Designation: D8092 – 17

Standard Test Method for
Field Determination of Kinematic Viscosity Using a Microchannel Viscometer[1]

This standard is issued under the fixed designation D8092; 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.

1. Scope

1.1 This test method describes a means for measuring the kinematic viscosity of transparent and opaque liquids such as new and in-service lubricating oils using a miniature microchannel viscometer at 40 °C in the range of 12.9 mm^2/s to 174 mm^2/s

1.2 The precision has only been determined for those materials and viscosity ranges, as indicated in Section 17 on Precision and Bias.

1.3 This test method is specifically tailored to obtaining a rapid, direct, temperature- stabilized measure of the kinematic viscosity of new and in-service lubricants in the field in real-time without the use of solvents or chemical cleaning agents. The measurement takes place at 40 °C and kinematic viscosity is directly obtained. No temperature extrapolations or density corrections are necessary.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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. Some specific hazards statements are given in Section 9 on Hazards.*

1.6 *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
D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

2.2 *ISO Standard:*[3]

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Hele-Shaw cell, n*—a liquid cell wherein Stokes flow is present between two parallel plates.

3.1.1.1 *Discussion*—The unbounded microchannel capillary acts as a Hele-Shaw cell in this test method, which enables a simple relationship between kinematic viscosity and fluid velocity to be established.

3.1.2 *loading funnel, n*—the cavity in Fig. 1 that the liquid is placed into upon sample introduction; the sample then travels out of this funnel and enters the capillary.

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[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 D02.07.0A on Newtonian Viscosity.

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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.

