,(dh/dt)}\left[M + \frac{\rho AL}{1.6}\right]\left[\frac{(1+\alpha_s T)^3}{(1+\alpha_g T)^4}\right] \quad (1)$$

where:

η	=	viscosity, Pa·s,
M	=	load (applied load + loading train), gms,
dh/dt	=	midpoint deflection rate of test beam, cm/s,
g	=	acceleration of gravity, 980 cm/s^2,
I_c	=	cross-sectional moment of inertia, cm^4,
ρ	=	density of glass, g/cm^3,
A	=	cross-sectional area of the beam, cm^2,
L	=	support span, cm, and
α_s and α_g	=	mean coefficient of linear thermal expansion of support stand and glass, respectively, 25°C to temperature of measurement, T, m/m/°C. See Note 1.

NOTE 1—The term $(1 + \alpha_s T)^3/(1 + \alpha_g T)^4$ corrects for thermal expansion changes of room temperature dimensions. It can be ignored when α_s and α_g are approximately equal. A fused silica support stand in combination with a high expansion glass can make this term 3 % in magnitude. Only an estimate of α_g is required, singe the correction is small. Use 1.5

[1] This test method is under the jurisdiction of ASTM Committee C-14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.04 on Physical and Mechanical Properties.

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[2] Hagy, H. E., "Experimental Evaluation of Beam Bending Method of Determining Glass Viscosities in the Range 10^8 to 10^{15} Poises", *Journal of the American Ceramic Society*, Vol 46, No. 2, 1963, pp. 95–97.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] The sole source of supply of flamebent hooks known to the committee at this time is Insaco Inc., P.O. Box 422, Quakertown, PA 18951. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

times the room temperature coefficient if data are unavailable.

4. Significance and Use

4.1 This test method is well suited for measuring the viscosity of glasses in ranges higher than those covered by parallel plate (see Test Method C 1351) and rotational viscometry (see Practice C 965) methods. This test method is useful for providing information related to the behavior of glass after it has been formed into an object of commerce and in research and development.

5. Apparatus

5.1 The apparatus shall consist of a furnace, a means of controlling its temperature and heating rate, specimen holders and loading rod, and a means of observing the rate of viscous deflection of the glass specimen.

5.2 *Furnace*:

5.2.1 The furnace shall be electrically heated by resistance elements. The dimensions and the details of the furnace construction are not critical; its cross-section can be circular of 75 mm (~3 in.) diameter or square with sides of 75 mm. The furnace should have a constant temperature zone that covers the specimen geometry, including the deflection range. Differences in temperature greater than 2°C within that constant temperature zone are unacceptable.

5.3 *Temperature Measuring and Indicating Instruments*:

5.3.1 For the measurement of temperature, there shall be provided a calibrated Type K, R, or S thermocouple. The thermocouple shall be housed in a double-bore alumina tube with its junction placed within 5 mm of the specimen near the axis of the furnace. The thermocouple shall be referenced to 0°C by means of an ice bath, and its emf measured with a calibrated potentiometer that can be read with a sensitivity of 0.1°C and an accuracy of ±0.5°C. Precautions shall be taken to ensure that the ice bath is maintained at 0°C throughout the test. Alternately, the output of the thermocouple can be measured on a calibrated, direct reading meter (electronic thermometer) that can be read with a sensitivity of 0.1°C and an accuracy of ±0.5°C. See Note 3 for temperature lag-lead corrections.

5.4 *Furnace Control*:

5.4.1 Suitable means shall be provided for maintaining the furnace temperature at a fixed control point and for controlling the heating and cooling rates. Commercially available programming equipment provides excellent control. A variable transformer with manual control is an inexpensive, but less adequate means of accomplishing the required control.

