Designation: D 4040 – 99

Standard Test Method for Viscosity of Printing Inks and Vehicles by the Falling-Rod Viscometer[1]

This standard is issued under the fixed designation D 4040; 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 falling-rod viscosity of printing inks, vehicles, and similar liquids that are essentially nonvolatile and unreactive under ordinary room conditions.

1.2 For printing inks, which are typically non-Newtonian, this test method is applicable in the apparent viscosity range from about 10 to 300 P at a shear rate of 2500 s^{-1}. For Newtonian liquids, the applicable viscosity range is about 10 to 1000 P (1 P = 0.1 Pa·s).

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.* For specific hazard statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *viscosity, V*—the ratio of shear stress to shear rate. The viscosity of a liquid is a measure of the internal friction of the liquid in motion. The cgs unit of viscosity is 1 g/cm·s (1 dyne·s/cm^2) and is called a poise. The SI unit is 1 N·s/cm^2 and is equal to 10 P.

3.1.2 *shear stress, S*—shearing force per unit area; the unit is 1 g/cm·s^2 (1 dyne/cm^2). In the falling-rod viscometer, shear stress is proportional to total weight *W* per unit of shearing area *A* times the gravitational constant *g*, in accordance with the equation: $S = Wg/A$.

3.1.3 *shear rate, D*—velocity gradient through the stressed liquid; the unit is 1/s or 1 s^{-1}. In the falling-rod viscometer, shear rate is inversely proportional to fall time *F* per unit distance *L* over which a unit thickness *x* of the liquid is stressed: $D = L/xF$.

3.1.4 *Newtonian*—refers to a liquid whose viscosity is constant at all shear rates.

3.1.5 *non-Newtonian*—refers to a liquid whose viscosity varies with shear rate. Such liquids may be either shear-thinning (pseudoplastic) or shear-thickening (dilatant). Most printing inks are shear-thinning.

3.1.6 *apparent viscosity, V_D*—the viscosity of a non-Newtonian fluid at a particular shear rate *D*. A shear rate of 2500 s^{-1} has been found useful for printing inks and is specified in this test method.

3.1.7 *yield stress, S_o*—the minimum shear stress required to initiate motion in a non-Newtonian liquid.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *power law*—a mathematical model that presumes that the viscosity of a liquid varies with shear rate in accordance with a power function as follows:

$$k = \frac{S}{D^N} \tag{1}$$

where:

k = a constant related to the viscosity of the liquid and
N = a constant describing the rate at which shear stress varies with shear rate.

The value of *N* is precisely 1.0 for a Newtonian fluid, less than 1.0 for a shear-thinning liquid, and greater than 1.0 for a shear-thickening liquid.

3.2.2 *power law plot*—a logarithmic plot of shear stress versus shear rate based on the expanded form of the power law equation:

$$\ln S = \ln k + N \ln D \tag{2}$$

For liquids conforming to the power law, the logarithmic plot of *S* versus *D* is linear over the shear rate range of interest. The slope of the line is the power law constant *N*.

3.2.3 *shortness*—the property of a non-Newtonian fluid that prevents it from being drawn into a filament.

3.3 *Symbols: (for Power-Law Calculations):*

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved June 10, 1999. Published August 1999. Originally published as D 4040 – 81. Last previous edition D 4040 – 96.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

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

B = intercept of a straight line.
F = measured fall time, s.
F_c = corrected fall time, s.
F_{2500} = fall time equivalent to a shear rate of 2500 s^{-1}, s.
K_{2500} = apparent viscosity constant at 2500 s^{-1}, $cm^{-1}s^{-1}$.
N = slope of the power law plot, a measure of non-Newtonianism, cm^2/dyne·s.
SF = shortness factor, s^{-1}.
S'_o = pseudo yield value, dyne/cm^2
S_o^L = Lehman yield value, dyne/cm^2.
T = measured specimen temperature,° C.
T_R = reference temperature, °C.
$V_{2.5}$ = apparent viscosity at 2.5 s^{-1}, P.
V_{2500} = apparent viscosity at 2500 s^{-1}, P.
W = total weight, g.
W_A = added weight, g.
W_R = weight of rod, g.
W_{2500} = weight required to obtain a shear rate of 2500 s^{-1}, g.

4. Summary of Test Method

4.1 This test method is based on measurements of the time required for a weighted rod to fall through an aperture containing the test specimen.

4.2 Fall times are corrected to a reference temperature of 25°C (or other mutually agreed-upon temperature). The test method specifies precise measurement of actual specimen temperature in order to detect fluctuations due to cooling by metal, heat of friction during shearing, and body heat of the operator.

4.3 Each specific instrument must be calibrated in order to establish the fall time that is equivalent to a shear rate of 2500 s^{-1}.

