Designation: C 1276 – 94 (Reapproved 1999)

Standard Test Method for Measuring the Viscosity of Mold Powders Above Their Melting Point Using a Rotational Viscometer[1]

This standard is issued under the fixed designation C 1276; 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 measurement of the viscosity of mold powders above their melting point through the use of a platinum spindle immersed in a platinum crucible containing the molten mold flux. Developed by differential angular velocity between the crucible and spindle, spindle torque is measured and used to calculate the viscosity. Data are generally taken as a function of temperature to describe the viscosity-temperature relationship for the molten mold flux.

1.2 This test method uses a high-temperature furnace and measurements on molten material. Personal protection equipment to wear include high-temperature resistant insulating gloves, coveralls, and a full-face shield.

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.* A specific warning statement is given in Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

C 965 Practices for Measurement of Viscosity of Glass Above the Softening Point[2]

C 1095 Practice for Calculating Precision Data on Refractories (C08) from Interlaboratory Test Results[3]

E 220 Method for Calibration of Thermocouples by Comparison Techniques[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

[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 15.02.

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

[4] *Annual Book of ASTM Standards*, Vol 14.02.

3. Significance and Use

3.1 The viscosity of a molten mold flux is a very important property for the correct functioning of a mold powder. This procedure is designed for producers and users of mold powders to develop the viscosity-temperature relationships of the molten mold flux for quality control or specification purposes, or both. Practices C 965 were developed for glasses that are significantly higher in viscosity than mold powders. Also, sample preparation is very different for the glass products covered.

NOTE 1—**Warning:** Use caution when discharging molten sample into water since this can cause an explosion.

4. Apparatus

4.1 *Electrically Heated Furnace*, equipped with a temperature controller, temperature measuring equipment, a spindle, a crucible, a device to rotate the spindle, and equipment to measure the torque or viscosity.

4.1.1 Any electrically heated furnace capable of providing a uniform and stable temperature in the testing zone of the furnace can be used. The furnace must be capable of maintaining a temperature 180°F (82°C) above the maximum test temperature. The atmosphere in the furnace should be air or oxidizing.

4.2 *Thermocouple*, calibrated in accordance with Method E 220. It is preferred that the thermocouple be immersed in the molten mold flux, in which case only the bare metal of the thermocouple should be immersed, since immersion of the thermocouple sheath may result in a reaction between the sheath and the molten flux. Alternatively, the thermocouple can be placed outside the crucible containing the molten mold flux, providing that the temperature difference between the flux and the position where the thermocouple is placed is no more than 9°F (5°C).

4.3 *Spindle and Crucible*, platinum or a platinum alloy.[5] The spindle is attached to the rotating device using platinum extension wires inside the furnace and other suitable wires outside the furnace, for example, stainless steel.

[5] An alloy such as zirconia grain-stabilized (ZGS) platinum has been used successfully.

4.3.1 The dimensions of the crucible and spindle shall be such that, when the crucible is filled with molten mold flux, the body of the spindle can be immersed fully to at least a depth of 0.4 in. (1 cm) and the distance between the spindle and the crucible bottom and all crucible walls shall be at least 0.4 in. (1 cm). Such minimum dimensions will eliminate wall effects. There are no limits on the maximum dimensions of the crucible. However, larger crucibles and consequently larger amounts of molten mold flux will take longer to stabilize at temperature. See Fig. 1 for a typical crucible/spindle configuration.

4.4 *Device to Rotate the Spindle*[6] *and Equipment to Measure the Spindle Torque*. This device should be accurate to within ± 1 % of the full-scale range of the spindle/speed combination in use. Reproducibility should be within ±0.2 %.

5. Preparation of Mold Flux Powder

5.1 Burn out any carbon in the mold powder by heating the sample in air at 900 to 1400°F (471 to 760°C). The required heating time will depend on the carbon content and types; experience has shown 3 to 24 h to be sufficient. Burning out the carbon is very important due to possible reaction with the platinum parts.

[6] Equipment such as the Brookfield RVT or HBT is commercially available.

