Standard Test Method for
Rheological Properties of Paste Printing and Vehicles by the Falling-Rod Viscometer[1]

1. Scope*

1.1 This test method covers the procedure for determining the falling-rod viscosity and degree of non-Newtonian behavior 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 test method uses a falling-rod viscometer in which shear conditions are altered by manually adding weight to the rod. A fully automatic instrument is described in Test Method D6606.

1.4 This test method, as does Test Method D6606, bases calculations on the power law model of viscosity. ISO 12644 covers not only the power law but also the Casson and Bingham models.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D6606 Test Method for Viscosity and Yield of Vehicles and Varnishes by the Duke Viscometer

2.2 *Other Standards:*

ISO 12644 Graphic Technology—Determination of rheological properties of paste inks and vehicles by the falling rod viscometer

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, V_D, n*—the viscosity of a non-Newtonian fluid at a particular shear rate *D.*

3.1.1.1 *Discussion*—A shear rate of 2500 s^{-1} has been found useful for printing inks and is specified in this test method.

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

3.1.3 *non-Newtonian, adj*—refers to a liquid whose viscosity varies with shear rate.

3.1.3.1 *Discussion*—Non-Newtonain liquids may be either shear-thinning (pseudoplastic) or shear-thickening (dilatant). Most printing inks are shear-thinning.

3.1.4 *shear rate, D, n*—velocity gradient through the stressed liquid; the unit is 1/s or 1 s^{-1}.

3.1.4.1 *Discussion*—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: $D = L/xF$.

3.1.5 *shear stress, S, n*—shearing force per unit area; the unit is 1 g/cm·s^2 (1 dyne/cm^2).

3.1.5.1 *Discussion*—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: $S = Wg/A$.

3.1.6 *viscosity, V, n*—the ratio of shear stress to shear rate.

3.1.6.1 *Discussion*—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.

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

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Lehman yield value, n*—calculated yield stress based on the Lehman chart.

3.2.2 *power law, n*—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.

3.2.2.1 *Discussion*—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.

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

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

3.2.3.1 *Discussion*—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.

3.2.4 *pseudo yield value, n*—calculated yield stress developed for use with the power law.

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

3.2.6 *shortness factor (also called shortness ratio), n*—ratio of yield value to viscosity.

3.3 *Symbols:*

3.3.1 *(for Power-Law Calculations):*

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. A 25-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.

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[3] Platform and timing device are standard on newer viscometer models. For equipping older models, see Bassemir, R, "Evaluation of the Laray Visocmeter," *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.

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 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. (**Warning**—*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 2—In accordance with Note 7, 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 3—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 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 4—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 depleted 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 5—Since each test involves replicate fall-time measurements at up to five weights, a single viscosity determination is usually considered adequate.

14. Calculation

NOTE 6—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.

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.

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:

$$\log W = B - N \log F_c \qquad (3)$$

where:

$$W = W_A + W_R \qquad (4)$$

and

$$F_c = F + 0.1 F (T - T_R) \qquad (5)$$

NOTE 7—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 8—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:

$$V_{2500} = K_{2500} W_{2500} \qquad (6)$$

where:

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

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

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

or

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

14.7 Optional—Compute the pseudo yield value as follows:

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

NOTE 9—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. The derivation of the

pseudo yield value (Eq 10) is given in Appendix X1.

NOTE 10—Calculations based on the Lehman chart define a yield value as follows:

$$S_o^L = 2.5 V_{2.5} \qquad (11)$$

For Newtonian fluids, Eq 11 gives a finite value for yield value that increases with increasing $V_{2.5}$, whereas Eq 10 correctly gives a value of zero. It should be noted that Eq 10 and 11 approach each other for fluids exhibiting a high degree of non-Newtonianism, that is, very high $V_{2.5}$ compared to V_{2500}.

14.8 Optional—Compute the shortness factor as follows:

$$SF = S'_o / V_{2500} \qquad (12)$$

15. Report

15.1 Report apparent viscosity at 2500 s^{-1}, degree of non-Newtonianism, reference temperature, and identifying code referring to the specific viscometer.