4.4 Fall times as a function of weight are extrapolated to 2500 s^{-1} by means of the power law (logarithmic) relationship between shear stress and shear rate. Apparent viscosity at 2500 s^{-1} and the degree of non-Newtonianism are determined by calculation or graphically. The calculation of several low shear parameters is also covered.

5. Significance and Use

5.1 Apparent viscosity at the relatively high shear rate of 2500 s^{-1} does not completely define the rheological properties of printing inks but is useful in the practical control of ink viscosity during production and the specification acceptance between supplier and purchaser.

5.2 The slope of the power law plot is the preferred measure of non-Newtonianism. The yield value, which is obtained by extrapolation of high-shear measurements to a shear rate approaching zero, does not conform to the definition of the true yield stress (see 3.1.7). The yield value and other low shear parameters are also subject to a high degree of variability (see the precision table in Section 16).

6. Apparatus

6.1 *Fall-Time Runs*:

6.1.1 *Falling-Rod Viscometer*, equipped with a swinging platform and automatic timing device[3] accurate to at least 0.1 s, preferably 0.01 s. A special lightweight rod is useful for liquids in the 10-P range.

6.1.2 *Set of Tapped or Slotted Weights*—Weights of 50 or 100 to 500 g are usually provided with the instrument. Extra 500-g weights, approximately 4, totaling about 2000 g are required to handle fluids at the upper end of the practical range. A25-g weight is useful for liquids in the 10-P range.

6.1.3 *A Thermostatically Controlled Cabinet or a Special Collar*, [4] through which water is circulated from a constant-temperature bath (both are optional if room conditioning is not available).

6.1.4 *Thermistor*, spanning the specified test temperature (usually 25°C), accurate to 0.01°C, and equipped with a probe having a response time of 3 to 6 s.

6.1.5 *Ring Stand and Clamp*, or other device for holding the thermistor probe in a suitable position.

6.1.6 *Small Plastic Spatula*—Metal spatulas are not suitable.

6.1.7 *Plastic Scraper*, consisting of a piece of flexible plastic, approximately 30 by 70 mm, having a semicircle cut out at one end; semicircle should fit the rod.

6.2 *Instrument Calibration*:

6.2.1 *Balance*, weighing to 0.1 g.

6.2.2 *Metric Rule or Scale*, at least 100 mm in length.

6.2.3 *Vernier Caliper*, accurate to 0.01 mm, having a capacity of at least 30 mm.

6.3 *Graphical Solutions*:

6.3.1 *Chart Paper*, logarithmic 2 × 2 to 2 × 3 cycles.

6.3.2 *Triangle*, 45°, with a hypotenuse length of at least 100 mm (approximately 8 in.).

6.3.3 *Protractor*.

7. Materials

7.1 *ASTM Standard Viscosity Oils*, [5] a minimum of two, preferably three, spanning the practical range of the falling-rod viscometer (used for calibration purposes only).

7.2 *Lithographic Varnish* or similar vehicle having a viscosity of about 200 P (for use in 12.3, if needed).

7.3 *Lint-and-Metal-Free Rags or Tissues*.

7.4 *Naphtha* or other low-boiling solvent in a wash bottle or closed metal container.

8. Hazards

8.1 *Safety Precautions*—Since solvents may be hazardous to the skin and eyes, in addition to other precautions, wear

[3] Platform and timing device are standard on newer viscometer models. For equipping older models, see Bassemir, R., "Evaluation of the Laray Viscometer," *American Ink Maker*, Vol 39, No. 4, April 1961, pp. 24–26 and 60.

[4] Collars are available as accessories from the respective manufacturers of falling-rod viscometers.

[5] 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. Table A1.2 in Test Method D 445 shows satisfactory oils including S-600 (16 P at 25°C), S-2000 (56 P), S-8000 (230 P), and S-30 000 (810 P). Viscosity at various temperatures is indicated on the label of each container.

rubber gloves and safety glasses during cleanup to avoid solvent contact with skin and eyes. In case of contact, wash skin with water; flush eyes for 15 min with water and call a physician. See supplier's Material Safety Data Sheets for further information on each solvent used.

8.2 *Instrument Cautions*:

8.2.1 Avoid any operation that will scratch the rod. Do not use a metal spatula. Never drop the rod through an empty aperture.

8.2.2 Weight loads in excess of 3000 g may cause bending of the rod.

8.2.3 To minimize heat buildup from body temperature during a run, avoid contacting the viscometer block with bare hands. When instructions call for holding the block steady, wear a glove or place a small cloth in the palm of the hand.

8.2.4 When making fall-time measurements, work quickly and without interruption so that the entire run is completed within 5 to 10 min.