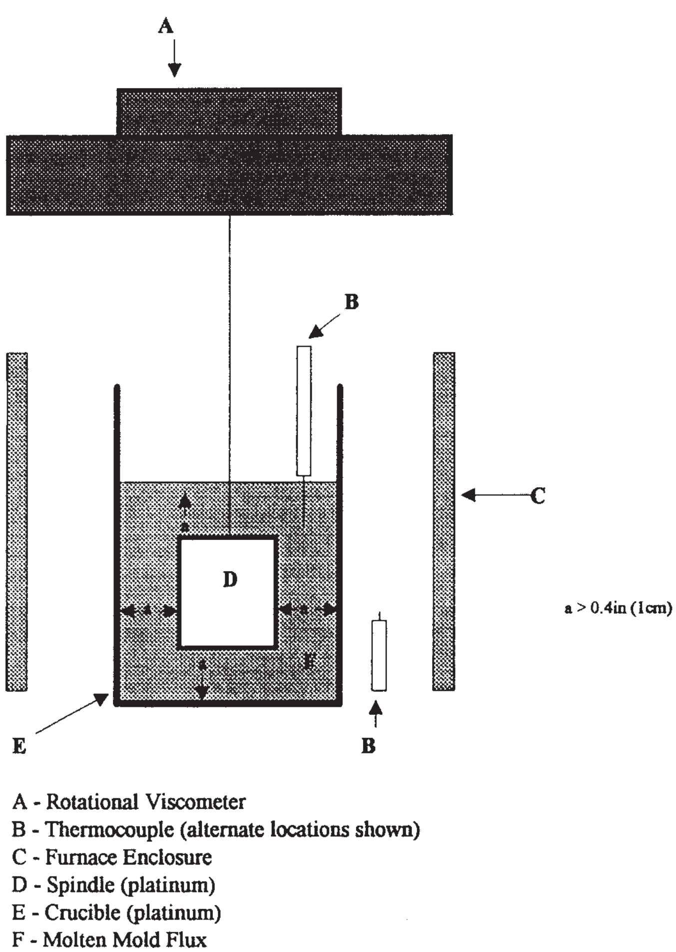

FIG. 1 Typical Crucible/Spindle Configuration

6. Calibration of the Torque Viscometer

6.1 Clean the spindle thoroughly prior to running this calibration test. Conduct this test out of the furnace at 77 ± 0.2°F (25.0 ± 0.1°C) using standard viscosity silicone oils. This calibration procedure must be run with silicone oils of various viscosities and using various spindle r/min. The types of silicone oils tested should have viscosities in the same range as those of the molten mold flux. Fill the crucible with the standard viscosity silicone oil, and stabilize the temperature by placing into a water bath set at 77 ± 0.2°F (25.0 ± 0.1°C).

6.2 Lower the spindle into the crucible, and position it in such a manner that it meets the dimensional tolerances as specified in 4.3.1.

6.3 Switch the viscometer on at a selected speed of rotation (r/min). Observe the reading on the viscometer.

6.4 Calculate the calibration constant from the following formula:

$$\text{calibration constant} = (\text{viscosity of standard})/(\text{viscometer reading}) \text{ at given r/min}$$

6.5 If the standard silicone oil has a viscosity of 3.0 poise and the viscometer reading at 30 r/min is 40, the constant is 0.075 at 30 r/min.

6.6 If the viscometer reading on the molten mold flux is 48 at 30 r/min, the calculated viscosity is 3.6 poise.

7. Procedure

7.1 Fill the crucible with decarburized (see 5.1) mold powder to within 0.4 in. (1 cm) of the top of the crucible. If the mold powder contains carbonates, it should be added in smaller increments to avoid possible boil out during the melting process.

7.2 The crucible containing the mold powder can be placed in the furnace either hot or cold. Control the furnace temperature to approximately 2372°F (1300°C), and allow the initial charge of mold powder to melt.

7.3 When the powder has melted completely, add more decarburized (see 5.1) mold powder to within 0.4 in. (1 cm) of the top of the crucible.

7.4 Repeat the step given in 7.3 until the volume of the molten flux is sufficient to meet the requirements of 4.3.1.

7.5 Increase the furnace temperature and control at 2552± 4°F (1400 ± 2°C).

7.6 Suspend the spindle above the crucible for 5 min to allow preheating.

7.7 Lower the spindle into the crucible and position it such that it meets the dimensional tolerances as specified in 4.3.1.