15.2 Optional—Report the viscosity at 2.5 s^{-1}, the pseudo yield value, and the shortness factor.

16. Precision

16.1 An interlaboratory study of this test method was conducted in which a single operator in each of nine laboratories made one run consisting of at least four replicated data points on four inks on two different days. The inks ranged in viscosity from 10 to 300 P. The results were calculated on a single programmable calculator. One laboratory was a consistent outlier and was deleted from the entire analysis, and the 10 P ink was deleted from analysis of the low shear parameters. The estimated standard deviations and the degrees of freedom are given in Table 2. (Since the standard deviation was 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:

16.1.1 Repeatability—Two results obtained by the same operator on different days should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

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

TABLE 2 Precision of Falling-Rod Viscosity Determinations

Test Results	Standard Deviation, % relative	Degrees of Freedom	Maximum Allowable Difference,% relative
Repeatability			
N	3.3	4	7.6
V_{2500}	5.4	4	15.3
$V_{2.5}$	11.5	4	31.4
S'_o	14.6	3	41.2
SF	17.7	3	50.1
Reproducibility			
N	4.6	28	12.9
V_{2500}	7.9	28	22.3
$V_{2.5}$	17.5	21	49.5
S'_o	22.3	21	63.1
SF	23.6	21	66.9

16.2 *Bias*—This test method requires that the viscometer be calibrated with up to three ASTM standard viscosity oils spanning the useful range of the instrument. Since test results are defined in reference to these oils, bias need not be determined.

17. Keywords

17.1 apparent viscosity; falling-rod viscometers ; inks; non-Newtonianism; power law viscosity; printing inks; shortness; vehicles; viscometers; viscosity; yield value

ANNEXES

(Mandatory Information)

A1. CALIBRATION OF FALLING-ROD VISCOMETERS

A1.1 *Determination of Instrument Constants:*

A1.1.1 Measure the distance between photocells of the timing device. Adjust to $100 + 0.5$ mm. Divide by 10 and record as L in centimetres in chart patterned after Table A1.1.

A1.1.2 Weigh the clean rod to 1 g. Record as W_R.

A1.1.3 Using a caliper, measure the diameter of the rod to 0.01 mm. Record as d in centimetres.

A1.1.4 With the caliper, measure the length of the block over which the liquid is sheared (total height of block minus ink well) to 0.01 mm. Record as h in centimetres.

A1.1.5 Calculate apparent shearing area from the equation $A = \pi dh$. Record as A in square centimetres.

NOTE A1.1—The shearing length of the block contains both a tapered and a parallel section; therefore, it is understood that A is not the true shearing area but an apparent shearing area.

A1.1.6 The thickness x of the test liquid is set by the clearance between the aperture and the rod. Since the aperture consists of two radii, both of which are difficult to measure, a value for a mean gap clearance $\bar{x}$ can be computed from fall-time runs on Newtonian oils in the following manner:

A1.1.6.1 Using the procedure described in Section 13, make fall-time runs on two or three standard oils in random sequence on two different days.

A1.1.6.2 For each added weight, compute the mean of fall times that agree within 2 %. Also compute the mean of the corresponding temperature. Record as $\bar{F}$ and $\bar{T}$ on worksheet (see Table A1.2). Correct each mean fall time according to Eq 5 (see 14.3). Record as F_c.

A1.1.6.3 Take the sum of each added weight and the rod weight. Record as the total weight, W.

A1.1.6.4 Multiply each total weight by the corresponding corrected fall time and record as WF_c. Examine trends in WF_c within each run. Values that change progressively with weight are indicative of inadequate temperature sensing or non-Newtonianism in the oil. Check the source of error or repeat with new oil if required.

TABLE A1.1 Instrument Constants for Falling-Rod Viscometers

Section	Symbol	Definition	Unit	Typical values	
				Laray	Thwing-Albert
A1.1.1	L	timed fall distance (distance between photocells)	cm	10.0	10.0
A1.1.2	W_R	weight of rod	g	130	130
A1.1.3	d	diameter of rod	cm	1.20	0.80
A1.1.4	h	height of shearing portion of aperture	cm	2.80	4.20
A1.1.5	A	apparent shearing area = $\pi\,dh$	cm^2	10.5	10.5
A1.1.6	$\bar{x}$	mean gap clearance	cm or μm	0.0045	0.0028
		$\bar{x} = [AL\,/g]\,[V/WF_c]$		45	28
	V	viscosity of calibrating oil at reference temperature	P		
	g	gravitational constant (approx. 980 cm/s^2)	cm/s^2		
	W	total weight = rod weight + added weight	g		
	F_c	corrected fall time	s		
A1.1.7	F_{2500}	fall time corresponding to shear rate of 2500 s^{-1} = $L/2500\,\bar{x}$	s	0.8	1.4
A1.1.8	K_{2500}	apparent viscosity constant at 2500 s^{-1} $K_{2500} = g/2500A$	cm^{-1} s^{-1}	0.0372	0.0372

