



Designation: ~~C336 – 71 (Reapproved 2010)~~ C336 – 71 (Reapproved 2015)

Standard Test Method for Annealing Point and Strain Point of Glass by Fiber Elongation¹

This standard is issued under the fixed designation C336; 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 determination of the annealing point and the strain point of a glass by measuring the viscous elongation rate of a fiber of the glass under prescribed condition.

1.2 The annealing and strain points shall be obtained by following the specified procedure after calibration of the apparatus using fibers of standard glasses having known annealing and strain points, such as those specified and certified by the National Institute of Standards and Technology (NIST)² (see [Appendix X1](#)).

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:*³

[C338 Test Method for Softening Point of Glass](#)

[C598 Test Method for Annealing Point and Strain Point of Glass by Beam Bending](#)

3. Definitions

3.1 *annealing point*—that temperature at which internal stresses in a glass are substantially relieved in a matter of minutes.^{4,5,6} During a test in accordance with the requirements of this method, the viscous elongation rate is measured by a suitable extensometer while the specimen fiber is cooling at a rate of $4 \pm 1^\circ\text{C}/\text{min}$. The elongation rate at the annealing point is approximately 0.14 mm/min for a fiber of ~~0.65 mm~~ 0.65 mm diameter.⁶

3.2 *annealing range*—the range of glass temperature in which stresses in glass articles can be relieved at a commercially desirable rate. For purposes of comparing glasses, the annealing range is assumed to correspond with the temperatures between the annealing point (AP) and the strain point (StP).

3.3 *strain point*—that temperature at which the internal stresses in a glass are substantially relieved in a matter of hours. The strain point is determined by extrapolation of the annealing point data and is the temperature at which the viscous elongation rate is 0.0316 times that observed at the annealing point.

4. Significance and Use

4.1 This test method provides data useful for (1) estimating stress release, (2) the development of proper annealing schedules, and (3) estimating setting points for seals. Accordingly, its usage is widespread throughout manufacturing, research, and development. It can be utilized for specification acceptance.

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

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² Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>. Publication 260.

³ 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.

⁴ Littleton, J. T., and Roberts, E. H., "A Method for Determining the Annealing Temperature of Glass," *Journal of the Optical Society of America*, Vol 4, 1920, p. 224.

⁵ Lillie, H. R., "Viscosity of Glass Between the Strain Point and Melting Temperature," *Journal of American Ceramic Society*, Vol 14, 1931, p. 502; "Re-Evaluation of Glass Viscosities at Annealing and Strain Points," *Journal of American Ceramic Society*, Vol 37, 1954, p. 111.

⁶ McGraw, D. A. and Babcock, C. L., "Effect of Viscosity and Stress Level on Rate of Stress Release in Soda-Lime, Potash-Barium and Borosilicate Glasses," *Journal of the American Ceramic Society*, Vol 42, 1959, p. 330.

5. Apparatus

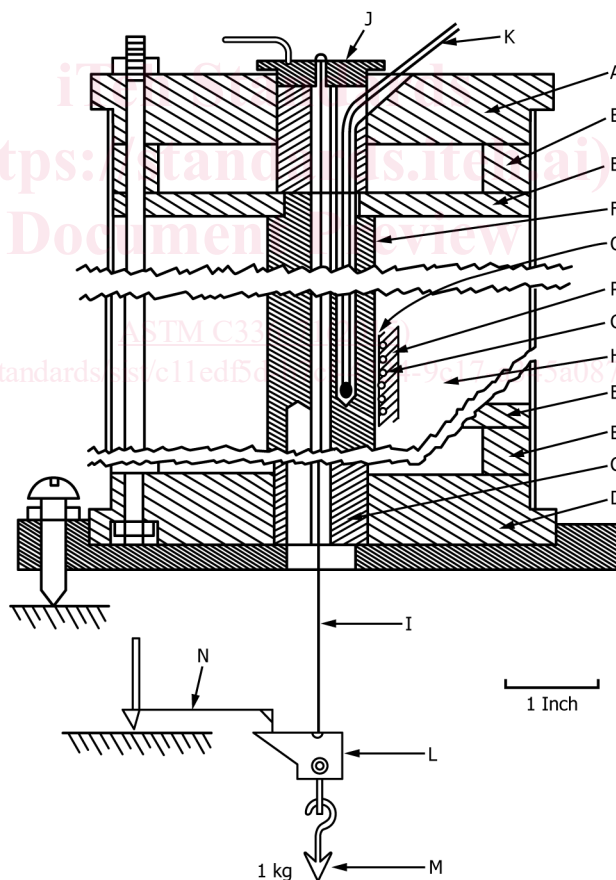
5.1 Furnace—The furnace shall be 368 mm (14½-in.) long and approximately 114 mm (4½ in.) in diameter and shall contain a copper core 305 mm (12 in.) long and 29 mm (1⅛-in.) in outside diameter, with inside diameter of 5.6 mm (7/32 in.). It shall be constructed substantially as shown in Fig. 1.

