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## Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)<sup>1</sup>

This standard is issued under the fixed designation D 445; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope\*

1.1 This test method specifies a procedure for the determination of the kinematic viscosity,  $\nu$ , of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity,  $\eta$ , can be obtained by multiplying the kinematic viscosity,  $\nu$ , by the density,  $\rho$ , of the liquid.

NOTE 1—For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods **D 2170** and **D 2171**.

NOTE 2—**ISO 3104** corresponds to Test Method D 445.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm<sup>2</sup>/s (see **Table A1.1**) at all temperatures (see **6.3** and **6.4**). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 appro-*

*priate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

- D 446** Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers
- D 1193** Specification for Reagent Water
- D 1217** Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
- D 1480** Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer
- D 1481** Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer
- D 2162** Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards
- D 2170** Test Method for Kinematic Viscosity of Asphalts (Bitumens)
- D 2171** Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
- D 6071** Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy
- D 6074** Guide for Characterizing Hydrocarbon Lubricant Base Oils
- D 6617** Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
- E 1** Specification for ASTM Liquid-in-Glass Thermometers
- E 77** Test Method for Inspection and Verification of Thermometers

#### 2.2 ISO Standards:<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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In the IP, this test method is under the jurisdiction of the Standardization Committee.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

**ISO 3104** Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity

**ISO 3105** Glass Capillary Kinematic Viscometers—Specification and Operating Instructions

**ISO 3696** Water for Analytical Laboratory Use—Specification and Test Methods

**ISO 5725** Accuracy (trueness and precision) of measurement methods and results.

**ISO 9000** Quality Management and Quality Assurance Standards—Guidelines for Selection and Use

**ISO 17025** General Requirements for the Competence of Testing and Calibration Laboratories

2.3 *NIST Standards*:<sup>4</sup>

**NIST Technical Note 1297**, Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results

**NIST GMP 11**

**NIST Special Publication 819**

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard*:

3.1.1 *automated viscometer, n*—apparatus which, in part or in whole, has mechanized one or more of the procedural steps indicated in Section 11 or 12 without changing the principle or technique of the basic manual apparatus. The essential elements of the apparatus in respect to dimensions, design, and operational characteristics are the same as those of the manual method.

3.1.1.1 *Discussion*—Automated viscometers have the capability to mimic some operation of the test method while reducing or removing the need for manual intervention or interpretation. Apparatus which determine kinematic viscosity by physical techniques that are different than those used in this test method are not considered to be Automated Viscometers.

3.1.2 *density, n*—the mass per unit volume of a substance at a given temperature.

3.1.3 *dynamic viscosity, n*—the ratio between the applied shear stress and rate of shear of a liquid.

3.1.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity or, simply, viscosity. Thus dynamic viscosity is a measure of the resistance to flow or deformation of a liquid.

3.1.3.2 *Discussion*—The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence.

3.1.4 *kinematic viscosity, n*—the resistance to flow of a fluid under gravity.

3.1.4.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density,  $\rho$ . For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity,  $\nu$ , where  $\nu = \eta/\rho$ , and  $\eta$  is the dynamic viscosity coefficient.

### 4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity (determined value) is the product of the measured flow time and the calibration constant of the viscometer. Two such determinations are needed from which to calculate a kinematic viscosity result that is the average of two acceptable determined values.

### 5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

### 6. Apparatus

6.1 *Viscometers*—Use only calibrated viscometers of the glass capillary type, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D 446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automated Viscometers*—Automated apparatus may be used as long as they mimic the physical conditions, operations or processes of the manual apparatus. Any viscometer, temperature measuring device, temperature control, temperature controlled bath or timing device incorporated in the automated apparatus shall conform to the specification for these components as stated in Section 6 of this test method. Flow times of less than 200 s are permitted, however, a kinetic energy correction shall be applied in accordance with Section 7 on Kinematic Viscosity Calculation of Specifications D 446. The kinetic energy correction shall not exceed 3.0 % of the measured viscosity. The automated apparatus shall be capable of determining kinematic viscosity of a certified viscosity reference standard within the limits stated in 9.2.1 and Section 17. The precision shall be of statistical equivalence to, or better (has less variability) than the manual apparatus.

NOTE 3—Precision and bias of kinematic viscosity measurements for flow times of less than 200 s has not been determined. The precision stated in Section 17 is not known to be valid for kinematic viscosity measurements with flow times less than 200 s.

