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.



Designation: D4212 – 16 (Reapproved 2023)

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

This standard is issued under the fixed designation D4212; 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 ( $\varepsilon$ ) 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 D2196, should be used. Under controlled conditions, comparisons of the viscosity of nonnewtonian materials may be helpful, but viscosity determination methods using controlled shear rate or shear stress are preferred.

1.3 The values stated in SI 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, 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:<sup>2</sup>

D1200 Test Method for Viscosity by Ford Viscosity Cup

- D2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer
- D4287 Test Method for High-Shear Viscosity Using a Cone/ Plate Viscometer
- E1 Specification for ASTM Liquid-in-Glass Thermometers

# 3. Terminology

3.1 Definitions:

3.1.1 *near-Newtonian liquid*, *n*—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.

3.1.2 *Newtonian liquid*, *n*—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 D1200), and the rotational viscometer (Test Methods D2196).

5.4 Certain higher shear rate devices such as cone/plate viscometers (Test Method D4287) 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.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints & Paint Materials.

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<sup>&</sup>lt;sup>2</sup> 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.



The nominal capacity of the cup is 44 mL, but may vary from 43 mL 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 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  $Cup^3$ —No. 1 through No. 6 Shell viscosity cups made of stainless steel with a capacity of 23 mL and a 25 mm (1 in.) 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 2<sup>1</sup>/<sub>2</sub> 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.

<sup>&</sup>lt;sup>3</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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.



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





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6.3 Calibration Thermometer—ASTM Saybolt Viscosity Thermometer 17F having a range of 66 °F to 80 °F and subdivisions of 0.2 °F, or 17C having a range of 19 °C to 27 °C and subdivisions of 0.1 °C, both conforming to the requirements of Specification E1. 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. In addition, temperature measuring devices such as non-mercury liquid-inglass thermometers, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range for thermometer 17C and 17F, may be used.

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.

#### 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 25 °C (77 °F) 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 temperature 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 s 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 s 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

TABLE 1 Approximate	<b>Viscosity Ranges</b>	s, cST (mm <sup>2</sup> /s) (Roughly	
Corresponding to 20 s to 80 s Flow Time)			

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Cup Number	Zahn Cup	Shell Cup
1^	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>A</sup> The lower limit for the Zahn No. 1 cup is 35 s rather than 20 s.

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 min to 5 min to attain thermal equilibrium. (Because of their greater mass, Shell cups should remain in the fluid for the full 5 min.)

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)