5.1.1 Such a furnace will cool naturally at approximately 4°C (7°F)/min at 500°C (932°F) and at a rate exceeding 3°C (5.5°F)/min at 400°C (752°F).

5.2 Temperature Measuring and Indicating Instruments—For the measurement of temperature there shall be provided a thermocouple, preferably platinum-platinum rhodium, inserted in the upper side hole of the copper core, as indicated in Fig. 1, so that its junction is located midway in the length of the core. The thermocouple wire shall not be allowed to directly contact the copper; this can be ensured by placing a 6-mm (¼-in.) length of ceramic tube in the bottom of the hole ahead of the couple. The cold junction of the thermocouple shall be maintained in an ice bath during tests.

5.2.1 The temperature-indicating instrument, preferably a potentiometer, shall be of such quality and sensitivity as to permit reading the thermocouple emf to an amount corresponding to 0.1°C (0.2°F), equivalent to about 1 μV for a platinum couple or to about 4 μV for a base-metal couple.

5.2.2 Provision shall be made for reading temperatures accurately at predetermined moments. One means of accomplishing this is to maintain the potentiometer setting at an electromotive force corresponding to a known temperature, near the annealing point and inferring the temperature from the deflection of a sensitive galvanometer, previously calibrated for the purpose. It is convenient to adjust the galvanometer shunt to a sensitivity of about 3°C (5.5°F)/cm of deflection and to somewhat less than critical damping. This technique for reading temperature changes is one of the preferred methods; in the following sections it will be assumed that this technique has been used, although any other equally sensitive and precise method of following the temperature of the thermocouple may be used.



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| A, B, C, D—Made of asbestos-cement (Transite or equivalent) | K—Thermocouple |
| E—Webbed asbestos-cement (Transite or equivalent) disk | L—Lever platform |
| F—Copper core 1⅛ in. (29 mm) outside diameter by 12 in. (305 mm) total length (Note) | M—Load |
| G—91 turns No. 22 Nichrome V wire (or equivalent) | N—Optical lever |
| H—Diatomaceous earth | O—Wrapping for electrical insulation (mica is suggested) |
| I—Sample fiber | P—Refractory cement |
| J—Stainless steel support disk | |

NOTE—A steel alloy, such as Inconel X, may be used as an alternative in place of copper and will allow measurements at higher temperatures.

FIG. 1 Apparatus for Determination of Annealing Point and Strain Point of Glass

5.3 *Furnace Control*—Suitable means shall be provided for idling the furnace, controlling its heating rate, and, in the case of very hard glasses, limiting the cooling rate to not more than 5°C (9°F)/min. A variable transformer is a convenient device for this purpose. The transformer can also be employed as a switch for interrupting the furnace current.

