Designation: ~~C1720 – 17$^{\varepsilon 1}$~~ C1720 – 21

Standard Test ~~Method~~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.

~~ε1 NOTE—Editorially corrected Fig. 9 title and 15.1.5 in January 2018.~~

1. Scope

1.1 These ~~practices~~ 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~~600 °C to ~~1600°C.~~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: ~~(A)~~ Gradient Temperature Furnace Method ~~(GT),~~(*BGT*~~)~~), Uniform Temperature Furnace Method ~~(UT), and~~ (~~C~~*UT*~~)~~), and Crystal Fraction Extrapolation Method ~~(CF).~~(*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 and health practices and determine the applicability of regulatory limitations prior to use.*~~

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[1] ~~This~~These test ~~method is~~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.

Current edition approved ~~Nov. 1, 2017~~Oct. 1, 2021. Published ~~December 2017~~December 2021. Originally approved in 2011. Last previous edition approved in ~~2011~~2017 as ~~C1720 – 11~~C1720 – 17$^{\varepsilon 1}$. DOI: ~~10.1520/C1720-17E01.~~10.1520/C1720-21.

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

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
~~D1129 Terminology Relating to Water~~
~~D1193 Specification for Reagent Water~~
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 ~~*Other Documents:*~~*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
~~Z540.3~~SRM-1976c ~~American National Standards Institute/National Conference of Standards Laboratories (ANSI/NCSL) Requirements for the Calibration of Measuring and Test Equipment~~Instrument Response Standard for X-Ray Powder Diffraction

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:* ~~(refer to Terminology C859)~~

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).

~~3.1.4 *ASTM Type I water*—purified water with a maximum total matter content including soluble silica of 0.1 g/m^3, a maximum electrical conductivity of 0.056 µΩ/cm at 25°C and a minimum electrical resistivity of 18 MΩ × cm at 25°C (see Specification D1193 and Terminology D1129).~~

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. ~~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.~~

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[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] ~~The boldface numbers in parentheses refer to a list of references at the end of this standard.~~Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

[5] ~~Webster's New Universal Unabridged Dictionary, 1979.~~Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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 ~~processes~~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—*~~*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; the term "radioactive component" refers to the actual radionuclides dispersed or suspended in the waste substance~~ respectively.~~(5).~~
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. ~~This is typically due to the crystal structure.~~
3.2.21.1 *Discussion—*
This is typically due to the crystal structure. **IUCr**[7]

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[6] Merriam-webster.com.

[7] IUCr Online Dictionary of Crystallography, 2011.

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, for example, α, β, and γ, by the disintegration of atomic nuclei; said of certain elements, such as radium, thorium, and uranium and their products.~~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]

~~3.1.27 *set of samples*—samples tested simultaneously in the same oven.~~

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.

~~3.1.29 *standard*—to have the quality of a model, gage, pattern, or type. **Webster's**[7]~~

~~3.1.30 *standardize*—to make, cause, adjust, or adapt to fit a standard (5); to cause to conform to a given standard, for example, to make standard or uniform. **Webster's**[7]~~

3.2.27 *surface tension*—a property, due to molecular forces, by which the surface film of all liquids tends to bring the contained volume into a form having the least possible area.

3.2.28 *test determination*—the value of a characteristic or dimension of a single test specimen derived from one or more observed values (see Terminology E2282).

3.2.29 *test method*—a definitive procedure that produces a test result (see Terminology E2282).

3.2.30 *test observation*—see *observation*.

3.2.31 *uniform temperature furnace*—a furnace in which the temperature is invariant over some defined volume and within some defined variance.

3.2.32 *vitrification*—the process of fusing waste with glass making chemicals at elevated temperatures to form a waste glass (see Terminology C162).

3.2.33 *volatility*—the act of one or more constituents of a solid or liquid mixture to pass into the vapor state.

