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.

Designation: D445 – 19

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Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)[1]

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 (ε) 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.

1. Scope*

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, ν, 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, η, can be obtained by multiplying the kinematic viscosity, ν, by the density, ρ, 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^2/s to 300 000 mm^2/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^2/s, and the SI unit used in this test method for dynamic viscosity is mPa·s. For user reference, 1 mm^2/s = 10^{-6} m^2/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 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.

1.6 *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:*[2]

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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, 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.

[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.

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

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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 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
D7962 Practice for Determination of Minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift
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
E644 Test Methods for Testing Industrial Resistance Thermometers
E1750 Guide for Use of Water Triple Point Cells
E2593 Guide for Accuracy Verification of Industrial Platinum Resistance Thermometers
E2877 Guide for Digital Contact Thermometers

2.2 *ISO Standards:*[3]
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

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

2.3 *NIST Standards:*[4]
NIST Technical Note 1297 Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results[5]
NIST GMP 11 Good Measurement Practice for Assignment and Adjustment of Calibration Intervals for Laboratory Standards[6]
NIST Special Publication 811 Guide for the Use of the International System of Units (SI)[7]
NIST Special Publication 1088 Maintenance and Validation of Liquid-in-Glass Thermometers[8]

3. Terminology

3.1 See also *International Vocabulary of Metrology*.[9]

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. These devices are sometimes referred to as "digital thermometers."

3.2.1.2 *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, η, 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

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.
[5] http://physics.nist.gov/cuu/Uncertainty/bibliography.html
[6] http://ts.nist.gov/WeightsAndMeasures/upload/GMP_11_Mar_2003.pdf
[7] http://www.nist.gov/pml/pubs/sp811/index.cfm
[8] http://www.nist.gov/pml/pubs/sp1088/index.cfm
[9] *International Vocabulary of Metrology — Basic and General Concepts and Associated Terms (VIM)*, 3rd ed., 2008, http://www.bipm.org/en/publications/guides/vim.html.

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, ν, n*—the ratio of the dynamic viscosity (η) to the density (ρ) 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 (m^2/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, ρ, 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, ν, where $\nu = \eta/\rho$, and η 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.

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 ±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 ±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 ±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 ±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 meeting the following requirements:

Criteria	Minimum Requirements
Nominal temperature ranges[A]	–80 °C to 0 °C
	0 °C to 100 °C
	Above 100 °C
Display resolution, minimum	0.01 °C
Accuracy,[B] minimum	0 °C to 100 °C: ±20 mK (±0.02 °C)
	–80 °C to 0 °C and above 100 °C: ±50 mK (±0.05 °C)
Sensor type	RTD, such as a PRT or thermistor
Immersion depth[C]	40 mm minimum by Practice D7962
Measurement drift[D]	less than 20 mK (0.02 °C) per year
Response time[E]	less than or equal to 8 s
Temperature calibration data	When the 'range-of-use' is less than 30 degrees Celsius, then at least 2 data points are required.
	When the 'range-of-use' is 30 degrees or greater and less than 90 degrees, then at least 3 data points are required.
	When the 'range-of-use' is 90 degrees or greater, then at least 4 data points are required.
	In all cases the calibration data is to be included in calibration report.
Temperature calibration report	The DCT shall have a report of temperature calibration traceable to a national calibration or metrology standards body issued by a competent calibration laboratory with demonstrated competency in temperature calibration. An ISO 17025 accredited laboratory with temperature calibration in its accreditation scope would meet this requirement.

[A] A device's minimum and maximum temperature may be different than the values shown provided the calibration requirements are met.
[B] Accuracy is the total combined accuracy of the DCT unit which includes the display, electronics, and sensor probe with correction factors.
[C] Minimum probe immersion depth as determined by Practice D7962, or an equivalent procedure, and is to be equal to or less than the value in the table. This is the minimum immersion needed to obtain an accurate temperature measurement thus the actual immersion will likely exceed this minimum.
[D] Drift is the difference between the corrected DCT temperature and a reference.
[E] *Response Time*—The time for a DCT to respond to a step change in temperature. This parameter is determined by the DCT provider following the procedure in Section 9 of Test Methods E644 or an equivalent method.

NOTE 4—See Guide E2877 for additional information regarding DCT parameters.

