Designation: D7394 – 17

# Standard Practice for Rheological Characterization of Architectural Coatings using Three Rotational Bench Viscometers[1]

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 (ε) 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:*[2]

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 $s^{-1}$), 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 to 1 $s^{-1}$), 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."

[1] 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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[2] 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.

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

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 cylindrical- or disc-type spindle viscometer operating at a low speed (at either 0.5 or preferably 0.3 r/min). The applicable shear rate for this viscometer/speed combination is in the range of 0.01 to 1 $s^{-1}$. 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 $s^{-1}$ 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 $s^{-1}$. 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 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.

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.

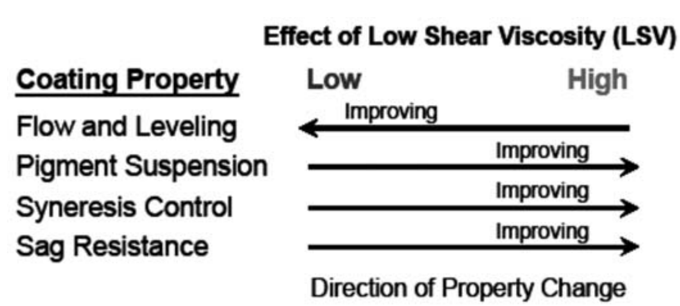


FIG. 1 Low Shear Viscosity (LSV)

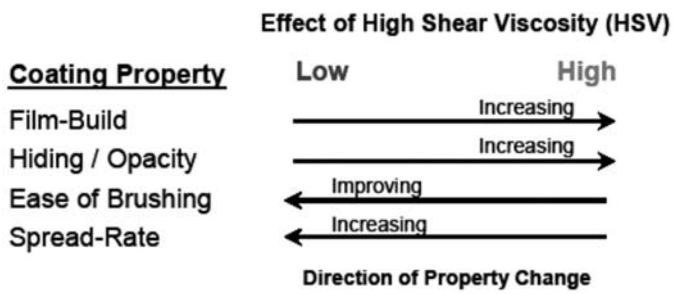


FIG. 2 High Shear Viscosity (HSV)

## 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 and 720 µN-m to measure the low-shear viscosity of a coating at 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:*

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/plate-type viscometer for high-shear viscosity (HSV). This test protocol is described briefly in the ASTM Paint and Coatings Testing Manual[3] and in more detail in the Handbook of Coatings Additives.[4] 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 to 120 KU ± 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 ±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 viscosity of each paint should be measured followed by appropriate thickener adjustments to obtain the same or similar KU before proceeding to obtain LSV or HSV. Sometimes paints have to be remade if the thickener amount is too high. Adjustment of the paints being tested to the same Krebs Unit

[3] *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.

[4] *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.

viscosity is optional, but it is highly recommended for meaningful comparison of the effects of coating variables on LSV and HSV.

8.3 *Running the Low-Shear Viscosity Test:*

8.3.1 After obtaining the MSV with a Krebs Unit viscometer and preferably after adjusting the KU to a constant value for the series of paints to be tested, the LSV is then obtained on each coating using Test Methods D2196 which is a test method for cylindrical- or disc-type spindle-type viscosity determination. The only constraint within Test Methods D2196 is that the test is run at the rotational speed of 0.3 to 0.5 r/min. Prior to making the LSV measurement, the sample can be stirred with a spatula to break up structure if present. Ideally, the same viscometer spindle should be used for all paints in a series of coatings being measured, but this is not always possible, as some coatings may be out of the preferred torque range on the viscometer. A recommended practice is to select the spindle that gives a torque reading between 10 to 100 % of full scale for each coating tested. If that is not possible, a spindle change can be made, but the rotational speed should always be the same to keep the shear rate relatively constant. The spindle should turn in the sample for 60 s (or a longer fixed time period) before taking the reading, and the same time period should be used for all measurements. Centipoises (cP) readings are converted to poise (P) by dividing by 100. Table 1 provides a guideline for performance prediction for architectural latex paints from 0.3 r/min LSV determinations with mid-shear consistency of about 95 KU. Any variation from these parameters, for example, measurement at 0.5 r/min or at a different KU value, will shift the correlation. For more refined predictions, correlation studies should be conducted for the specific type of coatings being examined and at the KU level specified for the coating. For leveling and sag resistance, Test Methods D4062 and D4400 may be used respectively.

