



Designation: D7279 – 20

# Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids by Automated Houillon Viscometer<sup>1</sup>

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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 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<sup>2</sup>/s to 2500 mm<sup>2</sup>/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<sup>2</sup>/s to 478 mm<sup>2</sup>/s at 40 °C for base oils, formulated oils, diesel oil, biodiesel, and biodiesel blends; 3 mm<sup>2</sup>/s to 106 mm<sup>2</sup>/s at 100 °C for base oils and formulated oils; 25 mm<sup>2</sup>/s to 150 mm<sup>2</sup>/s at 40 °C and 5 mm<sup>2</sup>/s to 16 mm<sup>2</sup>/s at 100 °C for used lubricating oils; 25 mm<sup>2</sup>/s to 2500 mm<sup>2</sup>/s at 50 °C and 6 mm<sup>2</sup>/s to 110 mm<sup>2</sup>/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 7.

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.*

<sup>1</sup> 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. Referenced Documents

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

- 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, Liquid Fuels, 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
- D8278 Specification for Digital Contact Thermometers for Test Methods Measuring Flow Properties of Fuels and Lubricants
- E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature
- E1750 Guide for Use of Water Triple Point Cells

### 2.2 ISO Standards:

- ISO 5725 Accuracy (Trueness and Precision) of Measurement Methods and Results<sup>3</sup>

<sup>2</sup> 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.

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

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

Sample Volume (μL)	Tube constant	Viscosity (mm <sup>2</sup> /s)																										
		Min	Max	2	3	7	10	15	20	30	35	45	50	60	70	75	100	120	150	200	210	250	300	450	500	700	1000	1500
90	0.07	2	7																									
	0.1	3	10																									
180	0.2	7	20																									
	0.3	10	30																									
	0.5	15	50																									
	0.7	20	70																									
	1	30	100																									
	1.2	35	120																									
360	1.5	45	150																									
	2	60	200																									
	2.5	75	250																									
	3	100	300																									
540	5	150	500																									
	7	210	700																									
	10	300	1000																									
	15	450	1500																									

 Most practical viscosity range

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

ISO/EC 17025 General Requirements for the Competence of Testing and Calibration Laboratories<sup>3</sup>

2.3 NIST Standard:

NIST Technical Note 1297 Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results<sup>4</sup>

3. Terminology

3.1 Definitions:

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

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

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

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

4. Summary of Test Method

4.1 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.

4.2 The kinematic viscosity is calculated using the formula:

$$v = C \times t \tag{1}$$

where:

- v* = the kinematic viscosity in mm<sup>2</sup>/s,
- C* = the viscometer tube constant in mm<sup>2</sup>/s<sup>2</sup>, and
- t* = the flow time in s measured during the test.

5. Significance and Use

5.1 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.

5.2 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.

5.3 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.

6. Apparatus

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

6.1.1 Viscometer Bath:

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

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

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

6.1.3 *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).

6.1.4 *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.

6.1.5 *Automated Viscometer Control System*—Suitable electronic processor capable of operating the apparatus, controlling the operation of the timers, regulating the bath temperature, cleaning the viscometer tubes, and recording and reporting the results.

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

6.1.7 *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  $\pm 0.07\%$  of the reading when tested over the minimum and maximum intervals of expected flow times.

6.1.8 *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.)

6.2 *Temperature Measuring Devices*—Use either calibrated liquid-in-glass thermometers, of an accuracy after correction of  $\pm 0.02$  °C or better, or other thermometric devices such as a digital contact thermometer as described in 6.2.1 with equal or better accuracy.

6.2.1 *Digital Contact Thermometer*—Use D02-DCT07 listed in Specification D8278.

6.2.2 *Measurement Drift*—The drift in calibration should be checked periodically and at least once per year. This can be accomplished using Practice D7962. 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 for the DCT since the last probe calibration. See Practice E563 or Guide E1750 for more information regarding checking calibrations.

6.2.3 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.

## 7. Reagents and Materials

7.1 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.

7.1.1 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.

7.2 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.)

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

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

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

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

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

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

## 8. Sampling and Test Specimens

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

8.2 *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.)

8.2.1 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.