NOTE 5—With respect to DCT probe immersion depth, a procedure is available in Test Methods E644, Section 7, for determining the minimum depth. With respect to an ice bath, Practice E563 provides guidance on the preparation of an ice bath, however, variance from the specific steps is permitted provided preparation is consistent as it is being used to track change in calibration.

6.4.2.1 Verify the calibration at least annually. The probe shall be recalibrated when the check value differs by more than 0.01 °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 ±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 ±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 ⅕ 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*—Use any timing device, spring-wound or digital, that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within ±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 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

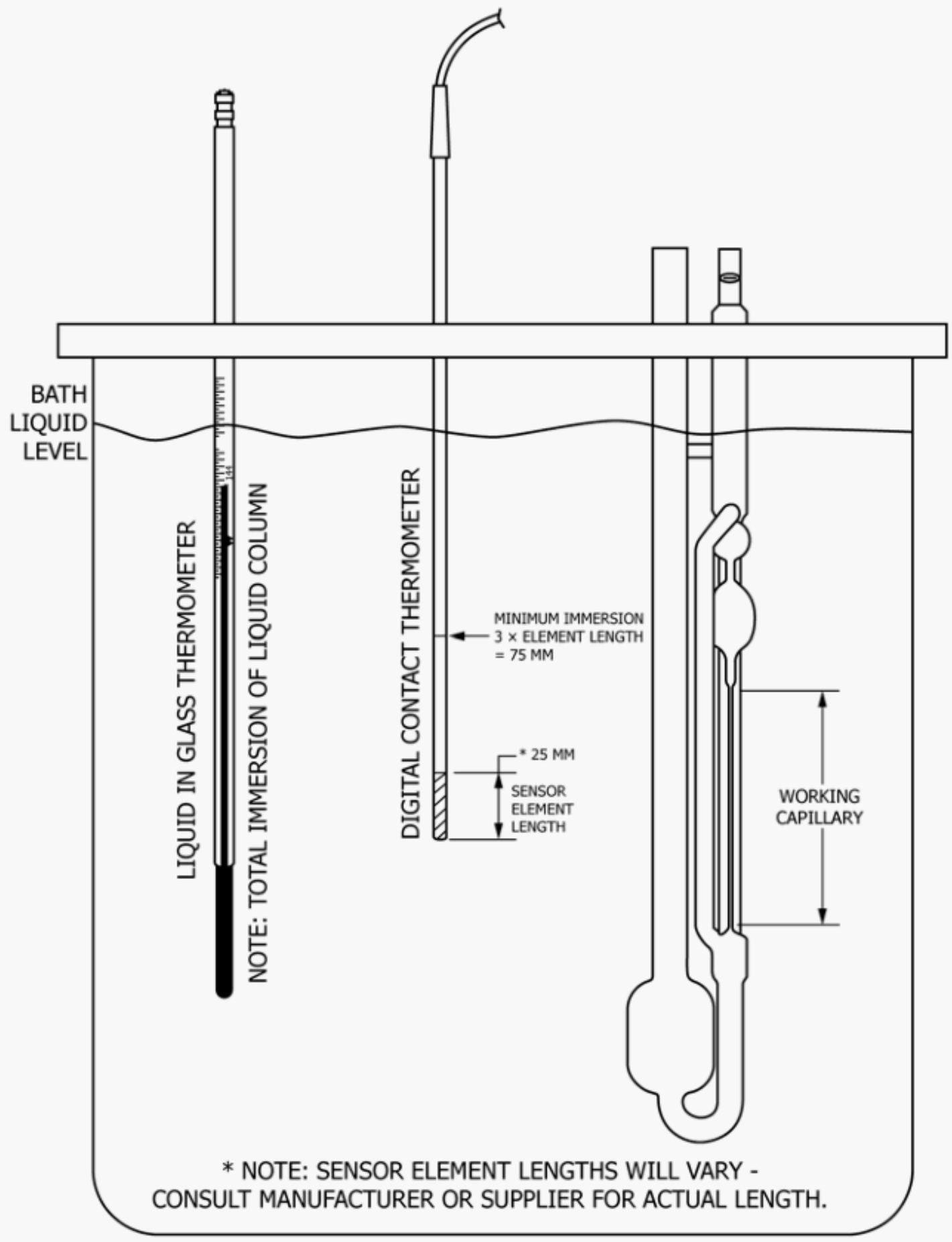


FIG. 1 Temperature Probe Immersion in Constant Temperature Bath

devices, such control can cause large errors in kinematic viscosity flow time measurements.