8.4 *Running the High-Shear Viscosity Test:*

8.4.1 The high-shear viscosity test is conducted using an analog dial scale or digital cone/plate-type viscometer per Test Method D4287, which covers the determination of the viscosities of paints and related products at a shear rate of 10,000 or 12,000 $s^{-1}$. The sampling procedure, apparatus preparation, and test procedure in Test Method D4287 should be followed as prescribed with the viscosity recorded in poise (P). Table 2 is an HSV guideline for typical architectural latex paints.

8.4.2 In the HSV table, B-D/F-B is an abbreviation for Brush-Drag/Film-Build which refers to the well-known relationship that increasing brush-drag results in increasing film-build. An increase in film build also naturally corresponds with a decrease in spread rate which is a measure of the amount of coating applied per unit area. For more refined HSV prediction capability, correlation studies should be conducted for the particular type of coating being examined and at the appropriate MSV. The following test methods are recommended for performance correlation with HSV. Film build can be determined by wet film thickness using Practice D4414 or by dry film thickness using Test Method D1005. If the area of coverage is known, spread rate can then be calculated. Hiding power by Test Method D2805 and subjecting brush-drag by Test Method D4958 are also directly related to film build.

**TABLE 1 LSV Guideline for Typical Architectural Latex Paints**

| LSV (Poise) | Coating Performance |
|---|---|
| <100 | will probably sag on a vertical surface |
| 100–150 | excellent leveling |
| 150–250 | very good leveling |
| 250–500 | good leveling |
| 500–900 | fair leveling |
| 900–1500 | poor leveling and some suspension |
| 1600–2400 | very poor leveling and good suspension |
| >2400 | no leveling very high suspension |

**TABLE 2 HSV Guideline for Typical Architectural Latex Paints**

| HSV (Poise) | Coating Performance |
|---|---|
| <0.5 | very low B-D/F-B, very high spread rate |
| 0.5–0.75 | low B-D/F-B, high spread rate |
| 0.75–1.0 | med B-D/F-B, moderate spread rate |
| 1.0–1.5 | med-high B-D/F-B, low spread rate |
| 1.5–2.0 | high B-D/F-B for one-coat coverage |
| 2.0–2.5 | very high B-D/F-B for high coat weight |
| >2.5 | excessive for brushing or rolling |

8.5 *A Practical Example for Assessing Thickener Performance using this Practice:*

8.5.1 This practice can be used to determine the effect of coating variables, (for example, thickeners, latex binders, coalescing agents, etc.) on coating rheology. One common example of this is determining the relative performance of different thickeners in the same paint formulation. The first step is the incorporation of each thickener at a level to give the same Krebs Unit coating viscosity. Water adjustments may be made to maintain constant coating solids. Once that is done, the mid-shear thickening efficiency of each thickener can be calculated from the solids content of each thickener to give lb active thickener/100 gal wet coating at the specified KU value. The next step is obtaining LSV with the spindle type viscometer at 0.3 or 0.5 r/min, and the HSV with the cone/plate-type viscometer. Paints with a combination of low LSV and high HSV are more Newtonian in rheology and predictably will exhibit excellent leveling and one-coat hiding performance. In contrast, paints with a combination of high LSV and low HSV would be very shear-thinning (pseudoplastic) giving poor flow but high spread-rate. Other properties can also be predicted based on the other guidelines provided in this practice.

## 9. Report

9.1 Report the following information:

9.1.1 Mid-shear Krebs viscosity of each paint in Krebs Units (KU),

9.1.2 Low-shear cylindrical- or disc-type spindle viscosity of each paint at 0.3 or 0.5 r/min,

9.1.3 High-shear cone/plate viscosity of each paint, and

9.1.4 Temperature of viscosity determinations.

## 10. Keywords

10.1 cone/plate-type viscometer; high-shear viscosity; Krebs unit viscometer; low-shear viscosity; mid-shear viscosity; paints; rheology; viscosity

## SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D7394-13) that may impact the use of this standard. (Approved February 1, 2017.)

*(1)* Changed the abbreviation "rpm" throughout the standard to the SI unit of "r/min."
*(2)* In sections 4.1, 7.2, 8.1.1, 8.3.1, 8.5.1, 9.1.2, and 10.1 changed from the trademarked brand name "Brookfield" to the technical equivalent "cylindrical- or disc-type spindle."