Designation: D7279 – 18ε1

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids by Automated Houillon Viscometer[1]

This standard is issued under the fixed designation D7279; 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.

ε[1] NOTE—Editorially corrected Eq 1 in December 2019.

1. Scope*

1.1 This test method covers the measurement of the kinematic viscosity of transparent and opaque liquids; such as base oils, formulated oils, diesel oil, biodiesel, biodiesel blends, residual fuel oils, marine fuels, and used lubricating oils using a Houillon viscometer in automated mode.

1.2 The range of kinematic viscosity capable of being measured by this test method is from 2 mm^2/s to 2500 mm^2/s (see Fig. 1). The range is dependent on the tube constant utilized. The temperature range that the apparatus is capable of achieving is between 20 °C and 150 °C, inclusive. However, the precision has only been determined for the viscosity range; 2 mm^2/s to 478 mm^2/s at 40 °C for base oils, formulated oils, diesel oil, biodiesel, and biodiesel blends; 3 mm^2/s to 106 mm^2/s at 100 °C for base oils and formulated oils; 25 mm^2/s to 150 mm^2/s at 40 °C and 5 mm^2/s to 16 mm^2/s at 100 °C for used lubricating oils; 25 mm^2/s to 2500 mm^2/s at 50 °C and 6 mm^2/s to 110 mm^2/s at 100 °C for residual fuels. As indicated for the materials listed in the precision section.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.* For specific warning statements, see Section 6.

1.5 *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]

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

D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

D7962 Practice for Determination of Minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift

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

E2877 Guide for Digital Contact Thermometers

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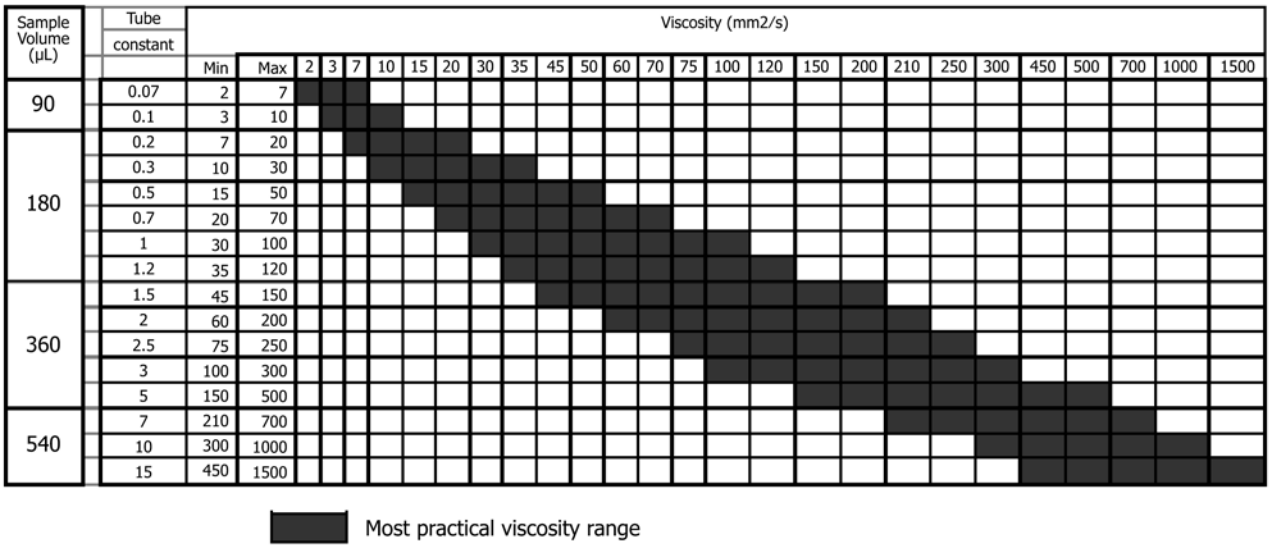
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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



NOTE 1—Viscosity range of a Houillon tube is based on most practical flow time of 30 s to 200 s.

FIG. 1 Houillon Viscometer Typical Viscosity Range of Tube Constants

2.2 *ISO Standards:*

ISO 5725 Accuracy (Trueness and Precision) of Measurement Methods and Results[3]

ISO/EC 17025 General Requirements for the Competence of Testing and Calibration Laboratories[3]

2.3 *NIST Standard:*

NIST Technical Note 1297 Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results[4]

3. Summary of Test Method

3.1 The kinematic viscosity is determined by measuring the time taken for a sample to fill a calibrated volume at a given temperature. The specimen is introduced into the apparatus and then flows into the viscometer tube which is equipped with two detection cells. The specimen reaches the test temperature of the viscometer bath and when the leading edge of the specimen passes in front of the first detection cell, the automated instrument starts the timing sequence. When the leading edge of the specimen passes in front of the second detection cell, the instrument stops timing the flow. The time interval thus measured allows the calculation of the kinematic viscosity using a viscometer tube constant determined earlier by calibration with certified viscosity reference standards.