6.2 *Viscometer Holders*—Use viscometer holders to enable all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0.3° in all directions (see Specifications D 446 and ISO 3105).

<sup>4</sup> Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D 446, see Operating Instructions in Annexes A1–A3. For those viscometers which have Tube L (see Specifications D 446) held vertical, vertical alignment shall be confirmed by using (1) a holder ensured to hold Tube L vertical, or (2) a bubble level mounted on a rod designed to fit into Tube L, or (3) a plumb line suspended from the center of Tube L, or (4) other internal means of support provided in the constant temperature bath.

6.3 *Temperature-Controlled Bath*—Use a transparent liquid bath of sufficient depth such, that at no time during the measurement of flow time, any portion of the sample in the viscometer is less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 *Temperature Control*—For each series of flow time measurements, the temperature control of the bath liquid shall be such that within the range from 15 to 100°C, the temperature of the bath medium does not vary by more than  $\pm 0.02^\circ\text{C}$  of the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature must not exceed  $\pm 0.05^\circ\text{C}$ .

6.4 *Temperature Measuring Device in the Range from 0 to 100°C*—Use either calibrated liquid-in-glass thermometers (Annex A2) of an accuracy after correction of  $\pm 0.02^\circ\text{C}$  or better, or any other thermometric device of equal or better accuracy.

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within  $0.04^\circ\text{C}$ .

6.4.2 Outside the range from 0 to 100°C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of  $\pm 0.05^\circ\text{C}$  or better, or any other thermometric device of equal or better accuracy. When two temperature measuring devices are used in the same bath, they shall agree within  $\pm 0.1^\circ\text{C}$ .

6.4.3 When using liquid-in-glass thermometers, such as those in Table A2.1, use a magnifying device to read the thermometer to the nearest  $\frac{1}{5}$  division (for example,  $0.01^\circ\text{C}$  or  $0.02^\circ\text{F}$ ) to ensure that the required test temperature and temperature control capabilities are met (see 10.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within  $\pm 0.07\%$  (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times.

6.5.1 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 kinematic viscosity flow time measurements.

## 7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution. (**Warning**—Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive, and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapor. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

7.2 *Sample Solvent*, completely miscible with the sample. Filter before use.

7.2.1 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

7.3 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.2) and water (see 7.4). Filter before use.

7.3.1 Acetone is suitable. (**Warning**—Extremely flammable.)

7.4 *Water*, deionized or distilled and conforming to Specification D 1193 or Grade 3 of ISO 3696. Filter before use.

## 8. Certified Viscosity Reference Standards

8.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D 2162.

8.2 The uncertainty of the certified viscosity reference standard shall be stated for each certified value ( $k = 2$ , 95% confidence). See ISO 5725 or NIST 1297.

## 9. Calibration and Verification

9.1 *Viscometers*—Use only calibrated viscometers, thermometers, and timers as described in Section 6.

9.2 *Certified Viscosity Reference Standards (Table A1.2)*—These are for use as confirmatory checks on the procedure in the laboratory.

9.2.1 If the determined kinematic viscosity does not agree within the acceptable tolerance band, as calculated from Annex A4, of the certified value, recheck each step in the procedure, including thermometer and viscometer calibration, to locate the source of error. Annex A1 gives details of standards available.

NOTE 4—In previous issues of Test Method D 445, limits of  $\pm 0.35\%$  of the certified value have been used. The data to support the limit of  $\pm 0.35\%$  cannot be verified. Annex A4 provides instructions on how to determine the tolerance band. The tolerance band combines both the uncertainty of the certified viscosity reference standard as well as the uncertainty of the laboratory using the certified viscosity reference standard.

9.2.1.1 As an alternative to the calculation in **Annex A4**, the approximate tolerance bands in **Table 1** may be used.

9.2.2 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

9.3 The calibration constant,  $C$ , is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the standardization laboratory together with the instrument constant. Where the acceleration of gravity,  $g$ , differs by more than 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \quad (1)$$

where the subscripts 1 and 2 indicate, respectively, the standardization laboratory and the testing laboratory.

## 10. General Procedure for Kinematic Viscosity

10.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in **6.3.1** taking account of the conditions given in **Annex A2** and of the corrections supplied on the certificates of calibration for the thermometers.

10.1.1 Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

10.1.2 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see **6.4**).

10.1.3 They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.

10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time for manual viscometers shall not be less than 200 s or the longer time noted in **Specifications D 446**. Flow times of less than 200 s are permitted for automated viscometers, provided they meet the requirements of **6.1.2**.

