Designation: C1720 − 21

Standard Test Methods for Determining Liquidus Temperature of Waste Glasses and Simulated Waste Glasses[1]

This standard is issued under the fixed designation C1720; 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 test methods cover procedures for determining the liquidus temperature (T_L) of nuclear waste, mixed nuclear waste, simulated nuclear waste, or hazardous waste glass in the temperature range from 600 °C to 1600 °C. This test method differs from Practice C829 in that it employs additional methods to determine T_L. T_L is useful in waste glass plant operation, glass formulation, and melter design to determine the minimum temperature that must be maintained in a waste glass melt to make sure that crystallization does not occur or is below a particular constraint, for example, 1 volume % crystallinity or $T_{1\%}$. As of now, many institutions studying waste and simulated waste vitrification are not in agreement regarding this constraint (**1**).[2]

1.2 Three methods are included, differing in (*1*) the type of equipment available to the analyst (that is, type of furnace and characterization equipment), (*2*) the quantity of glass available to the analyst, (*3*) the precision and accuracy desired for the measurement, and (*4*) candidate glass properties. The glass properties, for example, glass volatility and estimated T_L, will dictate the required method for making the most precise measurement. The three different approaches to measuring T_L described here include the following: Gradient Temperature Furnace Method (*GT*), Uniform Temperature Furnace Method (*UT*), and Crystal Fraction Extrapolation Method (*CF*). This procedure is intended to provide specific work processes, but may be supplemented by test instructions as deemed appropriate by the project manager or principle investigator. The methods defined here are not applicable to glasses that form multiple immiscible liquid phases. Immiscibility may be detected in the initial examination of glass during sample preparation (see 9.3). However, immiscibility may not become apparent until after testing is underway.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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
C829 Practices for Measurement of Liquidus Temperature of Glass by the Gradient Furnace Method
C859 Terminology Relating to Nuclear Materials
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2282 Guide for Defining the Test Result of a Test Method

2.2 *NIST Standards:*[4]

SRM-773 National Institute for Standards and Technology (NIST) Liquidus Temperature Standard
SRM-674b NIST X-Ray Powder Diffraction Intensity Set for Quantitative Analysis by X-Ray Diffraction (XRD)
SRM-1416 Aluminosilicate Glass for Liquidus Temperature
SRM-1976a NIST Instrument Response Standard for X-Ray Powder Diffraction
SRM-1976c Instrument Response Standard for X-Ray Powder Diffraction

[1] These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.13 on Spent Fuel and High Level Waste.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[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.

2.3 *Other Standard:*[5]

ISO/IEC 17025:2017 American National Standards Institute/National Conference of Standards Laboratories (ANSI/NCSL) General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 For terms not defined herein, refer to Terminology C859 and C162.

3.2 *Definitions:*

3.2.1 *air quenching*—to pour or place a molten glass specimen on a surface, for example, a steel plate, and cool it to the solid state.

3.2.2 *anneal*—to prevent or remove processing stresses in glass by controlled cooling from a suitable temperature, for example, the glass transition temperature (T_g) (modified from Terminology C162).

3.2.3 *annealing*—a controlled cooling process for glass designed to reduce thermal residual stress to an acceptable level and, in some cases, modify structure (modified from Terminology C162).

3.2.4 *cleaning glass*—glass or flux used to remove high viscosity glass, melt insolubles, or other contamination from platinum-ware.

3.2.5 *crystallize*—to form and/or grow crystals from a glass melt during heat-treatment or cooling.

3.2.6 *crystallization*—the progression in which crystals are first nucleated and then grown within a host medium.

3.2.6.1 *Discussion*—Generally, the host may be a gas, liquid, or another crystalline form. However, in this context, it is assumed that the medium is a glass melt.

3.2.7 *crystallization front*—the boundary between the crystalline and crystal-free regions in a test specimen that was subjected to a temperature gradient heat-treatment.

3.2.8 *furnace profiling*—the process of determining the actual temperature inside of a furnace at a given location; this involves different steps for different types of furnaces.

3.2.9 *glass*—an inorganic product of fusion that has cooled to a rigid condition without crystallizing (see Terminology C162); a noncrystalline solid or an amorphous solid (**2**).

3.2.10 *glass sample*—the material to be heat-treated or tested by other means.

3.2.11 *glass specimen*—the material resulting from a specific heat treatment.

3.2.12 *glass transition temperature (T_g)*—on heating, the temperature at which a glass transforms from a solid to a liquid material, characterized by the onset of a rapid change in several properties, such as thermal expansivity.

3.2.13 *gradient furnace*—a furnace in which a known temperature gradient is maintained between the two ends.

3.2.14 *inhomogeneous glass*—a glass that is not a single amorphous phase; a glass that is either phase separated into multiple amorphous phases or is crystallized.

3.2.15 *liquidus temperature (T_L)*—the maximum temperature at which thermodynamic equilibrium exists between the molten glass and its primary crystalline phase.

3.2.15.1 *Discussion*—T_L is the maximum temperature at which a glass melt crystallizes.

3.2.16 *melt insoluble*—a crystalline, amorphous, or mixed phase material that is not appreciably soluble in molten glass, for example, noble metals, noble metal oxides.

3.2.17 *mixed waste*—waste containing both radioactive and hazardous components regulated by the Atomic Energy Act (AEA) (**3**) and the Resource Conservation and Recovery Act (RCRA) (**4**), respectively.

3.2.17.1 *Discussion*—The term "radioactive component" refers to the actual radionuclides dispersed or suspended in the waste substance (**5**).

3.2.18 *mold*—a pattern, hollow form, or matrix for giving a certain shape or form to something in a plastic or molten state. **Webster's Dictionary**[6]

3.2.19 *nuclear waste glass*—a glass composed of glass-forming additives and radioactive waste.

3.2.20 *observation*—the process of obtaining information regarding the presence or absence of an attribute of a test specimen or of making a reading on a characteristic or dimension of a test specimen (see Terminology E2282).

3.2.21 *preferred orientation*—when there is a stronger tendency for the crystallites in a powder or a texture to be oriented more one way, or one set of ways, than all others.

3.2.21.1 *Discussion*—This is typically due to the crystal structure. **IUCr**[7]

3.2.22 *primary phase*—the crystalline phase at equilibrium with a glass melt at its liquidus temperature.

3.2.23 *radioactive*—of or exhibiting radioactivity; a material giving or capable of giving off radiant energy in the form of particles or rays. **American Heritage**[8] **Webster's**[6]

3.2.23.1 *Discussion*—Example of particles or rays formed by the disintegration of atomic nuclei are α, β, and γ; said of certain elements, such as radium, thorium, and uranium and their products.

3.2.24 *Round-Robin*—an interlaboratory and intralaboratory testing process to develop the precision and bias of a procedure.

3.2.25 *section*—a part separated or removed by cutting; a slice, for example, representative thin section of the glass specimen. **Webster's**[6]

3.2.26 *simulated nuclear waste glass*—a glass composed of glass forming additives with simulants of, or actual chemical species, or both, in radioactive wastes or in mixed nuclear wastes, or both.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[6] Merriam-webster.com.

[7] IUCr Online Dictionary of Crystallography, 2011.

[8] American Heritage Dictionary, 1973.