NOTE 1—Many modern printing inks and vehicles contain some solvent, and volatile loss during a run can seriously bias test results unless rigorous control of exposure time is exercised. Volatile loss can be detected if successive drops of the rod with the same weight result in increasingly longer fall times.

9. Preparation of Apparatus

9.1 Set the viscometer on a sturdy bench located in an area free of direct drafts, direct sunlight, and other sources of heat. Level the viscometer, using the adjustable feet.

9.2 Pass a hand over the upper and lower photocells to assure that the timer is activated and deactivated.

9.3 Attach the clamp to the ring stand and place next to or behind the viscometer. Drape the thermistor probe over the clamp; reset the clamp so that the probe end falls close to the viscometer block.

9.4 Clean the block and rod thoroughly with tissues wetted with naphtha. Remove residual solvent with clean dry tissue. Roll the clean dry rod over a flat surface to check for straightness. If rod is bent, discard and obtain a new rod/orifice set.

9.5 Examine the markings at the ends of the rod. Select one marking as an indication of the "proper" end to be always inserted into the aperture first.

10. Calibration

10.1 Determine instrument constants in accordance with the procedure given in Annex A1.

10.2 *Optional*—If a graphical method is to be used for direct conversion of test results to viscosity, prepare Master Sliding Scale Calibration Graph as in Annex A2.

10.3 Periodically check calibration as in A1.2.

11. Sample Preparation

11.1 Transport the sample to the test area and preserve in a closed container. Skin paper should be used for oxidative drying inks.

11.1.1 Ink samples should be uniform dispersions. If pigment settling is suspected, insert a spatula in the container and gently stir. Be careful not to introduce air bubbles.

11.1.2 Prior to the run, a portion of the sample may be transferred to a slab and gently spread out in order to remove bubbles, skin, or other debris.

NOTE 2—**Caution:** *Do not work the sample vigorously*; this practice causes a significant increase in sample temperature. Be sure to close the container immediately after removing the desired portion.

12. Conditioning

12.1 The temperature of the room (cabinet or collar) should be set at 23 ± 1°C (or 2°C below the reference temperature).

NOTE 3—In accordance with Note 8, the allowable range for specimen temperature is ±2°C from the reference temperature. However, during the course of testing, heat of shearing and body heat of the operator both contribute to continuous temperature rises in test specimens, notwithstanding room, cabinet, or collar conditions. To allow for inevitable temperature rises, temperature controls are set at the lower end of the allowable range.

12.2 Equilibration of test samples is not necessary. Specimen sizes are small (less than 2 mL); when spread out on a slab and applied to the viscometer, both hot and cold samples quickly reach the temperature of the metal.

12.3 If the viscometer has been idle for more than an hour, it may be necessary to bring it into equilibrium with the conditioning temperature (23°C or other specified in 12.1). Make preparations for an exploratory run (13.2-13.5) using a varnish if the test specimen contains volatiles. Read specimen temperature; if too low (a possibility considering that metal serves as a heat sink), add a 1000-g weight to the rod and make a few drops (13.7-13.9 but without recording time) until the specimen temperature reaches that of conditioning. Continue the run or, if a varnish was used, clean up.

13. Procedure for Fall-Time Runs

13.1 If required, prepare, level, and condition the instrument as described in 9.1 and 12.3.

13.2 With the proper end of the clean rod down, hold the rod vertically over the clean aperture and gently lower until it rests on the swinging platform.

13.3 Transfer a uniform specimen to the tip of a clean plastic spatula. The specimen size should be sufficient to fill the well of the viscometer.

13.4 Hold the rod with the fingertips and carefully raise about 20 mm. Transfer the specimen from the spatula to the rod as close as possible to the bottom of the well. Rotate the rod slowly to distribute the specimen around the well, ensuring that the well is full. Allow the rod to fall to the platform.

13.5 Place the thermistor probe in the well close to but not touching the rod. The probe can remain in the well throughout the run. Turn the thermistor on.

13.6 Using experience or the information in Table 1 as a guide, select a weight load that will produce a fall time as close to 1 or 2 s as is practical.

NOTE 4—For comparison of non-Newtonian liquids, runs must not be made at pre-specified rod weights. Rather, weights should be adjusted to obtain pre-specified fall times, the first of which corresponds as closely to a shear rate of 2500 s^{-1} as is practical.

13.7 Hold the block level and steady with one hand (see 8.2.3). With the other hand, carefully place the selected weights

TABLE 1 Weight/Fall-Time Relationships for Newtonian Liquids[A]
Rod weight = 130 g

Viscosity of Fluid, P	Fall Time, s					
	1	2	4	6	10	20
	Added Weight, g					
10	200	50	...	...	...	...
50	1 300	700	300	100	50	...
100	3 000	1 400	700	400	150	25
250	7 000 [B]	3 800 [B]	1 800	1 100	700	300
500	15 000 [B]	7 000 [B]	3 200 [B]	2 200	1 400	600
1000	25 000 [B]	16 000 [B]	8 000 [B]	5 500 [B]	3 000	900

[A] Weights required may be more or less depending on instrument type and degree of wear. Printing inks will require additional weight depending on degree of non-Newtonianism.