5.5 *Specimen Holder and Loading Rod*:

5.5.1 A diagram of the apparatus can be found in Test Method C 598.

5.5.2 A ceramic support stand and a ceramic loading rod shall be provided for supporting the specimen and applying the load to it. The thermal expansion characteristics of both members must be very similar so as to minimize motion of the loading rod due to expansion differences. A rectangular alumina muffle or circular tube that can be notched to define specimen position is a suitable support stand (see Note 2). The supporting surfaces of these notches shall be flat and lie in a plane perpendicular to the axis of the furnace. The inside edges of these notches define the support span once the specimen beam starts to deflect. A support span of about 5 cm (±2 in.) is recommended. A suitable loading rod can be provided by a single-crystal sapphire rod flame bent at one end in the form of a shepherd's crook.[4] This crook will contribute to the load on the specimen, so its weight should be kept to a minimum.

NOTE 2—Vitreous silica is a suitable material for both support stand and loading rod. It is not recommended for temperatures above 900°C.

5.6 *Extensometer for Measuring Midpoint Deflection*:

5.6.1 The means for observing the rate of deflection of the specimen shall allow reliable reading of total deflection of at least 10 mm. The extensometer shall permit direct reading of 0.010 mm and estimates of 0.0010 mm. Its accuracy shall be such that the error of indication will not exceed ±2 % for any measured deflection. This will limit the minimum deflection that may be used in calculation. A linearly variable differential transformer (LVDT) is suitable for this purpose, as is any other device (for example, optical or capacitive), provided that deflection is reliably measured as specified.

5.7 *Weights*:

5.7.1 A set of weights spanning the range from 1 to 500 g and accurate to 0.1 % relative is required.

5.8 *Micrometre Calipers*:

5.8.1 Micrometre calipers which can be read to an accuracy of at least 0.01 mm are required for measuring specimen dimensions.

5.9 *Analytical Balance*:

5.9.1 An analytical balance capable of weighing the shepherd's crook and loading train to an accuracy of 0.1 % relative.

6. Preparation of Test Specimen

6.1 Specimens may either be flame drawn or centerless ground into cylindrical form or diamond-saw cut and mill ground into rectangular form. Nonuniformity of any dimension along the length of the specimen shall not exceed 2 %. When nonuniformity of any dimension exists, an average value shall be used.

6.2 The numerical ratio of beam span to moment of inertia shall not be less than 60. The thickness or diameter to span ratio shall be less than 0.1.

7. Calibration

7.1 Direct calibration of the apparatus is accomplished by using standard glasses, such as those supplied and certified by the National Institute of Standards and Technology (NIST), having known temperature values over the viscosity range covered by this practice.[5] Bias should be corrected by overall instrument calibration:

7.1.1 Determine the viscosity using test beams of an SRM glass which cover a range of cross-sectional moments of inertia. Determine the viscosity over the viscosity range of 10^8 Pa·s to 10^{11} Pa·s by following the standard procedure described in Sections 8 and 9. Carry out tests keeping span and time-temperature function constant.

[5] Table 2, *Annual Book of ASTM Standards*, Vol 15.02 NIST Special Publication No. 260.

7.1.2 Mathematically fit resulting data to a convenient form (for example, polynomial or Fulcher[6] equation). Fit the data supplied for the glass SRM to a Fulcher equation.

7.1.3 Calculate the viscosities from both equations determined in 7.1.2 at 20°C minimum intervals over the measured range. Determine the viscosity ratio, $\eta_{SRM\ fit}/\eta_{measured\ fit}$ = fractional correction, and construct a calibration curve of fractional correction versus log viscosity (measured fit). This is used to correct experimental viscosity data. (See Note 3.) Corrections greater than 20 % are cause for concern and should initiate apparatus troubleshooting.

NOTE 3—If analyses are performed under some heating or cooling rate time-temperature function, the thermocouple temperature may lag or lead the actual sample temperature. If thermocouple lag or lead does occur, the calibration curve described in 7.1.3 would incorporate this temperature bias as well as any viscosity bias. To assess whether thermocouple lag or lead exists, viscosities for a glass SRM may be measured under isothermal conditions at several temperatures. Compare temperatures at equivalent viscosity levels from the analysis of the same glass SRM measured under the heating or cooling rate condition. Temperature differences indicate thermocouple lag or lead. The difference should be applied as a temperature correction to measured temperatures prior to generating the calibration curve (7.1.3) or applying the calibration correction to test data (Section 9).

