

Standard Test Methods for Viscosity of Adhesives¹

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This standard has been approved for use by agencies of the Department of Defense. These test methods replace Method 4021 or Federal Test Method Standard No. 175a.

1. Scope

1.1 These test methods cover the determination of the viscosity of free-flowing adhesives.

1.1.1 The limitation of this test method to self-leveling adhesives eliminates thixotropic and plastic materials whose viscosity is a function of the rate of stirring and previous history of the adhesive.

1.2 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.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

2. Referenced Documents

2.1 ASTM Standards:

- D 88 Test Method for Saybolt Viscosity²
- D 115 Test Methods for Varnishes Used for Electrical Insulation³
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 907 Terminology of Adhesives⁵
- D 1200 Test Method for Viscosity by Ford Viscosity Cup⁴
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁶
- D 1601 Test Method for Dilute Solution Viscosity of Ethylene Polymers⁷
- D 2556 Test Method for Apparent Viscosity of Adhesives Having Shear-Rate-Dependent Flow Properties⁵

3. Terminology

3.1 Definitions-Many terms in these test methods are

- ³ Annual Book of ASTM Standards, Vol 10.01.
- ⁴ Annual Book of ASTM Standards, Vol 06.01.
- ⁵ Annual Book of ASTM Standards, Vol 15.06.
- ⁶ Annual Book of ASTM Standards, Vol 06.03.

defined in Terminology D 907.

3.1.1 *viscosity*, *n*—*of a liquid*, the resistance to flow expressed as the ratio of the applied stress to the resulting rate of shearing strain (see Newtonian behavior, and thixotropy). (Compare consistency.)

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *viscosity*—the ratio of shear stress to shear rate. The viscosity of a liquid is a measure of the internal friction of the liquid in motion. The unit of dynamic viscosity is the poise, which is expressed as dyne-seconds per square centimetre. For a Newtonian liquid, the viscosity is constant at all shear rates. For a non-Newtonian liquid, viscosity will vary depending on shear rate.

4. Significance and Use

4.1 Several test methods are noted for viscosity determination of Newtonian and near Newtonian flow characteristics. (For adhesives not fitting into this category, Test Method D 2556 should be considered.) Four test methods are covered as follows:

4.1.1 *Method A* is applicable only to adhesives that will deliver 50 mL in a steady uninterrupted stream from one of the cups described in Section 5.

4.1.2 *Method B* is intended for measuring the viscosity of adhesives covering a range from 0.05 Pa \cdot s to 200 Pa \cdot s (50 to 200 000 cP) and is limited to materials that have or approach Newtonian flow characteristics.

4.1.3 *Method* C is intended primarily as a control method for determining the viscosity of adhesives that have or approach Newtonian flow characteristics.

4.1.4 *Method* D is intended primarily as a control method for determining the viscosity of materials that have an equivalent viscosity no greater than approximately 3000 cP and is limited to materials that have or approach Newtonian flow characteristics.

TEST METHOD A

5. Apparatus

5.1 The apparatus (Fig. 1) consists of a set of four viscosity cups so designed as to deliver 50 mL of the sample in from 30 to 100 s at a temperature of $23 \pm 0.5^{\circ}$ C (73.4 $\pm 0.9^{\circ}$ F).

5.1.1 Four consistency cups constitute a set as follows:

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² Annual Book of ASTM Standards, Vol 04.04.

⁷ Annual Book of ASTM Standards, Vol 08.01.

御》D 1084 60.33 MIN DIA 0.79 34.93 ± 0.40 ID (1 3/8 ± 1/64 in.) 3/8 in.) (2 (1/32 in.) APPROX. 12.7 (1/2 in.) APPROX. APPROX. NO 14 NONLEAK (0.0641) B. & S. GAGE JOINT 91.28 (3 19/32 in.) 90 mL 2.38 (3/32 in.) APPROX. TUBING HAVING 1.59 MIN WALL (1/16 in.) 0⊂ С NONLEAK JOINT 36.51 DIA. (7/16 in.) - APPROX.-50 mL 1.59 ± 0.03 (0.0625 ± 0.001 in.) 9.53 DIA (3/8 in.) PRESS FIT FOR STAINLESS STEEL ORIFICE

SECTION THROUGH DIAMETER OF CUP

FIG. 1 Consistency Cups and Apparatus Assembly

5.88 DIA 5/8 in.) APPROX.

(Test Method A)

Diameter of Orifice, in.
0.07 ± 0.0001
0.10 ± 0.0001
0.15 ± 0.0001
0.25 ± 0.0001

0.79 MIN (1/32 in.)

5.1.2 Cups made of brass or bronze. Orifice disks made of 18 % Chromium, 8 % Nickel stainless steel. The diameter of the Orifice pressed in the cup shall be stamped on the cup

5.1.3 Fractional dimensions subject to permissible variations of ± 0.01 in., unless otherwise specified.

6. Procedure

6.1 Bring the sample to be tested and the viscosity cup to a temperature of $23 \pm 0.5^{\circ}$ C (73.4 ± 0.9°F) (preferably in a constant-temperature room). Then mount the consistency cup in the clamp provided for the purpose and place the receiving cylinder in position. With the outlet of the cup closed by means of the finger, pour the sample into the cup until it is filled to overflowing. Strike off the excess with a straightedge, and then remove the finger from over the outlet and allow the sample to flow into the receiving cylinder. Determine the number of seconds from the time the finger is removed from the orifice until the top of the meniscus reaches to 50-mL mark on the cylinder by a stop watch and record as the viscosity of the material.

7. Report

7.1 Report the following:

7.1.1 Complete identification of the adhesive tested, including type, source, manufacturer's code numbers, form, date of test, date of manufacture, etc., 7.1.2 Conditioning procedure used for samples prior to testing,

203.2 (8 in.)

7.1.3 Number of tests made, and

7.1.4 Average consistency in seconds and the number of the viscosity cup used.

TEST METHOD B

8. Apparatus

8.1 Viscometer—The apparatus consists of a Brookfield synchrolectric viscosimeter,⁸ or an equivalent instrument. A series of spindles with various sized disks is provided with each instrument covering a standard range of viscosities. Scored, warped, or otherwise damaged spindles are not to be used. The calibration sleeve will be used when the values are to be reported in actual centipoise. This spindle guard ensures uniform edge effects. By mutual agreement, the instrument may be used without the calibration sleeve, but the values obtained must be reported in apparent centipoises. Use a standard container and take care to center the spindle in the container. A complete description of the container must be include in the report.

8.2 *Thermometer*—A precision thermometer with graduations not greater than 0.2° C (0.36° F) divisions.

9. Calibration

9.1 When used for referee purposes, calibrate the instrument

⁸ Manufactured by the Brookfield Engineering Laboratories, Stoughton, MA.

by measuring the viscosity of a standard oil,⁹ using the same spindle and speed of rotation employed to measure the sample. If the viscosity of the oil as read by the instrument differs by more than 2 % and less than 20 % from the certified viscosity of the oil, calculate the viscosity of the unknown by means of the appropriate correction factor. Investigate a difference of more than 20 %, and determine a calibration curve using at least two standard oils with viscosities on either side of the sample for the instrument.

10. Procedure

10.1 *Conditioning*—Bring the sample of the adhesive and the instrument to a temperature of $23 \pm 0.5^{\circ}$ C (73.4°F) (preferably in a constant-temperature room), and maintain the sample uniformly at this standard temperature throughout the test. Protect adhesives containing volatile solvents from evaporation during conditioning. If special conditioning methods are necessary, such as the use of a circulating water bath, report in 11.1.5.

10.2 *Adjustment*—Select a spindle suitable to the viscosity range of the material and firmly fit it into the shaft extension which comes down through the center of the dial casing.

10.3 Determination—Insert the spindle perpendicularly into the material to be tested until immersed to the depth indicated by the groove cut into the shaft. Press down the clutch lever and start the motor by snapping the toggle switch. Then release the lever and allow rotation to continue until the pointer has reached the position where it is stationary in relation to the rotating dial. Again press down the clutch lever and snap the switch off. If the pointer is not in view when the dial has come to rest, the motor should be started again and allowed to run until the pointer reaches the vision plate, keeping the clutch lever depressed. Take the reading at the pointer. A check reading can also be made by restarting the motor before releasing the clutch. If the pointer makes a complete turn of the dial, this indicates that the viscosity is too great for the capacity of the spindle used. A slower speed of rotation or a spindle with a greater range should be used. If the pointer moves less than 20 % of the way around the dial, this indicates that the viscosity is too low for accurate measurement with the spindle or rate of rotation used. A faster speed of rotation or a spindle with a lower range should be used. Make a minimum of three readings. Some instruments have two concentric scales, and great care should be taken to read the pointer on the correct scale as specified by the manufacturer for the spindle used.