6.6 *Ultrasonic Bath, Unheated—(optional),* with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤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 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 6—In previous issues of Test Method D445, limits of ±0.35 % of the certified value have been used. The data to support the limit of ±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 that 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.[A]

Viscosity of Reference Material, mm^2/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 %

[A]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 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 the 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 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 7—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 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 °C and 65 °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 °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, ν, 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.)

14. Calculation

14.1 Calculate each of the determined kinematic viscosity values, ν_1 and ν_2, from the measured flow times, t_1 and t_2, and the viscometer constant, C, by means of the following equation:

$$\nu_{1,2} = C \cdot t_{1,2} \tag{2}$$

where:

$\nu_{1,2}$ = determined kinematic viscosity values for ν_1 and ν_2, respectively, mm²/s,
C = calibration constant of the viscometer, mm²/s², and
$t_{1,2}$ = measured flow times for t_1 and t_2, respectively, s.

Calculate the kinematic viscosity result, ν, as an average of ν_1 and ν_2 (see 11.2.3 and 12.4.1).

14.2 Calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \tag{3}$$

where:

η = dynamic viscosity, mPa·s,
ρ = density, kg/m³, at the same temperature used for the determination of the kinematic viscosity, and
ν = kinematic viscosity, mm²/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 Method D1217, D1480, or D1481.

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 and Bias

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 °C[10]	0.0037 y	(0.37 %)
Base oils at 100 °C[10]	0.0036 y	(0.36 %)
Formulated oils at 40 °C[10]	0.0037 y	(0.37 %)
Formulated oils at 100 °C[10]	0.0036 y	(0.36 %)
Formulated oils at 150 °C[11]	0.015 y	(1.5 %)
Petroleum wax at 100 °C[12]	0.0080 y	(0.80 %)
Residual fuel oils at 50 °C[13]	0.0244 y	(2.44 %)
Residual fuel oils at 100 °C[13]	0.03 y	(3 %)
Additives at 100 °C[14]	0.00106 $y^{1.1}$	
Gas oils at 40 °C[15]	0.0013 (y+1)	
Jet fuels at –20 °C[16]	0.0018 y	(0.18 %)
Kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C[17]	0.0037 y	(0.37 %)

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

17.1.2 The determinability for used (in-service) formulated oils has not been determined, however use a limit of 1.0 % (see 12.4.1) for temperatures between 15 °C and 100 °C.[18]

[10] Supporting data has been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1788. These precision values were obtained by statistical examination of interlaboratory results for the following samples: Base Oils with viscosities between (12.0 and 476.0) mm^2/s at 40 °C tested in seven laboratories; Formulated Oils with viscosities between (28.0 and 472.0) mm^2/s at 40 °C tested in seven laboratories; Base Oils with viscosities between (2.90 and 32.0) mm^2/s at 100 °C tested in eight laboratories; Formulated Oils with viscosities between (6.50 and 107.0) mm^2/s at 100 °C tested in eight laboratories. Formulated Oils include automatic transmission fluids, hydraulic fluids, motor oils, gear oils, polymers in base oil and additives in base oil. The determinability, repeatability, and reproducibility results are for tests performed with manual viscometers. Determinability, repeatability, and reproducibility for automated/automatic instruments are no worse than that for the manual instruments. For the precision of specific automated/automatic instruments see Research Report RR:D02-1820.

[11] 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 mm^2/s to 19 mm^2/s at 150 °C, and first published in 1991. See Guide D6074.

[12] 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 mm^2/s to 16 mm^2/s at 100 °C, and were first published in 1988.

[13] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1837. These precision values were obtained by statistical examination of interlaboratory results from eleven laboratories on residual fuel oil samples conforming to D396 Grades 5 or 6 and/or ISO8217 RMG and RMK at 50 °C and 10 at 100 °C in the range from 27.34 mm^2/s to 2395 mm^2/s at 50 °C and 6.36 mm^2/s to 120.8 mm^2/s at 100 °C. These precision statements only refer to measurement of viscosity using manual viscometers.

[14] 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 mm^2/s to 1500 mm^2/s at 100 °C and were first available in 1997.