8. Procedure

8.1 Deflection data may be taken under isothermal conditions or heating or cooling at controlled rates not to exceed 5°C/min.

8.2 Identify the time-temperature function (for example, 5°C/min heating rate) to be used in the test. Use a sapphire or alumina specimen to generate a curve of background deflection against temperature, using the chosen time-temperature function intended for specimen measurement. The deflection of the test specimen is then determined by algebraic subtraction of this background curve from the measured curve.

8.3 Measure the dimensions of the test beam to the nearest 0.01 mm. Use these data to calculate the cross-sectional moment of inertia. (Formulae for common cross-sections are presented in Appendix X1 of Test Method C 598.)

8.4 To protect the support from reaction with the specimen and reduce friction between specimen and support, place a thin, platinum foil in each notch, then place the specimen beam across the support stand at the notch points. Place a thin, platinum foil between the loading rod and the specimen. All platinum foil must be the same thickness, and suitably thin (preferably 25 µm thick) so as to allow seating of the components in their required position.

8.5 Carefully engage the loading rod to the specimen and center it. Apply a weight to the hook on the end of the extensometer, adjusting the total, applied load (consisting of the specimen, loading rod, hooks, fixtures, and weight) so that a usable deflection rate is obtained. Adjust the position of the extensometer to the lower end of its measuring range. Start heating the furnace, using time-temperature function chosen for measurements.

8.6 When a usable deflection rate is attained, begin recording extensometer, time and temperature data to be used in data reduction. The collection interval should not exceed 1 min. Suitable means of accumulating data include computer-controlled data acquisition or plotting the deflection and temperature of the specimen with a two pen recorder operating on a convenient time base. (If such a recording device is not available and data must be taken manually, the deflection and temperature may be recorded by taking readings of both the extensometer and temperature alternately at 30-s intervals so that each will be read at 1-min. intervals. Because it is less accurate than the other methods, the user is discouraged from using this method to acquire data.) If the extensometer goes off range during the test, reset it. Total beam deflections greater than 10 mm are excessive.

9. Calculation

9.1 Use the corrected change in extensometer readings, *dh*, during a given time interval, *dt*, as the rate of midpoint deflection, *dh/dt*, at the temperature corresponding to the middle of that interval. Substitute those data into Eq 1 to calculate the viscosity, η. Correct viscosity using the calibration curve (see Section 7) by multiplying the viscosity by the fractional correction factor corresponding to that viscosity.

10. Report

10.1 At a minimum, report the following information:

10.1.1 Identification of the glass tested,

10.1.2 Manufacturing source and date,

10.1.3 Calibration reference,

10.1.4 Temperature and viscosity points,

10.1.5 Date of test and name of operator, and

10.1.6 Other observations (for example, sample crystallized during measurement).

11. Precision and Bias

11.1 *Precision*—In the absence of round robin testing, a specific precision statement cannot be made. However, Hagy's paper[3] describing the beam bending method can provide insight into the precision and bias of the test method. Precision can be estimated from data scatter in mathematical curve fitting of data.

11.2 *Bias*—In general, this procedure should yield viscosity data to ±10 % of referenced SRM values. Systematic departures may occur for values obtained near the beginning and end of the determination where the respective deflection rates are small and large. A rigid test of the apparatus is to calibrate with one NIST SRM glass and then measure other NIST SRM glasses based on this calibration. If the other standard glasses values are within ±10 % of certification, satisfactory performance has been established. If errors arise that increase or decrease with viscosity, a temperature measurement problem may exist or thermal gradients in the furnace may be too large. These should be corrected.

12. Keywords

12.1 beam bending; glass; viscosity

[6] Fulcher, G. S., *Journal of American Ceramic Society*, Vol 8, 1925.