

Designation: D7394 - 18 (Reapproved 2023)

# Standard Practice for Rheological Characterization of Architectural Coatings using Three Rotational Bench Viscometers<sup>1</sup>

This standard is issued under the fixed designation D7394; 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 practice describes a popular industry protocol for the rheological characterization of waterborne architectural coatings using three commonly used rotational bench viscometers. Each viscometer operates in a different shear rate regime for determination of coating viscosity at low shear rate, mid shear rate, and at high shear rate respectively as defined herein. General guidelines are provided for predicting some coating performance properties from the viscosity measurements made. With appropriate correlations and subsequent modification of the performance guidelines, this practice has potential for characterization of other types of aqueous and non-aqueous coatings.

1.2 The values in common viscosity units (Krebs Units, KU and Poise, P) are to be regarded as standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.4 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>

D562 Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer D869 Test Method for Evaluating Degree of Settling of Paint D1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers

- D1200 Test Method for Viscosity by Ford Viscosity Cup
- D2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer
- D2805 Test Method for Hiding Power of Paints by Reflectometry
- D4040 Test Method for Rheological Properties of Paste Printing and Vehicles by the Falling-Rod Viscometer
- D4062 Test Method for Leveling of Paints by Draw-Down Method
- D4287 Test Method for High-Shear Viscosity Using a Cone/ Plate Viscometer
- D4400 Test Method for Sag Resistance of Paints Using a Multinotch Applicator
- D4414 Practice for Measurement of Wet Film Thickness by Notch Gages
- D4958 Test Method for Comparison of the Brush Drag of Latex Paints

# 3. Terminology

3.1 Definitions:

3.1.1 *coating rheology, n*—the viscosity profile obtained for a fluid coating over a range of shear rates.

3.1.2 *high-shear viscosity (HSV), n*—the viscosity of a fluid coating at high shear rate (typically measured at  $10,000 \text{ s}^{-1}$  or  $12,000 \text{ s}^{-1}$ ), and for architectural coatings, it is often referred to as the "brush-drag" viscosity.

3.1.3 *leveling*, *n*—the ability of a wet coating to flow out to a smooth dry film after application, thereby minimizing or eliminating coating surface irregularities that occur during brushing, rolling or spraying (see also Test Method D4062).

3.1.4 *low-shear viscosity (LSV)*, *n*—the viscosity of a coating fluid at low shear rate (typically in the range of 0.001 s<sup>-1</sup> to  $1 \text{ s}^{-1}$ ), often referred to as the "leveling viscosity" or inversely as the "suspension viscosity."

3.1.5 *mid-shear thickener efficiency (MSTE), n*—the weight of active thickener per unit volume of wet coating required to give the target MSV, commonly expressed as lb active thickener/100 gal wet coating (or in g/L units).

<sup>&</sup>lt;sup>1</sup> This practice 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.

3.1.6 *mid-shear viscosity (MSV)*, *n*—the viscosity of a coating fluid at medium shear rate (typically in the range of 10 s<sup>-1</sup> to 1000 s<sup>-1</sup>), often referred to as the "consistency" or the "mixing viscosity."

3.1.7 *newtonian*, n—a rheological term describing a fluid that maintains constant viscosity over a range of shear rates (see also Test Method D1200 and Test Method D4040).

3.1.8 *rheometer*, *n*—an instrument capable of continuously measuring fluid viscosity over a range of shear rates or shear stresses, often capable of other types of rheological determinations, and ideally suited for research and well-defined characterization of fluid rheology.

3.1.9 *rotational viscometer*, *n*—an instrument that uses one or more turning surfaces in contact with a fluid to measure the fluid's viscosity, is capable of operating at one or more rotational speeds to provide different shear rates, is typically limited to one speed per measurement, is relatively simple to operate and ideally suited for quality control or routine lab determinations.

3.1.10 *settling*, n—the gradual sedimentation of pigment or other disperse phase particles, or both, that may occur during storage of a coating (see also Test Method D869).

3.1.11 *shear rate, n*—the change in velocity of a fluid per unit gap between shearing surfaces.

3.1.12 suspension, n—as defined in this practice, a coating formulation's ability to suspend pigment and other disperse phase particles, thereby inhibiting or preventing settling or syneresis, or both.

3.1.13 *syneresis, n*—the separation of a clear liquid layer at the top of coating in a container that may occur during storage.

3.1.14 *thixotropy*, *n*—a rheological term describing a nonnewtonian fluid that decreases in viscosity with time at a given shear rate, and then rebuilds viscosity with time when the shearing stops (see also Test Methods D2196).