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

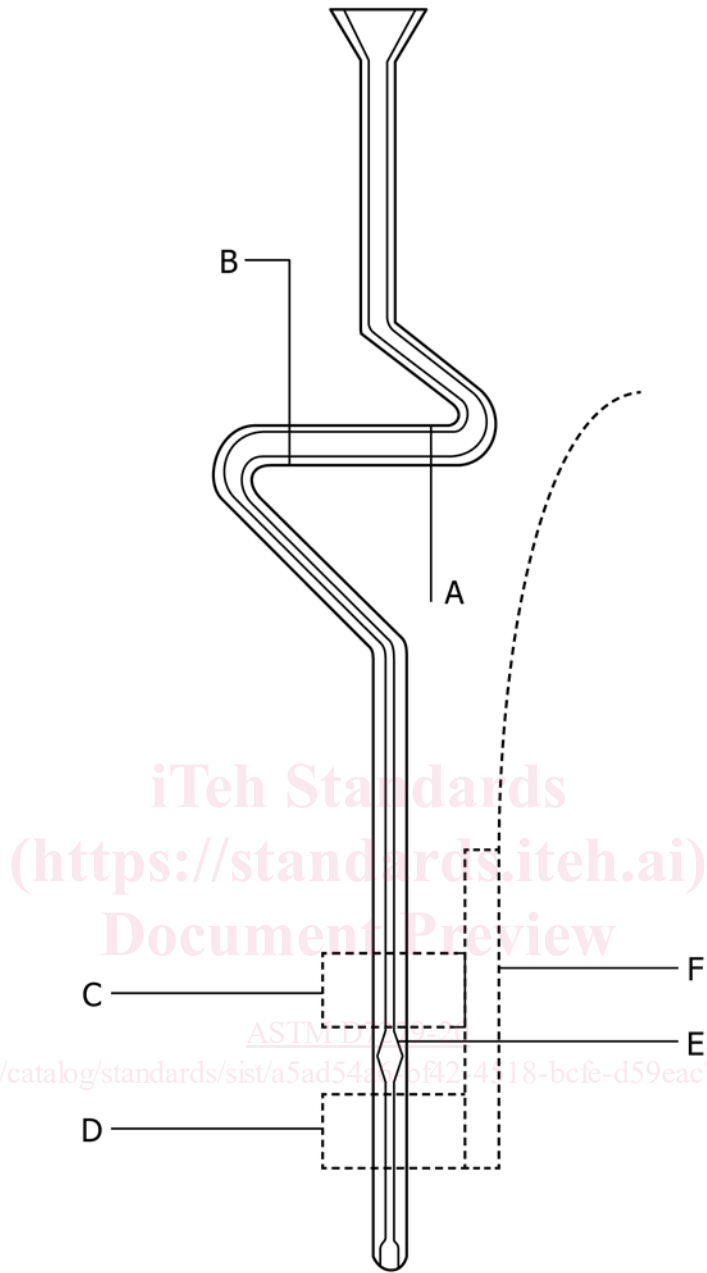
8.2.3 Remove the stirring rod and inspect for sludge or wax adhering to the rod. If there is sludge or wax adhering to the rod, continue stirring until the sample is homogeneous.

8.2.4 Recap each container tightly and shake vigorously for 1 min. Then loosen the cap, retighten to finger tight, then back off  $\frac{3}{4}$  turn to a full turn and place back into the sample pre-heat apparatus.

8.2.5 Upon completion of 8.2.4 for all samples in the batch, increase the sample pre-heat apparatus temperature to between 100 °C and 105 °C and continue heating for 30 min.

8.2.6 Remove each container from the sample pre-heat apparatus, close tightly, and shake vigorously for 60 s.

8.2.7 Using a volume delivery device such as a micropipette, introduce sufficient volume of the sample into the selected Houillon tube. The volume to be used is a function of the viscometer tube constant. The volume delivery device may be pre-warmed to facilitate transfer of highly viscous samples. (See Section 11.)



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

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Tube Filling Volume for a Measurement

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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

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**FIG. 2 Houillon Tube Schematic Diagram**

8.2.8 Analysis of all samples in the batch must be completed within 1 h from completion of 8.2.6.

## 9. Preparation of Apparatus

9.1 Place the automated viscometer on a stable and level horizontal surface. Make appropriate piping, drainage, and vacuum connections. Refer to the manufacturer's instructions.

9.2 If not already mounted, install the detection cells.

9.3 After installing and securing all viscometer tubes in the bath, fill the bath with appropriate amount of bath fluid (see 7.4).

