Designation: D 6606 – 00

Standard Test Method for Viscosity and Yield of Vehicles and Varnishes by the Duke Viscometer[1]

This standard is issued under the fixed designation D 6606; 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 test method covers the procedure for determining the viscosity of varnishes, ink vehicles, and similar liquids that are essentially nonvolatile and unreactive under ordinary room conditions using the Duke Automated high shear rod and collar viscometer.[2]

1.2 The instrument in this test method is similar in principle to the falling-rod viscometer described in Test Method D 4040 except that the collar is motor driven and the range of available shear stresses is considerably greater. This instrument is capable of measured and extrapolated viscosity and yield values provided the proper model is chosen for the given application. See Section 6 for the ranges of specified models.

1.3 *This standard does not purport to address all of the safety concerns, if any, that may be associated with its use. It is the responsibility of the user of this standard to establish any appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4040 Test Method for Viscosity of Printing Inks and Vehicles by the Falling Rod Viscometer[3]

3. Terminology

3.1 *Definitions:* Terms relative to this test method are defined in Test Method D 4040 except for those found in 3.2.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *yield value, n*—The Lehman's yield value that is defined as the stress at 2.5 s^{-1}

3.2.1.1 *Discussion*—This value can be either extrapolated or measured.

3.2.2 *shortness ratio, n*—a ratio to determine the shortness characteristic of the fluid where *SR* = yield value/apparent viscosity.

4. Summary of Test Method

4.1 This automated test method is based upon accurate measurement of the force that is transferred at a known temperature into a stationary vertical rod as a fluid such as printing ink/varnish moves through a precision measurement gap formed between the outside diameter of the rod and the inside diameter of collar of specific geometry as said collar moves upward along the length of the rod at specified velocities.

4.2 The operator of the instrument and the customer must agree on the preprogrammed test sequence, which is to be run for a given material and the desired test temperature. Supervisory personnel should know the specifics of each preprogrammed test sequence, how to change a given test sequence, and how to make a new test sequence.

4.3 This test method provides actual measured test data of fluids at specified shear rates, which can be programmed to relate. In addition to actual measured data, extrapolated data using the "power law mathematical model" is provided that correlates with manual testing methods as outlined in Test Method D 4040.

4.4 Specified test reference temperatures can range from 20–40°C. Allowable deviation from desired test reference temperature could be specified differently in each test sequence. Allowable programmable is within the range of 0.05 to 0.4°C.

5. Significance and Use

5.1 Actual direct measurements of apparent viscosity and stress at shear rates of interest can be useful in the practical control of ink viscosity during production and the specification acceptance between supplier and purchaser.

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[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 D01.37 on Ink Vehicles.

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[2] The sole source of supply of an automated high shear rod and collar viscometer known to the committee at this time is Duke Custom Systems, Inc., Pleasant View, TN. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[3] *Annual Book of ASTM Standards*, Vol 06.02.

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TABLE 1 Performance Ranges For Each Model

Model Number	Stress Range (Dynes/ cm^2)	Viscosity Range Poise at 2500sec^{-1}	Shear Rate Range (sec^{-1})	Temperature Range° C
D-2010 D-2010E D-2012 D-2012E	75 − 1.0×10^6	4 − 400	2.5 − 10,000	19–40
D-2020 D-2020E D-2022 D-2022E	150 − 2.0×10^6	10 − 800	2.5 − 10,000	19–40
D-2050 D-2050E D-2052 D-2052E	300 − 5.0×10^6	20 − 1,600	2.5 − 7,500	19–40
D-2100 D-2100E D-2102 D-2102E	600 − 10.0×10^7	40 − 3,200	2.5 − 7,500	19–40

5.2 Use of the Duke automated viscometer provides direct measurements for viscosity and yield value versus extrapolating data points that may be far from the desired shear rates.

6. Apparatus

6.1 Table 1 describes each model of the Duke automated high shear rod and collar viscometer with the appropriate ranges for the ink or varnish being tested.

7. Materials

7.1 *ASTM Standard Viscosity Fluids—(For calibration purposes only.)* At approximately 100, 200, and 400 poise, span the practical range of the instrument without inducing self-heating errors. The required fluids[4] are N4000 (100 poise at 25°C), S8000 (200 poise at 25°C), and N15000 (400 poise at 25°C).

7.2 *Lint and Metal-Free Rags or Tissues* of appropriate size, so that they may be pulled or pushed completely through the collar in a single pass.

7.3 *Naphtha or other Low-Boiling Solvent* in a wash bottle or closed metal container.

8. Hazards

8.1 **Warning:** Solvents may be hazardous to the skin and eyes. In addition to other precautions, always wear protective gloves and safety glasses during cleanup to avoid solvent contact with skin and eyes. See supplier's material safety data sheet for further information on each solvent used.

8.2 **Instrument Warning:** Never operate the instrument with the rod installed without fluid in the collar.