10.2.1 The specific details of operation vary for the different types of viscometers listed in **Table A1.1**. The operating instructions for the different types of viscometers are given in **Specifications D 446**.

10.2.2 When the test temperature is below the dew point, fill the viscometer in the normal manner as required in **11.1**. To ensure that moisture does not condense or freeze on the walls

of the capillary, draw the test portion into the working capillary and timing bulb, place rubber stoppers into the tubes to hold the test portion in place, and insert the viscometer into the bath. After insertion, allow the viscometer to reach bath temperature, and then remove the stoppers. When performing manual viscosity determinations, do not use those viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

10.2.2.1 The use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted, but not required. If used, the drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument.

10.2.3 Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers.

## 11. Procedure for Transparent Liquids

11.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75  $\mu\text{m}$  screen, either prior to or during charging (see **Specifications D 446**).

NOTE 5—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

11.1.1 In general, the viscometers used for transparent liquids are of the type listed in **Table A1.1**, A and B.

11.1.2 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

11.1.3 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring a flow time.

11.1.4 Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial.

11.1.4.1 Thirty minutes should be sufficient except for the highest kinematic viscosities.

11.1.5 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

11.2 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0.1 s, the time required for the meniscus to pass from the first to the second

**TABLE 1 Approximate Tolerance Bands**

NOTE—The tolerance bands were determined using Practice **D 6617**. The calculation is documented in Research Report RR: D02-1498.<sup>A</sup>

Viscosity of Reference Material, mm <sup>2</sup> /s	Tolerance Band
< 10	±0.30%
10 to 100	±0.32%
100 to 1000	±0.36%
1000 to 10 000	±0.42%
10 000 to 100 000	±0.54%
> 100 000	±0.73%

<sup>A</sup>Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1498.

timing mark. 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.

11.2.1 Repeat the procedure described in 11.2 to make a second measurement of flow time. Record both measurements.

11.2.2 From the two measurements of flow time, calculate two determined values of kinematic viscosity.

11.2.3 If the two determined values of kinematic viscosity calculated from the flow time measurements agree within the stated determinability figure (see 17.1.1) for the product, use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If not, repeat the measurements of flow times after a thorough cleaning and drying of the viscometers and filtering (where required, see 11.1) of the sample until the calculated kinematic viscosity determinations agree with the stated determinability.

11.2.4 If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C, use as an estimate of the determinability 0.20% and 0.35% for temperatures outside this range.

## 12. Procedure for Opaque Liquids

12.1 For steam-refined cylinder oils and black lubricating oils, proceed to 12.3 ensuring a thoroughly representative sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 12.1.1-12.2.2 shall be followed to minimize this.

12.1.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A1.1, C.

12.1.2 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

12.1.3 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

12.1.4 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

12.1.4.1 With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

12.2 Immediately after completing 12.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

12.2.1 Immerse the flask in a bath of boiling water for 30 min. (**Warning**—Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.)

12.2.2 Remove the flask from the bath, stopper tightly, and shake for 60 s.

12.3 Two determinations of the kinematic viscosity of the test material are required. For those viscometers that require a complete cleaning after each flow time measurement, two viscometers may be used. A single viscometer in which an immediate, repeat flow time measurement can be made without cleaning may also be used for the two measurements of flow time and calculation of kinematic viscosity. Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube

viscometers for opaque liquids, filter the sample through a 75- $\mu$ m filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use a preheated filter to prevent the sample coagulating during the filtration.

12.3.1 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

12.3.2 After 10 min, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see Specifications D 446).

12.3.3 Allow the charged viscometers enough time to reach the test temperature (see 12.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring flow time.

12.4 With the sample flowing freely, measure in seconds to within 0.1 s, the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the measurement.

12.4.1 In the case of samples requiring heat treatment described in 12.1 through 12.2.1, complete the measurements of flow time within 1 h of completing 12.2.2. Record the measured flow times.

12.5 Calculate kinematic viscosity,  $\nu$ , in mm<sup>2</sup>/s, from each measured flow time. Regard these as two determined values of kinematic viscosity.

12.5.1 For residual fuel oils, if the two determined values of kinematic viscosity agree within the stated determinability figure (see 17.1.1), use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If the calculated kinematic viscosities do not agree, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering of the sample. If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C use as an estimate of the determinability 1.0 %, and 1.5 % for temperatures outside this range; it must be realized that these materials can be non-Newtonian, and can contain solids which can come out of solution as the flow time is being measured.