3.2.34 *waste* ~~*glass*—~~*glass*[6]—a glass developed or used for immobilizing radioactive, mixed, or hazardous wastes.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *ASTM Type I water*—purified water with a maximum total matter content including soluble silica of 0.1 g/m^3, a maximum electrical conductivity of 0.056 $\mu\Omega^{-1}$/cm at 25 °C, and a minimum electrical resistivity of 18 MΩ × cm at 25 °C.

3.3.2 *set of samples*—samples tested simultaneously in the same oven.

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[8] American Heritage Dictionary, 1973.

3.3.3 *standard*—to have the quality of a model, gauge, pattern, or type. **Webster's**[6]

3.3.4 *standardize*—to make, cause, adjust, or adapt to fit a standard (**5**); to cause to conform to a given standard, for example, to make standard or uniform. **Webster's**[6]

3.4 *Abbreviations:*

3.4.1 *AEA*—Atomic Energy Act

3.4.2 *ANSI*—American National Standards Institute

3.4.3 *ASTM*—American Society for Testing and Materials

3.4.4 *CF*—crystal ~~fraction extrapolation~~extrapolation method

3.4.5 C_F—crystal fraction in a sample or specimen

3.4.6 *EDS*—energy dispersive spectrometry

3.4.7 η—viscosity

3.4.8 *FWHM*—full width of a peak at half maximum

3.4.9 *GF*—gradient temperature furnace

3.4.10 *GT*—gradient temperature furnace method

3.4.11 *HF*—hydrofluoric acid

3.4.12 *HLW*—high-level waste

3.4.13 *ID*—identification

3.4.14 *MSE*—mean squared error

3.4.15 *NBS*—National Bureau of Standards

3.4.16 *NCSL*—National Conference of Standards Laboratories

3.4.17 *NIST*—National Institute for Standards and Technology (formerly NBS)

3.4.18 *OM*—optical microscope or optical microscopy

3.4.19 *PDF*—powder diffraction file

3.4.20 *RCRA*—Resource Conservation and Recovery Act

3.4.21 *RIR*—relative intensity ratio

3.4.22 *RLM*—reflected light microscopy

3.4.23 *SD*—standard deviation

3.4.24 *SEM*—scanning electron microscope or scanning electron microscopy

3.4.25 *SRM*—Standard Reference Material

3.4.26 *SSE*—sum of squared errors

3.4.27 $T_{1\%}$—temperature where glass contains 1 volume % of a crystalline phase

3.4.28 T_a—primary UT measurement above T_L

3.4.29 T_c—primary UT measurement below T_L

3.4.30 T_g—glass transition temperature

3.4.31 T_L—liquidus temperature

3.4.32 *TLM*—transmitted light microscopy

3.4.33 T_M—melting temperature for glass preparations

3.4.34 *UF*—uniform temperature furnace

3.4.35 *UT*—uniform temperature furnace method

3.4.36 *WC*—tungsten carbide

3.4.37 *XRD*—X-ray diffraction

4. Summary of Test Method

4.1 ~~This procedure describes methods~~ These test methods describe methods for determining the T_L of waste or simulated waste glasses. ~~Temperature is defined as the maximum temperature at which equilibrium exists between the molten glass and its primary crystalline phase. In other words, T_L is the maximum temperature at which a glass melt crystallizes.~~ Fig. 1 illustrates an example T_L for a simple two-component liquid on an arbitrary binary phase diagram.

4.1.1 ~~*(A)*~~ *Gradient Temperature Furnace Method (GT)*—This method is similar to Practice C829, "Standard Practices for Measurement of Liquidus Temperature of Glass by the Gradient Furnace Method," although it has been modified to meet the

