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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Viscosity of Materials by a Falling Needle Viscometer[1]

This standard is issued under the fixed designation D 5478; 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 These test methods cover the measurement of the viscosity of Newtonian and non-Newtonian liquids. These test methods are applicable to liquids having viscosities in the range from 5×10^{-4} to 10^3 Pa·s (0.5 to 10^6 cP). The shear rate range is dependent upon the needle used and viscosity of the liquid and may vary from 10^{-4} to 10^3 s^{-1}.

1.2 The yield stress of liquids having this property may also be determined.

1.3 These test methods consist of determining liquid viscosities of Newtonian and non-Newtonian fluids (clear or opaque) by measuring the steady-state (constant) or terminal velocities of cylindrical needles as they fall through the test liquid under the influence of gravity. Yield stresses of non-Newtonian liquids may be measured using the same procedure.

1.4 *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:*

E 1 Specification for ASTM Thermometers[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *dilatant fluid (or shear thickening fluid)*— one in which the apparent viscosity increases with increasing shear rate.

3.1.2 *Newtonian and non-Newtonian fluids*—A Newtonian fluid is one in which the dynamic viscosity does not vary with shear rate but only with the temperature and pressure. A non-Newtonian fluid is one in which the dynamic viscosity varies with shear rate over at least some shear rate range.

3.1.2.1 *Discussion*—This viscosity is sometimes referred to as the "apparent viscosity" since it is not a true property of the fluid but a variable depending on the shear rate. The viscosity of most non-Newtonian fluids fits a power law expression. A power law fluid is defined by the following equation:

$$\eta_a = K(\dot{\gamma})^{n-1} \quad (1)$$

where:

η_a = apparent viscosity, dyne · s/cm^2 = P = 100 cP,
K = fluid consistency, dyne·s^n/cm^2,
$\dot{\gamma}$ = shear rate, 1/s, and
n = flow index, dimensionless.

3.1.3 *pseudoplastic fluid (or shear thinning fluid)*—one in which the apparent viscosity decreases with increasing shear rate.

3.1.4 *viscosity*—The ratio between an applied shear stress to the resulting shear rate (velocity gradient) is defined as the dynamic viscosity. It is a measure of the resistance to flow of a fluid.

3.1.4.1 *Discussion*—In the SI unit system the units of viscosity are Pa·s. One mPa·s is equal to one centipoise (cP).

3.1.5 *yield stress*—Some fluids when subjected to a shear stress behave as deformable solids until a certain critical shear stress is reached after which they behave as fluids. This critical shear stress is called the yield stress or yield value.

3.1.5.1 *Discussion*—Examples of such fluids include many paints and pigment pastes and certain food materials, for example, ketchup.

4. Summary of Test Methods

4.1 Test Method A consists of determining the viscosity of Newtonian liquids.

4.2 Test Method B consists of determining the apparent viscosity and shear rate of pseudoplastic and dilatant fluids in the power law region.

4.3 Test Method C consists of determining the apparent viscosity and shear rate of pseudoplastic and dilatant fluids outside of the power law region.

4.4 Test Method D consists of determining the yield stress of liquids that have such a property.

5. Significance and Use

5.1 These test methods are applicable for measuring the rheological properties of varnishes and paints. In particular, the low to moderate shear rate measurements provide information related to sag resistance, leveling, etc.

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[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

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[2] *Annual Book of ASTM Standards*, Vol 14.03.

