# Standard Test Method for Viscosity by Dip-Type Viscosity Cups<sup>1</sup>

This standard is issued under the fixed designation D 4212; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

# 1. Scope

- 1.1 This test method covers the determination of viscosity of paints, varnishes, lacquers, inks, and related liquid materials by dip-type viscosity cups. This test method is recommended for viscosity control work within one plant or laboratory and should be used to check compliance with specifications only when sufficient controls have been instituted to ensure adequate comparability of results.
- 1.2 Viscosity cups are designed for testing of Newtonian and near-Newtonian liquids. If the test material is non-Newtonian, for example, shear-thinning or thixotropic, another method, such as Test Methods D 2196, should be used. Under controlled conditions, comparisons of the viscosity of non-newtonian materials may be helpful, but viscosity determination methods using controlled shear rate or shear stress are preferred.
- 1.3 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:
- D 1200 Test Method for Viscosity by Ford Viscosity Cup<sup>2</sup> D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer<sup>2</sup>
- D 4287 Test Method for High Shear Viscosity Using the ICI Cone/Plate Viscometer<sup>2</sup>
- E 1 Specification for ASTM Thermometers<sup>3</sup>

#### 3. Terminology

- 3.1 Definitions:
- 3.1.1 *near-Newtonian liquid*—a liquid in which the variation of viscosity with shear rate is small and the effect on viscosity of mechanical disturbances such as stirring is negligible.

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3.1.2 *Newtonian liquid*—a liquid in which the viscosity is independent of the shear stress or shear rate. If the ratio of shear stress to shear rate is not constant, the liquid is non-Newtonian.

## 4. Summary of Test Method

4.1 The cup is completely immersed in the material to be tested, withdrawn, and the time for the material to flow through a hole in the base of the cup is measured.

#### 5. Significance and Use

- 5.1 Viscosity is a measure of the fluidity of a material. Viscosity data are useful in the determination of the ease of stirring, pumping, dip coating, or other flow-related properties of paints and related fluids.
- 5.2 This type of cup is used to measure viscosity because it is easy to use, robust, and may be used in tanks, reservoirs, and reactors.
- 5.3 There are other types of apparatus for measuring viscosity in the laboratory that provide better precision and bias, including the Ford viscosity cup (Test Method D 1200), and the Brookfield viscometer (Test Methods D 2196).
- 5.4 Certain higher shear rate devices such as cone/plate viscometers (Test Method D 4287) provide more information about sprayability, roll coatability, and other high-shear rate related properties of coatings.

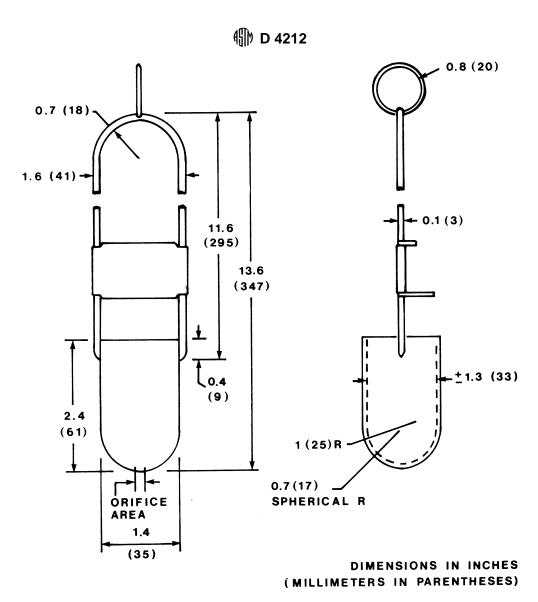
### 6. Apparatus

6.1 Zahn Viscosity Cup—No. 1 through No. 5 Zahn viscosity cups made of corrosion- and solvent-resistant materials. The nominal capacity of the cup is 44 mL, but may vary from 43 to 49 mL, depending on the manufacturer. A diagram of a Zahn cup is given in Fig. 1. The dimensions, including orifices, are only approximate because the cups are not made to a uniform specification. Each manufacturer produces a different cup and considerable variation between batches from some manufacturers has been noted in the past. This is a major reason why Zahn cups should not be referenced in specifications between producer and user only when controls sufficient to ensure adequate cup-to-cup and operator-to-operator comparison are included. (See Appendix X1 for additional information on Zahn Cups.)