# 4. Summary of Practice

4.1 This practice involves characterization of architectural coating rheology by measuring viscosity with three rotational bench viscometers to obtain low-shear viscosity (LSV), midshear viscosity (MSV) and high-shear viscosity (HSV), respectively. LSV is obtained with a cylindrical- or disc-type spindle viscometer operating at a low speed (at either 0.5 r/min (rpm) or preferably 0.3 r/min (rpm)). The applicable shear rate for this viscometer/speed combination is in the range of  $0.01 \text{ s}^{-1}$  to 1 s<sup>-1</sup>. The MSV or coating consistency is obtained using an analog or digital rotational paddle-type viscometer that measures viscosity in Krebs Units (KU). The applicable shear rate for this instrument is in the range of 10 s<sup>-1</sup> to 200 s<sup>-1</sup> for most architectural paints. The high-shear viscosity is obtained using a cone/plate-type viscometer with a fixed shear rate of either 10,000 s<sup>-1</sup> or 12,000 s<sup>-1</sup>. If coatings are to be characterized without any viscosity adjustments being made, measurements with the three viscometers can be conducted in any order. However, if a series of paints is being compared where it is desirable to have one of the three viscosities a constant, viscosity adjustments may be needed to achieve that. For example, it is quite common to have a specification for the Krebs Unit viscosity in architectural coatings. In this case, MSV would be the first viscosity measurement made, and any coatings out of specification would be adjusted (usually with the amount of thickener) to obtain the same or similar Krebs viscosity. With the Krebs viscosity constant, meaningful comparisons between coatings can then be made in the extreme shear rate regimes for LSV and HSV where many coatings properties are affected.

#### 5. Significance and Use

5.1 A significant feature of this practice is the ability to survey coating rheology over a broad range of shear rates with the same bench viscometers and test protocol that paint formulators and paint quality control (QC) analysts routinely use. By using this procedure, measurement of the shear rheology of a coating is possible without using an expensive laboratory rheometer, and performance predictions can be made based on those measurements.

5.2 Low-Shear Viscosity (LSV)—The determination of lowshear viscosity in this practice can be used to predict the relative "in-can" performance of coatings for their ability to suspend pigment or prevent syneresis, or both. The LSV can also predict relative performance for leveling and sag resistance after application by roll, brush or spray. Fig. 1 shows the predictive low-shear viscosity relationships for several coatings properties.

**5.3** *Mid-Shear Viscosity (MSV)*—The determination of MSV (coating consistency) in this practice is often the first viscosity obtained. This viscosity reflects the coatings resistance to flow on mixing, pouring, pumping, or hand stirring. Architectural coatings nearly always have a target specification for mid-shear viscosity, which is usually obtained by adjusting the level of thickener in the coating. Consequently, mid-shear viscosity is ideally a constant for a given series of coatings being tested to provide meaningful comparisons of low-shear and high-shear viscosity. With viscosities at the same KU value, MSV can also be used to obtain the relative Mid-Shear Thickener Efficiency (MSTE) of different thickeners in the same coating expressed as lb thickener/100 gal wet coating or g thickener/L wet coating.

5.4 *High-Shear Viscosity (HSV)*—High-shear viscosity in this practice is a measure of the coatings resistance to flow on application by brush or roller, which is often referred to as brush-drag or rolling resistance respectively. This viscosity

I	Effect of Low She	ar Viscosity (LSV)
Coating Property	Low	High
Flow and Leveling Pigment Suspension Syneresis Control Sag Resistance	Improving	
		Improving
		Improving
		Improving

Direction of Property Change

FIG. 1 Low Shear Viscosity (LSV)

relates to the coatings ability to provide one-coat hiding, its ease of application (brushing or rolling resistance), and its spread rate. Fig. 2 shows high-shear viscosity relationship predictions for relative coating performance.

#### 6. Reagents

6.1 *Viscosity Standards*—optional, for checking the accuracy of each of the three viscometers used in this practice.

# 7. Apparatus and Equipment

7.1 *Spatula or Lab Stirrer*—optional, for mixing coating samples prior to viscosity measurements.

7.2 Rotational Viscometer—with cylindrical- or disc-type spindle and torque constant between 65  $\mu$ N-m and 720  $\mu$ N-m to measure the low-shear viscosity of a coating at a low rotational speed of 0.3 r/min (rpm) standard, or 0.5 r/min (rpm) optional).

7.3 *Paddle-Type Rotational Viscometer*—digital or analog instrument to measure the mid-shear viscosity of the coating in Krebs Units (KU).