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

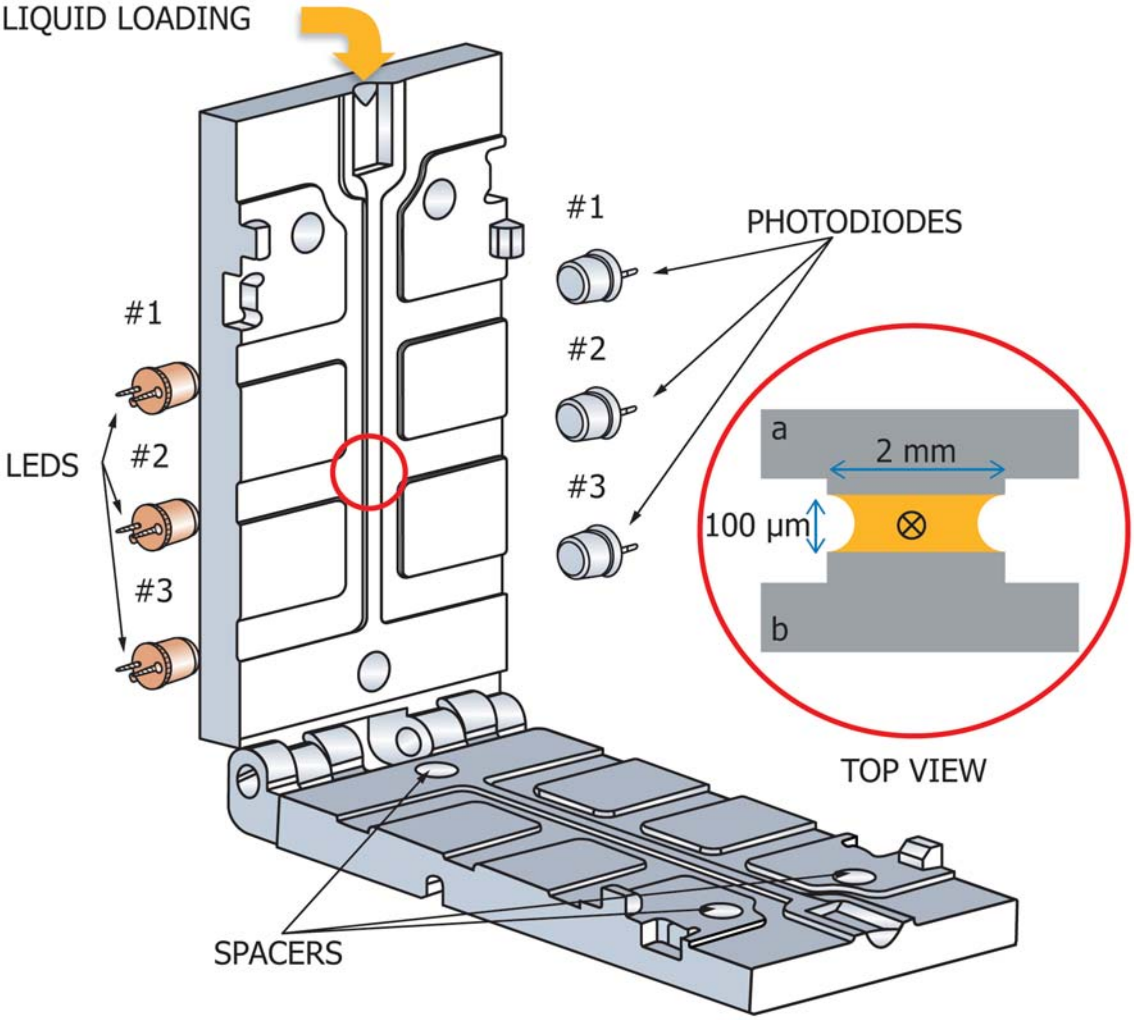

FIG. 1 Miniature Capillary Viscometer Schematic

3.1.3 *miniature capillary viscometer, n*—a viscometer, as shown in Fig. 1, which utilizes an unbounded microchannel capillary in order to enable the direct measurement of kinematic viscosity.

3.1.4 *unbounded microchannel capillary, n*—a rectangular channel, approximately 100 µm by 2 mm, which comprises the capillary for this test method.

3.1.4.1 *Discussion*—The channel, whose top view can be seen in Fig. 1, is unbounded because it is open to air on two sides. The sample stays in the capillary and does not leak through the unbounded portion due to the inherent surface tension at the boundary between the capillary and air. The capillary is repeatedly assembled for the purpose of the viscosity measurement and then disassembled immediately after the viscosity measurement to allow for cleaning. This is accomplished by having two mirror sides, which comprise the capillary, coupled by means of a clamshell arrangement. The integrity of the capillary is ensured by spacers which guarantee the distance between the two mirror sides when closing the clamshell.

4. Summary of Test Method

4.1 A liquid sample is placed into the loading funnel (see Fig. 1) of the miniature capillary viscometer and kinematic viscosity at 40 °C is determined by measuring the time in seconds (Δt) it takes this liquid to travel between the beam produced by LED (light-emitting diode) #1 and LED #3.

4.2 These times associated with the liquid passing each LED are determined by monitoring the voltage of the corresponding photodiodes: The starting time (t=0) is defined as when the operator initiates by means of button push the test sequence; a built-in clock then tracks the travel time. When the liquid enters the vicinity of the LED beam, the time, from the test starting time, at which the liquid is determined to have passed this beam is defined as the photodiode voltage falling by a factor of 0.5.

4.3 The liquid sample thermalizes rapidly to 40 °C as the entire unbounded microchannel capillary is constructed of aluminum which is stabilized at 40 °C. Thus when the 60 µL liquid is placed into the microchannel capillary it immediately comes into contact with the aluminum and the liquid flowing out of the loading funnel thermalizes to 40 °C within tenths of seconds.

4.4 The kinematic viscosity ν in square millimetres per second (mm^2/s) is determined a linear relationship with Δt, that is, $\nu = n_1 \cdot \Delta t + n_2$. The slope ($n_1$) in mm^2/s^2 and offset (n_2) in mm^2/s of this linear relationship are determined by calibration at the factory or at the point of use using certified viscosity reference standards.

5. Significance and Use

5.1 The significance of this test method is that it provides a means for a reliable field determination of kinematic viscosity at 40 °C without requiring solvents or chemicals for cleaning. Field use implies that the fluid may be very opaque, such as an in-service engine oil. The device may be cleaned with a disposable lint-free oil-absorbent material such as a clean cotton shop rag, and requires only 60 µL of sample for operation. As such the device provides a unique service to a range of industries where it is difficult or undesirable to obtain chemicals of any sort in order to determine the kinematic viscosity of their fluid of interest. Examples of such industries include many marine-based systems where a laboratory does not exist on-board, mines where equipment is needed for on-the-spot determination of asset viscosity, and large industrial plants where a walk-around inspection of oil sumps greatly increases efficiency. By using this test method, one can serve these crucial use-cases where a direct, immediate measure of kinematic viscosity at 40 °C may otherwise be difficult to obtain.