9.4 Add the appropriate amount of solvent(s) to the solvent reservoir(s).

9.5 Follow the manufacturer's instructions for the operation of the instrument.

9.6 Select a clean, dry, and calibrated viscometer tube having a range covering the estimated kinematic viscosity of the specimen to be tested, if known. The appropriate viscometer tube to use depends on the estimated viscosity of the sample to be tested. The calculation in 9.6.1 may be used to decide which tube to use.

9.6.1 Using Eq 1, the viscometer tube should be chosen so that its constant  $C$  falls between  $v/200 < C < v/30$  to give flow times,  $T$ , between 30 s and 200 s.

NOTE 1—In the interlaboratory study<sup>5</sup> conducted for the development of this test method, the flow times were between 30 s and 200 s.

9.6.2 If a viscosity estimate is not known, a second analysis may be necessary using a different viscometer tube after a first trial analysis.

## 10. Calibration

10.1 Calibrate according to the manufacturer's instructions. Calibrated tubes may be purchased but shall be verified as per 10.4.

10.2 Use certified viscosity reference standards (see 7.1).

10.3 Refer to Section 11 for general operation of the automated viscometer and to the manufacturer's instructions.

10.4 The determined kinematic viscosity should match the certified value within  $\pm 0.5\%$ . If it does not, then reanalyze the standard. If the value is still out of range, then check all control system settings for the viscometer tube, and recheck each step in the procedure, including the temperature measuring device, and viscometer calibration to locate the source of error.

NOTE 2—The most common sources of error are caused by particles of dust lodged in the capillary bore of the viscosity tube (particularly for used oils) and temperature measurement errors. Modification of the cleaning constants by increasing the number of cycles and increasing the aspiration time before and after passage of the solvent (see Section 12) may be required.

## 11. General Procedure for Kinematic Viscosity

11.1 Set and maintain the automated viscometer bath at the required test temperature.

11.1.1 Thermometers, if used, shall be held in an upright position under the same conditions of immersion as when calibrated.

11.2 Introduce a sufficient volume of sample to the Houillon tube, using a volume delivery device (see 6.1.8) such as a micro-pipette. The specimen volume to be used is a function of the viscometer tube constant (see Fig. 1). Fig. 2 shows the correct specimen levels during a measurement.

NOTE 3—Use a volume delivery device that is capable of introducing the entire specimen volume in one operation.

11.3 Introduce the specimen into the viscometer tube. Start the measurement sequence.

11.4 The automated viscometer processing system will measure the flow times, calculate the viscosity according to Eq 1, and record the result.

11.5 Start the cleaning sequence (see Section 12).

11.6 Allow the viscometer tube to reach the bath temperature (about 5 min) before performing a new measurement.

NOTE 4—In some units, the equilibration time required may be much less than 5 min.

## 12. Cleaning of Viscosity Tubes

12.1 The viscometer tubes are cleaned by vacuuming the specimen residue left in the viscometer tube followed by the use of a solvent to remove any traces of the specimen on the viscometer tube walls. The solvent is then removed by vacuum. Some units use a second solvent for drying the tube. This operation is repeated several times until the viscometer tube is clean. Periodically, the viscometer tube calibration constant should be checked.

12.2 The solvent(s) should have a boiling point appropriate for the bath temperature.

12.2.1 The solvent flow must be sufficient to clean the entire internal section of the viscometer tube. This can be achieved by adjusting the solvent flow(s) and the flow rate using appropriate controls.

12.3 The cleaning of one tube shall not be carried out while a test is being performed in another tube in the same bath.

NOTE 5—Some multiple tube systems, although not included in the interlaboratory study<sup>5</sup> conducted to obtain the precision and bias of this test method, are designed to clean tubes while other tubes are performing viscosity measurements. Hence, this cleaning does not interfere with the validity of the tests underway in the instrument.

12.4 To ensure good operation, regularly check the viscometer tubes for damage and cleanliness. The latter can be done by checking with the reference oils suitable for the particular tube being used. Use these check oils like regular samples. If the results obtained are different from the certified values of these oils, it is necessary to carry out a more vigorous cleaning of the tubes in question. Non-chromium containing cleaning solution (see 7.2) may have to be used.

12.5 The frequency at which the tubes should be checked using reference oils depends on the frequency with which the tubes are used for measurements.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1604. Contact ASTM Customer Service at service@astm.org.