Note 1—The various cup numbers are for identification of the viscosity ranges within the series only and should not be used for comparison between different kinds of cups, that is, a No. 2 Zahn cup has no

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 06.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 14.03.



Note 1—Dimensions are approximate only and may vary with the manufacturer and from batch to batch FIG. 1 Zahn Cup Nominal Dimensions

relationship whatsoever with a No. 2 Shell cup.

6.1.1 Nominal Zahn cup orifice diameters are listed in Table X2.1. Cup No. 1 with the smallest orifice is used for determining the viscosity of thin-bodied materials. Cup No. 2 is for use with clears, lacquers, enamels, and press-side adjustment of flexographic inks; cups Nos. 3 and 4 are for use with more viscous paints and inks (No. 3 for manufacturing of flexographic inks); and cup No. 5 is used for silk screen inks.

6.2 Shell Viscosity Cup<sup>4</sup>—No. 1 through No. 6 Shell viscosity cups made of stainless steel with a capacity of 23 mL and a 1-in. (25-mm) long capillary in the bottom and conforming to the dimensions shown in Fig. 2.

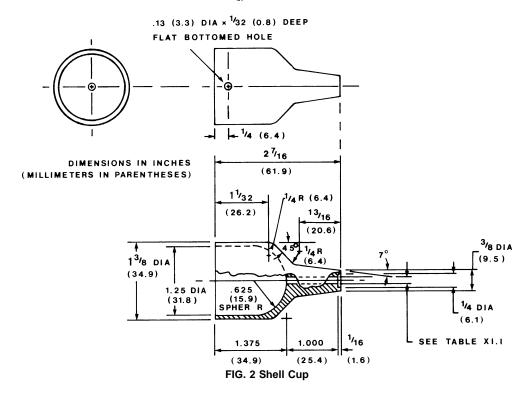
6.2.1 Nominal Shell cup orifice diameters are listed in Table X2.1. Cup Nos. 1 through 2½ are recommended for use with reduced rotogravure inks; No. 2 is for use with flexographic inks; Nos. 3 through 4 are used for industrial enamels, lacquers, flexographic, and gravure inks; Nos. 5 and 6 are used for heavy materials.

6.3 Calibration Thermometer—ASTM Saybolt Viscosity Thermometer 17F having a range of 66 to 80°F and subdivisions of 0.2°F, or 17C having a range of 19 to 27°C and subdivisions of 0.1°C, both conforming to the requirements of Specification E 1. Thermometers having subdivisions other than these may be used depending on the sensitivity of the material to be tested, the demands of the application, and the agreement between the purchaser and seller.

6.4 *Timer*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.1 s or better.

<sup>&</sup>lt;sup>4</sup> Shell cups may be obtained from the Norcross Corp., 255 Newtonville Ave., Newton, MA 02158. This committee is not aware of any other source for flow cups having properties similar enough to the Shell cup to be included in this test method. If you have knowledge of a cup that should be considered, please provide details to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, <sup>1</sup> which you may attend.





#### 7. Test Materials

7.1 The material to be tested should be visibly homogeneous and free from any foreign material or air bubbles.

# 8. Temperature of Testing

- 8.1 Measurements should be made at 77°F (25°C) unless otherwise specified. Temperature drift during the test should be kept to a minimum. The viscosities of paints and related materials are highly dependent on temperature. Differences in temperature between measurements can give substantially different viscosities (up to 5 % per °F). For careful work, the temperature should be taken in the efflux stream, but for process control (such as monitoring a dip tank), this is not necessary.
- 8.2 A temperature correction curve may be constructed for each liquid by plotting viscosity (seconds) against temperature over the expected temperature range. With this curve, a viscosity determined at one measured temperature may be converted quickly to a viscosity at another temperature.