9. Preparation of Apparatus

9.1 Set the viscometer up on a sturdy vibration-free bench capable of supporting at least 6.75 sq. m.

9.2 Locate the viscometer in an area free of direct sunlight to prevent sun-induced heating inside the clear temperature-controlled enclosure.

9.3 Level the viscometer by turning the adjustable feet up or down until the spirit level is centered and the instrument is free from wobble.

9.4 Clean the rod and collar thoroughly with tissues wetted with naphtha or other appropriate solvent. Remove residual solvent with clean dry tissue. Ensure rod and collar are free of lint or other particles from tissue or environment. **Warning:** Failure to clean instrument adequately will severely influence results.

9.5 Ensure identification code on rod and collar match.

9.6 Properly install and lock into viscometer the rod with rod cup and collar in accordance with manufacturer's recommendations.

9.7 Plug in collar/sample temperature probe.

9.8 Allow instrument to equilibrate at a desired test temperature.

10. Calibration

10.1 Ensure instrument is within calibration window by reading information contained in "Days to Next Calibration Window" on the computer screen. The number of days until the next factory-authorized calibration is displayed whenever the instrument is not in active use.

10.2 Periodically check calibration as in Section 17.

11. Sample Preparation

11.1 Transport sample to be tested to the test area and preserve in a suitable clean closed container. Skin paper should be used for oxidative fluids.

11.2 Samples should be uniform dispersions or structures throughout. Samples to be tested should be free of bubbles, skin, or other debris. If variations in structure are suspected, the sample must be remixed until it is of uniform consistency.

11.3 Fill pipet with the sample and place filled pipet into the temperature-controlled enclosure of viscometer. **Warning:** Do not work the sample vigorously. Be sure to close the sample container immediately after removing the desired sample to be tested.

12. Conditioning

12.1 The test sequence and reference temperatures are programmable and are protected by password security to prevent unauthorized changes from desired temperatures. The reference temperature for this test method is 25.0° and the enclosure temperature for this test method is 24.95°C.

12.2 The pipet containing the sample should remain inside the temperature-controlled enclosure a minimum of one minute before the sample is placed on the rod. Samples are approximately 2.2 ml and will quickly reach equilibrium.

[4] The sole source of supply of the certified standard viscosity oil known to the committee at this time is Cannon Instrument Company, P.O. Box 16, State College, PA 16801. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

13. Procedure for Test Runs

13.1 Allow instrument to equilibrate to desired test temperature.

13.2 Ensure sample to be tested in pipette is placed in its holder inside the temperature-controlled enclosure.

13.3 Select the desired test sequence from a list of preprogrammed test sequences. (Press F1 key for list). See Section 18 for details of test sequence specific to this test method.

13.4 Start selected test sequence. (Press F2 key.)

13.5 Enter Test ID name. This is mandatory. (Name must be DOS compatible.) (Press Enter key.)

13.6 Enter Operator ID (Optional). (Press Enter key.)

13.7 Enter Formula ID (Optional). (Press Enter key.)

13.8 Enter Batch ID (Optional). (Press Enter key.)

13.9 Follow the instructions on computer screen.

13.10 Open doors. Rod will start rotating once doors open.

13.11 Place sample onto rotating rod just above the collar. Ensure sample is distributed completely around rotating rod.

NOTE 1—Do not place sample on upper 2 in. of the rod.

13.12 Close doors. The rod will stop rotating.

13.13 Press the Enter key to proceed with the selected test sequence.

13.14 No additional operator action is required until test is completed. The instrument will proceed automatically following preprogrammed specifications of the selected test sequence. (Rates of shear and allowable temperature deviations.)

13.15 After completion of the test, the operator must remove rod and collar assembly for cleaning.

13.15.1 Open doors of temperature-controlled enclosure.

13.15.2 Unplug collar/sample temperature sensor cable.

13.15.3 Unlock collar assembly by pulling outwards on collar on collar lock slide assembly.

13.15.4 Unlock rod assembly by pressing down on rod lock ring. While rod lock ring is depressed, lift rod assembly approximately 12.3 mm. Release pressure on rod lock ring.

13.15.5 With one hand on top and the other hand at bottom of rod, lift rod and collar assembly up through collar mount. Remove your hand from bottom of rod long enough for bottom to clear collar mount.

13.15.6 Take rod and collar assembly to designated cleanup station.

13.15.7 Before cleaning rod and collar assembly, if available, insert secondary rod and collar assembly into the primary position in instrument by reversing steps 13.15.7-13.15.1.

13.15.8 Close doors on temperature-controlled enclosure.

13.15.9 Return to cleanup station, the thoroughly clean rod and collar assembly using appropriate solvents and tissues or rags.

13.15.10 Return clean rod and collar assembly to temperature-controlled enclosure. If secondary rod and collar are inserted, place this rod and collar assembly on storage rack inside left side of enclosure. If secondary rod and collar are not inserted into instrument, replace this rod and collar assembly into primary position by reversing steps 13.15.7-13.15.1.