[B] Weights are impractical or not recommended.

on top of the rod. If weights are slotted, evenly distribute the slots around the circumference of the rod. Make certain that the rod is vertical. (If weights tend to make the viscometer unsteady, retain the hand on the block so that the rod falls smoothly in 13.8.)

13.8 Set the timer. Release the platform and allow the rod to fall naturally. If the fall time is within the desired range (for example, 1 to 2 s for the first weight, etc.), record the added weight W_A, fall time F, and specimen temperature T on worksheet.

13.9 Remove the weights from the rod. Pull the rod up slowly with the fingertips of one hand while holding the viscometer block firmly with the other hand. Rest the rod on the swinging platform. Using the plastic scraper, scrape the "collar" of specimen from the top to the bottom of the rod where it enters the block. Gently rotate the rod in the well to redistribute the specimen.

13.10 Repeat the drop (13.7-13.9) with the same or adjusted weights until two fall times with a specific set of weights agree within 2 % (0.04 s at a 2-s fall time, 0.2 at a 10-s fall time, etc.).

13.11 Make additional measurements (13.7-13.10) with succeedingly lighter sets of weights, each approximately 50 % of the previous set, but do not exceed a fall time of 20 s.

NOTE 5—Newtonian liquids may be run with only one or two sets of weights. Non-Newtonian liquids require at least four or five.

13.12 If the specimen is deplenished during the run, clean up and start over from 13.1, preferably using ink fresh from the container. Make certain that the quantity of specimen is sufficient.

13.13 Immediately after completing the run, turn the thermistor off, remove the probe from the well, and clean the probe, the viscometer orifice, and the rod thoroughly.

NOTE 6—Since each test involves replicate fall-time measurements at up to five weights, a single viscosity determination is usually considered adequate.

14. Calculation

NOTE 7—This section covers calculations by computer or programmable calculator. The list of symbols is given in 3.3. The procedure for graphical solution of test results is described in the Annex A2.

14.1 Enter into the computer the values for the instrument constants, W_R, F_{2500} and K_{2500} and the reference temperature T_R.

14.2 Enter data from fall-time runs in sets of W_A, replicate values of F that agree within 2 %, and the corresponding values of T.

14.3 Compute the non-Newtonianism parameter N by simultaneous solution of the following general equation:

$$\log W = B - N \log F_c \tag{3}$$

where:

$$W = W_A + W_R \tag{4}$$

and

$$F_c = F + 0.1\,F\,(T - T_R) \tag{5}$$

NOTE 8—Eq 5 corrects each measured fall time by 10 % per degree differential between the measured temperature and the reference temperature and is applicable only within 2°C of T_R. As noted in the third column of Table A1.2, specimen temperature increases progressively during a run. For accurate results, it is important that the temperature correction be applied to the fall time corresponding to each added weight.

NOTE 9—In some software programs, average temperature is being used to correct viscosity results. This procedure may introduce significant error into the slope of the power low plot. Because of the long extrapolation from 2500 to 2.5 s^{-1}, all low-shear parameters cited in 14.6-14.8 are especially prone to error.

14.4 Examine (by computer) the value of N. Any value over 1.0 is improbable for a printing ink or a vehicle and suggests error in the test measurements; check data or repeat runs. Alternatively, treat any value between 1.0 and 1.05 as 1.0.

14.5 Compute the viscosity at 2500 s^{-1} as follows:

$$V_{2500} = K_{2500} W_{2500} \tag{6}$$

where:

$$W_{2500} = \text{antilog}\,(B - N\,log\,F_{2500}) \tag{7}$$

14.6 *Optional*—Compute the viscosity at 2.5 s^{-1} from either Eq 8 or Eq 9 as follows:

$$V_{2.5} = V_{2500}\,(1000^{1-N}) \tag{8}$$

or

$$V_{2.5} = 1000\,K_{2500}\,antilog\,(B - N\,log\,1000\,F_{2500}) \tag{9}$$

14.7 *Optional*—Compute the pseudo yield value as follows:

$$S'_o = 2.5\,(V_{2.5} - V_{2500}) \tag{10}$$

NOTE 10—Since the logarithmic nature of the power law precludes a zero shear rate, Eq 10 was derived to approximate the Bingham yield value, which is normally determined by extrapolating the linear portion of a shear stress/shear rate plot to zero rate of shear.