7.8 Start rotation of the spindle. Allow 20 min for the temperature to stabilize. Measure and record the torque readings and corresponding temperature once the temperature is stable (this will be indicated by a stable reading on the measuring apparatus).

7.9 Decrease the temperature to 2372 ± 4°F (1300 ± 2°C). Allow 15 min for stabilization. Repeat the steps given in 7.8.

7.10 Decrease the temperature to 2282 ± 4°F (1250 ± 2°C). Allow 15 min for stabilization. Repeat the steps given in 7.8.

7.11 After completion of the temperature and viscosity measurements, increase the furnace temperature to 2372°F

(1300 °C) for 10 min. Raise the spindle and allow the molten flux to drip back into the crucible. Remove the spindle and crucible from the furnace. The molten mold flux can be poured into a sand bucket or a bucket containing a large volume of water.

7.12 Ensure that the spindle and crucible are cleaned prior to the next test in order to eliminate test-to-test contamination.

8. Report

8.1 Report the following information:

8.1.1 Viscosity standard used and calibration constant found.

8.1.2 Designation of the mold flux powder, source, and date.

8.1.3 Viscosity readings at the three temperatures.

8.1.4 Date of the test and name of the operator.

9. Precision and Bias

9.1 *Ruggedness Data*—Ruggedness tests were conducted in 1988. Ruggedness test factors included the following: temperature, viscometer position, submerged depth, sample weight, and spindle wobble. The sample weight and temperature were determined to be non-rugged. These two variables were subsequentially compensated for in the final protocol. A research report covering the ruggedness results is on file at ASTM Headquarters.

9.2 *Interlaboratory Data*—An interlaboratory round robin was conducted in 1989 in which three laboratories determined the viscosity of mold powder sample at three temperatures; two laboratories obtained three test results, while one laboratory obtained two test results. The precision statistics from this study are given in Table 1. Refer to Practices E 691 and C 1095 for calculation and explanation of the various components. A research report covering the interlaboratory study is on file at ASTM Headquarters.

9.3 *Precision*:

9.3.1 *Repeatability*—The maximum permissible difference due to test error between two test results, obtained by one operator on the same material using the same test equipment, is given by the repeatability interval and relative repeatability interval (coefficient of variation). The 95 % repeatability intervals are given in Table 1. Two test results that do not differ by more than the repeatability interval will be considered to be from the same population, and, conversely, two test results that do differ by more than the repeatability interval will be considered to be from different populations.

9.3.2 *Reproducibility*—The maximum permissible difference due to test error between two test results, obtained by two operators in different laboratories on the same material using the same test equipment, is given by the reproducibility interval and relative reproducibility interval (coefficient of variation). The 95 % reproducibility intervals are given in Table 1. Two test results that do not differ by more than the reproducibility interval will be considered to be from the same population, and, conversely, two test results that do differ by more than the reproducibility interval will be considered to be from different populations.

9.4 *Bias*—No justifiable statement of bias can be made since the true value for mold powder viscosity cannot be established by an accepted reference standard.

10. Keywords

10.1 flux; mold powder; refractories; viscosity

TABLE 1 Precision Statistics

Mold Powder 1	Precision Data				
Temperature (°C)	Average Viscosity (*P*)	Standard Deviation Within	Standard Deviation Between	95 % Repeatability Interval	95 % Reproducibility Interval
	$\bar{x}$	*S*(*r*)	*S*(*R*)	*r*	*R*
1250	4.53	0.10	0.29	0.27	0.80
1300	3.25	0.06	0.21	0.17	0.60
1400	1.91	0.05	0.06	0.13	0.18

Mold Powder 1	Relative Precision Data				
Temperature (°C)	Average Viscosity (*P*)	Within Laboratory	Between Laboratory	Relative Repeatability	Relative Reproducibility
	$\bar{x}$	*V*(*r*)	*V*(*R*)	% *r*	% *R*
1250	4.53	2.15	6.31	6.01	17.66
1300	3.25	1.92	6.60	5.37	18.49
1400	1.91	2.47	3.30	6.91	9.24