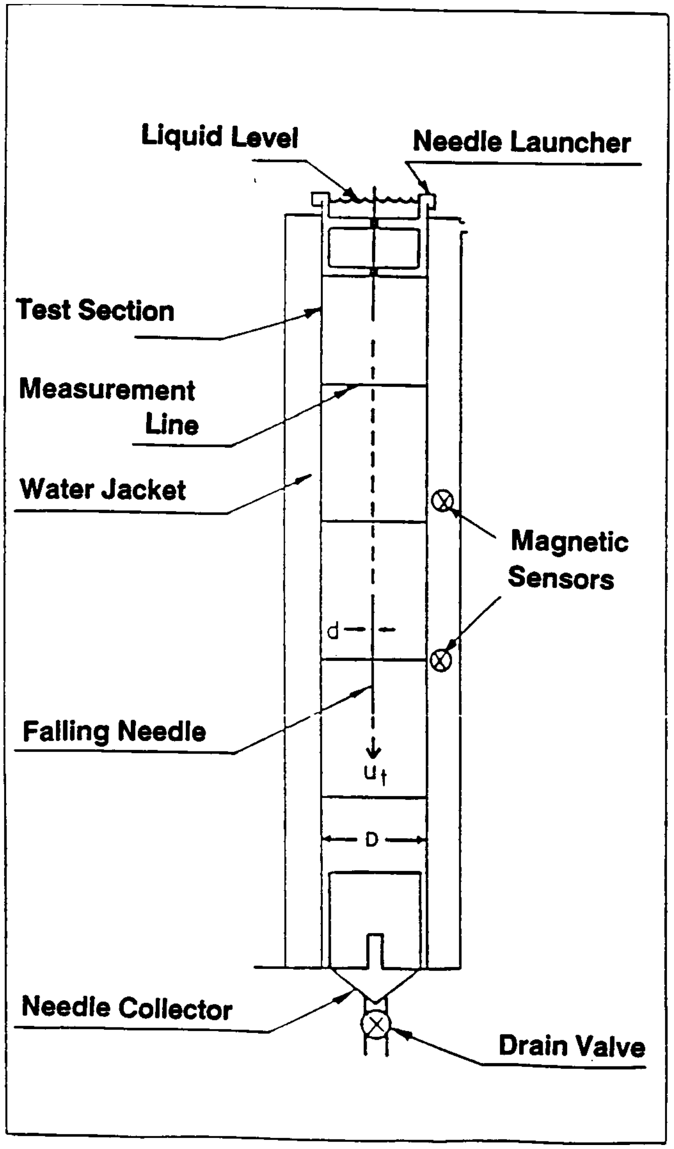

FIG. 1 Schematic of Falling Needle Viscometer

6. Apparatus

6.1 *Viscometer*, falling-needle-type and associated equipment listed as follows:

6.1.1 *Falling Needle Viscometer* [3,4,5]—A schematic of the falling needle viscometer is shown in Fig. 1. The viscometer consists of a vertical cylindrical test section of diameter *D*. The liquid specimen is placed in the test section and the specimen's temperature is maintained constant by means of a constant temperature bath that circulates a liquid through another cylindrical container (water jacket) that is coaxial to the test section. A thin hollow cylinder of length *L* with hemispherical ends and diameter *d* (the needle) is aligned with the axis of the test section and allowed to fall under the influence of gravity. The needle has a small weight in its forward end that may be varied to change its density. After the needle has attained its constant terminal velocity, this velocity is measured by determining the needle transit time between two circumferential marks a known distance apart on the test section (for opaque liquids this can be done by an automatic sensing device, such as a magnetic sensor, etc.). With a knowledge of the terminal velocity, the liquid and needle densities, the geometric constants of the system (*L, D, d*), the viscosity of a Newtonian fluid can be calculated from the instrument theory. For a non-Newtonian fluid whose viscosity depends upon the shear rate, a series of needles are dropped. The falling needle is an absolute method of viscosity measurement that does not need any instrument calibration. However, it may be checked through use of known certified viscous fluids such as standard oils.

6.1.2 *Thermometer*—A thermometric device calibrated to 0.1°C whose accuracy, precision, and sensitivity are equal to or better than the ASTM thermometer described in Specification E 1.

6.1.3 *Circulating Liquid Bath*, capable of maintaining the test specimen temperature to ±0.1°C.

6.1.4 *Stopwatch or Electronic Device*, capable of measuring to ±0.01 s or an automatic sensing device with the same accuracy.

7. Preparation of Specimen

7.1 After opening the specimen container, mix the fluid gently with a glass rod for 5 min.

7.2 Pour the specimen carefully into the test section so as to minimize the formation of air bubbles. If available, a syringe is useful for this purpose.

7.3 Remix the specimen in the test container using the needle retriever rod by pushing it up and down four times at a velocity of approximately 4 cm/s.

7.4 Allow the specimen to remain at rest in the test section for a minimum of 5 min or until any air bubbles have risen to the surface. Longer rest times may be used in the case of yield stress measurements.

TEST METHOD A—NEWTONIAN FLUIDS VISCOSITY MEASUREMENTS

8. Procedure

8.1 Level the viscometer so that the central vertical axis of the test section is parallel to the gravity vector by using either a bubble level or a plumb bob.

