National Institute of Standards & Technology

Certificate

Standard Reference Material® 717a

High Boron Glass Viscosity

This Standard Reference Material (SRM) is intended primarily to check test methods and to calibrate equipment for the determination of the viscosity of glass in accordance with ASTM Procedure C 965-81 [1]. A unit of SRM 717a consists of a borosilicate glass block with nominal dimensions: 40 mm x 40 mm x 150 mm and a nominal mass of 570 g.

The certified viscosity values as a function of temperature were obtained from the results of seven cooperating laboratories, used to calculate a consensus fit of the Fulcher equation as follows:

$$\text{Log}_{10}\,[\text{viscosity (Pa·s)}] = -2.5602 + 4852.2/(t - 192.462) \quad (1)$$

Where t is the temperature expressed in degrees Celsius.

Certified Values: From the consensus fit of the Fulcher equation (1), the certified viscosity values versus temperature were calculated and are listed in Table 1.

Information Values: Noncertified viscosity values for the temperature range of 834 °C to 540 °C and borosilicate glass fixpoint temperatures are given for information only in Tables 2 and 3. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 717a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Cautions to User"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certificate: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Cautions to User: This SRM, like many borosilicates, is susceptible to volatilization at high temperature, especially above 1400 °C. Care should be taken to ensure that the SRM not be exposed for prolonged periods of time to temperatures near or above 1400 °C since there may be some time dependence to the volatilization. Experience has shown that volatilization will also occur with repeated re-melting of the glass, re-melting will invalidate the certification.

Overall direction and coordination of the analytical measurements leading to the certification of this SRM were performed by M.J. Cellarosi formerly of NIST and with the support of A.E. Siefert, ASTM C14.91 Research Associate.

Statistical analysis was provided by L.M. Gill of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Richard D. Holbrook, Chief
Materials Measurement Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steve J. Choquette, Director
Office of Reference Materials

PREPARATION AND ANALYSIS[1]

The glass for this SRM was obtained from Corning, Inc., (Corning, NY). The interlaboratory measurements leading to certification were performed under the auspices of ASTM Subcommittees C14.04 on Physical and Mechanical Properties of Glass and C14.91 on Glass Reference Materials.

Certified Values: From the consensus fit of the Fulcher equation (1), the certified viscosity values versus temperature were calculated and are listed in Table 1. The certified uncertainties are the 95 % simultaneous confidence intervals for the Fulcher equation. Metrological traceability is to the SI derived unit for temperature, expressed in degrees Celsius.

Table 1. Certified Viscosity

Log_{10} [viscosity (Pa·s)][a]	Temperature (°C)
1.00 ± 0.06	1555
1.25 ± 0.06	1466
1.50 ± 0.07	1388
1.75 ± 0.08	1318
2.00 ± 0.10	1256
2.25 ± 0.11	1201
2.50 ± 0.12	1151
2.75 ± 0.13	1106
3.00 ± 0.14	1065
3.25 ± 0.14	1028
3.50 ± 0.15	993
3.75 ± 0.15	961
4.00 ± 0.16	932
4.25 ± 0.16	905
4.50 ± 0.17	880

[a] The SI unit for viscosity is Pa·s [2]. To convert to poise from Pa·s multiply by ten. The viscosity in Table 1 is expressed in the customary manner as Log_{10} viscosity. If Log_{10}[viscosity (Pa·s)] = 1.0 ± 0.06, then Log_{10}[viscosity (poise)] = 2.0 ± 0.06.

Information Values: The following laboratory data provided by one of the round robin participants is given for information purposes and is not certified. Fulcher fit of beam bending [3] and parallel plate viscometry [4] data using a 5 °C per min heating rate for the range 1×10^{11} Pa·s was:

$$Log_{10}\,[\text{viscosity (Pa·s)}] = -3.012 + 5495.3/(t - 148.1) \qquad (2)$$

Table 2. Noncertified Viscosity

Log_{10} [viscosity (Pa·s)]	Temperature (°C)
5	834
6	758
7	697
8	647
9	606
10	570
11	540

[1]Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

The information fixpoint temperatures as measured by ATM Test Methods C 336 [5], C 338 [6], and C 598 [7] are:

Table 3. Information Fixpoint Values

Fixpoint	Temperature (°C)
Softening Point	719 ± 5
Annealing Point	513 ± 6
Strain Point	470 ± 9

The uncertainties give for the fixpoint temperatures are the 95 % confidence intervals of the interlaboratory mean temperatures.

Table 4. Information Glass Nominal Composition Values

Component	Mass Fraction (%)
SiO_2	68.0
B_2O_3	18.5
K_2O	8
Na_2O	1.0
Al_2O_3	3.5
Li_2O	1.0

Index of Refraction $N_D = 1.487$.

Cooperating Laboratories;

Corning Inc. (Corning, NY)
Ferro Corp. (Independence, OH)
Monarch Analytical Laboratories Inc. (Toledo, OH)
OSRAM/Sylvania Inc. (Danvers, MA)
Owens Corning Fiberglass (Granville, OH)
PPG Industries Inc. (Pittsburgh, PA)
Schuller Corp. (Littleton, CO)

REFERENCES

[1] ASTM Standard C 965-81; *Standard Practices for Measurement of Viscosity of Glass Above the Softening Point*, Annual Book of ASTM Standards, ASTM, Philadelphia, PA, Vol. 15.02 (1990).

[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Dec 2018).

[3] Hagy, H.E.; *Experimental Evaluation of Beam-Bending Method of Determining Glass Viscosities in the Range 10^8 to 10^{15} Poise*; J. Am. Ceram. Soc., Vol. 46, Issue 2, p. 93 (1963),

[4] Fontana, E.H.; *A Versatile Parallel Plate Viscometer for Glass Viscosity Measurement to 1000 °C*; Bull. Am. Ceram. Soc., Vol. 49, Issue 6, p. 595 (1970).

[5] ASTM Standard C 336-71; *Standard Practices for Annealing Point and Strain Point of Glass by Fiber Elongation*, Annual Book of ASTM Standards, Vol. 15.02 (1991).

[6] ASTM Standard C 338-93; *Standard Test Method for Softening Point of Glass*, Annual Book of ASTM Standards, Vol. 15.02 (1993).

[7] ASTM Standard C 598-93. *Test Method for Annealing Point and Strain Point of Glass by Beam Bending*; Annual Book of ASTM Standards, Vol. 15.02 (1993).

Certificate Revision History: 14 December 2018 (Corrected unit size dimensions; editorial changes); 03 April 2018 (Editorial changes); 18 September 1996 (Editorial changes); 07 August 1995 (Original certificate date).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm