



Designation: ~~D445 – 21<sup>e2</sup>~~ D445 – 23

British Standard 2000: Part 71: Section 1: 1996

## 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 D445; 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 U.S. Department of Defense.*

~~<sup>1</sup> NOTE—Editorially corrected tables in Section 17 in December 2021 to align with research reports.~~

~~<sup>2</sup> NOTE—Editorially removed joint designation in October 2022.~~

### 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 [D2170](#) and [D2171](#).

NOTE 2—ISO 3104 corresponds to Test Method D445 – 03.

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 mm<sup>2</sup>/s 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 standard. The SI unit used in this test method for kinematic viscosity is mm<sup>2</sup>/s, and the SI unit used in this test method for dynamic viscosity is mPa·s. For user reference, 1 mm<sup>2</sup>/s = 10<sup>-6</sup> m<sup>2</sup>/s = 1 cSt and 1 mPa·s = 1 cP = 0.001 Pa·s.

1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

<sup>1</sup> This test method is under the jurisdiction of Committee [D02](#) on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee [D02.07](#) on Flow Properties.

Current edition approved ~~May 15, 2021~~ Nov. 1, 2023, Published ~~June 2021~~ November 2023. Originally approved in 1937. Last previous edition approved in ~~2019~~ 2021 as ~~D445 – 19a~~ D445 – 21<sup>e2</sup>. DOI: ~~10.1520/D0445-21E02~~ 10.1520/D0445-23.

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

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *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.*

## 2. Referenced Documents

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

- D396 Specification for Fuel Oils
- D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers
- D1193 Specification for Reagent Water
- D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
- D1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer
- D1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer
- D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards
- D2170 Test Method for Kinematic Viscosity of Asphalts
- D2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
- D6071 Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atomic Absorption Spectroscopy
- D6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
- D6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
- D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
- D8278 Specification for Digital Contact Thermometers for Test Methods Measuring Flow Properties of Fuels and Lubricants
- E1 Specification for ASTM Liquid-in-Glass Thermometers
- E77 Test Method for Inspection and Verification of Thermometers
- E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature
- E1750 Guide for Use of Water Triple Point Cells
- E2593 Guide for Accuracy Verification of Industrial Platinum Resistance Thermometers

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

- 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<sup>5</sup>
- NIST GMP 11 Good Measurement Practice for Assignment and Adjustment of Calibration Intervals for Laboratory Standards<sup>6</sup>
- NIST Special Publication 811 Guide for the Use of the International System of Units (SI)<sup>7</sup>
- NIST Special Publication 1088 Maintenance and Validation of Liquid-in-Glass Thermometers<sup>8</sup>

<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, <http://www.ansi.org>.

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

<sup>5</sup> <http://physics.nist.gov/cuu/Uncertainty/bibliography.html>

<sup>6</sup> [http://ts.nist.gov/WeightsAndMeasures/upload/GMP\\_11\\_Mar\\_2003.pdf](http://ts.nist.gov/WeightsAndMeasures/upload/GMP_11_Mar_2003.pdf)

<sup>7</sup> <http://www.nist.gov/pml/pubs/sp811/index.cfm>

<sup>8</sup> <http://www.nist.gov/pml/pubs/sp1088/index.cfm>

### 3. Terminology

3.1 See also *International Vocabulary of Metrology*.<sup>9</sup>

#### 3.2 Definitions:

3.2.1 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

##### 3.2.1.1 Discussion—

This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device.

##### 3.2.1.2 Discussion—

The devices are often referred to as a “digital thermometers,” however the term includes devices that sense temperature by means other than being in physical contact with the media.

##### 3.2.1.3 Discussion—

PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

#### 3.3 Definitions of Terms Specific to This Standard:

3.3.1 *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.

##### 3.3.1.1 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.

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

3.3.3 *dynamic viscosity,  $\eta$ , n*—the ratio between the applied shear stress and rate of shear of a material.

##### 3.3.3.1 Discussion—

It is sometimes called the coefficient of dynamic viscosity or absolute viscosity. Dynamic viscosity is a measure of resistance to flow or deformation which constitutes a material’s ability to transfer momentum in response to steady or time-dependent external shear forces. Dynamic viscosity has the dimension of mass divided by length and time and its SI unit is pascal times second (Pa·s). Among the transport properties for heat, mass, and momentum transfer, dynamic viscosity is the momentum conductivity.

3.3.4 *kinematic viscosity,  $\nu$ , n*—the ratio of the dynamic viscosity ( $\eta$ ) to the density ( $\rho$ ) of a material at the same temperature and pressure.

##### 3.3.4.1 Discussion—

Kinematic viscosity is the ratio between momentum transport and momentum storage. Such ratios are called diffusivities with dimensions of length squared divided by time and the SI unit is metre squared divided by second ( $\text{m}^2/\text{s}$ ). Among the transport properties for heat, mass, and momentum transfer, kinematic viscosity is the momentum diffusivity.

##### 3.3.4.2 Discussion—

Formerly, kinematic viscosity was defined specifically for viscometers covered by this test method as the resistance to flow under gravity. More generally, it is the ratio between momentum transport and momentum storage.

##### 3.3.4.3 Discussion—

For gravity-driven flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density,  $\rho$ , if the density of air is negligible compared to that of the liquid. For any particular viscometer covered by this test method, the time of flow of a fixed volume of liquid is directly proportional to its kinematic viscosity,  $\nu$ , where  $\nu = \eta/\rho$ , and  $\eta$  is the dynamic viscosity.

### 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.

<sup>9</sup> *International Vocabulary of Metrology — Basic and General Concepts and Associated Terms (VIM)*, 3rd ed., 2008, <http://www.bipm.org/en/publications/guides/vim.html>.

## 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 [D446](#) 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 [D446](#). 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 has been determined for automated viscometers tested on the sample types listed in [17.3.1](#) and is no worse than the manual apparatus (that is, exhibits the same or less variability).

NOTE 3—Precision and bias of kinematic viscosity measurements for flow times as low as 10 s have been determined for automated instruments tested with the sample types listed in [17.3.1](#).

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 [D446](#) and ISO 3105).

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 [D446](#), see Operating Instructions in Annexes A1–A3. For those viscometers which have Tube L (see Specifications [D446](#)) 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 °C to 100 °C, the temperature of the bath medium does not vary by more than  $\pm 0.02$  °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  $\pm 0.05$  °C.

### 6.4 *Temperature Measuring Devices:*

6.4.1 *Liquid-in-glass Thermometers*—Use calibrated thermometers noted in [Annex A2](#). Devices with a nominal temperature range from 0 °C to 100 °C will have an accuracy after correction of  $\pm 0.02$  °C or better. When the nominal temperature range is outside the 0 °C to 100 °C range, they will have an accuracy after correction of  $\pm 0.05$  °C or better.

6.4.1.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. When the temperature

range is from 0 °C to 100 °C, the two thermometers, with corrections applied, shall agree within 0.04 °C. When the temperature range is outside 0 °C to 100 °C, the two thermometers, with corrections applied, shall agree to within 0.1 °C.

6.4.2 *Digital Contact Thermometer*—Use the indicated DCT for the following nominal temperature ranges:

Nominal Temperature Range	Specification <b>D8278</b> DCT Id
–80 °C to 0 °C	D02-DCT04
0 °C to 100 °C	D02-DCT05
100 °C to 175 °C	D02-DCT06

6.4.2.1 Verify the calibration at least annually. The probe shall be recalibrated when the check value differs by more than 0.02 °C from the last probe calibration. Verification can be accomplished with the use of a water triple point cell, an ice bath, or other suitable constant temperature device which has a known temperature value of suitable precision. See Practice **E563** and Guides **E1750** and **E2593** for more information regarding checking calibrations.

6.4.2.2 In the case of constant temperature baths used in instruments for automatic viscosity determinations, the user is to contact the instrument manufacturer for the correct DCT that has performance equivalence to that described here.

6.4.3 Outside the range from 0 °C to 100 °C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of  $\pm 0.05$  °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  $\pm 0.1$  °C.

6.4.4 *Temperature Device Placement:*

6.4.4.1 *Liquid-in-glass Thermometer*, shall be suspended vertically and positioned so that the top of the liquid column is just below the surface of the bath fluid. See **Fig. 1**.

6.4.4.2 *DCT Probe*, shall be immersed by more than its minimum immersion depth in a constant temperature bath so that the center of the probe’s sensing region is at the same level as the lower half of the working capillary provided the probe’s minimum immersion depth is met and is no less than indicated on calibration certificate. See **Fig. 1**. The end of the probe sheath shall not extend past the bottom of the viscometer. It is preferable for the center of the sensing element to be located at the same level as the lower half of the working capillary as long as the minimum immersion requirements are met.

6.4.5 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 °C or 0.02 °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—Device for Manual Viscometers*—Use any timing device, ~~spring-wound or~~ mechanical (spring-wound or motor driven) or digital, that is capable of taking readings with a discrimination of ~~0.1 s~~ 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 Timing devices powered by alternating electric current motors 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.

6.5.2 Timing devices employed in automated viscometers are an integral part of the apparatus and typically are digital (using a precision crystal oscillator) with discriminations of 0.01 s or better. As such, the timing devices are included within the overall system calibration of automated viscometers. Follow the manufacturer’s instructions for ensuring the automated viscometer is properly calibrated using CRM’s such that it is capable of determining kinematic viscosity of a certified viscosity reference standard within the limits stated in 9.2.1 and Section 17 (see 6.1.2).

6.6 *Ultrasonic Bath, Unheated—(optional)*, with an operating frequency between 25 kHz to 60 kHz and a typical power output of  $\leq 100$  W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating

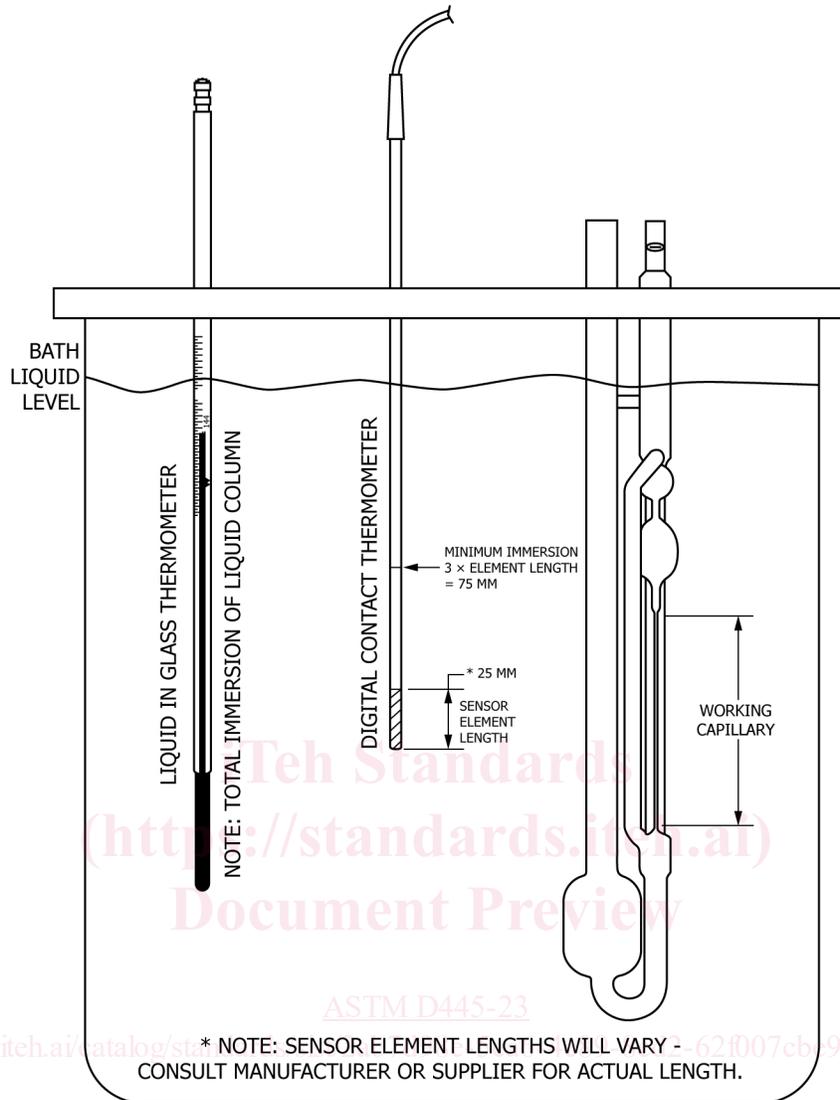


FIG. 1 Temperature Probe Immersion in Constant Temperature Bath

frequencies and power outputs outside this range, however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

## 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 **D1193** 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 Practice **D2162**.

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 D445, 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.

**TABLE 1 Approximate Tolerance Bands**

NOTE 1—The tolerance bands were determined using Practice **D6617**. 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	$\pm 0.30\%$
10 to 100	$\pm 0.32\%$
100 to 1000	$\pm 0.36\%$
1000 to 10 000	$\pm 0.42\%$
10 000 to 100 000	$\pm 0.54\%$
> 100 000	$\pm 0.73\%$

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

## 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 D446. 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 D446.