11. Report

11.1 Report the following:

11.1.1 Complete identification of the adhesive tested, including type, source, manufacturer's code numbers, form, date of test, date of manufacture, etc.,

11.1.2 Name and model number of the instrument used,

11.1.3 Number of the spindle used, and specify use of spindleguard,

11.1.4 Speed used,

11.1.5 Conditioning procedure employed, including details of the container and time elapsed between various operations

used in the preparation of the adhesive mix,

11.1.6 Temperature of the sample at the conclusion of the test,

11.1.7 Number of tests made, and

11.1.8 Viscosity in actual or apparent centipoises, together with the range of the observation in centipoises.

TEST METHOD C

12. Apparatus

12.1 *Viscometer*—A Stormer viscosimeter with double flag paddle-type rotor as specified in Test Method D 562, or an equivalent apparatus, in good repair and a spindle that spins freely when started without the paddle.

12.2 *Containers*—Round friction-top metal cans having a capacity of 1 qt (0.9 L). Nonstandard containers may be used by mutual agreement, when necessary to prevent corrosion or other deleterious effects of metal cans.

12.3 *Thermometer*—A precision thermometer with graduations not greater than 0.2° C (0.36° F) divisions.

12.4 *Timer*—A stop watch or suitable timer measuring to 0.2 s.

13. Procedure

13.1 Conditioning—Bring the containers holding the samples to be tested and the paddle to be used to a temperature of 23 ± 0.5 °C (73.4°F) (preferably in a constant-temperature room), and maintain the sample uniformly at this standard temperature throughout the test. Protect adhesives containing volatile solvents from evaporation during conditioning. If special conditioning methods are necessary, such as the use of a circulating water bath. Report this in 14.1.2.

13.2 Adjustment—Prepare the instrument for use by raising the weight to the top by winding up the cord with the ratchet provided for that purpose. Set the revolution counter at 10 revolutions below the zero mark. Use a falling weight of such magnitude that a minimum time of 20 s is required for 100 revolutions of the paddle when the test is conducted as specified in 13.2. Choose the weight from the series 50, 100, 200, 500, and 1000 g. Fasten the paddle securely on the shaft, place the container on the platform, and raise until the surface of the adhesive just reaches the mark on the stem of the paddle. This ensures uniform depth of immersion of the paddle.

13.3 *Determination*—Release the brake on the large cog wheel, and start the timing device when the needle on the revolution counter passes the zero mark. Note the time for 100 revolutions accurately to the closest 0.2 s. Make a minimum of three determinations.

14. Report

14.1 Report the following:

14.1.1 Complete identification of the adhesive tested, including type, source, manufacturer's code numbers, form, date of test, date of manufacture, etc.,

14.1.2 Conditioning procedure employed, including time elapsed between various operations used in the preparation of the adhesive mix,

14.1.3 Details of nonstandard containers, if used,

14.1.4 Temperature of the sample at the conclusion of the test,

⁹ Available from the Cannon Instrument Co., Box 16, State College, PA 16801.

14.1.5 Weight used,

14.1.6 Number of tests made, and

14.1.7 Average viscosity in seconds for 100 revolutions, together with the range of the observations.

TEST METHOD D

15. Apparatus

15.1 The apparatus (Fig. 2) consists of a set of five Zahn viscosity $cups^{10}$ so designed as to allow a sample to flow through the calibrated orifice in approximately 1 min or less, best results being obtained when the flow time is between 20 and 40 s.

15.1.1 Five viscosity cups constitute a set as follows:

	Diameter of Orifices		
	in. + 0.0003	mm + 0.0076	
	- 0.0002	-0.0051	
First cup	0.0788	2.002	
Second cup	0.1082	2.748	
Third cup	0.1487	3.777	
Fourth cup	0.1684	4.277	
Fifth cup	0.2072	5.263	

15.1.2 Cups shall be made of stainless steel, 0.050 in. (1.27 mm) thick. The number of the cup shall be stamped on a plate on the cup.

