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

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures.

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

1. Scope

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, 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 D 2170 and D 2171.

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

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm²/s (see Table A1.1) at all temperatures (see 6.3 and 6.4). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the*

responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers²

D 1193 Specification for Reagent Water³

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer²

D 1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer²

D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer²

D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)⁴

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer⁴

D 6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils⁵

E 1 Specification for ASTM Thermometers⁶

E 77 Test Method for the Inspection and Verification of Thermometers⁶

2.2 ISO Standards:⁷

ISO Guide 25—General Requirements for the Calibration and Testing Laboratories

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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

² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 04.03.

⁵ *Annual Book of ASTM Standards*, Vol 05.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.03.

⁷ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

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

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

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

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

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

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

4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

5. Significance and Use

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

6. Apparatus

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

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D 446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method

to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automation*—Automated viscometers, which have been shown to determine kinematic viscosity within the limits of precision given in Section 16 are acceptable alternatives. Apply a kinetic energy correction (see Specifications D 446 and ISO 3105) to kinematic viscosities less than 10 mm²/s and flow times less than 200 s.

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

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D 446, see Operating Instructions in Annex A1, Annex A2, and Annex A3. For those viscometers which have Tube L (see Specifications D 446) held vertical, vertical alignment shall be confirmed by using (1) a holder ensured to hold Tube L vertical, or (2) a bubble level mounted on a rod designed to fit into Tube L, or (3) a plumb line suspended from the center of Tube L, or (4) other internal means of support provided in the constant temperature bath.

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

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

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

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within 0.04°C.

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

6.4.3 When using liquid-in-glass thermometers, such as those in Table A2.2, use a magnifying device to read the thermometer to the nearest 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 9.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better, and has an accuracy within $\pm 0.07\%$ (see Annex A3) of the reading when tested over intervals of 200 and 900 s.

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

7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution.

NOTE 2—**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.

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

8. Calibration and Verification

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

8.2 *Certified Viscosity Reference Standards*⁸ (Table A1.2)—These are for use as confirmatory checks on the procedure in the laboratory.

8.2.1 If the determined kinematic viscosity does not agree within $\pm 0.35\%$ 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.

8.2.2 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.

8.3 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.

9. General Procedure for Kinematic Viscosity

9.1 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.

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

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

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

9.2 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 shall not be less than 200 s or the longer time noted in Specifications D 446.

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

9.2.2 When the test temperature is below the dew point, affix loosely packed drying tubes to the open ends of the viscometer. The drying tubes must fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument. Carefully flush the moist room air from the viscometer by applying vacuum to one of the drying tubes. Finally, before placing the viscometer in the bath, draw up the sample into the working capillary and timing bulb and allow to drain back as an additional safeguard against moisture condensing or freezing on the walls.

9.2.3 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.

10. Procedure for Transparent Liquids

10.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity

⁸ The ASTM Viscosity Oil Standards are available in 1-pt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA 16804. Shipment will be made as specified or by best means.

with that employed when the instrument was calibrated. If the sample contains solid particles, filter during charging through a (75- μm) filter (see Specifications D 446). 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 D 446).

NOTE 3—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.

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

10.1.2 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.

10.1.3 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 a viscometer while any other viscometer is in use for measuring a flow time.

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

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

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

10.2 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 9.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

10.2.1 Repeat the procedure described in 10.2 to make a second measurement of flow time. Record both measurements.

10.2.2 If the two determinations of kinematic viscosity, calculated from the flow time measurements, agree within the stated determinability figure (see 16.1) for the product, use the average of these determinations to calculate the kinematic viscosity result to be reported. Record the result. If the determinations of kinematic viscosity do not agree within the stated determinability, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering (where required, see 10.1) of the sample. If the material or temperature, or both, is not listed in 16.1, for temperatures between 15 and 100°C use as an estimate of the determinability 0.20 %, and 0.35 % for temperatures outside this range.

11. Procedure for Opaque Liquids

11.1 For steam-refined cylinder oils and black lubricating oils, proceed to 11.3 ensuring a thoroughly representative

sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 11.1.1-11.2.2 shall be followed to minimize this.

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

11.1.2 Heat in the original container, in an oven, at $60 \pm 2^\circ\text{C}$ for 1 h.

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

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

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

11.2 Immediately after completing 11.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

11.2.1 Immerse the flask in a bath of boiling water for 30 min.

NOTE 4—**Precaution:** Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.

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

11.3 Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75- 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.

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

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

11.3.3 Allow the charged viscometers enough time to reach the test temperature (see 11.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw a viscometer while any other viscometer is in use for measuring flow time.

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

11.4.1 In the case of samples requiring heat treatment described in 11.1-11.2.1, complete the measurements of flow time within 1 h of completing 11.2.2. Record the measured flow times.

11.5 Calculate kinematic viscosity, ν , in mm^2/s , from each measured flow time.

11.5.1 For residual fuel oils, if the two determinations of