



Standard Test Methods for Viscosity of Materials by a Falling Needle Viscometer¹

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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} Pa·s to 10^3 Pa·s (0.5 cP 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} s⁻¹ to 10^3 s⁻¹. With an extension bar and applied weight, a shear rate of 10^4 s⁻¹ may be achieved.

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

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

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2. Referenced Documents

2.1 *ASTM Standards:*²

E1 Specification for ASTM Liquid-in-Glass Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions:*

3.1.1 *dilatant or shear thickening fluid, n*—fluid in which the apparent viscosity increases with increasing shear rate.

3.1.2 *Newtonian fluid, n*—fluid in which the dynamic viscosity does not vary with shear rate but only with the temperature and pressure.

3.1.3 *Non-Newtonian fluid, n*—fluid in which the dynamic viscosity varies with shear rate over at least some shear rate range.

3.1.3.1 *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:

$$\eta_a = K(dy/dt)^{n-1} \quad (1)$$

where:

η_a = apparent viscosity, Pa·s (or dyne·s/cm² = P), mPa·s = cP,

K = fluid consistency, Pa·sⁿ (or dyne·sⁿ/cm²),

dy/dt = shear rate or velocity gradient, 1/s, and

n = flow behavior index, dimensionless.

3.1.4 *pseudoplastic or shear thinning fluid, n*—fluid in which the apparent viscosity decreases with increasing shear rate.

3.1.5 *viscosity, n*—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.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

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

3.1.6 *yield stress, n*—some fluids when subjected to a shear stress behave as deformable solids until a certain critical shear stress (yield stress or yield value) is reached after which they behave as fluids.

3.1.6.1 *Discussion*—Examples of such fluids include many paints and pigment pastes and certain food materials such as 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.

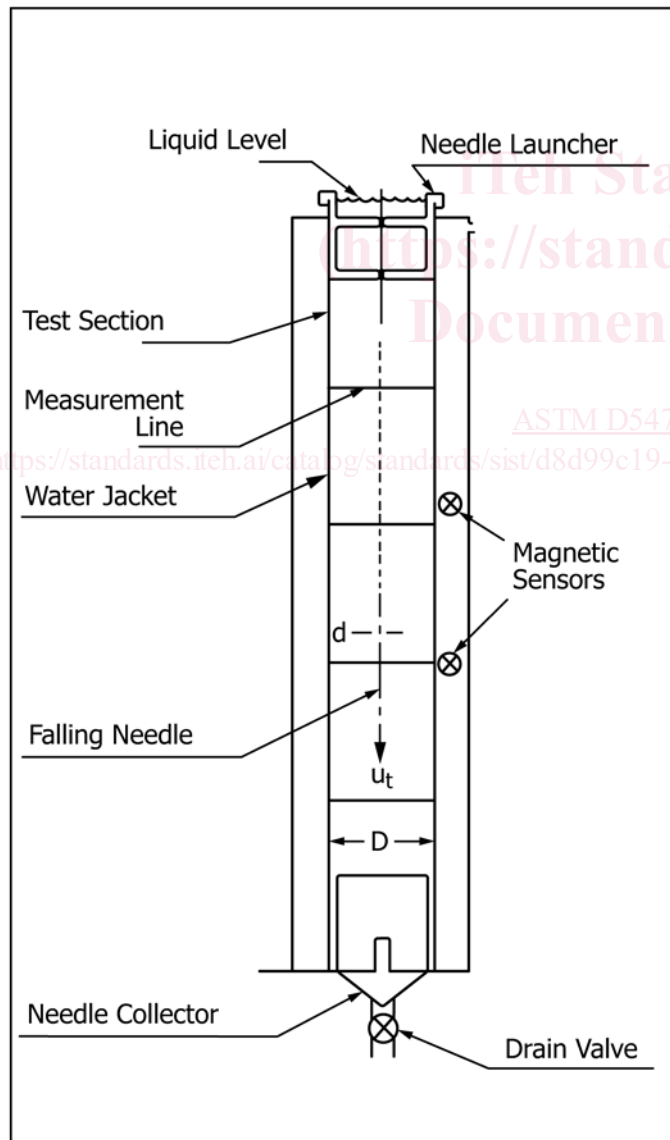


FIG. 1 Schematic of Falling Needle Viscometer

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.

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. Another type of needle is connected at the top with an extension bar and a weight holder so that external weights may be added to increase the effective density of the needle and the maximum achievable shear rate. With any needle, terminal 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

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., 914 Fillmore Rd., Norristown, PA 19403. If you are aware of alternative suppliers, please provide this information to ASTM International 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.

or better than the ASTM thermometers described in Specifications **E1** or **E2251**. The use of non-mercury thermometers or other devices is highly recommended.

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 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:

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

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, mPa·s (= 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.

where:

η = dynamic viscosity, mPa·s (= cP),
 g = local acceleration of gravity, cm/s²,
 ρ_s = needle density, g/cm³,
 ρ_l = test specimen density, g/cm³,
 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, mPa·s (= 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 mPa·s (cP) 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.

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**.