[15] 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 mm^2/s to 13 mm^2/s at 40 °C and were first available in 1997. Kerosine and diesel fuel samples, which can be considered as gas oils, were included in a dataset to determine the precision for kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C (RR:D02-1780). The precision stated in RR:D02-1780 was developed in a more recent interlaboratory study than the precision stated in RR:D02-1422. Therefore, the gas oil precision statements do not apply to kerosine and diesel fuels and a user should refer to the precision statements for kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C.

[16] 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 mm^2/s to 5.6 mm^2/s at –20 °C and were first available in 1997.

[17] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1780. These precision values were obtained by statistical examination of interlaboratory results from seven samples including kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends (RR:D02-1780) in the range from 2.06 mm^2/s to 4.50 mm^2/s at 40 °C. The determinability, repeatability, and reproducibility results are for tests performed with manual viscometers. Determinability, repeatability, and reproducibility for automated/automatic instruments are no worse than that for the manual instruments. For the precision of specific automated/automatic instruments see Research Report RR:D02-1820.

[18] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1852. The precision values were obtained by statistical examination of interlaboratory results from ten used (in-service) formulated oil samples. These consisted of steam turbine, gas turbine, diesel engine, hydraulic, and gasoline engine oil samples which were analyzed by ten laboratories using both manual and automated apparatuses. The kinematic viscosities of these samples ranged from 25 mm^2/s to 125 mm^2/s at 40 °C, and from 6 mm^2/s to 16 mm^2/s at 100 °C. The statistical output is based on ten laboratories and eight samples at 40 °C and ten laboratories and ten samples at 100 °C.

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 °C[10]	0.0101 x	(1.01 %)
Base oils at 100 °C[10]	0.0085 x	(0.85 %)
Formulated oils at 40 °C[10]	0.0074 x	(0.74 %)
Formulated oils at 100 °C[10]	0.0084 x	(0.84 %)
Formulated oils at 150 °C[11]	0.0056 x	(0.56 %)
Petroleum wax at 100 °C[12]	$0.0141\ x^{1.2}$	
Residual fuel oils at 80 °C[13]	0.013 (x + 8)	
Residual fuel oils at 100 °C[13]	0.08088 x	(8.08 %)
Residual oils at 50 °C[13]	0.07885 x	(7.88 %)
Additives at 100 °C[14]	$0.00192\ x^{1.1}$	
Gas oils at 40 °C[15]	0.0043 (x+1)	
Jet fuels at –20 °C[16]	0.007 x	(0.7 %)
Kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C[17]	0.0056 x	(0.56 %)
Used (in-service) formulated oils at 40 °C[18]	$0.000233\ x^{1.722}$	
Used (in-service) formulated oils at 100 °C[18]	$0.001005\ x^{1.4633}$	

where: x is the average of results being compared.

17.2.1.1 The degrees of freedom associated with the repeatability estimate for the kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C round robin study are 16. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual repeatability may be significantly different than these estimates.

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.

Base oils at 40 °C[10]	0.0136 x	(1.36 %)
Base oils at 100 °C[10]	0.0190 x	(1.90 %)
Formulated oils at 40 °C[10]	0.0122 x	(1.22 %)
Formulated oils at 100 °C[10]	0.0138 x	(1.38 %)
Formulated oils at 150 °C[11]	0.018 x	(1.8 %)
Petroleum wax at 100 °C[12]	$0.0366\ x^{1.2}$	
Residual fuel oils at 80 °C[13]	0.04 (x + 8)	
Residual fuel oils at 100 °C[13]	0.1206 x	(12.06 %)
Residual oils at 50 °C[13]	0.08461 x	(8.46 %)
Additives at 100 °C[14]	$0.00862\ x^{1.1}$	
Gas oils at 40 °C[15]	0.0082 (x+1)	
Jet fuels at –20 °C[16]	0.019 x	(1.9 %)
Kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C[17]	0.0224 x	(2.24 %)
Used (in-service) formulated oils at 40 °C[18]	$0.000594\ x^{1.722}$	
Used (in-service) formulated oils at 100 °C[18]	$0.003361\ x^{1.4633}$	

where: x is the average of results being compared.

17.2.2.1 The degrees of freedom associated with the reproducibility estimate for the kerosine, diesel fuels, biodiesel fuels, and biodiesel fuel blends at 40 °C round robin study are 19. Since the minimum requirement of 30 (in accordance with Practice D6300) is not met, users are cautioned that the actual reproducibility may be significantly different than these estimates.