## 13. Cleaning of Viscometer

13.1 Between successive determinations of kinematic viscosity, clean the viscometer thoroughly by several rinsings with the sample solvent, followed by the drying solvent (see 7.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

13.2 Periodically clean the viscometer with the cleaning solution (**Warning**—see 7.1), for several hours to remove residual traces of organic deposits, rinse thoroughly with water (7.4) and drying solvent (see 7.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits by hydrochloric acid treatment before the use of cleaning acid, particularly if the presence of barium salts is suspected. (**Warning**—It is essential that alkaline cleaning solutions are not used as changes in the viscometer calibration can occur.)

## 14. Calculation

14.1 Calculate each of the determined kinematic viscosity values,  $\nu_1$  and  $\nu_2$ , from the measured flow times,  $t_1$  and  $t_2$ , and the viscometer constant,  $C$ , by means of the following equation:

$$\nu_{1,2} = C \cdot t_{1,2} \quad (2)$$

where:

$\nu_{1,2}$  = determined kinematic viscosity values for  $\nu_1$  and  $\nu_2$ , respectively,  $\text{mm}^2/\text{s}$ ,

$C$  = calibration constant of the viscometer,  $\text{mm}^2/\text{s}^2$ , and

$t_{1,2}$  = measured flow times for  $t_1$  and  $t_2$ , respectively, s.

Calculate the kinematic viscosity result,  $\nu$ , as an average of  $\nu_1$  and  $\nu_2$  (see 11.2.3 and 12.5.1).

14.2 Calculate the dynamic viscosity,  $\eta$ , from the calculated kinematic viscosity,  $\nu$ , and the density,  $\rho$ , by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \quad (3)$$

where:

$\eta$  = dynamic viscosity,  $\text{mPa}\cdot\text{s}$ ,

$\rho$  = density,  $\text{kg}/\text{m}^3$ , at the same temperature used for the determination of the kinematic viscosity, and

$\nu$  = kinematic viscosity,  $\text{mm}^2/\text{s}$ .

14.2.1 The density of the sample can be determined at the test temperature of the kinematic viscosity determination by an appropriate method such as Test Methods D 1217, D 1480, or D 1481.

## 15. Expression of Results

15.1 Report the test results for the kinematic or dynamic viscosity, or both, to four significant figures, together with the test temperature.

## 16. Report

16.1 Report the following information:

16.1.1 Type and identification of the product tested,

16.1.2 Reference to this test method or a corresponding international standard,

16.1.3 Result of the test (see Section 15),

16.1.4 Any deviation, by agreement or otherwise, from the procedure specified,

16.1.5 Date of the test, and

16.1.6 Name and address of the test laboratory.

## 17. Precision

17.1 *Comparison of Determined Values:*

17.1.1 *Determinability (d)*—The difference between successive determined values obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C <sup>5</sup>	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C <sup>6</sup>	0.0013 y	(0.13 %)
Formulated oils at 150°C <sup>7</sup>	0.015 y	(1.5 %)
Petroleum wax at 100°C <sup>8</sup>	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C <sup>9</sup>	0.011 (y + 8)	
Residual fuel oils at 50°C <sup>9</sup>	0.017 y	(1.7 %)
Additives at 100°C <sup>10</sup>	0.00106 y <sup>1.1</sup>	

Gas oils at 40°C <sup>11</sup>	0.0013 (y+1)	
Jet fuels at -20°C <sup>12</sup>	0.0018 y	(0.18 %)

where:  $y$  is the average of determined values being compared.

17.2 *Comparison of Results:*

17.2.1 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:

Base oils at 40 and 100°C <sup>5</sup>	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C <sup>6</sup>	0.0026 x	(0.26 %)
Formulated oils at 150°C <sup>7</sup>	0.0056 x	(0.56 %)
Petroleum wax at 100°C <sup>8</sup>	0.0141 x <sup>1.2</sup>	
Residual fuel oils at 80 and 100°C <sup>9</sup>	0.013 (x + 8)	
Residual oils at 50°C <sup>9</sup>	0.015 x	(1.5 %)
Additives at 100°C <sup>10</sup>	0.00192 x <sup>1.1</sup>	
Gas oils at 40°C <sup>11</sup>	0.0043 (x+1)	
Jet fuels at -20°C <sup>12</sup>	0.007 x	(0.7 %)

where:  $x$  is the average of results being compared.