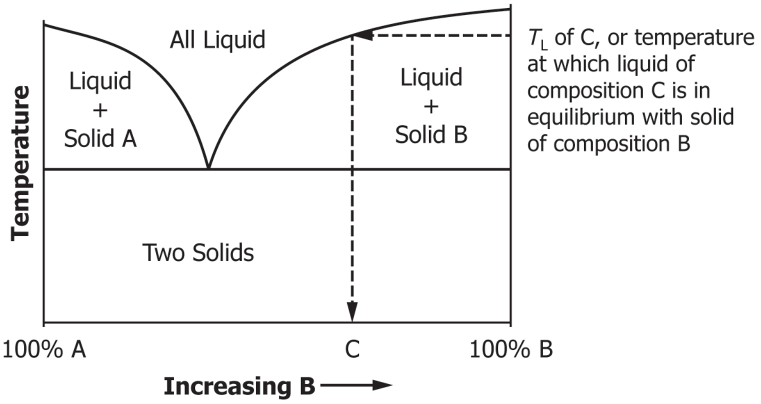


FIG. 1 Binary Phase Diagram of Components A and B with T_L of Composition C Highlighted

specific needs of waste and simulated waste glass measurements. The most pronounced differences between this method and the Practice C829 "boat method" are the sample preparation and examination procedures.

4.1.1.1 Samples are loaded into a boat, for example, platinum alloy (Fig. 2) with a tight-fitting lid, and exposed to a linear temperature gradient in a gradient furnace (Fig. 3) for a fixed period of time. The temperature, as a function of distance, *d*, along the sample, is determined by the location within the ~~GF,~~ gradient furnace, and the T_L is then related to the location of the crystallization front in the heat-treated specimen (Fig. 4).

4.1.1.2 Following the heat-treatment, the specimen should be annealed at, or near, the glass ~~transition,~~ transition temperature, T_g, of the glass (this should be previously measured or estimated) to reduce specimen cracking during cutting and polishing.

4.1.1.3 The specimen should then be scored or marked to signify the locations on the specimen located at different depths into the gradient furnace, that is, locations heat-treated at specific temperatures.

4.1.1.4 If the specimen is optically transparent, it can be observed with transmitted light microscopy (TLM) or reflected light microscopy (RLM) to look for bulk or surface crystallization, respectively. If the specimen is not optically transparent or is barely optically transparent (for example, in glasses with high quantities of Fe_2O_3), a cut or fractured section of the glass can be polished very thin (that is, a thin section can be made) to allow for observation. Another option for surface observations is scanning electron microscopy (SEM). This method provides a quick measurement of T_L in the absence of convective flow of glass in the ~~GF,~~ gradiant furnace, which distorts the location of the crystallization front (that is, the crystallization front is likely not constant at a given temperature (see Fig. 4)).

4.1.1.5 The temperature gradient and increased volatility at higher temperatures cause gradients in surface tension, which in turn cause convective flow. This method is ideal for glasses with a T_L less than roughly ~~1000°C~~ 1000 °C or glasses with a low volatility near the T_L. If the temperature range spanned by the crystallization front is too high for the desired tolerance, ~~the~~*UT* or *CF* ~~methods (Method B or C)~~ should be used for a more precise T_L measurement. ~~*Method*~~*GT* ~~A~~ is not easily used to measure the T_L on radioactive glasses because of the size of the ~~GF~~ gradient furnace and the complicated sample analysis required. This method is not recommended for glasses with a T_L in a temperature range of very low glass viscosity (that is, $\eta < 50$ Pa·s).

4.1.2 ~~*(B)*~~ *Uniform Temperature Furnace Method (UT)*—This method is similar to the methods used in phase diagram determination and can be used for making more precise measurements than those determined with ~~*(A) Gradient Temperature Furnace Method (GT).*~~*GT.*

4.1.2.1 In this method, a glass sample is loaded into a crucible (for example, platinum alloy, see Fig. 5) with a tight-fitting lid and subjected to temperatures for a fixed period of time (for example, ~~24~~24 h ± 2 ~~hrs).~~h). Following heat-treatment, the specimen can be observed by optical microscopy (OM) for the appearance or absence of crystalline or other undissolved materials with methods similar to those previously described (4.1.1). Crystalline material present in the meniscus (that is, in the upper corners of the heat-treated specimen) can be an artifact of this process and should be reported separately. The locations of the crystals within the heat-treated specimen need to be reported (that is, the melt-crucible interface, meniscus, melt-air interface, or the bulk) on the *liquidus temperature data sheet* (see Appendix X1). The crystal locations used to define T_L should be clearly documented when reporting T_L. Typically, crystals in any location except for the meniscus (where composition can be ~~effected~~affected by volatility) are used. In some circumstances, surface crystallization can be excluded from T_L determination.