5.4 *Device for Measuring Elongation*—The means of observing the rate of elongation of the fiber should be such as to indicate reliably over a range of about ~~6 mm~~ 6 mm (1/4-in.) change in fiber length with an uncertainty not greater than about 0.01 mm (0.0004 in.). A convenient method is shown in Fig. 1, where the arm of the optical lever, *N*, bears upon a platform, *L*, incorporated in the loading linkage. The fulcrum of the lever should be mounted on a rigid (but height-adjustable) member, substantially free of vibration. With an optical lever arm about ~~38 mm~~ 38 mm (1½ in.) long and a scale distance of about 1 m (40 in.), the multiplying factor is about 50. Readings can be made to 0.5 mm on the scale and, if the scale is 508 mm in length, a sufficient range is attained. The scale is curved with its center of curvature at the mirror location. The system may be calibrated by mounting a micrometer screw in place of the platform, *L*.

5.4.1 Any other extensometer arrangement, such as a linearly variable differential transformer (LVDT) or a travelling microscope, is suitable for measuring elongation, provided that length changes are reliably measured as specified.

5.5 *Micrometer Calipers*, with a least count of 0.005 mm, for measuring specimen fiber diameters.

6. Test Specimen

6.1 *Drawing the Fiber*—Draw a suitable fiber from 2 cm³ (more or less) of glass in any form such as a fragment, cane, flat strip, or tubing. Stick the piece to handles of glass or other suitable material, such as refractory or metal, and then work it into a ball, using a flame adjustment found suitable for the particular kind of glass. When the ball is in a uniform state of proper temperature, and while it is still in the fire, slightly elongate it into a pear shape. Then, remove the ball from the fire, and draw it down to a convenient length.

6.2 *Measurement of Fiber Dimensions*—Measure the fiber with micrometer calipers at ~~51 mm~~ 51 mm (2-in.) intervals, and select a ~~508 mm~~ 508 mm (20-in.) length that is substantially circular in cross section, has a diameter of 0.65 ± 0.10 mm (0.025 ± 0.004 in.), and is uniform to 0.015 mm (±0.0006 in.). The fiber used in Method B of this test procedure may be between 102 mm (~~4 in.~~) and 203 mm (8 in.) in length; once established, such a short fiber specimen length must be maintained within ± 2 mm for all further calibration and testing.

6.3 *Fiber Preparation*—Prepare the selected length of fiber for the test by melting both its ends down into spherical form about 2.5 mm (0.1 in.) in diameter, taking care that the balls are centered on the fiber axis. Starting 25.4 mm (1 in.) from one end, which is thereafter to be regarded as the top end, remeasure the fiber for diameter at 1 in. intervals over the remaining fiber length or up to a maximum of 305 mm (12 in.). Record the average diameter for subsequent calculations.

7. Calibration with Standard Glass

7.1 *Calibration*—Prepare at least four fibers of the calibrating or standard glass,² with diameters covering the diameter range 0.55 to 0.75 mm. In accordance with procedures in Sections ~~8.8 and 9.1~~ and ~~9.1~~, determine the elongation rates at the specified annealing point temperature, and make a calibration plot as in Fig. 2, of the rate of elongation versus the reciprocal square of the fiber diameter. Then use this calibration plot to determine the annealing points of unknown glasses with similar annealing ranges. It is recommended that the apparatus be calibrated periodically depending upon usage.

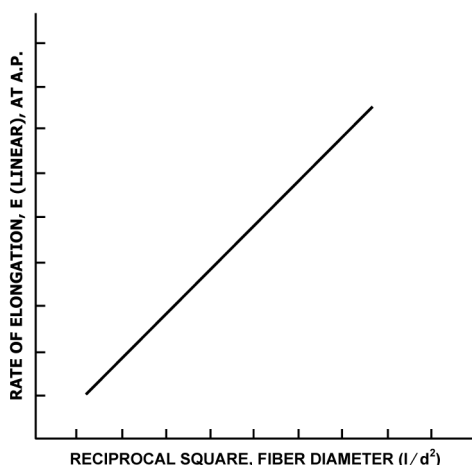


FIG. 2 Calibration