6. Interferences

6.1 Possible interferences for this test method include the presence of large particles in the liquid which would tend to get stuck in the capillary and impede the liquid flow. Since the capillary is only approximately 100 µm in its smallest dimension this must be carefully considered. To address this in the field, a middle LED/photodiode arrangement (#2, see Fig. 1) validates the linearity of the flow. If the flow is found to be non-linear, the operator is instructed that a probable invalid measurement has occurred and a re-test is indicated. This re-test involves cleaning the capillary and thereby removing any trapped particulate. If the liquid being analyzed commonly has a large population of particulate expected in the 60 µL of drawn sample, letting the sample settle and sampling towards the top of a bottle as described in Practice D4057 may be followed in order to significantly reduce the probability that this occurs.

7. Apparatus[4]

7.1 The miniature capillary viscometer (see Fig. 1) consists of the following components:

7.1.1 Temperature-controlled aluminum plates coupled together in a clamshell arrangement. These plates serve as a heat sink for bringing the liquid to a fixed temperature rapidly upon the sample being loaded into the device.

7.1.2 Temperature control of the aluminum plates is accomplished using a built-in PID controller with insulated strip heaters placed on the back side of each plate. Two PID (proportional integral derivative) loops control each plate separately. The thermistor is also placed into each plate, on the back side but as near as possible to the center of the liquid flow through the capillary. Such an arrangement controls temperature of the liquid to within 0.1 °C.

7.1.3 An unbounded microchannel capillary is machined into the temperature-controlled aluminum plates in the center of the two plates.

7.1.4 Miniature emitters operating at a center wavelength of 880 nm are used for the LEDs. Center wavelength 850 nm silicon photodiodes are used for detecting the LED illumination. Using a real-time operating system, the voltage on these photodiodes is monitored at millisecond resolution and is used determine the time, relative to when the test is initiated, when the liquid has passed the threshold of each LED beam. This time detection system is mounted in a measurement chamber which receives the aluminum plates. This measurement chamber uses a permanent-magnet holder to ensure the alignment between the aluminum plates (which contain the capillary) and the time detection system.

7.1.5 The viscometer control system comprises embedded electronics which allow the user to control the system and perform the viscosity measurement.

7.1.6 A micropastuerette, or micropipette with disposable tip, is used to dispense 60 µL of fluid into the loading funnel.

8. Reagents and Materials

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is pure enough to be used without lessening the accuracy of the determination.

8.2 *Certified Viscosity Reference Standards*—These are for use as confirmatory checks on the procedure in the laboratory. Certified viscosity reference standards shall be certified by a laboratory, which has shown to meet the requirements of ISO/IEC 17025 or a corresponding national standard by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D2162. The accepted reference value for the Certified Reference Material must be known with a relative error no greater than 0.25 % with a 95 % confidence.

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[4] The sole source of supply of the apparatus known to the committee at this time is Spectro Scientific, Inc., Chelmsford, MA, USA. These viscometers are covered under US Parents 8,661,878, 9,234,829, and related international patents and filings. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.3 A microdispenser (such as a pastuerette) which can accurately (within ±10 µL) dispense 60 µL of sample liquid is required.

8.4 A lint free, oil-absorbent material should be used to clean the viscometer. Some suitable examples would be polypropylene industrial wipes or a clean cotton shop rag.

9. Hazards

9.1 The device may utilize a certified Li-Ion battery.

9.2 Since the viscometer employs metal plates heated to 40 °C which come into contact with the operator when wiping clean the viscometer, the PID control is designed so that under no circumstances does the temperature of the plates reach elevated temperatures which may be hazardous to the human touch.