17.2.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1331 and RR:D02-1132. These precision values were obtained by statistical examination of interlaboratory results from six mineral oils (base oils without additive package) in the range from 8 to 1005  $\text{mm}^2/\text{s}$  at 40°C and from 2 to 43  $\text{mm}^2/\text{s}$  at 100°C, and were first published in 1989. Request See Guide D 6074.

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1332. These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range from 36 to 340  $\text{mm}^2/\text{s}$  at 40°C and from 6 to 25  $\text{mm}^2/\text{s}$  at 100°C, and were first published in 1991. See Guide D 6071.

<sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1333. These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range from 7 to 19  $\text{mm}^2/\text{s}$  at 150°C, and first published in 1991. See Guide D 6074.

<sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1334. These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range from 3 to 16  $\text{mm}^2/\text{s}$  at 100°C, and were first published in 1988.

<sup>9</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1198. These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range from 30 to 1300  $\text{mm}^2/\text{s}$  at 50°C and from 5 to 170  $\text{mm}^2/\text{s}$  at 80 and 100°C, and were first published in 1984.

<sup>10</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1421. These precision values were obtained by statistical examination of interlaboratory results from eight additives in the range from 145 to 1500  $\text{mm}^2/\text{s}$  at 100°C and were first available in 1997.

<sup>11</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1422. These precision values were obtained by statistical examination of interlaboratory results from eight gas oils in the range from 1 to 13  $\text{mm}^2/\text{s}$  at 40°C and were first available in 1997.

<sup>12</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1420. These precision values were obtained by statistical examination of interlaboratory results from nine jet fuels in the range from 4.3 to 5.6  $\text{mm}^2/\text{s}$  at -20°C and were first available in 1997.

Base oils at 40 and 100°C <sup>5</sup>	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C <sup>6</sup>	0.0076 x	(0.76 %)
Formulated oils at 150°C <sup>7</sup>	0.018 x	(1.8 %)
Petroleum wax at 100°C <sup>8</sup>	0.0366 x <sup>1,2</sup>	
Residual fuel oils at 80 and 100°C <sup>9</sup>	0.04 (x + 8)	
Residual oils at 50°C <sup>9</sup>	0.074 x	(7.4 %)
Additives at 100°C <sup>10</sup>	0.00862 x <sup>1,-1</sup>	
Gas oils at 40°C <sup>11</sup>	0.0082 (x+1)	
Jet fuels at –20°C <sup>12</sup>	0.019 x	(1.9 %)

where:  $x$  is the average of results being compared.

17.3 The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

17.4 The precision for specific automated viscometers has not been determined. However, an analysis has been made of a

large data set including both automated and manual viscometers over the temperature range of 40 to 100°C. The reproducibility of automated viscometer data is not statistically significantly different than the reproducibility of manual viscometer data. It is also shown that there is no bias of the automated data in comparison to the manual data.<sup>13</sup>

## 18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

<sup>13</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1498.

## ANNEXES

### (Mandatory Information)

#### A1. VISCOMETER TYPES AND CERTIFIED VISCOSITY REFERENCE STANDARDS

##### A1.1 Viscometer Types

A1.1.1 **Table A1.1** lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications, operating instructions, and calibration, refer to specifications in Specifications **D 446**.

A1.1.2 **Table A1.2** lists certified viscosity reference standards.

**TABLE A1.1 Viscometer Types**

Viscometer Identification	Kinematic Viscosity Range, <sup>A</sup> mm <sup>2</sup> /s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine <sup>B</sup>	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube <sup>B</sup>	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL <sup>B</sup>	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch <sup>B</sup>	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL <sup>B</sup>	3.5 to 100 000
BS/IP/SL(S) <sup>B</sup>	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde <sup>B</sup>	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic <sup>B</sup>	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution <sup>B</sup> (B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

<sup>A</sup> Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Specifications **D 446**.

<sup>B</sup> In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

**TABLE A1.2 Certified Viscosity Reference Standards**

Designation	Approximate Kinematic Viscosity, mm <sup>2</sup> /s					
	20°C	25°C	40°C	50°C	80 °C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30 000	...	81 000	23 000	11 000	...	...