17.3 The precision for specific automated and automatic viscometers has been determined for sample types and temperatures listed in 17.3.1. An analysis has been made of a large dataset including both automated/automatic and manual viscometers over the temperature range of 40 °C to 100 °C for the sample types listed in 17.3.1. The determinability, repeatability, and reproducibility of automated/automatic viscometer data are no worse than the determinability, repeatability, and reproducibility for the manual instruments. It is also shown in the research reports that no statistically significant bias was observed between the automated/automatic data in comparison to the manual data.[19] For the precision of specific automated/automatic instruments, see RR:D02-1820.[20]

17.3.1 The determinability, repeatability, and reproducibility have been determined for automated/automatic viscometers for the following sample types and temperatures:

Distillates, fatty acid methyl esters, and distillates containing fatty acid methyl esters at 40 °C

Base oils at 40 °C and 100 °C

Formulated oils at 40 °C and 100 °C

For these sample types, determinability, repeatability, and reproducibility for automated/automatic instruments are no worse than that for the manual instruments. For the precision of specific automated/automatic instruments see Research Report RR:D02-1820.

The precision has been determined for automated viscometers and the range of r and R values for automated instruments is shown in RR:D02-1820. For the samples listed in RR:D02-1820, precision for automated instruments is no worse than that for the manual instruments.[21]

17.3.1.1 *Degree of Agreement Between Results by Manual and Automated Instruments in Test Method D445*—Results for the sample types listed in RR:D02-1820 produced by manual and automated instruments in this test method have been assessed in accordance with procedures outlined in Practice D6708.

17.3.1.2 The findings are: Results from manual and automated instruments in Test Method D445 may be considered to be practically equivalent, for sample types listed in RR:D02-1820. No sample-specific bias, as defined in Practice D6708, was observed for the materials studied. Differences between results from manual and automated instruments in Test Method D445, for the samples listed in RR:D02-1820, are expected to exceed the following between methods reproducibility: 1.91 % for distillates, fatty acid methyl esters, and distillates containing fatty acid methyl esters at 40 °C; 1.27 % for base oils at 40 °C; 1.23 % for formulated oils at 40 °C; 1.70 % for base oils

[19] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1498. Contact ASTM Customer Service at service@astm.org.

[20] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1820. Contact ASTM Customer Service at service@astm.org.

[21] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1787. These precision values were obtained by statistical examination of interlaboratory results from seven samples including distillates, fatty acid methyl esters, and distillates containing fatty acid methyl esters (RR:D02-1790) in the range from (2.06 to 4.50) mm^2/s at 40 °C. These seven samples were tested in 21 different Cannon and Herzog instruments to obtain the precision values shown.

at 100 °C; 1.21 % for formulated oils at 100 °C, as defined in Practice D6708, about 5 % of the time. These percent differences are based upon the highest calculated combined method reproducibilities (R_{xy} in Practice D6708).[22]

[22] Supporting data has been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1787. These precision values were obtained by statistical examination of interlaboratory results for the following samples: Base Oils with viscosities between (12.0 and 476.0) mm^2/s at 40 °C tested in 22 laboratories; Formulated Oils with viscosities between (28.0 and 472.0) mm^2/s at 40 °C tested in 22 laboratories; Base Oils with viscosities between (2.90 and 32.0) mm^2/s at 100 °C tested in 21 laboratories; Formulated Oils with viscosities between (6.50 and 107.0) mm^2/s at 100 °C tested in 21 laboratories. Formulated Oils include automatic transmission fluids, hydraulic fluids, motor oils, gear oils, polymers in base oil, and additives in base oil.

18. Keywords

18.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

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 D446.

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

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm^2/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B] (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

[A] 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 D446.
[B] 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^2/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 Liquid-in-Glass Thermometer

A2.1.1 Use a short-range specialized liquid-in-glass 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. As an alternative, use a digital contact thermometer (DCT) as defined in 6.4.2.

A2.1.2 The difference in the designs of the liquid-in-glass thermometers 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 When using a liquid-in-glass thermometer, it shall have a report of temperature calibration traceable to a national calibration or metrology standards body issued by a competent calibration laboratory with demonstrated competency in temperature calibration. An ISO 17025 accredited laboratory with temperature calibration in its accreditation scope would meet this requirement. The calibration report shall include data for a series of temperatures which are appropriate for its intended use.

