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Standard Test Method for Testing of Glass Exudation from AZS Fusion-Cast Refractories[1]

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 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water[2]

3. Significance and Use

3.1 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.

3.2 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.

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

4. Apparatus and Materials

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

4.2 *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).

4.3 *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.

4.4 *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.

5. Test Specimens and Sampling

5.1 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 by 1 in. (25.4 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.

5.2 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.

5.3 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.

5.4 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

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[1] This test method is under the jurisdiction of ASTM Committee C-8 on Refractories and is the direct responsibility of Subcommittee C08.10 on Refractories for Glass.

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[2] *Annual Book of ASTM Standards*, Vol 15.01.

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.

5.5 Regular-cast AZS blocks, approximately 8 to 12-in. (203 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).

5.5.1 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.

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

5.5.3 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.)

5.6 For smaller regular-cast blocks less than 8-in. (203-mm) thick, specimen length and location are determined by 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.

5.7 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.

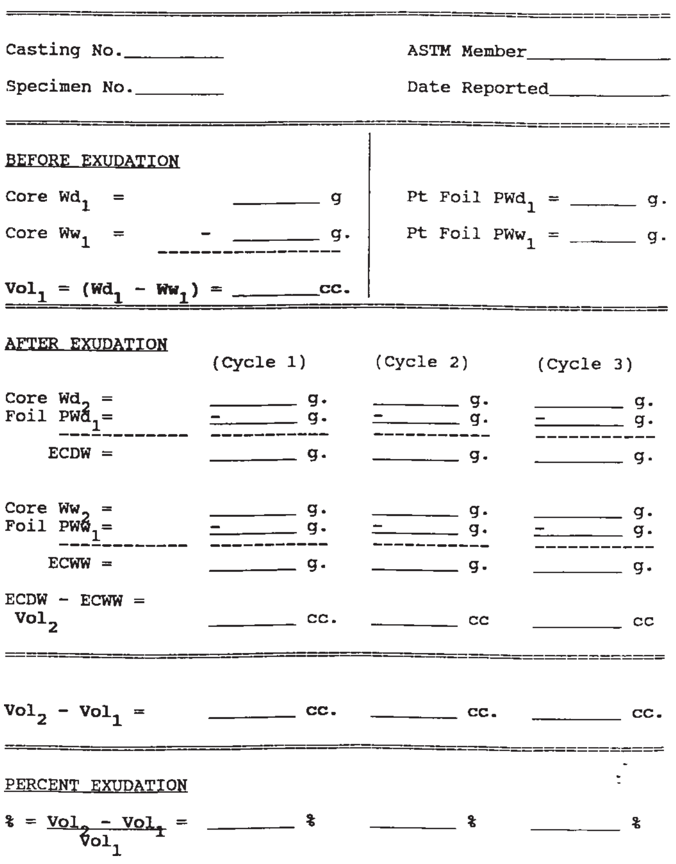
5.8 Large, vertically-cast blocks, such as those that are used commonly in high-wear glass-contact applications, may be sampled perpendicularly to any of the four major vertical surfaces, with the following restrictions: sampling should be at least 4 in. (102 mm) from any edge, and the entire bottom 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" region, as when the casting is inverted, has been found to be not representative of the overall casting.

6. Procedure

6.1 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).

6.2 To account for the possible presence of surface-connected porosity in specimen cores, the treatments (drying and boiling) as specified by Test Methods C 20 must be applied, as described as follows:

6.3 Dry the specimen cores to constant weight by heating to 220 to 230°F (105 to 110°C), and determine the dry weight (*Wd*$_1$) to the nearest 0.01 g.



Casting No.________ ASTM Member____________

Specimen No.________ Date Reported__________

BEFORE EXUDATION

Core Wd_1 = ________ g

Core Ww_1 = - ________ g.

$Vol_1 = (Wd_1 - Ww_1)$ = ________cc.

Pt Foil PWd_1 = ______ g.

Pt Foil PWw_1 = ______ g.

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 = Vol_2	________ cc.	________ cc	________ cc
$Vol_2 - Vol_1$ =	________ cc.	________ cc.	________ cc.

PERCENT EXUDATION

$\% = \frac{Vol_2 - Vol_1}{Vol_1}$ = ________ % ________ % ________ %

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

6.4 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.

6.5 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.

6.6 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.

6.7 This weighing is usually accomplished by suspending the specimen in a loop or halter of copper wire (such as AWG Gage 22, 0.643 mm) hung from one arm, or from the underside of the balance. The balance shall be 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.

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

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

6.10 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.

6.11 *Heating Cycle*:

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

6.11.2 Maintain the test temperature for 4 h.

6.11.3 Shut the power off; let the furnace coil.

6.11.4 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.

6.12 Prepare the specimen cores (with foil attached) for wet weighing by first boiling again as described in 6.4 and 6.5. Care should be taken to avoid turbulent boiling, which might cause fracture and loss of exudate.

6.13 Determine the specimen core-plus-foil wet weight (Ww_2) after boiling, and while suspended in water, to the nearest 0.01 g.

6.14 Dry the specimen core-plus-foil to constant weight by heating to 220 to 230°F (105 to 110°C), and determine the dry weight (Wd_2) to the nearest 0.01 g.

NOTE 2—The drying of specimens to constant weight can be avoided as a procedural step if specimens are weighed (Wd_2) shortly after being removed from the test furnace.

7. Calculation

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

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

$$\text{volume}_1 = (Wd_1 - Ww_1) = \text{ cc} \tag{1}$$

$$\text{exuded core dry weight (ECDW)} = (Wd_2 - PWd_1) = \text{ g} \tag{2}$$

$$\text{exuded core wet weight (ECWW)} = (Ww_2 - PWw_1) = \text{ g} \tag{3}$$

$$\text{volume}_2 = \text{ECDW} - \text{ECWW} = \text{ cc} \tag{4}$$

7.3 Thus,

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

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

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

8. Retesting (Cycling) for Additional Exudation

8.1 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.

8.2 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.

9. Adjustments

9.1 It is acknowledged that the volume changes in AZS specimens that occur during heating are not entirely the result of exudation. Other variables, such as the zirconia (ZrO2) hysteresis and high-temperature creep, are known to have an effect on volume, albeit minor compared to that of exudation.

10. Precision and Bias

10.1 *Interlaboratory Data*—An interlaboratory study was conducted in 1991 in which specimen cores drilled from a single AZS (33 % ZrO_2) casting were tested for exudation. Five laboratories each received a randomized set of four cores. Each laboratory tested the specimen cores for cumulative exudation over three temperature cycles (see Section 8).

10.2 *Precision*—Precision and relative precision data at the 95 % confidence level are given in Table 1.

10.3 *Bias*—No justifiable statement on bias can be made since the true value cannot be established from an accepted reference sample.

11. Keywords

11.1 AZS; casting; exudation; fusion-cast; glass (glass phase); refractories

TABLE 1 Precision Statistics

Precision	Cycle 1	Cycle 2
Average, x	2.98	8.73
Standard within, S_r	0.376	0.816
Deviation between, S_R	0.706	0.841
Repeatability interval, r	1.05	2.29
Reproducibility interval, R	1.98	2.35
Relative Precision		
Coefficient of variation:		
within laboratory, V_r	12.6	9.35
between laboratories, V_R	23.7	9.63
Relative repeatability, % r	35.3	26.2
Relative reproducibility, % R	66.3	26.9