Designation: C829 – 81 (Reapproved 2022)

Standard Practices for Measurement of Liquidus Temperature of Glass by the Gradient Furnace Method[1]

This standard is issued under the fixed designation C829; 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 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 Terminology C162.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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:*[3]

C162 Terminology of Glass and Glass Products

2.2 *Other Document:*

NIST Certificate for Liquidus Temperature, SRM 773[4]

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 Figs. 1-3 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 Figs. 4 and 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 °C/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

[1] These practices are under the jurisdiction of ASTM Committee C14 on Glass and Glass Productsand are the direct responsibility of Subcommittee C14.04 on Physical and Mechanical Properties.

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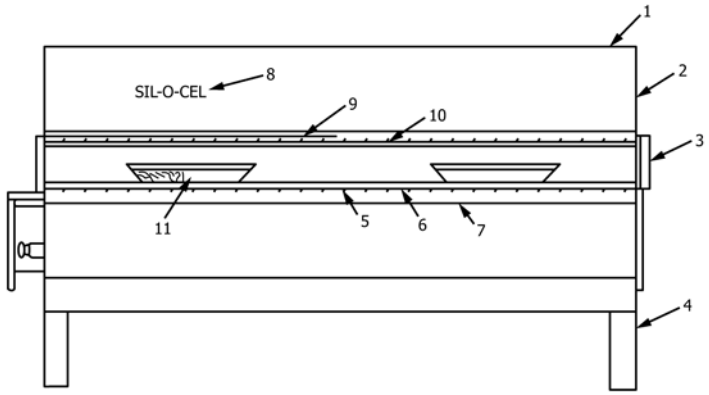
[2] From *NBS Research Paper RP2096,* Vol 44, May 1950, by O. H. Grauer and E. H. Hamilton, with modification and improvement by K. J. Gajewski, Ford Motor Co., Glass Research and Development Office (work unpublished).

[3] 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.

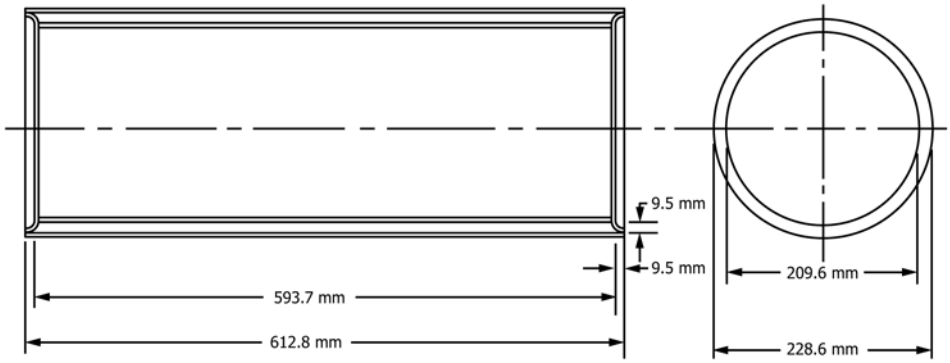
[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.



NOTE 1—See A1.1 for further description.

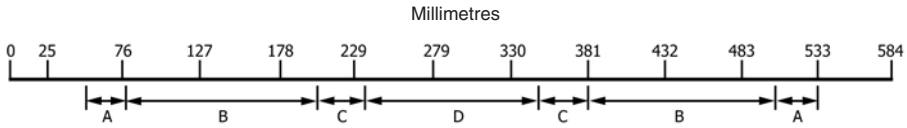
1. Outer shell (stainless steel)
2. End plate (Transite)[4]
3. End plate (quartz)
4. Stand
5. Inner protection tube
6. Heating element tube
7. Outer protection tube
8. Sil-O-Cel[5] insulation
9. Control thermocouple (platinum/rhodium)
10. Heating element wire
11. Specimen tray

FIG. 1 Liquidus Furnace (Method A)



Material: 26-gauge stainless steel

FIG. 2 Liquidus Furnace Shell (Method A)



No. of Turns
A: 6 turns—4.8 mm spacing
B: 13 turns—9.5 mm spacing
C: 5 turns—6.4 mm spacing
D: 24 turns—4.8 mm spacing

FIG. 3 Recommended Liquidus Furnace Winding (Method A)

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 thermocouples in conjunction with a calibrated potentiometer, or other comparable instrumentation, capable of measurements within 0.5 °C. In addition to control thermocouples, Method A requires an unshielded supported thermocouple for insertion into the furnace chamber to determine temperature gradients, and Method B requires five thermocouples mounted in the specimen support fixture as shown in Fig. 6. An alternative method is to attach (spot weld) the thermocouples to a fixed platinum or platinum alloy plate which supports the tray or perforated plate. A solid-state digital thermometer capable of the measurement accuracy specified may be used for temperature measurement.