A2.2.2 The scale correction of liquid-in-glass thermometers can change during storage and use. Therefore, regular in-house ice-point verifications are required, and can be achieved within the working laboratory, using an ice melting bath. However, these checks may not be an adequate means of recalibration as the use of a single-point recalibration at the ice point adds an additional uncertainty to the updated thermometer calibration result at every temperature but the ice point. The user must determine if the thermometer requires complete re-calibration to continue to meet the expanded measurement uncertainty requirements of this section. NIST Special Publication 1088, Section 7.9 on determining uncertainty of correction, may be helpful to the user in making this decision. If in-house ice-point verification brings the expanded measurement uncertainty out of the requirements of A2.2.1, complete recalibration at a laboratory meeting the requirements of A2.2.1 is required.

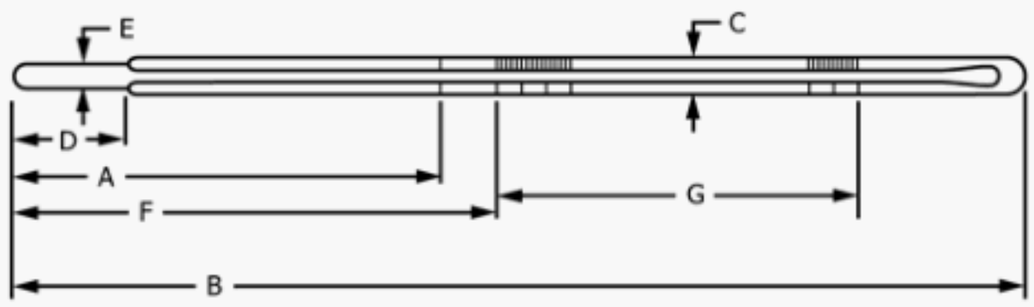
TABLE A2.1 General Specification for Thermometers

NOTE 1—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 E1 and Test Method E77.

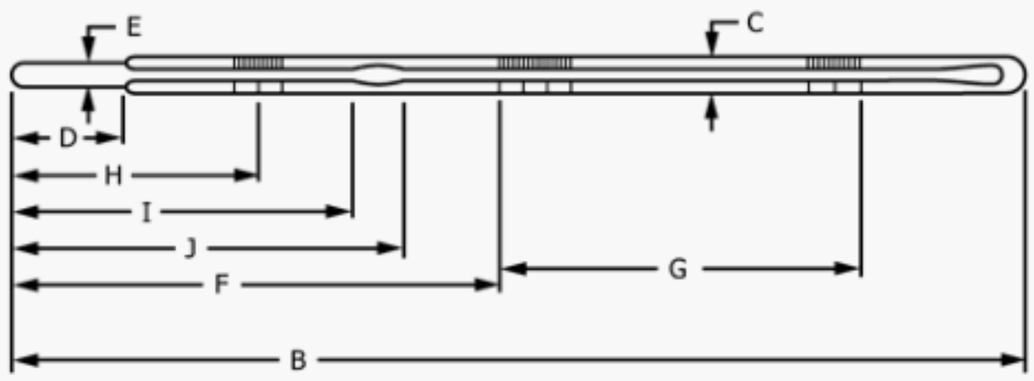
Immersion		Total
Scale marks:		
Subdivisions	°C	0.05
Long lines at each	°C	0.1 and 0.5
Numbers at each	°C	1
Maximum line width	mm	0.10
Scale error at test temperature, max	°C	0.1
Expansion chamber:		
Permit heating to	°C	105 up to 90, 120 between 90 and 95 130 between 95 and 105, 170 above 105
Total 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