TABLE A1.2 Typical Worksheet for Falling-Rod Viscometer

		Test sample	Calibrating Oil S-2000, V = 45.4		Date of run	1-10-80
		Instrument	Laray LV #1		Rod weight	130 g
Run #	GG32	Reference temperature	25°C		Room temp.	22.8°C

Test Measurements			Mean Results		Corrected Results			
Added Load W_A, g	Recorded Fall Time F, s	Specimen Temperature T,°C	Mean Fall Time[A] $\bar{F}$, s	Mean Specimen Temperature[A] $\bar{T}$,°C	Temperature Difference $(\bar{T}$-25$)$[B] ,°C	Corrected Fall Time[C] F_c, s	Total Weight[D] W, g	Newtonian Viscosity Multiplier[E] WF_c, g·s
600	1.45	24.49						
	1.55	24.55	1.57	24.61	−0.39	1.51	730	1102
	1.59	24.66						
400	2.02	24.70						
	2.02	24.82	2.02	24.76	−0.24	1.97	530	1044
200	3.35	24.97						
	3.27	24.98	3.35	24.99	−0.01	3.35	330	1105
	3.35	25.00						
100	4.72	25.03						
	4.85	25.03	4.73	25.03	+ 0.03	4.74	230	1090
	4.75	25.04						
25	7.27	25.11						
	7.25	25.07	7.26	25.08	+ 0.08	7.28	155	1126
								Mean WF_c 1097

[A] Using results only for fall times that agree within 2 %.
[B] The number 25 refers to the reference temperature. Change figure if appropriate.
[C] Formula: $F_c = \bar{F} + 0.1\ \bar{F}\ (\bar{T} - 25)$.
[D] Weight of rod plus added load.
[E] Complete this column only if sample is a calibrating oil or a Newtonian fluid.

A1.1.6.5 Compute a mean WF_c for each run and divide into the viscosity of the oil at 25°C (or other reference temperature). The resulting values of V/WF_c should be essentially the same for all oils. Compute the mean V/WF_c.

NOTE A1.2—If the label on the oil container does not list viscosity specifically at the reference temperature, the desired viscosity may be obtained from plots of log viscosity versus reciprocal of temperature on semi-logarithmic chart paper.

A1.1.6.6 Compute the mean gap clearance $\bar{x}$ by multiplying V/WF_c by AL/g. Record to three significant figures in centimetres (10^{-4} cm = 1 µm).

A1.1.7 Calculate the fall time corresponding to a shear rate of 2500 s^{-1} from the equation: $L/2500\ \bar{x}$. Record as F_{2500} in seconds to three significant figures.

A1.1.8 Compute g/2500 A and record as the "apparent viscosity constant at 2500 s^{-1}," K_{2500}, in cm·s^{-1}.

A1.2 *Recalibration:*

A1.2.1 Periodically determine several corrected fall times with one oil used for the original calibration.

A1.2.2 Calculate values for Wt_c and compare with those originally obtained for the oil.

A1.2.3 If the new values do not reasonably agree with the original ones, use the new fall time results to calculate a new value for the mean gap clearance $\bar{x}$. If $\bar{x}$ increases by less than 5 % of the original value, the existing calibration may be retained. If the increase is between 5 and 20 %, repeat the entire calibration process. If gap clearance increases by 20 % or more, replace the rod and aperture.