8.2 Circulate the liquid from the constant temperature bath until the test specimen temperature is constant at the specified value with a variation of ±0.1°C.

8.3 To determine the viscosity, drop a needle along the central axis of the test section and measure its velocity by the

[3] Park, N. A., and Irvine, T. F., Jr., "Measurements of Rheological Fluid Properties with the Falling Needle Viscometer," *Review of Scientific Instruments*, Vol 59, 1988, pp. 2051–2058.

[4] Park, N. A., and Irvine, T. F., Jr., "The Falling Needle Viscometer, A New Technique for Viscosity Measurements," *American Laboratory*, Vol 20, November 1988, pp. 57–63.

[5] "The sole source of supply of the falling needle viscometer known to the committee at this time is Stony Brook Scientific, Ltd., P.O. Box 147, 914 Filmore Rd., Norristown, PA 19403. 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." This instrument may be interfaced with a computer for data collection and analysis. A computer program is available for data analysis for instruments that are not interfaced.

amount of time taken to move between two of the measurement lines. This may be done by using a stopwatch or an automatic sensing device. The measurement lines should be at least a test section diameter from the top and bottom of the liquid.

8.4 Record the values of the needle velocity, the liquid and needle densities, the test specimen temperature, the local acceleration of gravity and the test section, and needle dimensions *D*, *L*, and *d*.

8.5 Drop additional needles of different densities to establish whether the fluid is Newtonian. If the measured viscosity is essentially constant using the different density needles, then the fluid is Newtonian.

9. Calculation

9.1 Calculate the Newtonian fluid viscosity for any needle drop as follows:

$$\eta = \frac{g(\rho_s - \rho_l)}{U_t G} \quad (2)$$

where:

η = dynamic viscosity, P = 100 cP,
g = local acceleration of gravity, cm/s^2,
ρ_s = needle density, g/cm^3,
ρ_l = test specimen density, g/cm^3,
U_t = measured needle terminal velocity, cm/s, and
G = geometric constant depending upon the test section and needle dimensions *D*, *L*, and *d* that is furnished by the instrument manufacturer. Table 1 lists several typical geometric constants.

10. Report

10.1 Report the following information:

10.1.1 Name of the test specimen,

10.1.2 Temperature of the test specimen, °C, and

10.1.3 Viscosity of the test specimen, cP (Note 1).

NOTE 1—If the same needle is dropped more than once, report the minimum, maximum, and average viscosity values. If needles of different densities are dropped, report the individual viscosity measurements.

11. Precision and Bias

11.1 *Precision*—In an interlaboratory study, six operators in six laboratories measured (four replicates) viscosities of three Newtonian oils and one essentially Newtonian spar varnish. These materials covered a viscosity range of 100 to 1440 mPa.s (cP). The within-laboratory coefficient of variation was found to be 2.70 or 0.5 % of the average viscosity. The corresponding between-laboratories coefficient was 4.58 or 0.9 % of the average viscosity. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results of individual viscosity measurements obtained by the same operator at different times should be considered suspect if they differ by more than 1.4 % relative.

11.1.2 *Reproducibility*—Two results of individual viscosity measurements obtained by operators in different laboratories should be considered suspect if they differ by more than 2.4 % relative.

11.2 *Bias*—Bias has not been determined for this test method.

TABLE 1 Geometric Constants (*G*) for Several System Diameters[A] (*D*) and Needle Lengths (*L*)

System Diameter, cm	Needle Length, cm	*G*, 1/cm^2	Viscosity Range, cP
1.905	10.2	80.89	50–10^6
0.8044	4.2	529.8	10–10^4
0.4996	4.2	12,816	0.5–20

[A] Needle diameter = 0.3980 cm.

TEST METHOD B—APPARENT VISCOSITY AND SHEAR RATE OF PSEUDOPLASTIC AND DILATANT FLUIDS IN POWER LAW REGIONS

12. Procedure

12.1 Follow the procedures in accordance with 8.1 and 8.2.

12.2 Drop a series of needles of the same geometry but different densities along the central axis of the test section and measure their velocities by the amount of time taken to travel between two of the measurement lines. This may be done by using a stopwatch or an automatic sensing device. The measurement lines should be at least a test section diameter from the top and bottom of the liquid.