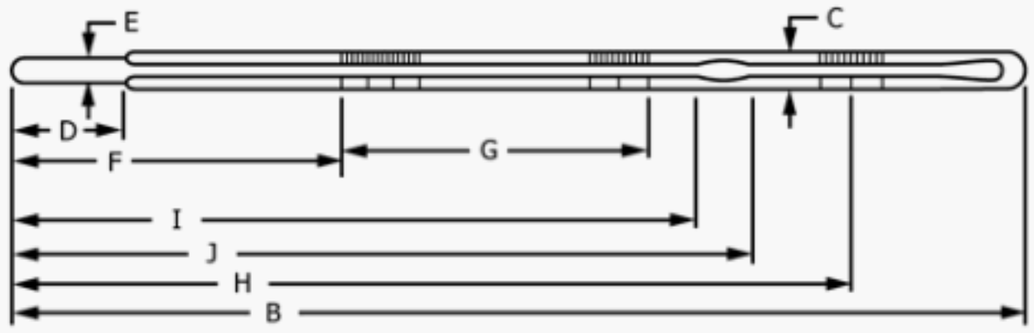
Thermometer No.	Test Temperature		Thermometer No.	Test Temperature	
	°C	°F		°C	°F
ASTM 132C, IP 102C	150		ASTM 128C, F/IP 33C	0	32
ASTM 110C, F/IP 93C	135	275	ASTM 72C, F/IP 67C	–17.8	0
ASTM 121C/IP 32C	98.9,	210,	ASTM 127C/IP 99C	–20	–4
	100	212	ASTM 126C, F/IP 71C	–26.1	–20
ASTM 129C, F/IP 36C	93.3	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			



(a)



(b)



(c)

FIG. A2.1 Thermometer Designs

A2.2.2.1 For liquid-in-glass thermometers, the interval for ice-point verification 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.

A2.2.2.2 Keep records of all recalibration.

A2.2.3 *Procedure for Ice-point Verification 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. NIST Special Publication 1088 from the NIST website describes an effective procedure for ice-point verification, including all formulae necessary for calculations of change of correction and measurement uncertainty. Alternatively, use Practice E563 or those in A2.2.3.2 through A2.2.3.5.

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 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. See A2.2.1 for discussion on expanded measurement uncertainty changes when changing correction values from ice-point verifications.

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 0.01 °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

NOTE A4.1—These calculations are based on Practice D6617.

A4.1 Determine the standard deviation for site uncertainty, σ_{site}, from a laboratory quality control program. (See Practice D6299.)

A4.1.1 If the standard deviation for site uncertainty, σ_{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. In this test method, the CRM is the Certified Viscosity Reference Standard (CVRS) as defined in 9.2.

NOTE A4.2—Combined Extended Uncertainty (CEU) is equivalent to Expanded Uncertainty (*U*). See NIST Technical Note 1297.

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

$$SE_{ARV} = \frac{CEU_{ARV}}{k} \quad (A4.1)$$

NOTE A4.3—Standard Error (SE_{ARV}) is equivalent to Combined Standard Uncertainty (U_C). See NIST Technical Note 1297.

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

A4.4 Construct the acceptable tolerance zone:

$$TZ = \pm 1.44 \sqrt{\sigma_{site}^2 + SE_{ARV}^2} \quad (A4.2)$$

A4.5 Worked out example for kinematic viscosity zone (band):

$$\sigma_{site} = 0.19\,\% \text{ (default value from A4.1.1)} \quad (A4.3)$$

$$CEU_{ARV} = 0.22\,\% \text{ (from the CVRS label or documentation)}$$

$$k = 2 \text{ (from the CVRS label or documentation)}$$

$$SE_{ARV} = \frac{0.22}{2} = 0.11$$

$$TZ = \pm 1.44\sqrt{0.19^2 + 0.11^2} = \pm 1.44\sqrt{0.0361 + 0.0121} = \pm 0.32\,\%$$

A4.5.1 In this example, the tolerance zone will be ±0.32 % of the certified viscosity reference standard value on the report of test or bottle label. If this site uses a CVRS (for example) with a kinematic viscosity of 33.98 mm^2/s, the TZ = 33.87 mm^2/s to 34.09 mm^2/s, with 95 % certainty. Viscosity measurements made with this CVRS at that site should fall within that tolerance zone (band) 19 out of 20 times.

APPENDIX

(Nonmandatory Information)

X1. TROUBLESHOOTING GUIDE

X1.1 There are many factors that can impact the precision of this test method. Subcommittee D02.07 has developed a troubleshooting guide to assist users in determining which relevant sections in this test method may require closer attention. See Table X1.1.