Note 2—When dip cups are used for original purposes, that is thinning or monitoring of materials in tanks, coaters, etc., temperature is not important. This is because the key to good operation is to maintain the fluid within a certain range of dip cup-seconds regardless of the temperature of the fluid.

## 9. Checking and Calibration of Cups

- 9.1 Cups should be checked in accordance with the procedure described in Appendix X2. The frequency of this depends upon the amount of use and care that the individual cup receives, and the level of precision required.
- 9.2 Cups may be calibrated with standard fluids according to the procedure in Appendix X3. However, because the viscosity of standard fluids can vary significantly with tem-

perature and due to difficulty in obtaining adequate temperature control with dip cups, calibration is a difficult procedure that must be done with great care and knowledge.

#### 10. Procedure

10.1 Choose the proper cup so that the time of efflux will be between 20 and 80 s. See Table 1 for viscosity ranges for the various cups.

Note 3—The formulas used in this test method to describe the conversion from Zahn seconds to stokes are linear, the actual cup response is not. The range of 20 to 80 s covers the most linear portion of each cup. In addition, below 20 s, turbulent flow may cause additional inconsistencies. Above 80 s, factors that may impact on the precision include; loss of solvent (and therefore varying viscosity), "skinning" of the liquid in the cup, intermittent flow.

10.2 Immerse the cup in the container, which may be a can or beaker, but is more likely to be a thinning or mixing tank or even a resin reactor. Stir or agitate the fluid well to give uniform temperature and density. Allow the cup to remain in the fluid for 1 to 5 min to attain thermal equilibrium. (Because of their greater mass, Shell cups should remain in the fluid for the full 5 min.)

TABLE 1 Approximate Viscosity Ranges, cST (mm²/s) (Roughly Corresponding to 20 to 80 s Flow Time)

Cup Number	Zahn Cup	Shell Cup
1 <sup>A</sup>	5–60	2–20
2	20-250	10–50
21/2		20–80
3	100-800	30–120
31/2		40–170
4	200-1200	70–270
5	400-1800	125-520
6		320-1300

<sup>&</sup>lt;sup>A</sup>The lower limit for the Zahn No. 1 cup is 35 s rather than 20 s.



Note 4—Dip cups are not recommended for use with thixotropic (time dependent) materials but if used for them (such as gravure or flexographic inks), more vigorous agitation will be necessary to break up the structure before the measurement is made.

10.3 Lift the cup vertically out of the material in a quick, steady motion. As the top edge of the cup breaks the surface, start the timer. During the time of flow, hold the cup vertically no more than 15.2 cm (6 in.) above the level of the liquid. Stop the timer at the first definite break in the stream at the base of the cup. The efflux time in seconds constitutes the viscosity. It is common to make only a single measurement, but for greater precision and accuracy the mean of two or more measurements should be taken.

Note 5—The cup should not be held by the loop handle during the measurement process. Most manufacturers equip the cup with a ring through the loop handle. Holding the cup by this ring will help to ensure that the cup hangs vertically.

## 11. Care of Cups

11.1 Following each determination, clean the cup with a suitable solvent and a soft brush. Use no metal tools in contact with the instrument as nicks or wear of the drilled orifice affect the accuracy of the cup.

## 12. Report

12.1 Report the efflux time to the nearest 0.2 s for Zahn or Shell cup No. \_\_\_\_, manufactured by \_\_\_\_\_, (in the case of Zahn cups) the temperature of the fluid (where measured), and whether the result is from a single measurement or the mean of two of more measurements.

