Designation: D4486 – 10

Standard Test Method for Kinematic Viscosity of Volatile and Reactive Liquids[1]

This standard is issued under the fixed designation D4486; 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 measurement of kinematic viscosity of transparent, Newtonian liquids which because of their reactivity, instability, or volatility cannot be used in conventional capillary kinematic viscometers. This test method is applicable up to 2×10^{-5} N/m^2 (2 atm) pressure and temperature range from −53 to +135°C (−65 to +275°F).

1.1.1 For the measurement of the kinematic viscosity of other liquids, see Test Method D445.

1.2 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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 warning statements, see 7.2, 7.3, 7.4, and Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *density*—the mass per unit volume of the liquid.

3.1.1.1 *Discussion*—The cgs unit of density (ρ) has the dimensions of grams per cubic centimetre. The SI unit of density has the dimensions of kilograms per cubic metre.

3.1.2 *kinematic viscosity*—The ratio of the viscosity to the density of the liquid.

3.1.2.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density ρ. For any particular viscometer, the time of flow of a fixed volume of liquid is directly proportional to η/ρ. This ratio is the kinematic viscosity coefficient (ν). The cgs unit of kinematic viscosity is the stoke and has the dimensions of centimetre squared per second: the centistoke (0.01 St) is frequently used. The SI unit of kinematic viscosity has the dimensions of metre^2/second, and is equivalent to 10^4 St.

3.1.3 *viscosity*—the ratio between the applied shear stress and rate of shear.

3.1.3.1 *Discussion*—This ratio is called the coefficient of viscosity. The coefficient of viscosity (η) is thus a measure of the resistance to flow of the liquid. This is commonly called the viscosity of the liquid. The cgs unit of viscosity is the poise. P, which has the dimensions of dyne-seconds per square centimetre: the centipoise (0.01 poise) is frequently used. The SI unit of viscosity has the dimensions of newton second/metre^2, and is equivalent to 10 P.

3.1.4 *vulnerable liquid*—a liquid which by reason of its volatility, instability or reactivity in the presence of air or any other specific gaseous medium may undergo physical or chemical changes that may affect its viscosity.

4. Summary of Test Method

4.1 The time is measured, in seconds, for a fixed volume of liquid to flow under gravity through the capillary of the viscometer under a reproducible driving head and at a closely

[1] This test method is under the jurisdiction of Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.L0.07 on Engineering Sciences of High Performance Fluids and Solids (Formally D02.1100).

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[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.

TABLE 1 Approximate Values of the ASTM Viscosity Standards

Viscosity Standard Conforming to ASTM Standards[A]	Approximate Kinematic Viscosity, cSt								
	At −53.89°C (−65°F)	At – 40°C (−40°F)	At 20°C (68°F)	At 25°C (77°F)	At[B] 37.78°C (100°F)	At 40°C (104°F)	At 50°C (122°F)	At[B] 98.89°C (210°F)	At 100°C (212°F)
S-3	300	80	4.6	4.0	3.0	2.9	...	1.2	1.2
S-6	...	...	11	8.9	6.0	5.7	...	1.8	1.8
S-20	...	...	44	34	20	18	...	4.0	3.9
S-60	...	...	170	120	60	54	...	7.4	7.2
S-200	...	...	640	450	200	180	...	17	17
S-600	...	...	2400	1600	600	520	280	33	32
S-2000	...	...	8700	5600	2000	1700	...	78	75
S-8000	...	...	37 000	23 000	8000	6700	...	...	...
S-30000	...	...	...	81 000	27 000	23 000	11 000	...	...

[A] The actual values for the standards listed above are established and annually reaffirmed by cooperative tests. In 1971, tests were made using 15 different types of viscometers in 26 laboratories located in 9 countries.
[B] Standardizations at 37.78 °C and 98.89 °C are to be discontinued Jan 1, 1977.

controlled temperature. The kinematic viscosity is calculated from the measured flow time and the calibration constant of the viscometer.

5. Significance and Use

5.1 Kinematic viscosity is a physical property which is of importance in the design of systems in which flowing liquids are used or handled.

6. Apparatus

6.1 *Viscometer Thermostat*—Any transparent liquid or vapor bath of sufficient depth such that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath may be used. The temperature control must be such that for the range from 15 to 100 °C (60 to 212 °F) the temperature of the bath medium does not vary by more than 0.02 °F (0.01 °C) over the length of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the variation must not exceed 0.05 °F (0.03 °C).