10. Sampling, Test Specimens, and Test Units

10.1 A sample of the liquid should be obtained. At minimum a quantity of 60 µL is needed. If obtained directly from the equipment such as with a dipstick or from a bottle that contains a recent sample, the sample should be representative of the system and no further actions are required. The sample should be at temperatures that are compatible with the specifications of the microdispenser used when drawn for analysis. If such equipment is available, the sample may be obtained as described in Practice D4057.

10.2 If the sample is obtained from a source, such as an analysis bottle, that may be non-homogeneous, or clearly appears to be non-homogeneous, the sample should be rehomogenized to ensure that a representative sample will be obtained. Follow Practice D5854 and ensure that the sample is gently inverted 20 times before drawing a sample in these cases.

11. Preparation of Apparatus

11.1 Ensure that the viscometer resides upright on a flat, hard surface.

11.2 The microchannel is cleaned with a lint free, oil-absorbent material by wiping it along the capillary flow area, from the center of the Hele-Shaw cell towards the loading funnel, removing liquid and debris.

11.3 The microchannel is cleaned again with a lint free, oil-absorbent material by pressing it firmly on the capillary flow area and rubbing it for at least five (5) strokes, or until no visible sample residue remains.

11.4 Make a final cleaning stroke using the lint free, oil-absorbent material along both capillary flow areas on each half of the Hele-Shaw cell.

11.5 Inspect the aluminum plates and in particular the unbounded microchannel capillary area to ensure that no visible liquid is present on the plates.

12. Calibration and Standardization

12.1 Use only a calibrated apparatus as described in Section 7. The calibration shall be checked periodically using certified reference standards as described in Section 8. The recommended interval for viscosity calibration check is once per day or before a new operator uses the apparatus. For temperature control, the recommended calibration interval is once per year. For calibration procedures, follow the instructions of the manufacturer of the apparatus. The pass/fail criteria for the calibration check is that the obtained value should be within the repeatability of the apparatus (as described in 17.1.1) as compared to the reference value. If not, the calibration check shall be run again. If this also fails, the apparatus must be recalibrated. A nominal reference sample of 100 mm^2/s is typically used for this calibration check.

12.1.1 Calibration measurements are performed per normal device operating instructions as delineated in Section 14.

12.2 The microdispenser should be calibrated per manufacturer's instructions.

13. Conditioning

13.1 Gather approximately 60 µL (±10 µL) of sample to be measured using the microdispenser and apply this amount of sample evenly along both capillary flow areas on each half of the Hele-Shaw cell.

13.2 Apply cleaning steps described in 11.1, 11.2, and 11.3 one final time. At this point in time, the viscometer is conditioned and ready for measurement.

13.3 Place the aluminum plates into the loading chamber.

13.4 The plates will take approximately 90 s to 120 s to reach temperature stability at 40 °C when placed in the system at room temperature (25 °C).

13.5 Follow the manufacturer's instructions for the operation of the apparatus.

14. Procedure

14.1 When prompted by the viscometer control system, draw and place a fresh 60 µL (±10 µL) of sample into the loading funnel using the microdispenser.

14.2 The viscometer control system will automatically measure the flow times, and calculate kinematic viscosity according to 4.4. An estimated total measurement time is one second per mm^2/s, so if the viscosity of the sample at 40 °C is 100 mm^2/s, the measurement time will be approximately 100 s.

14.3 When a measurement is completed, the plates are pulled out of the measurement chamber and the Hele-Shaw cell is opened and cleaned by repeating steps 11.1, 11.2, and 11.3.

14.4 The viscometer is then conditioned before the next measurement by following Section 13.

14.5 Once cleaned and the aluminum plates are placed back into the measurement chamber, the viscometer will be ready for the next measurement sequence in approximately 1 min. This will be indicated automatically by the viscometer control system.

14.6 If, upon completion of a measurement, a bad measurement is indicated on the liquid crystal display (LCD) screen, take appropriate action. This may arise from either a non-linear flow profile or a calculated viscosity which falls outside the range of the apparatus: If the liquid appears to be outside the measurement range, the sample may not be measured using the viscometer. If a non-linear flow or otherwise bad measurement is indicated, prepare the apparatus as described in Section 11 and condition the apparatus as described in Section 13, then re-measure. A non-linear flow may be indicated in the following way: Δt/2 is compared to the time difference between LED #1 and LED #2. If these two quantities are different by more than 5 %, a re-measure is indicated.