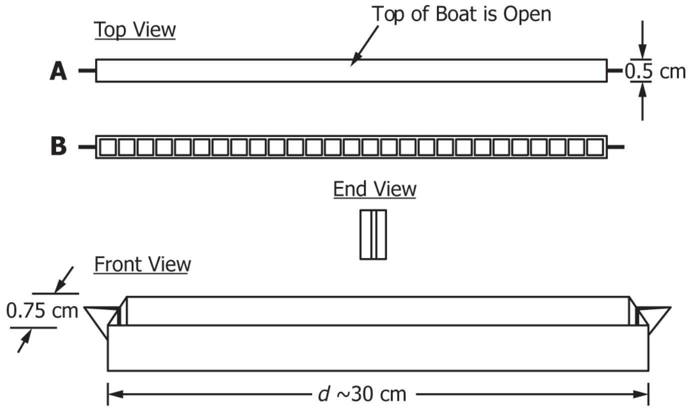


FIG. 2 GF Boat Diagram: (*A*) Single Chamber Crucible Design (*B*) Single Chamber Design Loaded with a Set of Samples (that is, Smaller Crucibles)

FIG. 3 Photograph of Typical Gradient Temperature Furnace

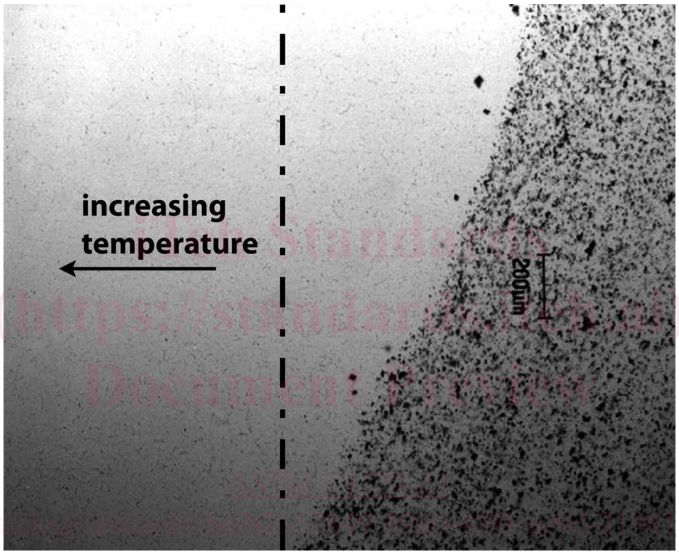
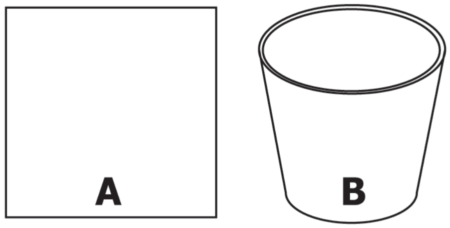