6.2 *Temperature Measuring Device*—A resistance thermometer (RTD) capable of measurement to ± 0.01ºC (0.02ºF) is the preferred device for temperature measurement. The use of suitable liquid-in-glass Kinematic Viscosity Test Thermometers covering the range of test temperatures indicated in Table 1 as listed in Specification E1, is permitted provided they have been standardized before use (see 8.2). The use of an RTD is preferred because the thermometers listed in Specification E1 contain mercury.

6.3 *Timing Device*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 s or better, and that it has an accuracy within ±0.07 % when tested over intervals of 15 min.

6.3.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

7. Reagents and Materials

7.1 *Viscosity Oil Standards,* conforming to ASTM viscosity oil standards having the approximate kinematic viscosity shown in Table 1. Certified kinematic viscosity values are compared by annual cooperative tests by a number of laboratories and are supplied with each portion.

7.2 *Chromic Acid (Cleaning Solution)*—(**Warning—**Causes severe burns. A recognized carcinogen. Strong oxidizer, contact with organic material may cause fire. Hygroscopic. See A1.2.)

7.2.1 Other suitable cleaning solutions[3] are available. In referee testing situations, glassware shall be cleaned with a cleaning solution agreed upon by the parties involved.

7.3 *Acetone*—(**Warning—**Extremely flammable. Vapors may cause flash fire. See Annex A1.3.)

7.4 *Hydrochloric Acid (Concentrated)*—(**Warning—** Poison. Corrosive. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled. See Annex A1.4.)

8. Standardization

8.1 *Viscometers*—Only calibrated viscometers standardized as described in Annex A2 shall be used.

8.2 *Temperature*—Temperature measuring devices shall be checked to the nearest 0.01ºC (0.02ºF) by comparison to a suitable standardized instrument. Liquid-in-glass thermometers shall be standardized at "total immersion," which means immersion to the top of the liquid column with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature; do not submerge the expansion bulb at the top of the thermometer. It is essential that the ice point of the standardized thermometers be determined periodically and the official corrections be adjusted to reflect the change in the ice point.

8.3 *Timers*—Standard time signals available in some nations may be used in checking the accuracy of timing devices. In the

[3] Other suitable chromium free, sulfuric acid-based cleaning solutions are available.

United States of America, time signals, as broadcast by the National Bureau of Standards, Station WWV, Washington, DC 20234, at 2.5, 5, 10, 15, 20, 25, 30, and 35 MHz are a convenient and primary standard reference for calibrating timing devices; the signals are broadcast 24 h daily. Station CHU from Ottawa, Canada, at 3.330, 7.335, and 14.670 MHz or Station MSF at Rugby, United Kingdom, at 2.5, 5, and 10 MHz may be received better in some locations.

8.4 Viscosity standards may also be used to check the over-all kinematic viscosity procedure in a laboratory. If the measured kinematic viscosity does not agree within ±0.35 % of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration to locate source of error.

9. Cleaning of Viscometer

9.1 Between successive determinations, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the viscometer with vacuum attached to Tube *A* or by placing viscometer in a vacuum oven.

9.2 Periodically clean the instrument with chromic acid (**Warning—**See 7.2 and A1.2.) to remove organic deposits, rinse thoroughly with distilled water and acetone (**Warning—** See 7.3 and A1.3.), and dry with clean dry air. Inorganic deposits may be removed by hydrochloric acid (**Warning—** See 7.4 and A1.4.) treatment before use of cleaning acid, particularly if barium salts are suspected.

NOTE 1—Do not allow chromic acid or hydrochloric acid to stand in contact with the Kovar fitting on the viscometer. Use a glass pipet to introduce these acids into the viscometer in such a manner that contact with the metal fittings is kept to an absolute minimum.

NOTE 2—Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, should be reserved for the exclusive use of those fluids except when standardizing. Such viscometers should be subjected to standardization checks at frequent intervals.

10. Procedure for Kinematic Viscosity

10.1 Maintain the bath at the test temperature within the limits given in 6.1. Apply the necessary corrections, if any, to all thermometer readings.

10.2 Select a clean, dry, calibrated viscometer that will give a flow time not less than the minimum specified for the viscometer (see Table 2), or 200 s, whichever is the greater.

10.3 Charge the viscometer through Tube *A* (see Fig. 1) until Bulb *B* is half filled.

10.4 Test samples that are not stable in the presence of air at the test temperature must have the air in the viscometer purged by a working gas that does not react with the test sample.