3.2 The kinematic viscosity is calculated using the formula:

$$v = C \times t \quad (1)$$

where:

v = the kinematic viscosity in mm^2/s,
C = the viscometer tube constant in mm^2/s^2, and
t = the flow time in s measured during the test.

4. Significance and Use

4.1 Many petroleum products and some non-petroleum products are used as lubricants in the equipment, and the correct operation of the equipment depends upon the appropriate viscosity of the lubricant being used. Additionally, 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.

4.2 The viscosity of used oils is a commonly determined parameter in the oil industry to assess the effect of engine wear on the lube oils used, as well as the degradation of the engine parts during operation.

4.3 The Houillon viscometer tube method offers automated determination of kinematic viscosity. Typically a sample volume of less than 1 mL is required for the analysis.

5. Apparatus

5.1 *Automated Viscometer*—The system shall consist of the following components:

5.1.1 *Viscometer Bath:*

5.1.1.1 *Bath,* to ensure optimal thermal equilibration of the system, the bath is filled with mineral or silicone oil and equipped with a stirring device.

5.1.2 *Temperature Regulation System,* to control the bath temperature to within 0.02 °C.

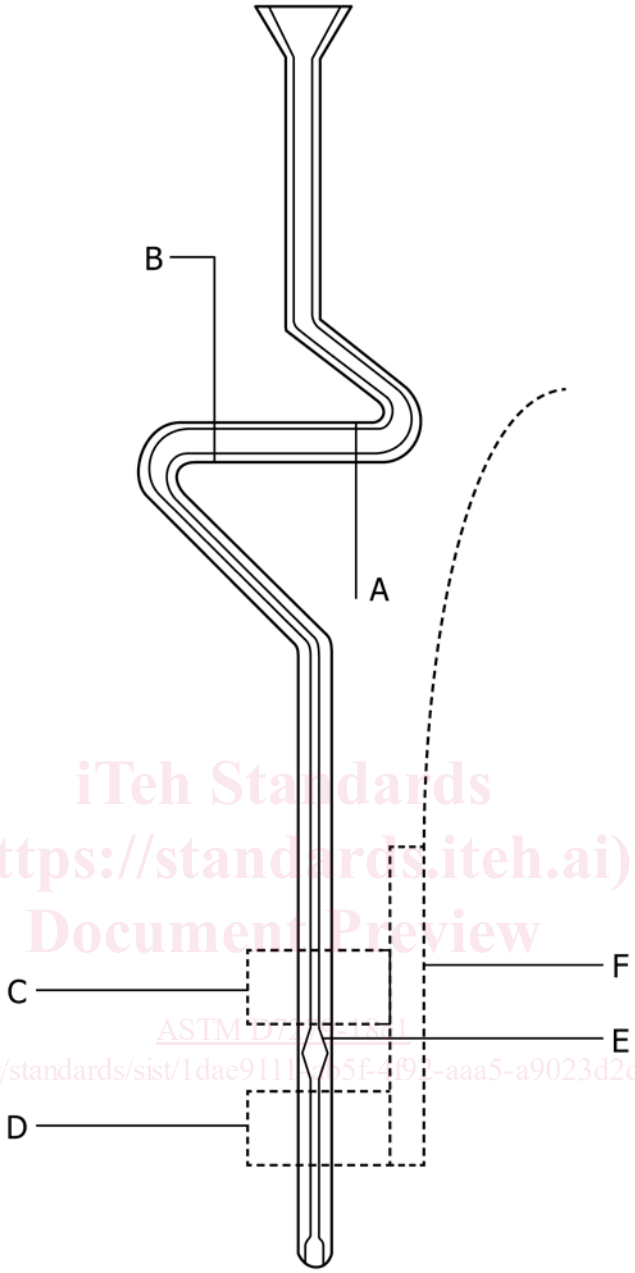
5.1.3 *Houillon Viscometer Tubes,* made of glass with a calibrated volume which varies depending on the tube size (see Fig. 2). This technique allows the viscosity to be measured over a wide range of values (see Fig. 1).

5.1.4 *Cleaning/Vacuum System,* consisting of one or more solvent reservoirs to transport the solvent(s) to the viscometer tubes, dry the viscometer tubes after the flushing cycle, to remove the sample, and for drainage of waste products.

5.1.5 *Automated Viscometer Control System*—Suitable electronic processor capable of operating the apparatus, controlling

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

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.



A and B = sample reservoir
C and D = calibrated volume—measurement zone
E = bulb
F = detection cell

Tube Filling Volume for a Measurement
The filling volume is OK when:
At the beginning of a measurement:
Sample lower meniscus is on C (start timing)
Sample upper meniscus should be below A
At the end of a measurement:
Sample lower meniscus is on D (stop timing)
Sample upper meniscus should be above B

FIG. 2 Houillon Tube Schematic Diagram

the operation of the timers, regulating the bath temperature, cleaning the viscometer tubes, and recording and reporting the results.