#### 13. Precision and Bias

- 13.1 The most satisfactory results when using dip cups are obtained when viscosity is being controlled at a single location only. However, when comparisons between locations are made, cups from the same manufacturer must be used or other action taken to ensure compatibility of results. The following criteria can be used for judging the acceptability of results at the 95 % confidence level:
- 13.1.1 Zahn Cups—Precision was determined on the basis of an interlaboratory test in which six laboratories used new Zahn cups (all from the same set from the same manufacturer) to test eight paints covering a broad range of viscosities. The within-laboratory coefficient of variation was 3.7 % and the

between-laboratories coefficient of variation was 11.5 %. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

- 13.1.1.1 *Repeatability*—Two results, each the mean of two measurements, obtained by the same operator should be considered suspect if they differ by more than 11 % of their mean value.
- 13.1.1.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 33 % of their mean value.

Note 6—The values used to determine the precision were obtained under ideal conditions (a single set of cups), reproducibility in practice can be just as good, by employing strict controls and good techniques.

13.1.1.3 *Bias*—Bias does not apply to this test method as no acceptable standards exist.

Note 7—Since the precision values were obtained under ideal conditions (a single set of cups), reproducibility in practice probably is poorer than that given (perhaps as bad as  $50\,\%$ ).

- 13.1.2 Shell Cups—Precision was determined on the basis of an interlaboratory test in which four laboratories tested seven paints covering a broad range of viscosities. The within-laboratory coefficient of variation was 3.2 % and the between-laboratories coefficient of variation was 6.3 %. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:
- 13.1.2.1 *Repeatability*—Two results, each the mean of two measurements, obtained by the same operator should be considered suspect if they differ by more than 9 % of their mean value.
- 13.1.2.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 18 % of their mean value.
- 13.1.2.3 *Bias*—Bias does not apply to this test method as no acceptable standards exist.

## 14. Keywords

14.1 dip cup(s); flow cup(s); Shell cup(s); viscosity; Zahn cup(s)



#### **APPENDIXES**

(Nonmandatory Information)

## X1. ZAHN CUP DESIGN, MANUFACTURE, AND USE

X1.1 Zahn cups were designed (and manufactured by General Electric) as simple flow property devices for use in dip tanks, flow coat reservoirs, etc. They were not designed to be viscometers or to be used as such. Zahn cup use usually involves thinning or maintaining a coating, adhesive, or ink at a certain consistency (so many Zahn-seconds) regardless of temperature. This is the beauty of dip cups. The operator knows that his or her tank, bath, or coater runs well over a certain range of Zahn-seconds whether the plant temperature is 50°F or 100°F. The operator does whatever is necessary to keep the fluid within the range. In such an application the viscosity at 25.0°C (77.0°F) is not important.

X1.2 Zahn cup patents have expired and Zahn-type cups are available from several sources. Each manufacturer makes cups that are somewhat different from those made by others. Considerable batch-to-batch difference has been noted in the past from some manufacturers. Cup variations are not a problem for consistent control of a bath or tank as long as appropriate comparisons are made with old cups, if required, to maintain continuity of data. However, cup differences can cause great difficulty if cups are used to set producer-user specifications. Comparisons under such conditions require considerable attention to detail and practiced expertise.

#### X2. DIP CUP CHECKING PROCEDURES

X2.1 The viscosity versus efflux time formulas published here are in wide use, however, not all Zahn-type cups are designed to comply with these formulas. Techniques for ensuring continued calibration of cups that do not claim compliance with these formulas include; comparison of current flow times for a standard fluid against the original flow time, and comparison against a verified standard cup. The same calibration verification methods of comparing a cup against one in known condition applied to Zahn-type cups can be used with Shell cups.

X2.2 Monitoring Cup Characteristics—A useful checking technique is to measure the efflux time for a new cup with a given standard fluid, then check the cup periodically with the same oil at the same temperature to determine if the efflux time has changed. If the time changes more than 20 % (or more than your process tolerance permits), the cup should be replaced. If the change is small, it may be appropriate to use the cup as is, or by applying a correction factor to subsequent efflux times, depending on the degree of precision required. The correction factor is equal to original standard—fluid efflux time divided by the current one. Recommended viscosities for standard fluids for such tests are given in Table X2.1.

