Designation: D1343 – 95 (Reapproved 2011)

Standard Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method[1]

This standard is issued under the fixed designation D1343; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method describes the apparatus and general procedure for making ball-drop viscosity measurements on solutions of various cellulose derivatives. Instructions for sample preparation, solution concentration, and other details are discussed in the ASTM methods for the respective cellulose derivatives.

1.2 This test method is applicable to solutions of various cellulose derivatives having viscosities greater than 10 P, by using balls of various diameters and densities. Viscosity results are expressed preferably in poises.

1.3 In commercial practice, viscosities are often expressed in seconds using 2.38-mm (3⁄32-in.) stainless steel balls.[2] When the viscosity is outside the practical range for these balls (75 to 300 P), the measurement can be made using a calibrated pipet viscometer or a different ball and calculating the observed viscosity to the corresponding time for a 2.38-mm (3⁄32-in.) ball, even though it is a small fraction of a second.

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

1.5 *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:*[3]

D301 Test Methods for Soluble Cellulose Nitrate (Withdrawn 2011)[4]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D817 Test Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate

D871 Test Methods of Testing Cellulose Acetate

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 A solution of the cellulose derivative is made in a suitable solvent and allowed to equilibrate at a chosen temperature. A stainless steel or aluminum ball is dropped into the solution, and the time required for it to cover a measured distance in its fall is recorded. The viscosity of the solution can then be calculated in poise or recorded in seconds.

NOTE 1—The choice of solvent has significant influence on viscosity.

4. Significance and Use

4.1 This test provides an easy method of determining the viscosity of cellulose derivatives in a given solvent. The answers are in units commonly used in industrial practice. Such information is needed for cellulose derivatives that are to be extruded, molded, sprayed, or brushed as is or in solution.

5. Apparatus

5.1 *Constant-Temperature Water Bath,* glass-walled.

5.1.1 For routine testing, an aquarium viscometer is recommended. This viscometer is a rectangular glass enclosure with front and rear walls that have etched horizontal parallel lines 50.8 mm (2.00 ± 0.02 in.) apart. The bottles containing the

[1] 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 D01.36 on Cellulose and Cellulose Derivatives.

Current edition approved June 1, 2011. Published June 2011. Originally approved in 1954. Last previous edition approved in 2006 as D1343 – 95 (2006). DOI: 10.1520/D1343-95R11.

[2] When a 3⁄32-in. stainless steel ball is used, the viscosities in seconds should be practically the same as those obtained using the apparatus described in Section 11 of Test Methods D871 – 48, and in Section 10 of Test Methods D301 – 50, which last appeared in the *1952 Annual Book of ASTM Standards*, Part 4.

[3] 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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

TABLE 1 Bottles

Bottle	Round	Square
Capacity, oz	16	16
Weight, oz	12	12
Height, in.	6.7	7
Inside diameter, cm	6.4	
Side to side, cm	...	6.0
Corner to corner, cm	...	7.2

samples solutions are set inside the viscometer at a level such that the upper etched line of the viscometer is at least 12.7 mm (½ in.) below the upper surface of the solution in the bottle, and the lower etched line of the viscometer is at least 12.7 mm (½ in.) above the bottom of the sample bottle. Suitable lighting is provided to enable the observer to sight across the parallel etched lines, through the sample bottle and solution, avoiding in this manner parallax errors. With this viscometer no timing markers need to be provided on the bottles.

5.2 *Bottles and Caps:*

5.2.1 Bottles, round or square, conforming to the dimensional requirements shown in Table 1, shall be used. Screw caps of metal or phenolic plastic in sizes to fit the bottles and having aluminum foil or cardboard and cellophane liners may be used to close the bottles. Alternatively, rubber stoppers covered with aluminum or tin foil, may also be used as closures. In this latter case, solvent loss during measurement of viscosity can be minimized by removing the stopper, leaving the foil in place, and making a small hole in the center of the foil through which the balls may be dropped.

5.2.2 Timing marks shall be provided around each bottle or on the front and back of the glass-walled constant-temperature water bath, to avoid parallax errors. The lower timing mark shall be at least 13 mm (0.5 in.) above the base of the bottle, and the upper mark shall be 50.8 ± 0.5 mm (2.00 ± 0.02 in.) above the lower mark. A practical means of marking consists of wrapping a 50.8-mm (2-in.) strip of transparent sheeting around the water bath at the proper location. The edges of the sheeting may be darkened with crayon. A light located back of the water bath aids in observing the ball during its fall.

5.3 *Balls*—Unless specifically directed otherwise, balls of varying size and density shall be used, depending on the viscosity of the solution. Table 2 gives the useful ranges, approximate apparatus constants, and dimensions of several such balls. The exact diameter, weight, and density shall be determined accurately for each lot of balls used.

5.4 *Stop Watch*—A stop watch reading to 0.2 s.

6. Calibration

6.1 Calculate the apparatus constant, K, using the following equation and exact dimensions of the bottle and balls used:

$$K = 2gr^2[1 - 2.104(d/D) + 2.09(d/D)^3]/9L$$

where:

g = acceleration of gravity in cgs units
r = ball radius, cm,
d = ball diameter, cm,
D = bottle diameter, cm (in the case of square bottles the average of the side to side and corner to corner diameters shall be used), and
L = distance of ball drop, cm.

7. Procedure

7.1 *Preparation of Solution*—Dry the sample and prepare a solution as specified for the particular material. Such instructions are given in the viscosity sections of Test Methods D301, D871, and D817. Weigh into the bottle an appropriate amount of dry sample and specified solvent, accurate to 0.1 g, to make about 350 mL of solution. The accurate and precise make up of the solution is a necessity (example: 60.00 g of cellulose acetate and 240.00 g solvent). Close the bottle tightly. Allow to stand a short time for the solvent to penetrate the sample. Then tumble or shake until a uniform solution is obtained. For some samples this may require several days. Transfer to the water bath at 25 ± 0.1°C, and allow the solution to come to temperature. A practical method to determine possible solvent loss during this time involves weighing the bottle immediately after adding the components, and again before performing the ball drop.

7.2 *Viscosity Determination*—Drop a 2.38-mm (3⁄32-in.) stainless steel ball through the center of the column of solution and time its fall through the marked 50.8-mm (2-in.) distance, using a stop watch and taking precautions to avoid parallax errors. If the observed time is less than 15 s or greater than 100 s repeat the measurement, unless directed otherwise, using a different ball (see Table 2) which has a time of fall within these limits. If the solution is known to be thixotropic in nature or if the times of fall for successive balls vary significantly, use freshly prepared solutions for duplicate measurements or measurements with balls of other sizes.

7.3 *Determination of Lower Viscosities*—If the viscosity of the solution is too low to measure satisfactorily using one of the balls, use a calibrated pipet as described in Test Method

TABLE 2 Balls

Ball	Viscosity Range, P	Typical Data			
		Apparatus Constant, K	Diameter, cm	Weight, g	Density, g/cm^3, *a*
1.59-mm (1⁄16-in.) (aluminum)	10 to 50	0.256	0.1588	0.00591	2.82
1.59-mm (1⁄16-in.) (stainless steel)	35 to 150	0.256	0.1588	0.01605	7.66
2.38-mm (3⁄32-in.)	75 to 300	0.560	0.2380	0.0542	7.68
3.18-mm (⅛-in.)	125 to 600	0.965	0.3170	0.1277	7.68
5.56-mm (7⁄32-in.)	350 to 1800	2.70	0.5556	0.6897	7.68