A2. GRAPHICAL SOLUTION OF VISCOSITY

A2.1 *Construction of Sliding Scale Calibration Graph:*

A2.1.1 On a sheet of 2 by 2 to 2 by 3 cycle logarithmic chart paper, label the 2-cycle scale "Corrected fall time, s." (See Fig. A2.1.) Add one zero to the second cycle. Mark position of F_{2500} (from A1.1.7) on time scale. If below 1.0 s, the position can be located using another sheet of log paper as a guide. Draw a horizontal line across the entire sheet of paper and label "2500 s^{-1}" at the far end.

A2.1.2 Along the 3-cycle axis, mark the start of the cycles as 100, 1000, and 10 000. Label the scale "Total weight, g" so as not to interfere with the line previously drawn for "2500 s^{-1}."

A2.1.3 Using the results from fall-time runs made with standard oils (A1.1.6.1) and found satisfactory in accordance with A1.1.6.4, plot corrected fall time versus total weight for each oil-day combination. Using the triangle, draw the best 45° line through the points so that each plot line intercepts the line labeled "2500 s^{-1}."

A2.1.4 From a second sheet of the same type of chart paper, cut a piece along the 3-cycle axis so that the resulting strip contains the scale plus one or two graph divisions. Add zeros to the scale so that the second cycle starts at 10 and the third at 100; if required for high-viscosity fluids, a fourth cycle starting at 1000 may be obtained by pasting two strips together. Label the strip "Viscosity at 25°C, P." Locate the 25°C viscosity of each calibrating oil on the scale and draw vertical lines to the top. If three oils were used, there should be three lines on the strip.

A2.1.5 Position the top of the strip directly beneath the 2500 s^{-1} line drawn on the first sheet of chart paper so that the viscosity lines on the strip match as closely as possible the intercepts at 2500 s^{-1}. Paste securely in place. Cut off portions of the strip that extend beyond the edges of the chart paper. Relabel "Total weight" scale if strip hides pertinent coordinates.

A2.1.6 The chart paper containing the viscosity strip represents the "Master Sliding-Scale Calibration Graph" for a specific instrument. Prepare an overlay containing the desired identification information and paste in a suitable place (see example in Fig. A2.1). Make sufficient copies of the master for normal use of the instrument. Be sure to preserve the original.

NOTE A2.1—Use a duplication procedure that reproduces copy exactly. Office copiers and "quick" printing methods are prone to provide distorted copy.

NOTE A2.2—The Master Sliding-Scale Calibration Graph is essentially similar to the Inmont-Lehman chart, which is also based on the power law but presumes that a shear rate of 2500 s^{-1} corresponds to a fall time of 1.0 s in all instruments. The charts issued by viscometer manufacturers are Cartesian (1/F versus W) and are based on Bingham.

A2.2 *Graphical Solution of Viscosity:*

A2.2.1 Make the fall-time run on the test sample according to Section 13. Compute the corrected mean fall times and the total weights according to A1.1.6.2 and A1.1.6.3.

A2.2.2 On a copy of the Master Sliding Scale Calibration Graph (A2.1), plot corrected fall time versus total weight for the test material.
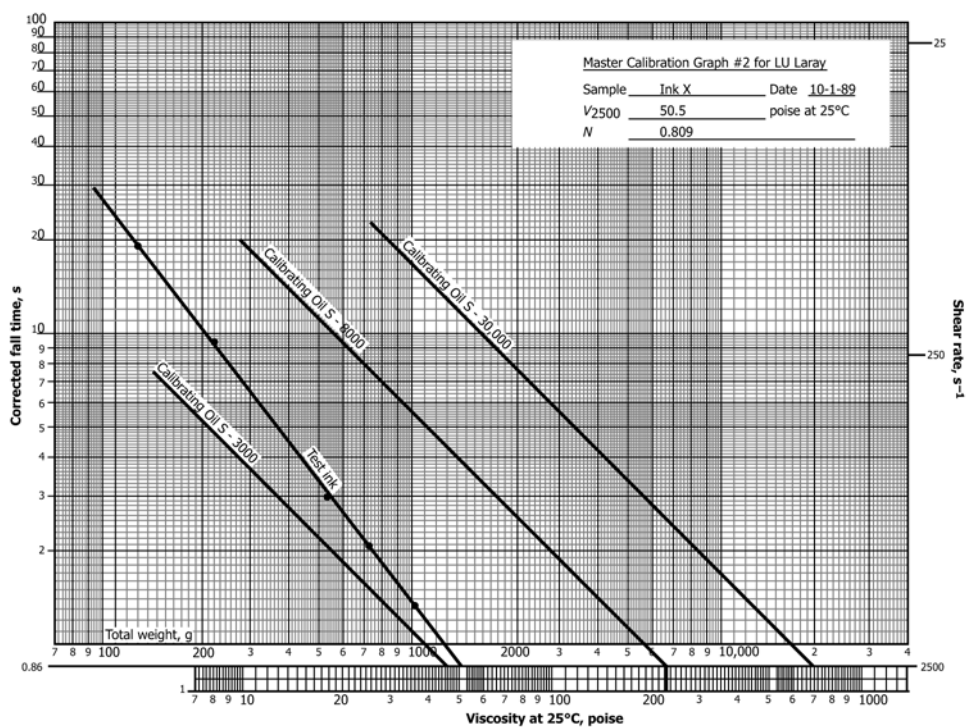