FIG. 4 OM Micrograph of the Crystallization Front in a GT Specimen

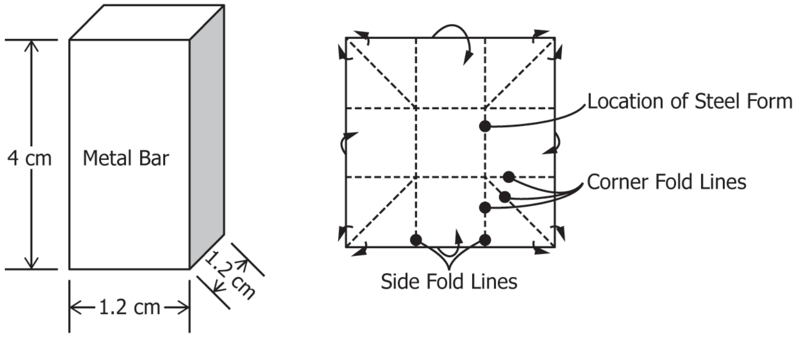
4.1.2.2 The T_L is then given by the temperature range between the highest temperature at which a specimen contains crystals (T_c) and the lowest temperature without crystals in the specimen (T_a); the T_L is then typically defined as the average of T_a and T_c.

4.1.2.3 This method is more time consuming as it requires more heat-treatments than ~~*the*~~*GT,* ~~GT method,~~ although it minimizes the effects of volatility and eliminates the convection-driven uncertainty in crystallization front measurements. This method is used for high precision measurements (on the order of ~~±5°C),~~ ±5 °C), is more easily applied to radioactive glasses, and can be used to measure T_L values as high as ~~1600°C~~ 1600 °C with typical high-temperature furnaces (for example, furnaces with $MoSi_2$ heating elements), and even higher with specialized equipment and high-temperature crucibles. This method may be used for glasses with a high volatility near T_L under certain circumstances.

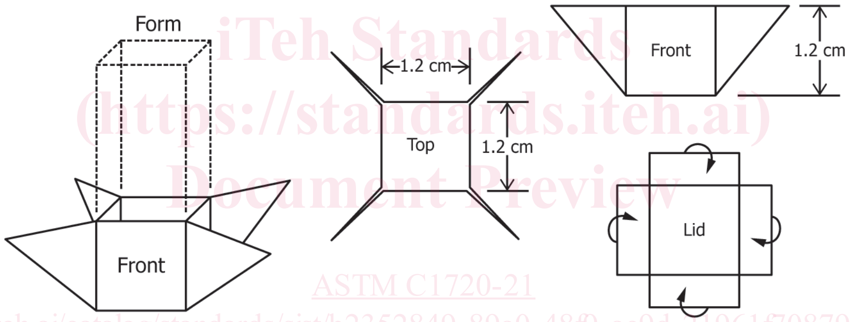
4.1.3 ~~*(C)*~~ *Crystal Fraction Extrapolation Method (CF)*—This method is an alternate method that uses a UT specimen to measure the crystal fraction, C_F (in mass % or volume %), of a crystalline phase or phases in a sample heat-treated at multiple temperatures, $T << T_L$. The C_F at each temperature is measured with XRD, RLM, TLM, SEM, or combinations thereof, by mass and/or volume %, and then T_L is achieved by extrapolating C_F as a function of temperature to zero crystals. This method is more suited for glasses with a higher volatility near the T_L than the previous methods. When multiple crystalline phases are present, XRD is an effective method for quantifying C_F as a function of temperature and is very effective at determining the T_L of each phase independently; this would be more difficult ~~with~~by ~~*Methods*~~*GT* ~~A~~and ~~*or*~~*UT.CF* ~~B. The C_F method~~ yields the additional benefit of equilibrium crystal fractions as a function of temperature, which can sometimes tend to be non-linear at $C_F > 5$ mass % to 10 mass % crystallinity for most crystalline phases. Different techniques for ~~the *C*~~*CF*$_F$ ~~method~~ are described below.



(1) 3.6- × 3.6-cm square piece of foil (A) or conical thimble (B) made of inert material, for example, platinum alloy



(2) For the cubic crucible, take foil in (1) and draw an "×" pattern from corner to corner (excluding the center square) to help locate the form used and where to fold the corners.



(3) Fold the corners along the diagonal lines. Then fold the sides up next to the form.