TABLE X1.1 Troubleshooting Guide

Observed Error	Possible Causes	Resolution	Reference
Flow times don't meet determinability of D445 or are drifting	Foreign particle lodged in the viscometer capillary	Visually inspect for foreign particles in capillary and back flush or clean with mild detergent or acid	13.2
	Organic or inorganic deposits on viscometer capillary wall		
	Particulates in sample	Filter new samples to remove visible particles. For used oil samples, the largest particle size should not exceed one half the capillary diameter. Select a larger viscometer tube range.	11.1, 12.2, Note 6
	Air bubbles in the sample	Allow sample to settle or use ultrasonic bath to remove air	6.6, 11.1
	Water in sample (such as with in-service engine oils)	Remove water from sample prior to testing	
	Samples tested at sub-ambient temperatures may have wax precipiates which may clog the capillary during measurement	Test sample at warmer temperature or remove wax from the sample prior to testing	12.1, 12.1.4, 12.1.5
	Sample is mixed phases or non-homogeneous	Precision in D445 is not known for these types of samples	11.2.4, 12.4.1
	Temperature of bath not regulating within method requirements	Contact bath supplier to confirm/verify equipment performance specifications meet the apparatus section of method. If bath set point temperature is near ambient, an external chiller might be required.	6.3, 6.4
	Bath has temperature gradients	Verify bath liquid is circulating and the bath temperature uniformity using a second reference temperature measuring device. Verify the proper viscosity of bath fluid is being used as per the manufacturer's recommendations for the specific test temperature being used.	6.3, 6.4, Fig. 1
	Sample thermal equilibrium time too short	Allow sample to remain in viscometer for 30 min prior to measurement	11.1.3 – 11.1.5, 12.2.3
	Sample requires thermal pretreatment prior to measurement		11.1.2, 12.1
	Inaccurate flow time measurement	Verify timing device meets specifications or operator technique is proper	6.5
Short flow times <200 s	Viscometer range too large	Select smaller range viscometer	10.2
	Actual bath temperature warmer than desired target temperature	Verify bath temperature set point	6.3, 6.4
		Verify reference temperature measuring device calibration	6.3, 6.4
		Verify that any temperature calibration offset (see calibration certificate) is being applied	6.4.1, 6.4.4
Excessively long flow times	Viscometer range too small	Select a larger range viscometer	10.2
	Actual bath temperature colder than desired target temperature	Verify bath temperature set point	6.3, 6.4
		Set point temperature is close to ambient temperature and the viscosity bath needs to be connected to an external chiller	
		Verify reference temperature measuring device calibration	6.3, 6.4
		Verify that any temperature calibration offset (see calibration certificate) is being applied	6.4.1, 6.4.4

TABLE X1.1 *Continued*

Observed Error	Possible Causes	Resolution	Reference
Measured results with certified viscosity reference standard (CVRS) or check standard exceed the published repeatability	Temperature of bath is not at target temperature	Verify bath temperature set point	6.3, 6.4
		Verify reference temperature measuring device calibration	6.3, 6.4
		Verify that any temperature calibration offset (see calibration certificate) is being applied	6.4.1, 6.4.4
	Viscometer tube is not properly cleaned or dried	Verify solvent system compatibility with sample chemistry	7.2, 7.3, 7.4, 10.2.3
	Contaminated CVRS or check standard	Verify with alternate CVRS or verify check standard in different viscometer	9.2
	Viscometer tube calibration error or not in accord with D446	Verify viscometer meets the specifications of D446 and that calibration was performed by using reference viscometers or by certified viscosity reference standards with traceability in accord with ISO 17025. Verify that the calibration constant on the viscometer calibration certificate is appropriate for the test temperature being used and the gravitational correction is applied as required. Note additional sources of error may arise from using certified viscosity reference standards: These may arise from contamination, exposure to sunlight, expired shelf life, or only one standard being used instead of the mandatory two standards, whereby the second standard has a flow time of at least 50 % longer than the first oil and incorrect bath temperature during the calibration.	See 6.2 and 6.3 of D446; see 9.3 of D445
	Viscometer tube does not meet D446 specification	Verify with supplier that viscometer is constructed in accord with the dimensions and tolerances noted in D446, otherwise the viscometer precision may deviate from the method precision	See D446

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D445 – 18) that may impact the use of this standard. (Approved June 1, 2019.)

(1) Revised subsection 6.4.2.
(2) Added new Appendixes (nonmandatory information) section and new Appendix X1.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D445 – 17a) that may impact the use of this standard. (Approved Nov. 1, 2018.)

(1) Revised subsection 6.4 and Fig. 1; added new subsection 6.4.4.

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