15. Calculation or Interpretation of Results

15.1 The results are calculated automatically by the viscometer control system by means of the equation described in 4.4 and displayed to the user. If a bad measurement occurs this is also displayed to the user. Interpretation of results depends on the nature of the liquid being analyzed. Any subsequent actions are determined separately.

16. Report

16.1 The kinematic viscosity at 40 °C is reported by the viscometer by means of human-readable LCD interface. These results are reported to three (3) significant figures.

17. Precision and Bias[6]

17.1 The precision of this test method was determined by statistical examination of results on 12 in-service oil samples ranging in viscosity from 12.9 mm^2/s to 174 mm^2/s at 40 °C. These samples comprised a range of in-service hydraulic, gear, transmission, aero-derived turbine, and standard diesel engine lubricants. They were run in duplicates by six different laboratories. The determined precision is as follows:

17.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

$$r = 0.2253X^{0.8071} \tag{1}$$

where:

X = the mean of two results, in mm^2/s at 40 °C.

17.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

$$R = 0.2525X^{0.8071} \tag{2}$$

where:

X = the mean of two results, in mm^2/s at 40 °C.

17.2 *Bias*—No information can be presented on the bias of the procedure in this test method, because no material having an accepted reference value has been tested.

17.2.1 *Between-Method Bias*—Degree of agreement between results by Test Method D445 and Test Method D8092:

17.2.1.1 Results on the same materials produced by Test Method D445 and Test Method D8092 have been assessed in accordance with procedures outlined in Practice D6708. The findings are: The degree of agreement between results from Test Method D445 and Test Method D8092 can be further improved by applying correction equation C1 as listed below (see Research Report for Test Method D8092, Annex E). Sample-specific bias, as defined in Practice D6708, was observed for some samples after applying the bias-correction, for the material types and property range listed below. Ten (10) in-service oil samples ranging in viscosity from 12.9 mm^2/s to 174 mm^2/s at 40 °C were examined for between-method bias by means of two (2) interlaboratory studies (see Research Report for Test Method D8092, Annex E). These samples comprised a range of in-service hydraulic, gear, transmission, aero-derived turbine, and standard diesel engine lubricants. One interlaboratory study was carried out for the Test Method D8092 Precision Statement discussed in 17.1. The other was carried out by means of Test Method D445 running at a different set of seven (7) laboratories on this same set of samples.

17.2.1.2 *Correction Equation C1:*

$$\text{bias-corrected X} = \text{predicted Y} = bX + a;\ b = 1.060269;\ a = -1.46325 \tag{3}$$

where:

X = result by Test Method D8092,
Bias-corrected X = Predicted Y,
Predicted Y = result that would have been obtained by Test Method D445 on the same sample, and
b, a = parameter estimates for a linear correction as defined in this test method.

Differences between bias-corrected results from Test Method D8092 and Test Method D445, for the sample types and property ranges studied, are expected to exceed the following between methods reproducibility (R_{XY}) as defined in Practice D6708, about 5 % of the time.

17.2.1.3 *Kinematic Viscosity at 40 °C:*

$$\text{between-methods}\ (R_{XY}) = (0.96\ R_X^2 + 0.85\ R_Y^2)^{0.5} \tag{4}$$

As a consequence of sample-specific biases, R_{XY} may exceed the reproducibility for D8092 (R_X), or the reproducibility for Test Method D445 (R_Y), or both. Users intending to use D8092 as a predictor of Test Method D445, or vice versa, are advised to assess the required degree of prediction agreement relative to the estimated R_{XY} to determine the fitness-for-use of the prediction.

18. Keywords

18.1 capillary; field-based device; handheld device; Hele-Shaw flow; kinematic viscosity; viscometer

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1856. Contact ASTM Customer Service at service@astm.org.

RELATED MATERIAL

"Hand-Portable Kinematic Viscometer," ASTM STP 1564 (2013).

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