FIG. 5 ~~*UF*~~*UT* and *CF* Crucible Schematic

4.1.3.1 *Volume Fraction of Crystal(s) in the Specimen* (12.4.2)—With TLM, RLM, or SEM as well as image analysis software, it is possible to measure the area fraction of crystals in an image or micrograph of the specimen. The area fraction is then equivalent to the volume fraction if the image is representative of the bulk of the specimen, and the effective depth of the image is insignificant. If this process is done at different temperatures, the T_L can be extrapolated as a function of temperature.

NOTE 1—The mass fraction of crystals in the specimen can be estimated if the densities of the glass and the crystal(s) are known.

4.1.3.2 *Number Fraction of Crystal(s) in the Specimen* (12.4.3)—In the same fashion as described in 4.1.3.1, count the number of crystals in an image or micrograph of the specimen at different temperatures. If this process is done at different temperatures, the T_L can be extrapolated as a function of temperature.

4.1.3.3 *Mass Fraction of Crystal(s) in the Specimen by Adding a Known Crystalline Phase* (12.4.4)—Adding a known mass fraction of a known, standard crystalline material (for example, NIST SRM-674b) allows the standardization of the XRD pattern. The standards and the unknown specimen should be run ~~in~~ independently before mixing to verify that there is not overlap between the peaks of the standard and the peaks in the unknown specimen because this will make quantification difficult and less accurate. The standardized pattern can then be used to generate quantitative (if the crystal structure has been refined) or semi-quantitative (if the crystal structure has not been refined) C_F analysis with Rietveld (**6-8**) refinement software or the relative intensity ratio (RIR) method (12.4.5).

4.1.3.4 *Mass Fraction of Crystal(s) in the Specimen by Comparing it to the Calibration Curve* (12.4.5)—In this method, samples with known concentrations of the crystalline phases being analyzed are prepared and tested using XRD. The peak area's (full width at half maximum or FWHM, total crystal peak area, or highest peak area) and known crystal fractions are used to generate a calibration curve. The peak area of the unknown specimen is then used in the calibration equation to determine a quantitative (if interpolated) or semiquantitative (if extrapolated) crystal fraction.

4.1.3.5 *Volume Fraction of Crystal(s) in the Specimen With C_F Data From XRD Analysis*—Commonly, melter constraints are in terms of a volume % of crystallinity, for example, $T_{1\%}$. Once C_F data are obtained in mass % by XRD, the remaining mass of glass, m_g, is calculated as a difference given by

$$m_g = m_t - \sum_{i=1}^{N} m_{c,i} \quad (1)$$

where:

m_t = the total mass (that is, the value is normalized to one and thus component values are mass fractions), and
$m_{c,i}$ = the mass fraction of the *i*-th crystalline phase observed and quantified by XRD.

By converting the mass fractions of the *i*-th component additives, m_i, into mole fractions, M_i, the density of glass, ρ_g, can be computed with the following expression:

$$\rho_g = \frac{\sum_{i=1}^{N} M_i m_{m,i}}{\sum_{i=1}^{N} M_i V_{M,i}} \quad (2)$$

where:

$m_{m,i}$ = the molecular mass of the *i*-th oxide, and
$V_{M,i}$ = the molar volume of the *i*-th component additive explained elsewhere (**9**).

The total volume of each heat treatment, V_{HT}, is calculated with

$$V_{HT} = \frac{m_g}{\rho_g} + \frac{\sum_{i=1}^{N} m_{c,i}}{\rho_{c,i}} \quad (3)$$

where:

$\rho_{c,i}$ = the density of the *i*-th crystalline component.

The volume % of the *i*-th crystalline component, $V_{c,i}$, in the heat-treated specimen is denoted by

$$V_{c,i} = 100 \times \frac{m_{c,i}}{(\rho_{c,i} \times V_{HT})} \quad (4)$$

The values of $V_{c,i}$ can then be plotted as a function of temperature and a linear correlation fit to the data with

$$V_{c,i} = m \times T + b \quad (5)$$

where:

$T_{1\%}$ = $(V_{c,i} - b)/m$ when $V_{c,i} = 1$ ($T_{1\%}$)

5. Significance and Use

5.1 This procedure can be used for (but is not limited to) the following applications:
(1) support glass formulation development to make sure that processing criteria are met,
(2) support production (for example, processing or troubleshooting), and
(3) support model validation.