X2.3 The viscosity of many standard fluids is very sensitive to temperature variations, therefore, the temperature of the fluid must be controlled closely during calibration testing. It is recommended that the fluid (and the cup) be held at the test temperature for at least 15 min prior to testing.

Note X2.1—Silicone fluid viscosity standards are not recommended. Although they exhibit very little viscosity change with temperature, they

TABLE X2.1 Viscosity Standards Recommended for Checking

Cups				
Cup Number	Nominal Orifice Diameter, (mm)	Approximate Oil Viscosity at 77°F (25°C), cST (mm²/s)		
Zahn				
1	2.0	20		
2	2.7	120		
3	3.8	480		
4	4.3	480		
5	5.3	900, 1600		
Shell				
1	1.8	9		
2	2.4	9, 20		
21/2	2.7	35		
3	3.1	35, 120		
31/2	3.5	120		
4	3.8	120		
5	4.6	120, 480		
6	5.8	480		

have a tendency to contaminate cups and containers and to change the drainage characteristics of cups (especially cups with capillaries).

X2.4 Comparing Cups—It sometimes is necessary to compare one Zahn-type cup with another in order to settle a dispute, determine whether a new cup will give similar times to an old one, etc. The most usual technique is to dip both cups into the same container of standard fluid at the same time and once the temperature of both cups has stabilized, pull them out together, timing both of them. The efflux times are compared and a correction factor can be calculated. Sometimes paint or another material is substituted for the oil.

#### X3. CALIBRATION OF DIP CUPS

X3.1 Select the appropriate standard oil for the cup to be checked (see Table X2.1).

X3.2 Bring the cup and the standard fluid to a constant temperature as close as possible to  $77.0^{\circ}F$  ( $25.0^{\circ}C$ ). Some fluids sold as viscosity standards can vary in viscosity by 2 to 18 % per degree centigrade, higher viscosities typically having the higher rate of change. Determine the time of efflux to the nearest 0.2 s using the procedure detailed in Section 10. Record the temperature of the efflux stream. If it is not  $77^{\circ}F$ , correct the viscosity of the standard fluid to the actual temperature. Temperature versus viscosity data is available from most suppliers of viscosity standards.

X3.3 Convert the time of flow in seconds to kinematic viscosity as follows:

$$V = K(t - c) \tag{X3.1}$$

where:

 $V = \text{kinematic viscosity, cST (mm}^2/\text{s})$ 

t = efflux time, s, and,

K, c = appropriate constants (from Table X3.1).

X3.3.1 These equations represent linear or relatively linear portions of the overall viscosity-time curves for the cups. The linear equations have been chosen because it is much more straightforward to make time corrections based on linear equations than on nonlinear ones and they are adequate for most applications. Zahn cup nonlinear equations that better fit viscosity curves for some cups better may be found in the

TABLE X3.1 Constants<sup>A</sup> for Use with Viscosity Formulas

Cup	К	С	
Zahn			
1	1.1	29	
2	3.5	14	
3	11.7	7.5	
4	14.8	5	
5	23	0	
Shell			
1	0.226	13	
2	0.576	5	
21/2	0.925	3	
3	1.51	2	
31/2	2.17	1.5	
4	3.45	1	
5	6.5	1	
6	16.2	0.5	

<sup>A</sup>Cup constants from Patton, T. C., *Paint Flow and Pigment Dispersion*, second edition, John Wiley & Sons, New York, 1979, p. 82.

literature. <sup>5,6</sup> No equations for Shell cups have been published.

X3.4 Calculate the correction factor by dividing the true kinematic viscosity of the standard fluid by the kinematic viscosity calculated from the efflux time. This factor may then be used to correct viscosity readings taken with the cup. The product of the factor and an efflux time gives a corrected viscosity in Zahn or Shell seconds.

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<sup>&</sup>lt;sup>5</sup> Euverard, M., ASTM Bulletin, Vol 162, No. 67, October 1950.

<sup>&</sup>lt;sup>6</sup> Pierce, P. E., Journal of Paint Technology, Vol 42, No. 533, 1969, p. 383.