¹⁰ Available from Paul N. Gardner Co., Pompano Beach, FL.

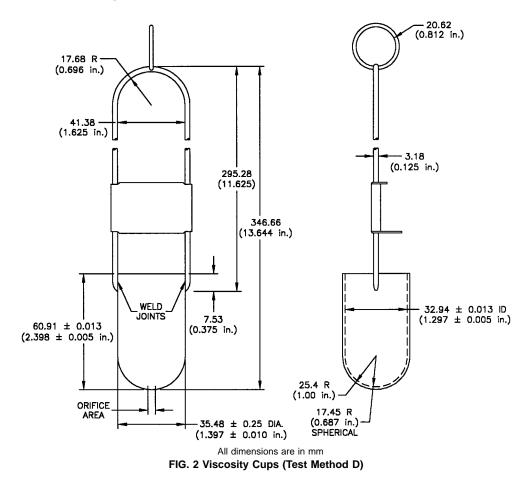
15.2 The following figures are given only for the purpose of selecting the proper viscometer. They are not intended for use in converting centipoises to Zahn seconds.

Range Pa · S	(Range, cP)	Range, Zahn's
0.02 to 0.085	(20 to 85)	40 to 85
0.03 to 0.170	(30 to 170)	20 to 70
0.120 to 0.550	(170 to 550)	25 to 60
0.200 to 0.900	(200 to 900)	20 to 65
0.250 to 1.200 (and above)	(250 to 1200) (and above)	15 to 60
	0.02 to 0.085 0.03 to 0.170 0.120 to 0.550 0.200 to 0.900 0.250 to 1.200	0.02 to 0.085 (20 to 85) 0.03 to 0.170 (30 to 170) 0.120 to 0.550 (170 to 550) 0.200 to 0.900 (200 to 900) 0.250 to 1.200 (250 to 1200)

16. Procedure

16.1 *Conditioning*—Bring the sample of the adhesive and the cup to a temperature of $23 \pm 0.5^{\circ}$ C ($73.4 \pm 0.9^{\circ}$ F), preferably in a constant-temperature room, and maintain the sample uniformly at this standard temperature throughout the test. Other temperatures of test are optional. Protect adhesives containing volatile solvents from evaporation during conditioning. If special conditioning methods are necessary, such as the use of a circulating water bath, they shall be specified in 17.1.2.

16.2 *Determination*—Hold the viscometer in a vertical position (by means of a small ring at the end of the handle) and completely immerse the cup into the sample being tested. Lift the viscometer out of the sample (Note 1). Determine by means of a stop watch the number of seconds from the time the top edge of the viscometer cup breaks the surface until the steady flow from the orifice first breaks, and record this time as the consistency of the material. Record the temperature of the



sample and the number of the cup.

NOTE 1—Time of removal must be rapid. Unless build-up of adhesive on the side of the cup occurs, the time of removal should be about 1 s.

17. Report

17.1 Report the following:

17.1.1 Complete identification of the adhesive tested, including type, source, manufacturer's code numbers, form, date of test, date of manufacture, etc.,

17.1.2 Conditioning procedure employed for samples prior to testing,

17.1.3 Number of tests made, and

17.1.4 Average viscosity in seconds and the number of the viscosity cup used.

PRECISION AND BIAS

18. Precision and Bias

18.1 At the present time there is no basis for a statement of precision and bias concerning the reproducibility of results among laboratories.

19. Keywords

19.1 adhesive; viscosity

APPENDIX

(Nonmandatory Information)

X1. REFERENCE VISCOSITY METHODS

X1.1 Reference viscosity methods giving equipment used are shown on the following table:

Test Method for	Refers to	Equipment Used
Viscosity of Transparent Liquids by Bubble Time Method	D 1545	Gardner-Holdt tubes
Saybolt Viscosity	D 88	Saybolt viscometer
Dilute Solution Viscosity of Ethylene Polymers	D 1601	Modified Ubbelohde viscometer
Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup	D 1200	Ford cup
Varnishes Used for Electrical Insulation	D 115	MacMichael viscosimeter

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