FIG. A2.1 Typical Sliding Scale Calibration Graph

A2.2.3 Using the 45° triangle as a guide, check whether the best straight line through the plotted points is 45° or greater to the weight axis. A line less than 45° is improbable for a printing ink or a vehicle and suggests error in the test measurements; check calculations or repeat the run.

A2.2.4 Draw the best straight line through the points, making certain that the angle is at least 45° to the weight axis. Extend the line so that it intersects the horizontal axis at 2500 s^{-1}. Read viscosity as the intercept on the sliding scale. Record as V_{2500}, apparent viscosity at 2500 s^{-1}.

A2.2.5 Center the protractor at the intersept and read the angle of the plot line. Subtract the measured angle from 90° and obtain the tangent from mathematical tables or a suitable calculator. Record as the non-Newtonianism parameter N.

NOTE A2.3—By turning Fig. A2.1 90° to the left, the graph can be viewed as a logarithmic plot of shear stress versus shear rate, and the shear thinning nature of the test ink made more evident. The tangent of the angle with respect to the time (shear rate) axis is the power law constant N.

A2.2.6 *Optional*—Calculate $V_{2.5}$, S'_o, and *SF* according to Eq 8 (see 14.6), Eq 10 (see 14.7) and Eq 12 (see 14.8).

APPENDIX

(Nonmandatory Information)

X1. DERIVATION OF THE PSEUDO YIELD VALUE

X1.1 According to the Bingham Model

$$S = S_o + V_{PL}D \qquad (X1.1)$$

where:
S = shear stress
S_o = shear stress yield value
V_{PL} = plastic viscosity
D = shear rate

X1.2 Apparent viscosity, V_D, is defined as:

$$V_D = \frac{S}{D} \qquad (X1.2)$$

X1.3 Combining the two equations gives

$$V_D = \frac{S_o}{D} + V_{PL} \qquad (X1.3)$$

X1.4 If two sets of data, $V_{2.5}$ and V_{2500} are available, one has two equations:

$$V_{2.5} = \frac{S_o}{2.5} + V_{PL} \qquad (X1.4)$$

$$V_{2500} = \frac{S_o}{2500} + V_{PL} \qquad (X1.5)$$

X1.5 Subtracting Eq X1.5 from Eq X1.4 gives

$$V_{2.5} - V_{2500} = \frac{S_o}{2.5} - \frac{S_o}{2500} \qquad (X1.6)$$

X1.6 Since $S_o/2500$ is 1000 times smaller than $S_{2.5}/2.5$, it can be deleted from Eq X1.6, giving

$$S_o = 2.5\,(V_{2.5} - V_{2500}) \qquad (X1.7)$$

NOTE X1.1—The import of Eq X1.7 is that it correctly gives a value of zero for Newtonian fluids.

X1.7 In Test Method D4040, we simply use S'_o instead of S_o and call it a pseudo yield value.

Originally derived by
Dr. Eugene Allen
Retired Professor of Chemistry
at Lehigh University
Independently derived by
Dr. Gary Poehlein
Former Professor of Chemical Engineering
at Lehigh University

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D4040 - 05) that may impact the use of this standard. (Approved February 1, 2010.)

(1) Addition of the "Appendix. Derivation of the Pseudo Yield Value" and a reference to this addition at the end of 14.7, Note 9.