



Designation: C1223 – 19 (Reapproved 2023)

Standard Test Method for Testing of Glass Exudation from AZS Fusion-Cast Refractories¹

This standard is issued under the fixed designation C1223; 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 This test method covers a procedure for causing the exudation of a glassy phase to the surface of fusion-cast specimens by subjecting them to temperatures corresponding to glass furnace operating temperatures.

1.2 This test method covers a procedure for measuring the exudate as the percent of volume increase of the specimen after cooling.

1.3 *Units*—The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.3.1 *Exception*—The balance required for this test method uses only SI units (Section 7).

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*:²

C20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water

¹ This test method is under the jurisdiction of ASTM Committee C08 on Refractories and is the direct responsibility of Subcommittee C08.10 on Refractories for Glass.

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² 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.

3. Terminology

3.1 *Acronyms*:

3.1.1 *AZS*—Alumina-Zirconia-Silica

4. Significance and Use

4.1 This test method was developed for use both by manufacturers as a process control tool for the production of AZS fusion-cast refractories, and by glass manufacturers in the selection of refractories and design of glass-melting furnaces.

4.2 The results may be considered as representative of the potential for an AZS refractory (specifically, in the tested region) to contribute to glass defect formation during the furnace production operation.

4.3 The procedures and results may be applied to other refractory types or applications (that is, reheat furnace skid rail brick) in which glass exudation is considered to be important.

5. Apparatus and Materials

5.1 *Scale*—A laboratory scale or balance rigged for suspension of specimens for dry/wet weight determinations to an accuracy of 0.01 g.

5.2 *Kiln*—An electric kiln to accommodate several 4-in. (102 mm) specimen cores placed vertically on end, and for service at 2750 °F (1510 °C), with a variation of <10 °F (6 °C).

5.3 *Foil*—Cups formed from 2¼-in. (56 mm) squares of platinum foil (Pt, 5 % Au alloy, 0.003 in. (0.076 mm) thick). One cup required per specimen.

5.4 *AZS Casting*—A virgin casting having no prior thermal history except that of its own formation, and of a size and casting process equivalent to the intended application (such as an arch block) in which exudation potential is of interest.

6. Test Specimens and Sampling

6.1 Specimens may be removed from the original casting either as drilled cores or as sawed bars, depending on laboratory capability. Specimen cores or bars should be 4 in. (102 mm) long and either 1 in. (25.4 mm) in diameter or 1 in. by 1 in. (25.4 mm by 25.4 mm) in cross section. The length dimension of the specimen should be perpendicular to the surface of the block from which it is removed.

6.2 The dimensions of the prepared specimen core are not critical but should be maintained as specified, with minimal specimen-to-specimen variation. Excessive thickness can prevent isothermal heating within the specimen. Height and width can affect the positioned stability of the specimen in the kiln during heating.

6.3 The size of the original casting may influence the results. Evaluations of the product should be made relative to only the intended application. For example, a conveniently sized bottom paver might not be representative of a larger superstructure casting because (for example) casting mold types and solidification rates may have been different during manufacture.

6.4 The location and depth of specimens within the original casting can influence the results. Regions closely underlying the surface of the casting (particularly near the corners and edges) are thermally quenched and have aligned microstructures that are atypical of more slowly cooled regions. Deeper in a casting, glass-phase pockets and crystal sizes are larger, and certain shifts in chemical stratification exist due to fractional crystallization during solidification. No single point in an AZS casting represents the whole entirely.

6.5 Regular-cast AZS blocks, approximately 8 in. to 12 in. (203 mm to 305 mm) thick, such as is typical of furnace superstructure and sidewall sizes, are sampled by drilling or plunge-cutting perpendicularly to the bottom surface (the surface opposite the casting scar).

6.5.1 The location of entry (by drilling or sawing) should be at least 4 in. (102 mm) away from any edge, yet not immediately under the casting scar.

6.5.2 Drill or cut deeper than specified, then break the specimen out from the casting and saw square to 4-in. (102 mm) length, retaining the mold skin (original surface of the block) on one end of the specimen by cutting off the end opposite it.

6.5.3 The quantity of specimens per casting is not specified. (Correlation coefficients of 10 % to 20 % have been obtained by this procedure on large specimen populations taken from single castings.)

6.6 For smaller, regular-cast blocks less than 8 in. (203 mm) thick, specimen length and location are determined by the original casting size. That is, the proximity of specimen location to any edge should be no less than half the casting thickness. The specimen length should be approximately half the casting thickness.

6.7 Solid-cast tile (3 in. (76 mm)) should be sampled perpendicularly to a major face, with the proximity to any edge being no less than half the thickness of the casting. The specimen length should be either half the thickness or full surface-to-surface thickness.

6.8 Large, vertically cast blocks, such as those that are used commonly in high-wear glass-contact applications, may be sampled perpendicularly to any of the four major vertical surfaces, with the following restrictions: sampling should be at least 4 in. (102 mm) from any edge, and the entire bottom region should be avoided up to 8 in. (203 mm) from the bottom

(as cast). This lower region, which often becomes the top “metal line” when the casting is inverted, has been found to be not representative of the overall casting.

7. Procedure

7.1 Weights must be obtained individually for both the untested specimen cores and the foil squares on which the cores will be placed. This is because each core and its foil will usually be fused together at the end of testing and cannot be separated before weighing without risk of lost exudate. Once paired, each set of core-and-foil must remain together throughout testing and subsequent calculation of data (see Fig. 1).