5.1.6 *PC-compatible Computer System,* may be used for data acquisition, as per manufacturer's instructions.

5.1.7 *Timing Devices*—Use any timing device that is capable of taking readings with a discrimination of 0.01 s or better with an accuracy within ±0.07 % of the reading when tested over the minimum and maximum intervals of expected flow times.

5.1.8 *Volume Delivery Device,* such as a micropipette, capable of delivering a sufficient volume of sample to the Houillon tube being used. (See Fig. 1 for approximate sample volumes.)

5.2 *Temperature Measuring Devices*—Use either calibrated liquid-in-glass thermometers, of an accuracy after correction of ±0.02 °C or better, or other thermometric devices such as a digital contact thermometer as described in 5.2.1 with equal or better accuracy.

5.2.1 *Digital Contact Thermometer Requirements:*

Parameter	Requirement
Nominal temperature range[A]	20 ° C to +150 ° C
Display resolution	0.01 °C minimum
Accuracy[B]	±20 mK (±0.02 °C)
Sensor type	Platinum Resistance Thermometer (PRT), thermistor
Sensor sheath[C]	7 mm OD maximum
Sensor length[D]	Less than 18 mm
Immersion depth[E]	Less than 40 mm per Practice D7962
Measurement Drift[E]	less than 10 mK (0.01 °C) per year
Response time[F]	less than or equal to 8 s per footnote F
Calibration error	less than 10 mK (0.01 °C) over the range of intended use
Calibration range	Consistent with temperature range of use
Calibration data	Two data points when the "range of use" is less than 30 °C. At least three data points when the "range of use" is 30 °C to 90 °C. At least four data points when "range of use" is greater than 90 °C. When more than 2 data points, they shall be evenly distributed over the calibration range. The calibration data is to be included in calibration report.
Calibration report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body

[A] The nominal temperature range may be different from the values shown provided the calibration and accuracy criteria are met.
[B] Accuracy is the combined accuracy of the DCT unit, which is the display and sensor. See Guide E2877 for more information on selecting a DCT.
[C] Sensor sheath is the tube that holds the sensing element.
[D] The physical length of the temperature sensing element.
[E] As determined by Practice D7962 or an equivalent procedure.
[F] *Response Time* —The time for a DCT to respond to a step change in temperature. The response time is 63.2 % of the step change time as determined per Section 9 of Test Method E644. The step change evaluation begins at 20 °C ± 5 °C air to 77 °C ± 5 °C with water circulating at 0.9 m/s ± 0.09 m/s past the sensor.

5.2.2 *Measurement Drift*—The drift in calibration should be checked periodically and at least once per year. This can be accomplished using Practice D7962 or Test Methods E644. When a DCT's calibration drifts in one direction over several checks against a reference temperature, such as the ice point, it may be an indication of deterioration of the DCT. The probe is to be recalibrated when the check value differs by more than the drift listed in 5.2.1 since the last probe calibration. See Practice E563, Test Methods E644, or Guide E1750 for more information regarding checking calibrations.

5.2.3 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. Reagents and Materials

6.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO/EC 17025 by independent assessment. The certified viscosity reference standards shall be traceable to master viscometer procedures described in Test Method D2162.

6.1.1 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.

6.2 Non-chromium-containing, strongly oxidizing acid cleaning solution. (**Warning—**Non-chromium-containing, strongly oxidizing acid cleaning solutions are highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

6.3 Solvent(s) for cleaning, drying, reagent grade. Refer to manufacturer's recommendations. Filter before use if necessary. Typical solvent(s) include:

6.3.1 Toluene. (**Warning—**Flammable. Vapor harmful.)

6.3.2 Petroleum spirit or naphtha. (**Warning—**Flammable. Health hazard.)

6.3.3 Acetone. (**Warning—**Extremely flammable. Health hazard.)

6.3.4 Heptane. (**Warning—**Flammable. Health hazard.)

6.4 Technical grade silicone oil or white oil of appropriate viscosity (for example, about 100 mm^2/s @ 25 °C or equivalent) to maintain the test temperature.

7. Sampling and Test Specimens

7.1 Obtain a representative test specimen in accordance with Practice D4057 or Practice D4177.

7.2 *Instructions for Residual Fuel Oils*—(**Warning—**Exercise care as vigorous boil-over can occur when opaque liquids that contain high levels of water are heated to high temperatures. Wear appropriate personal protective equipment for handling hot materials.)

7.2.1 Place the first batch of resid samples to be analyzed for the day in their original containers in a sample pre-heat apparatus that is held between 60 °C and 65 °C for 1 h. Ensure the cap on each container is tightly closed.

7.2.2 Rigorously stir each sample for approximately 20 s with a glass or steel rod of sufficient length to reach the bottom of the container.