13.16 Remove printout of test data from printer.

13.17 Instrument is ready for next test.

14. Test Data Available

14.1 This instrument can provide these values for each stroke of the test sequence performed: measured temperature, measured stress, measured rate of shear, measured grams, and elapsed seconds. The instrument can provide temperature-corrected measured stress and viscosity if the sequence was not performed at exactly the desired reference temperature.

14.2 In addition, this instrument can compute apparent viscosity at 2500 s^{-1}, extrapolated yield stress at 2.5 s^{-1}, and shortness factor. These values are derived from the Power Law Model.

15. Report

15.1 Report measured and apparent viscosity at 2,500 s^{-1}, measured yield stress at 2.5 s^{-1}, measured shortness factor, reference temperature, test sequence number, instrument model number, and instrument serial number.

16. Precision

16.1 *Precision*—An interlaboratory study of this method was conducted in which a single operator in each of seven laboratories made one run consisting of four varnishes and one ink. The samples ranged in viscosity from 250 to 1200 P. The varnishes were of different rheological character to reflect a range of shortness ratios applicable to those seen in the graphic arts industry. The estimated standard deviations and degrees of freedom are given in Table 2. (Since the standard deviations are proportional to the test value, precision statements are made in terms of percent of the observed value.) Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

16.2 *Reproducibility*—Two results, each the mean of results obtained on different days by operators in different laboratories should be considered suspect if they differ more than the allowable differences indicated in Table 2.

17. Keywords

17.1 apparent viscosity; automated viscometers; falling-rod viscometers; non-Newtonian; power law model; rod and collar viscometers; shortness; vehicles; viscometers; viscosity; yield value

TABLE 2 Reproducibility Round Robin Results

Test Results	Standard Deviation % Relative	Degrees of Freedom	Maximum Allowable Difference, ± % Relative
	Reproducibility		
Measured Viscosity at 2500 s^{-1}	4.68	6	4.33
Extrapolated Viscosity at 2500 s^{-1}	4.69	6	4.34
Measured Yield Stress at 2.5 s^{-1}	5.26	6	4.86
Measured Shortness Ratio	6.79	6	6.28

ANNEXES

(Mandatory Information)

A1. CHECKING INSTRUMENT CALIBRATION

A1.1 Thoroughly clean rod and collar assembly using appropriate solvent and tissues or rags.

A1.2 Thoroughly clean all mechanicals inside temperature-controlled enclosure. Use appropriate solvents and tissues or rags. Remove all lint, dust, and visible particulate matter using a vacuum cleaner.

A1.3 Thoroughly clean pipet and place inside temperature-controlled enclosure to equilibrate to desired test temperature (25°C).

A1.4 Install rod and collar assembly. Allow rod and collar to equilibrate to the desired test temperature before proceeding.

A1.5 Run a minimum of five tests utilizing certified Viscosity Standard N4000[4] on each rod and collar assembly of the instrument. Repeat utilizing Viscosity Standard[4] S8000 and N15000.

NOTE A1.1—This calibration procedure is defined by the manufacturer for use on this instrument.

A1.6 Viscosity at 2.5 s^{-1}. Calculate the difference, in percent, between the actual certified viscosity of each viscosity standard and the apparent viscosity at 2,500 s^{-1} that is reported on the instrument. The allowable error is less than 2.5 %. If the error is greater than 2.5 %, the instrument is out of calibration and in need of service.

A1.7 Yield stress at 2.5 s^{-1}. Calculate the yield stress at 2.5 s^{-1} for each viscosity standard by multiplying the certified viscosity standard in poise at 25°C by 2.5. This figure represents the certified stress at 2.5 s^{-1} for this particular viscosity standard. Subtract the "measured yield stress at 2.5 s^{-1}" that was reported from the instrument from the "certified stress at 25°C." Express this difference in dynes/cm^2. If the difference is greater than 150 dynes/cm^2 on models D-2010, 2012, 2020, 2022, 2050, or 2052, or greater than 300 dynes/cm^2 on models D-2100 or D-2102, the instrument is out of calibration and in need of service.

A2. DETAILS OF TEST SEQUENCE

A test sequence that has shown to be particularly beneficial in determining variations and differences in varnishes is as follows:

A2.1	Type Instrument	Viscometer
A2.2	Allowable Temperature	0.2
A2.3	Rod Rotation	1000
A2.4	Rod Rotation Speed	200
A2.5	Bottom Dwell time	10
A2.6	Top Dwell Time	5
A2.7	Non-Test Collar Speed	12000
A2.8	Number of Conditioning Strokes	0
A2.9	Conditioning Stroke Time	0
A2.10	Number of Strokes at Each Time	1
A2.11	Stroke # 01 Time	83.333
A2.12	Stroke # 02 Time	16
A2.13	Stroke # 03 Time	4
A2.14	Stroke # 04 Time	2
A2.15	Stroke # 05 Time	1
A2.16	Stroke # 06 Time	0

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