7.2 To account for the possible presence of surface-connected porosity in specimen cores, the treatments (drying and boiling) as specified by Test Methods C20 must be applied, as described as follows:

7.3 Dry the specimen cores to constant weight by heating to 220 °F to 230 °F (105 °C to 110 °C), and determine the dry weight (Wd_1) to the nearest 0.01 g.

7.4 Place the specimen cores in water and boil for 2 h. Keep the specimens entirely covered with water during the boiling period, and permit no contact with the heated bottom of the container.

7.5 After the boiling period, cool the test specimens to room temperature, while still covered completely with water, for a minimum of 12 h before weighing.

7.6 Determine the specimen core wet weight (Ww_1) of each specimen core after boiling and while suspended in water to the nearest 0.01 g.

7.7 This weighing is usually accomplished by suspending the specimen in a loop or halter of copper wire (such as AWG Gauge 22, 0.643 mm) hung from one arm, or from the underside of the balance. The balance shall be tared or counter-balanced previously with the wire in place and immersed in water to the same depth as is used when the refractory specimens are in place.

7.8 Determine the platinum foil dry weight (PWd_1) to the nearest 0.01 g.

7.9 Determine the platinum foil wet weight (PWw_1) to the nearest 0.01 g.

7.10 Stand the specimen cores on foil squares in the test furnace with the sawed ends facing downward. Form the foil into crude cups so that any rundown of exudation will be contained. Failure to use foil may result in disappearance of exudate into the furnace floor.

7.11 Heating Cycle:

7.11.1 Over 12 h, attain 2750 °F ± 10 °F (1510 °C ± 6 °C).

7.11.2 Maintain the test temperature for 4 h.

7.11.3 Shut the power off; let the furnace cool.

7.11.4 Remove the specimen cores with adhered foil; allow to cool for 24 h.

NOTE 1—A stable, uninterrupted test temperature is essential; it has been found that cooling and reheating of AZS specimens can cause a significant increase in exudation.

Casting No. _____	ASTM Member _____
Specimen No. _____	Date Reported _____

BEFORE EXUDATION

Core Wd ₁ = _____ g	Pt Foil PWD ₁ = _____ g.
Core Ww ₁ = _____ g.	Pt Foil PWW ₁ = _____ g.
Vol ₁ = (Wd ₁ - Ww ₁) = _____ cc.	

AFTER EXUDATION

	(Cycle 1)	(Cycle 2)	(Cycle 3)
Core Wd ₂ =	_____ g.	_____ g.	_____ g.
Foil PWD ₁ =	_____ g.	_____ g.	_____ g.
ECDW =	_____ g.	_____ g.	_____ g.
Core Ww ₂ =	_____ g.	_____ g.	_____ g.
Foil PWW ₁ =	_____ g.	_____ g.	_____ g.
ECWW =	_____ g.	_____ g.	_____ g.
ECDW - ECWW =	_____ cc.	_____ cc.	_____ cc.
Vol ₂	_____ cc.	_____ cc.	_____ cc.

Vol ₂ - Vol ₁ =	_____ cc.	_____ cc.	_____ cc.
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PERCENT EXUDATION

$$\% = \frac{\text{Vol}_2 - \text{Vol}_1}{\text{Vol}_1} = \quad \% \quad \% \quad \%$$

FIG. 1 Worksheet – Round Robin No. 2 for AZS Exudation

7.12 Determine the dry weight of the specimen with the foil attached (Wd₂) to the nearest 0.01 g.

7.13 Prepare the specimen cores (with foil attached) for wet weighing by first boiling again as described in 7.4 and 7.5. Care should be taken to avoid turbulent boiling, which might cause fracture and loss of exudate.

7.14 Determine the specimen core-plus-foil wet weight (Ww₂) after boiling, and while suspended in water, to the nearest 0.01 g.

NOTE 2—Alternately, the weight (Wd₂) of the specimen core plus foil can be determined after wet weighing by drying to constant weight at 220 °F to 230 °F (105 °C to 110 °C).

8. Calculations and Reporting

8.1 Exudate is defined as the percent increase in original volume of the specimen core.

8.2 Calculations are simplified by first converting dry-versus-wet weight differences into volumes, and by correcting for the weight of attached foil, as follows:

$$\text{volume}_1 = (Wd_1 - Ww_1) = \text{_____cc} \quad (1)$$

$$\begin{aligned} \text{exuded core dry weight (ECDW)} &= (Wd_2 - PWD_1) \quad (2) \\ &= \text{_____g} \end{aligned}$$

$$\begin{aligned} \text{exuded core wet weight (ECWW)} &= (Ww_2 - PWW_1) \quad (3) \\ &= \text{_____g} \end{aligned}$$

$$\text{volume}_2 = \text{ECDW} - \text{ECWW} = \text{_____cc} \quad (4)$$

8.3 Thus,

$$\% \text{ exudation} = \frac{\text{volume}_2 - \text{volume}_1}{\text{volume}_1} \times 100$$

8.4 Observations may be made concerning the clarity and color of exudate, and the extent of beading or rundown of exudate on the specimen.

8.5 Gain or loss of specimen dry weight after testing may be noted as a check upon the accidental loss of exudate.

9. Retesting (Cycling) for Additional Exudation

9.1 Prior work has shown that the reheating (temperature cycling) of oxidized AZS specimen cores produces additional exudation considerably above a level that the increased time-at-temperature could explain. Reheating has been found to have more effect on total exudation than the variables of time, temperature, casting size, or specimen location. This phenomenon may have application in understanding of the relatively poor performances of intermittently operated glass melting furnace.

9.2 To obtain a measure of reheat exudation, repeat testing two more times on the same specimens, starting each time at room temperature. Calculate the incremental and cumulative volume increases after each test. Changes as measured should be relative to the original (untested) volume.