10.4.1 For the vulnerable liquid viscometer (Fig. 1), attach Tube *A* to a controlled source of a working gas. Tilt the charged viscometer until the liquid sample no longer covers the end of Tube *C*. Pressure purge the viscometer with working gas. Release the pressure and repeat the purge at least four times.

10.4.2 Multiple purges are not required in the case of vulnerable samples that are volatile but stable in the presence of air at the test temperature.

TABLE 2 Dimensions for Vulnerable Liquid and Tilting Viscometers

Size No.	Approximate Constant, cSt/s	Viscosity Range, cSt	Inside Diameter of Tube D, mm (±2 %)		Volume Bulb F mL (±5%)	
			Liquid	Tilting	Liquid	Tilting
25	0.002	0.5[A] to 2	0.31	0.31	1.5	1.5
50	0.004	0.8 to 4	0.44	0.37	3.0	1.5
75	0.008	1.6 to 8	0.54	0.46	3.0	1.5
100	0.015	3 to 15	0.63	0.52	3.0	1.5
150	0.035	7 to 35	0.78	0.65	3.0	1.5
200	0.1	20 to 100	1.01	0.84	3.0	1.5
300	0.25	50 to 250	1.26	1.05	3.0	1.5

[A] 250 s minimum flow time.

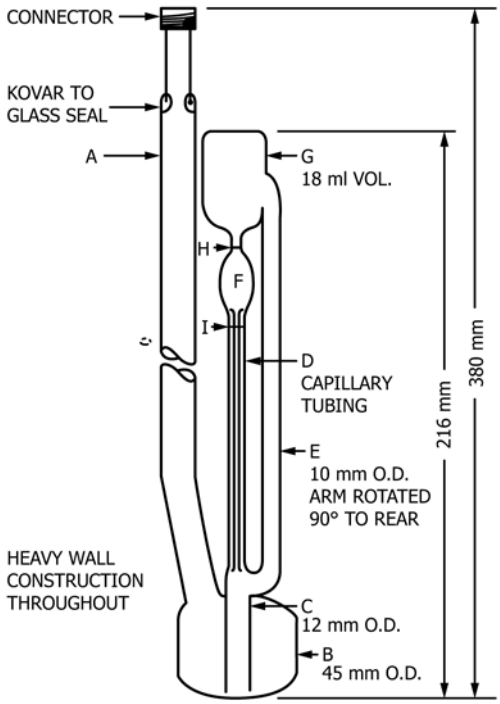

FIG. 1 Viscometer for Vulnerable Liquids

10.5 *Adjustment of Pressure:*

10.5.1 In the vulnerable-liquid viscometer, adjust the pressure in the viscometer to the predetermined test pressure.This pressure may be equal to the ambient in the case of reactive samples or it may be an elevated pressure sufficient to repress the boiling of a volatile sample. In any case the pressure inside the viscometer must not be permitted to exceed 2×10^{-5} kPa (2 atm).

10.6 Mount the viscometer in the viscometer holder, and place assembly in the constant-temperature bath. Ensure that the Working Capillary *D* is held vertical. Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time may vary with the viscosity of the sample and test temperature, establish a safe temperature equilibrium time by trial.

10.7 Fill the timing Bulb *F* with sample as follows: Increase the pressure in the viscometer by an amount sufficient to force the sample upward through Tubes *D* and *E* to fill Bulb *F*

entirely and Bulb *G* partially. Since the inside diameter of Tube *E* is greater than that of Tube *D*, Bulb *F* is filled mainly by liquid that flows through Tube *E* and Bulb *G*. Restore the pressure in the system to the prechecked test pressure. As this is done the sample begins to drain from the capillary and upper bulbs of the viscometer. Tubes *E* and *C* are sufficiently large in a diameter that they drain completely before the liquid level in Bulb *G* reaches the upper timing mark.

10.8 With the sample flowing freely, measure in seconds, to within 0.2 s (see 6.3), the time required for the meniscus to pass from the first timing mark to the second. If this flow time is less than the specified minimum (see 10.2) select a viscometer with a capillary of smaller diameter and repeat the operation.

10.9 Repeat the procedure described in 10.7 and 10.8 to obtain a second determination of the flow time. If the two measurements agree with 0.4 % use the average for calculating the reported kinematic viscosity. If the required agreement is not obtained, reject the results and repeat the test.