## A2. KINEMATIC VISCOSITY TEST THERMOMETERS

### A2.1 Short-Range Specialized Thermometer

A2.1.1 Use a short-range specialized thermometer conforming to the generic specification given in [Table A2.1](#) and [Table A2.2](#) and to one of the designs shown in [Fig. A2.1](#).

A2.1.2 The difference in the designs rests mainly in the position of the ice point scale. In Design A, the ice point is within the scale range, in Design B, the ice point is below the scale range, and in Design C, the ice point is above the scale range.

### A2.2 Calibration

A2.2.1 Use liquid-in-glass thermometers with an accuracy after correction of 0.02°C or better, calibrated by a laboratory meeting the requirements of [ISO 9000](#) or [ISO 17025](#), and carrying certificates confirming that the calibration is traceable to a national standard. As an alternative, use thermometric devices such as platinum resistance thermometers, of equal or better accuracy, with the same certification requirements.

A2.2.2 The scale correction of liquid-in-glass thermometers can change during storage and use, and therefore regular re-calibration is required. This is most conveniently achieved in a working laboratory by means of a re-calibration of the ice point, and all of the main scale corrections altered for the change seen in the ice point.

**TABLE A2.1 General Specification for Thermometers**

NOTE—[Table A2.2](#) gives a range of ASTM, IP, and ASTM/IP thermometers that comply with the specification in [Table A2.1](#), together with their designated test temperatures. See Specification [E 1](#) and Test Method [E 77](#).

	Immersion	Total
Scale marks:		
Subdivisions	°C 0.05	
Long lines at each	°C 0.1 and 0.5	
Numbers at each	°C 1	
Maximum line width	mm 0.10	
Scale error at test temperature, max	°C 0.1	
Expansion chamber:		
Permit heating to	°C 105 up to 90, 120 between 90 and 95 130 between 95 and 105, 170 above 105	
Total length	mm 300 to 310	
Stem outside diameter	mm 6.0 to 8.0	
Bulb length	mm 45 to 55	
Bulb outside diameter	mm no greater than stem	
Length of scale range	mm 40 to 90	

**TABLE A2.2 Complying Thermometers**

Thermometer No.	Test Temperature		Thermometer No.	Test Temperature	
	°C	°F		°C	°F
	ASTM 132C, IP 102C	150			ASTM 128C, F/IP 33C
ASTM 110C, F/IP 93C	135	275	ASTM 72C, F/IP 67C	-17.8	0
ASTM 121C/IP 32C	98.9,	210,	ASTM 127C/IP 99C	-20	-4
	100	212	ASTM 126C, F/IP 71C	-26.1	-20
ASTM 129C, F/IP 36C	93.3	200	ASTM 73C, F/IP 68C	-40	-40
ASTM 48C, F/IP 90C	82.2	180	ASTM 74C, F/IP 69C	-53.9	-65
IP 100C	80				
ASTM 47C, F/IP 35C	60	140			
ASTM 29C, F/IP 34C	54.4	130			
ASTM 46C F/IP 66C	50	122			
ASTM 120C/IP 92C	40				
ASTM 28C, F/IP 31C	37.8	100			
ASTM 118C, F	30	86			
ASTM 45C, F/IP 30C	25	77			
ASTM 44C, F/IP 29C	20	68			

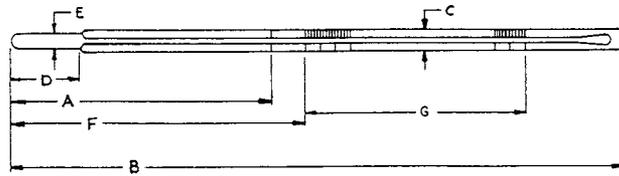
A2.2.2.1 The interval for ice-point recalibration shall be no longer than six months (see [NIST GMP 11](#)). For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, and it may be out of calibration. Such thermometers shall be removed from service until inspected, or recalibrated, or both. A complete recalibration of the thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to this design thermometer (see [NIST Special Publication 819](#)). Any change in ice-point correction shall be added to the other corrections of the original Report of Calibration.

A2.2.2.2 Other thermometric devices, if used, will also require periodic recalibration. Keep records of all recalibration.

