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### 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 covers 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 and health practices and determine the applicability of regulatory limitations prior to use.

#### 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 or 12,000  $\sec s^{-1}$ ), and for architectural coatings, it is often referred to as the "ICI" or "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).

\*A Summary of Changes section appears at the end of this standard

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<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 and 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'sstandard's Document Summary page on the ASTM website.

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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 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).

3.1.6 *mid-shear viscosity (MSV), n*—the viscosity of a coating fluid at medium shear rate (typically in the range of 10 to 1000  $s^{-1}$ ), 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 non-newtonian 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), mid-shear viscosity (MSV) and high-shear viscosity (HSV), respectively. LSV is obtained with a Brookfield-type cylindrical- or disc-type spindle viscometer operating at its lowesta low speed (at either 0.5 or preferably 0.3 rpm).r/min). The applicable shear rate for this viscometer/speed combination is in the range of 0.01 to  $1 \text{ s}^{-1}$ . 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 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 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  $\frac{QC}{QC}$  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 low-shear 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.

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### Effect of Low Shear 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)

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 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 *Brookfield-Type*<u>Rotational</u> Viscometer—with cylindrical- or disc-type spindle and torque constant between 65 and 720 μN-m to measure the low-shear viscosity of a coating at the lowest instrument rpm (Brookfield LVT at 0.3 rpm is standard, LVT at 0.5 rpm is a low rotational speed of 0.3 r/min standard, or 0.5 r/min 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 or 12,000  $s^{-1}$ , depending on whether the electrical system is 50 or 60 Hz.

7.5 *Thermometer (ASTM 49C or equivalent of 0.1C 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 viscosity 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:

Coating Property	Low	High
Film-Build		Increasing
Hiding / Opacity		Increasing
Ease of Brushing	Improving	
Spread-Rate	Increasing	
opread-rate	•	

### Effect of High Shear Viscosity (HSV)

Direction of Property Change

FIG. 2 High Shear Viscosity (HSV)