10.10 Clean the viscometer as described in Section 9.

11. Calculation and Report

11.1 Calculate the kinematic viscosity as follows:

$$\text{Kinematic Viscosity, cSt} = Ct \quad (1)$$

where:

C = calibration constant of the viscometer, cSt/s, and
t = flow time, s.

11.2 Report both the kinematic viscosity and the temperature of test.

12. Precision and Bias

12.1 The precision and bias are expected to be substantially identical to that given in Test Method D445 because the functional part of the viscometer in this test method is identical to the Ubbelohde viscometer specified in Test Method D445.

13. Keywords

13.1 kinematic viscosity; reactive liquid; viscosity; volatile liquid

ANNEXES

(Mandatory Information)

A1. WARNING STATEMENTS

A1.1 Compressed Gases (Helium, Nitrogen)

A1.1.1 **Warning—**Compressed gas under high pressure. Gas reduces oxygen available for breathing.

Keep cylinder valve closed when not in use.

Use with adequate ventilation.

Do not enter storage areas unless adequately ventilated.

Always use a pressure regulator. Release regulator tension before opening cylinder.

Do not transfer to cylinder other than one in which gas is received.

Do not mix gases in cylinders.

Never drop cylinder. Make sure cylinder is supported at all times.

Stand away from cylinder outlet when opening cylinder valve.

Keep cylinder out of sun and away from heat.

Keep cylinder from corrosive environment.

Do not use cylinder without label.

Do not use dented or damaged cylinders.

For technical use only. Do not use for inhalation purposes.

A1.2 Chromic Acid (Cleaning Solution)

A1.2.1 **Warning—**Causes severe burns. A recognized carcinogen. Strong oxidizer, contact with organic material may cause fire. Hygroscopic.

Do not get in eyes, on skin, or on clothing.

Avoid breathing vapor or mist.

Keep container closed.

Use with adequate ventilation.

Do not take internally.

Wash thoroughly after handling.

Use protective clothing and goggles when handling.

A1.3 Acetone

A1.3.1 **Warning—**Extremely flammable. Vapors may cause flash fire.

Keep away from heat, sparks, and open flame.

Keep container closed.

Use with adequate ventilation.

Avoid build-up of vapors, and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.

Avoid prolonged breathing of vapor or spray mist.

Avoid contact with eyes or skin.

A1.4 Hydrochloric Acid (Concentrated)

A1.4.1 **Warning—**Poison. Corrosive. May be fatal if swallowed. Liquid and vapor cause severe burns. Harmful if inhaled.

Do not get in eyes, on skin, or on clothing.

Do not breathe vapor, spray, or mist.

Dilute by addition of acid to water.

Keep in tightly closed container in approved acid storage cabinet.

Keep cool.

Loosen closure carefully when opening.

Use with adequate ventilation.
Keep container closed when not in use.

Use protective clothing and goggles when handling.
Wash thoroughly after handling.

A2. CALIBRATION OF VISCOMETERS

A2.1 Using Liquid Standards

A2.1.1 Select from Table 2 a liquid standard having a kinematic viscosity at the calibration temperature (preferably 100 °F) in excess of the minimum shown in Table 1. Determine the flow-time to the nearest 0.2 s in accordance with Section 10, and calculate the viscometer constant, C, as follows:

$$C = v/t \quad \text{(A2.1)}$$

where:

v = Kinematic viscosity for the standard liquid, cSt, and
t = flow time, s.

A2.2 Using Standard Viscometers

A2.2.1 Select any petroleum oil that will have a flow time of at least 200 s in both the standard and to be standardized viscometers. Some viscometers, as listed in Table 1, require that the oil have a flow time in excess of 200 s in order that the kinetic correction will be less than 0.2 %.

A2.2.2 Select a standard viscometer of known constant C. This viscometer may be a master viscometer that has been calibrated by the "step-up" procedure using viscometers of successively larger diameters starting with distilled water as the basic viscosity standard or a routine viscometer of the same type that has been calibrated by comparison with a master viscometer. Mount the standard viscometer together with the viscometer to be calibrated in the same bath and determine the flow times of the oil in accordance with Section 10.

A2.2.3 Calculate the constant C as follows:

$$C_t = (t_t \times C_t)/t_t \quad \text{(A2.2)}$$

where:

C_t = C constant of the viscometer being calibrated,
t_t = flow time, to the nearest 0.2 s, in the viscometer being calibrated,
C_t = C constant of the standard viscometer, and
t_t = flow time, to the nearest 0.2 s, in the standard viscometer.