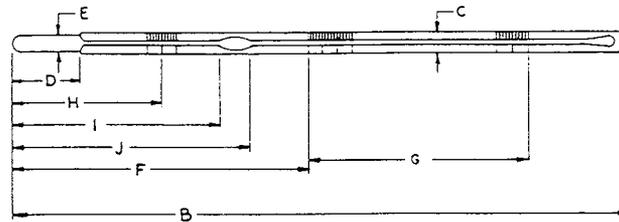
A2.2.3 *Procedure for Ice-point Recalibration of Liquid-in-glass Thermometers.*

A2.2.3.1 Unless otherwise listed on the certificate of calibration, the recalibration of calibrated kinematic viscosity thermometers requires that the ice-point reading shall be taken within 60 min after being at test temperature for not less than 3 min.

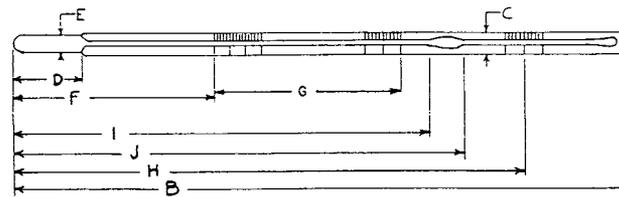
A2.2.3.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the



(a)



(b)



(c)

**FIG. A2.1 Thermometer Designs**

crushed ice and add sufficient water to form a slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer, and pack the ice gently about the stem, to a depth approximately one scale division below the 0°C graduation.

A2.2.3.3 After at least 3 min have elapsed, tap the thermometer gently and repeatedly at right angles to its axis while making observations. Successive readings taken at least 1 min apart shall agree within 0.005°C.

A2.2.3.4 Record the ice-point readings and determine the thermometer correction at this temperature from the mean reading. If the correction is found to be higher or lower than that corresponding to a previous calibration, change the correction at all other temperatures by the same value.

A2.2.3.5 During the procedure, apply the following conditions:

(1) The thermometer shall be supported vertically.

(2) View the thermometer with an optical aid that gives a magnification of approximately five and also eliminates parallax.

(3) Express the ice-point reading to the nearest 0.005°C.

A2.2.4 When in use, immerse the thermometric device to the same depth as when it was fully calibrated. For example, if a liquid-in-glass thermometer was calibrated at the normal total immersion condition, it shall be immersed to the top of the mercury column with the remainder of the stem and the expansion volume at the uppermost end exposed to room temperature and pressure. In practice, this means that the top of the mercury column shall be within a length equivalent to four scale divisions of the surface of the medium whose temperature is being measured.

A2.2.4.1 If this condition cannot be met, then an extra correction may be necessary.

### A3. TIMER ACCURACY

A3.1 Regularly check timers for accuracy and maintain records of such checks.

A3.1.1 Time signals as broadcast by the National Institute of Standards and Technology are a convenient and primary standard reference for calibrating timing devices. The following can be used to an accuracy of 0.1 s:

WWV	Fort Collins, CO	2.5, 5, 10, 15, 20 MHz
-----	------------------	------------------------

WWVH	Kauai, HI	2.5, 5, 10, 15, MHz
CHU	Ottawa, Canada	3.33, 7.335, 14.67 MHz

A3.1.2 Radio broadcast of voice and audio on a telephone line at phone 303-499-7111. Additional time services are available from the National Institute of Standards and Technology.

### A4. CALCULATION OF ACCEPTABLE TOLERANCE ZONE (BAND) TO DETERMINE CONFORMANCE WITH A CERTIFIED REFERENCE MATERIAL

A4.1 Determine the standard deviation for site uncertainty,  $\sigma_{\text{site}}$ , from a laboratory quality control program.

A4.1.1 If the standard deviation for site uncertainty,  $\sigma_{\text{site}}$ , is not known, use the value 0.19%.

A4.2 Determine the combined extended uncertainty (CEU) of the accepted reference value (ARV) of the certified reference material (CRM) from the supplier's label or included documentation.

A4.3 Calculate the standard error of the accepted reference value (SEARV) by dividing the CEU by the coverage factor,  $k$ , listed on the supplier's label or included documentation.

A4.3.1 If the coverage factor,  $k$ , is not known, use the value 2.

A4.4 Construct the acceptable tolerance zone:

$$TZ = \pm 1.44 \sqrt{\sigma_{\text{site}}^2 + SE_{\text{ARV}}^2}$$

### SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 445-04<sup>e2</sup>) that may impact the use of this standard.

- |  |                           |
|--|---------------------------|
| (1) Revised <b>3.1.1</b> .                         | (3) Revised <b>10.2</b> . |
| (2) Revised <b>6.1.2</b> and added <b>Note 3</b> . | (4) Added <b>Note 2</b> . |

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