12.3 Record the values of the needle velocities, needle densities, the test specimen temperature, the local acceleration of gravity and the test section, and needle dimensions *D*, *L*, and *d*.

13. Calculation

13.1 Plot a graph of $\text{Log}_e(\rho_s - \rho_l)$ versus $\text{Log}_e U_t$. If the points form a straight line then the shear rate is in the power law region (see section 13.3) and the slope of the straight line is the flow index, *n*.

13.2 Calculate the shear rate $\dot{\gamma}$ from the following equation:

$$\text{Log}_e(\gamma/U_t) = A_1 + B_1 \text{Log}_e n + C_1 (\text{Log}_e n)^2 + D_1 (\text{Log}_e n)^3 + E_1 (\text{Log}_e n)^4 + F_1 (\text{Log}_e n)^5 \quad (3)$$

where the values of A_1, B_1, C_1, D_1, E_1, and F_1 are given in Table 2 for the same representative systems and needles as in Table 1.

13.3 Calculate the apparent viscosity from the following equation:

$$\text{Log}_e\left[\frac{\eta_a U_t}{g(\rho_s - \rho_l)}\right] = A_2 + B_2 \text{Log}_e n + C_2 (\text{Log}_e n)^2 \quad (4)$$

$$+ D_2 (\text{Log}_e n)^3 + E_2 (\text{Log}_e n)^4 + F_2 (\text{Log}_e n)^5 \quad (5)$$

where the values of A_2, B_2, C_2, D_2, E_2, and F_2 are given in Table 2 for the same representative systems and needles as in Table 1.

14. Report

14.1 Report the following information:

14.1.1 Name of the test specimen,

14.1.2 Temperature of the test specimen during the needle drops, °C,

14.1.3 Graph of $\text{Log}_e(\rho_s - \rho_l)$ versus $\text{Log}_e U_t$ and the value of *n* as determined by the slope,

14.1.4 Calculated values of η_a and $\dot{\gamma}$, cP, and 1/s respectively in accordance with 13.2 and 13.3, and

TABLE 2 Values of the Constants for Calculating $\dot{\gamma}$ and η_a of Pseudoplastic and Dilatant Fluids[A]

D[B]	L[C]	A_1	B_1	C_1	D_1	E_1	F_1
1.905	10.2	1.997959	(−)0.7730782	0.2205017	3.775071×10^{-3}	(−)1.947361×10^{-2}	(−)4.960324×10^{-3}
0.8044	4.2	3.465018	(−)0.5415453	0.1626492	(−)5.57856×10^{-3}	(−)1.032115×10^{-2}	(−)1.852084×10^{-3}
0.4996	4.2	5.653115	(−)0.3778035	0.1115236	(−)3.95266×10^{-3}	(−)6.785062×10^{-3}	2.911597×10^{-5}
D	L	A_2	B_2	C_2	D_2	E_2	F_2
1.905	10.2	(−)4.393038	0.7893092	(−)0.2289874	(−)1.754509×10^{-4}	2.037374×10^{-2}	4.745022×10^{-3}
0.8044	4.2	(−)6.272388	0.5850253	(−)0.1755108	4.676713×10^{-3}	1.165808×10^{-2}	1.973054×10^{-3}
0.4996	4.2	(−)9.458938	0.4070585	(−)0.1172265	2.299561×10^{-3}	7.390045×10^{-3}	1.161545×10^{-5}

[A] Needle diameter = 0.3980 cm.
[B] D—System diameter, cm.
[C] L—Needle length, cm.

14.1.5 Flow curve (graph) of apparent viscosity η_a versus $\dot{\gamma}$ in Log-Log coordinates including the temperature and test specimen information.

15. Precision and Bias

15.1 *Precision*—In an interlaboratory study, six operators in six laboratories measured (four replicates) viscosities of four materials comprising a spar varnish, a rust inhibitive primer, a latex semigloss, and an alkyd gloss enamel, that covered a reasonable range of viscosities. The varnish was very slightly dilatant (shear thickening) and the paints all were shear thinning. Measurements were taken with at least three needles in each case and as many as six (equivalent to six operators testing 18 paints). Because Test Method B does not require single point viscosities, but rather viscosity-shear rate curves, and slight differences in specified needles give different shear rates and, therefore, viscosities, it was decided not to try to determine precision on the basis of individual viscosities. We were not comfortable with trying to determine the precision of curves either, so we decided to normalize the values obtained by multiplying the viscosities by the shear rates to produce shear stresses and use these to determine the precision. The within-laboratory coefficient of variation was found to be 0.31 or 0.2 % of the average shear stress. The between-laboratories coefficient was 3.22 or 2.2 % of the average shear stress. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

15.1.1 *Repeatability*—Two shear stress results calculated from shear rates and viscosities measured by the same operator at different times should be considered suspect if they differ by more than 0.6 % relative.