6. Apparatus

6.1 *Equipment for ~~the GT Method:~~ GT:*

6.1.1 Resistance-heated tubular gradient furnace capable of achieving temperatures of ~~550~~550 °C to ~~1150°C~~ 1150 °C with gradients in the range of roughly ~~1°C/mm~~ 1 °C/mm (Fig. 3). For glasses with an estimated T_L > ~~1150°C,~~ 1150 °C, furnaces with elements capable of high temperatures need be used, for example, $MoSi_2$.

6.1.2 Calibrated thermocouple and temperature readout device appropriate for the estimated temperature range that will be used for testing. Type K thermocouples can be used within ~~95~~95 °C to ~~1260°C,~~ 1260 °C, Type R thermocouples can be used within ~~870~~870 °C to ~~1450°C,~~ 1450 °C, and Type S thermocouples can be used within ~~980~~980 °C to ~~1450°C~~ 1450 °C without special calibrations or qualifications.

6.1.3 Resistance furnace and controller used for annealing (capable of maintaining constant temperatures between ~~400~~400 °C and ~~~900°C)~~ ~900 °C) with a temperature accuracy of ~~10°C.~~10 °C.

6.1.4 Specimen boat made of material inert to the sample (for example, platinum alloy) with approximate dimensions of 0.5 cm × 1 cm × 10 cm to 30 cm (width × height × length), respectively; an example specimen boat is shown in Fig. 2. If the test glass viscosity is below 5 Pa·s at the measurement temperature, it is recommended that a round-based crucible be used. A separate option with Method A is to fill the long boat with several small individual boats with individual lids (Fig. 2~~-B)~~-B).

6.1.5 Diamond cutoff saw.

6.1.6 Variable speed polisher.

6.1.7 Silicone rubber mold for mounting of GT glass specimen in epoxy.

6.1.8 OM for TLM and/or RLM.

6.1.9 SEM/EDS.

6.1.10 XRD.

6.2 *Equipment Needed for ~~the UT Method:~~ UT:*

6.2.1 Resistance furnace capable of maintaining constant temperatures T ~~~550~~~550 °C to ~~1600°C~~ 1600 °C (that is, $MoSi_2$ heating elements) or furnace capable of $T \leq$ ~~1200°C~~ 1200 °C for glasses with $T_L \leq$ ~~1150°C.~~ 1150 °C.

6.2.2 Calibrated thermocouple and temperature readout device appropriate for the estimated temperature range that will be used for testing (6.1.2).

6.2.3 Specimen boat (or crucible) and tight fitting lid made of material compatible with the sample (for example, platinum alloy) with suggested dimensions of 1.2 cm × 1.2 cm × 1.2 cm (width × height × length, respectively) (Fig. 5-1A). Another option is a round-bottom, thimble-shaped crucible (Fig. 5-1B).

6.2.4 Diamond cutoff saw.

6.2.5 Variable speed polisher.

6.2.6 OM for TLM and/or RLM.

6.2.7 SEM/EDS.

6.2.8 XRD.

6.3 ~~Equipment needed for the CF method includes the same equipment as described previously in 6.2 because a UT specimen is required for the measurement technique, although additional materials are also required.~~*Equipment Needed for CF:*

6.3.1 This includes the same equipment as described previously in 6.2 because a UT specimen is required for the measurement technique, although additional materials are also required.

6.3.2 Image analysis software for measuring the C_F present in a micrograph collected with OM, SEM, etc.

6.3.3 Crystal structure/unit cell refinement software for quantifying crystal fractions by spiking in a known mass% of a known crystalline material.