7.4 *Cone/Plate-Type Viscometer*—to measure the high-shear viscosity of the coating at a fixed shear rate of  $10,000 \text{ s}^{-1}$  or  $12,000 \text{ s}^{-1}$ , depending on whether the electrical system is 50 Hz or 60 Hz.

7.5 Thermometer (ASTM 49C or equivalent of 0.1 °C accuracy per Test Method D562 or Test Methods D2196)—to record and adjust the coating sample temperature.

7.6 *Leveling Draw-Down Blade*—optional, to determine the relative leveling of coatings for comparison and correlation with low-shear viscosity measurements.

7.7 *Sag Bar*—optional, to determine the sag resistance of coatings for comparison and correlation with low-shear viscos-ity measurements.

7.8 *Paint Brush*—optional, for brushing out paints for relative brush drag, wet film thickness, hiding power, and leveling of brush marks for comparison and correlation with low-shear and high-shear viscosity measurements.

7.9 *Paint Roller*—optional, for rolling out paints for relative rolling resistance and for measuring wet film thickness, hiding power, and leveling of roller tracking marks for comparison and correlation with low-shear and high-shear viscosity measurements.

# 8. Procedure

8.1 Background and Testing Protocol:

#### Effect of High Shear Viscosity (HSV)

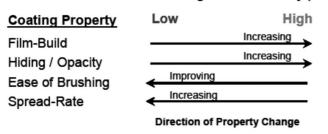


FIG. 2 High Shear Viscosity (HSV)

8.1.1 A common practice in many architectural coatings labs is the examination of coating viscosity in three shear rate regimes using three different rotational bench viscometers: a cylindrical- or disc-type spindle viscometer at low rotational speed for low-shear viscosity (LSV), a fixed speed paddle-type viscometer for mid-shear viscosity (MSV), and a cone/platetype viscometer for high-shear viscosity (HSV). This test protocol is described briefly in the ASTM Paint and Coatings Testing Manual<sup>3</sup> and in more detail in the Handbook of Coatings Additives.<sup>4</sup> Although controlled shear rate and controlled shear stress rheometers do provide more complete coating rheology profiles, have well defined shear rates and shear stresses, are often more accurate in their measurements, and can provide other rheological information such as elastic properties etc., many routine decisions about relative coating rheology performance predictions are made using the test protocol of this practice.

8.1.2 The mid-shear Krebs Unit viscosity is a primary specification for nearly all architectural coatings. Consequently this is usually the first viscosity measurement made. If the Krebs Unit viscosity is not on target, a common practice is to adjust thickener level to bring the coating into the correct KU specification range. Krebs Unit viscosity specifications for architectural coatings can range from about 70 KU to 120 KU  $\pm$  3 KU, depending on the type of coating and application. For a typical house paint with a midpoint specification of 100 KU viscosity, the specification range would be  $\pm 3$  %. Since MSV is often a primary specification, a series of coatings being compared for rheology will often have the same or similar KU viscosity, and this is actually advantageous and important for meaningful comparisons of LSV and HSV. The reason for this is that an increase in MSV for a coating will result in a corresponding increase in its LSV and HSV. If the coatings do not have the same MSV, viscosity comparisons at low and high shear cannot be made on an equal basis.

8.1.3 In some protocols, LSV or HSV can be a primary specification for a coating with MSV having secondary priority. In those instances, LSV or HSV are adjusted to constant value with thickeners or rheology modifiers, or both, and the other two viscosities are then determined for comparison

8.2 Adjustment of the Mid-Shear Rate Krebs Unit Viscosity:

8.2.1 A first step based on the preferred test protocol outlined above in this practice is the determination of the Krebs viscosity (or just KU viscosity) of the coating using a paddle-type analog or digital viscometer that measures viscosity in Krebs Units. Test Method D562 is the test method recommended for this determination. All setup and operational criteria should be followed. As some paints are thixotropic, it is a good practice to pre-stir the paint with a spatula or lab mixer to break up any structure prior to making a viscosity measurement. If the mid-shear Krebs viscosity is a specification or is a fixed value for the paints being tested, the Krebs

<sup>&</sup>lt;sup>3</sup> ASTM Paint and Coatings Testing Manual, 14th Edition of the Gardner-Sward Handbook, ASTM Manual Series MNL 17, Joseph V. Koleske, Editor, Chapter 30, "Thickeners and Rheology Modifiers," G. D. Shay, pp. 268–285, June 1995.

<sup>&</sup>lt;sup>4</sup> *Handbook of Coatings Additives*, Second Edition, J. J. Florio and D. J. Miller Editors, Chapter 12, Rheology Modifiers for Waterborne Coatings: Formulating, G. D. Shay and M. C. Kaufman, pp. 405–467, 2004.