15.1.2 *Reproducibility*—Two shear stress results calculated from shear rates and viscosities measured by operators in different laboratories should be considered suspect if they differ by more than 6.1 % relative.

15.2 *Bias*—Bias has not been determined for this test method.

TEST METHOD C—APPARENT VISCOSITY AND SHEAR RATE OF PSEUDOPLASTIC OR DILATANT FLUIDS OUTSIDE OF THE POWER LAW REGION

16. Procedure

16.1 Follow the procedures in accordance with 12.1, 12.2, and 12.3.

17. Calculation

17.1 Plot a graph of $\text{Log}_e(\rho_s - \rho_l)$ versus $\text{Log}_e U_t$. If the points do not form a straight line, the shear rate is outside the power law region.

17.2 The local value of n at any value of U_t is equal to the slope of the curve at that value of U_t. Determine the slope either graphically or by a least squares curve fit and differentiation.

17.3 Using this local value of n, calculate η_a and $\dot{\gamma}$ in accordance with 13.2 and 13.3.

18. Report

18.1 Report the following information:

18.1.1 Name of the test specimen,

18.1.2 Temperature of the test specimen during the needle drops, °C,

18.1.3 Graph of $\text{Log}_e(\rho_s - \rho_l)$ versus $\text{Log}_e U_t$,

18.1.4 Local value of n at each value of U_t,

18.1.5 Calculated values of η_a and $\dot{\gamma}$, cP, and 1/s respectively in accordance with 13.2 and 13.3, and

18.1.6 Flow curve graph of apparent viscosity η_a versus $\dot{\gamma}$ in Log-Log coordinates including the temperature and test specimen information.

19. Precision and Bias

19.1 *Precision*—Precision has not been determined for Test Method C, but would be expected to be similar to that of Test Method B.

19.2 *Bias*—Bias has not been determined for this test method.

TEST METHOD D—YIELD STRESS DETERMINATION

20. Procedure

20.1 Follow the procedures in accordance with 14.1, 14.2, and 14.3.

21. Calculation

21.1 Plot a graph of $(\rho_s - \rho_l)$ versus U_t (Note 2).

NOTE 2—Choose needle densities close to the test specimen density to attain the lowest values of U_t.

21.2 Extrapolate the curve to $U_t = 0$ and determine $(\rho_s - \rho_l)$ at $U_t = 0$. This is indicated by $(\rho_s - \rho_l)_0$. If $(\rho_s - \rho_l)_0$ exists, the fluid has a yield stress. The extrapolation process may either be made graphically or analytically by a least squares curve fit.

21.3 Calculate the yield stress from the following equation:

$$\tau_y = \frac{gd(\rho_s - \rho_l)_0\,(1 + 2/(3L^+))}{4 + \pi/L^+} \tag{6}$$

where:

τ_y = yield stress, dyne/cm^2,
g = local acceleration of gravity, cm/s^2,
d = needle diameter, cm,
ρ_s = needle density, g/cm^3,
ρ_l = test specimen, density, g/cm^3, and
L^+ = dimensionless needle length, $((L - d)/d)$).

22. Report

22.1 Report the following information:

22.1.1 Name of the test specimen,

22.1.2 Temperature of the specimen during the needle drops,° C,

22.1.3 Graph of $(\rho_s - \rho_l)$ versus U_t showing the extrapolation line to $U_t = 0$, and

22.1.4 Value of the yield stress τ_y, dyne/cm^2.

23. Precision and Bias

23.1 *Precision*—No attempt has been made to determine the precision of the measurement of yield stress because this parameter is dependent on the material, its shear history, and other variables.

23.2 *Bias*—Bias has not been determined for this test method.

24. Keywords

24.1 falling needle viscometer; Newtonian fluid; non-Newtonian fluid; rheological property; rheometer; viscometer; viscosity; yield stress