



Standard Practices for Measurement of Liquidus Temperature of Glass by the Gradient Furnace Method¹

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1. Scope

1.1 These practices cover procedures for determining the liquidus temperature (Note 1) of a glass (Note 1) by establishing the boundary temperature for the first crystalline compound, when the glass specimen is held at a specified temperature gradient over its entire length for a period of time necessary to obtain thermal equilibrium between the crystalline and glassy phases.

NOTE 1—These terms are defined in Definitions C 162.

1.2 Two methods are included, differing in the type of sample, apparatus, procedure for positioning the sample, and measurement of temperature gradient in the furnace. Both methods have comparable precision. Method B is preferred for very fluid glasses because it minimizes thermal and mechanical mixing effects.

1.2.1 *Method A* employs a trough-type platinum container (tray) in which finely screened glass particles are fused into a thin lath configuration defined by the trough.

1.2.2 *Method B* employs a perforated platinum tray on which larger screened particles are positioned one per hole on the plate and are therefore melted separately from each other.²

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 162 Terminology of Glass and Glass Products³

E 220 Method for Calibration of Thermocouples by Comparison Techniques⁴

2.2 Other Document:

NIST Certificate for Liquidus Temperature, SRM 773⁵

3. Significance and Use

3.1 These practices are useful for determining the maximum temperature at which crystallization will form in a glass, and a minimum temperature at which a glass can be held, for extended periods of time, without crystal formation and growth.

4. Apparatus

4.1 The apparatus for determining the liquidus temperature shall consist essentially of an electrically heated gradient furnace, a device for controlling the furnace temperature, temperature measuring equipment, and other items listed.

4.1.1 Furnace:

4.1.1.1 *Method A*—Horizontal temperature gradient, electrically heated furnace, tube type, as illustrated in Fig. 1, Fig. 2, and Fig. 3 and described in A1.1.

4.1.1.2 *Method B*—An alternative furnace detail employing pregrooved Al_2O_3 cores and dual windings, as illustrated in Fig. 4 and Fig. 5, and described in A1.2.

4.1.1.3 Equivalent temperature gradient conditions may also be obtained with furnaces having multiple windings equipped with separate power and control, or a tapped winding shunted with suitable resistances. *For high precision, temperature gradients in excess of $10^\circ\text{C}/\text{cm}$ should be avoided.*

4.1.2 Furnace Temperature Control:

4.1.2.1 *Method A*—A suitable temperature controller shall be provided to maintain a fixed axial temperature distribution over the length of the furnace.

4.1.2.2 *Method B*—A rheostat shall be used to supply power to the outer winding. A separate rheostat and controller shall be used for the inner core winding. The basic furnace temperature level is achieved by controlling power to both inner and outer core windings. The slope of the gradient is achieved by adjusting power input to the outer core winding only. The established temperature gradient is then maintained by controlling power to the inner core winding only.

4.1.3 *Temperature-Measuring Equipment*—Furnace temperatures shall be measured with calibrated Type R or S

¹ These practices are under the jurisdiction of ASTM Committee C-14 on Glass and Glass Products and are the direct responsibility of Subcommittee C14.04 on Physical and Mechanical Properties.

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² From NBS Research Paper RP2096, Vol 44, May 1950, by O. H. Grauer and E. H. Hamilton, with modification and improvement by K. J. Gajewski, Ford Motor Co., Glass Research and Development Office (work unpublished).

³ Annual Book of ASTM Standards, Vol 15.02.

⁴ Annual Book of ASTM Standards, Vol 14.03.

⁵ National Institute of Standards and Technology, Office of Standard Reference Materials, Room B311, Chemical Building, Gaithersburg, MD 20899.