10.2.2 When the test temperature is below the ambient dew point, the use of loosely-packed drying tubes affixed to the open ends of the viscometer is permitted but not mandatory. These are designed to prevent water condensation. It is essential that they do not set up a pressure differential and affect the rate of flow. Before the first use of drying tubes, it is recommended that a certified viscosity reference standard is used to verify the correct use of the viscometer with and without drying tubes in order to ensure that there is no restriction in the flow. When the test temperature is below the dew point, fill the viscometer in the normal manner as required in 11.1. It is recommended to charge the viscometer outside the bath. 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.

10.2.3 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 Although not mandatory, for some transparent liquid sample types such as viscous oils that are prone to having entrained air or gas bubbles present in the sample, the use of an ultrasonic bath (see 6.6) without the heater turned on (if so equipped) has been found effective in homogenizing and dissipating bubbles typically within 5 min prior to taking a test specimen for analysis, with no material impact on results. 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 µm screen, either prior to or during charging (see Specifications D446).

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; however, for the manual measurement of kinematic viscosity of jet fuels at –20 °C only suspended-level type viscometers as noted in Table A1.1 shall be used. The suspended level type viscometer types used for jet fuel do not require a correction to the calibration constant for the test temperature being used.

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, however a minimum of 30 min is specifically required for manual analysis of jet fuels at  $-20\text{ }^{\circ}\text{C}$ .

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 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, use 1.5 % as an estimate of the determinability.

## 12. Procedure for Residual Fuel Oils and Opaque Liquids

12.1 For steam-refined cylinder oils and black lubricating oils, proceed to 12.2 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 to 12.1.8 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 the sample in the original container at a temperature between  $60\text{ }^{\circ}\text{C}$  and  $65\text{ }^{\circ}\text{C}$  for 1 h.

12.1.3 Place the BS/IP/RF U-tube reverse-flow, or Zeitfuchs Cross-arm, or Lantz-Zeitfuchs type reverse-flow viscometer for the samples to be tested in the viscometer bath(s) at the required test temperature. If the viscometers are to be charged prior to insertion in the viscometer bath, for example, Cannon Fenske Opaque, see 12.2.1.

12.1.4 Upon completion of step 12.1.2, vigorously stir each sample for approximately 20 s with a glass or steel rod of sufficient length to reach the bottom of the container. For samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above  $65\text{ }^{\circ}\text{C}$  to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

12.1.5 Remove the stirring rod and inspect for sludge or wax adhering to the rod. Continue stirring until there is no sludge or wax adhering to the rod.

12.1.6 Recap the container tightly and shake vigorously for 1 min to complete the mixing. To protect the integrity of the sample should a repeat analysis be required, pour sufficient sample to fill two flasks and loosely stopper. (Each flask should hold sufficient sample to fill two viscometers in order to obtain two determinations. The second flask is required to carry out a repeat analysis.) If a repeat analysis is not a consideration the next steps can be performed using the original container, loosely capped.

12.1.7 Heat the first sample flask or sample container between 100 °C and 105 °C for 30 min.

12.1.8 Remove the first sample flask or sample container from the heat, close tightly, and shake vigorously for 60 s.

12.2 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 must be used. These two determinations are used to calculate one result. Charge two viscometers in the manner dictated by the design of the instrument. For example, for the Lantz-Zeitfuchs Cross-arm or the BS/IP/RF U-tube reverse-flow viscometers for opaque liquids, filter the sample through a 75 µm 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.

12.2.1 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.

12.2.2 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 [D446](#)).

12.2.3 Allow the charged viscometers enough time to reach the test temperature (see [12.2.1](#)). 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.

12.3 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.

12.3.1 In the case of samples requiring heat treatment described in [12.1](#) through [12.1.8](#), complete the measurements of flow time within 1 h of completing [12.1.8](#). Record the measured flow times.

12.4 Calculate kinematic viscosity,  $\nu$ , in millimetres squared per second, from each measured flow time. Regard these as two determined values of kinematic viscosity.

12.4.1 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. This constitutes one analysis. Record the result. If a second value (repeat) is required, then repeat the analysis after thorough cleaning and drying of the viscometers starting from sample preparation steps [12.1.6](#) using the second flask. If the original container has been conditioned using steps [12.1.2](#) to [12.1.8](#), then this is not suitable for a repeat analysis. 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 °C 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 If periodic verification of the viscometer calibration using certified viscosity reference standards (see [9.2](#)) is outside of the acceptable tolerance band, the viscometer may need to be cleaned. 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.)