Standard Test Method for
Determination of Dynamic Viscosity and Derived Kinematic Viscosity of Liquids by Constant Pressure Viscometer

1. Scope*

1.1 This test method covers the measurement of dynamic viscosity and density for the purpose of derivation of kinematic viscosity of petroleum liquids, both transparent and opaque. The kinematic viscosity, $v$, in this test method is derived by dividing the dynamic viscosity, $\eta$, by the density, $\rho$, obtained at the same test temperature.

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 rate are proportional (Newtonian flow behavior).

1.3 The range of kinematic viscosity covered by this test method is from 0.5 mm²/s to 1000 mm²/s in the temperature range between −40 °C to 120 °C; however the precision has been determined only for fuels and oils in the range of 2.06 mm²/s to 476 mm²/s at 40 °C and 1.09 to 107 mm²/s at 100 °C (as stated in Section 12 on Precision and Bias). The precision has only been determined for those materials, viscosity ranges, and temperatures as indicated in Section 12 on Precision and Bias. The test method can be applied to a wider range of materials, viscosity, and temperature. For materials not listed in Section 12 on Precision and Bias, the precision and bias may not be applicable.

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.

2. Referenced Documents

2.1 ASTM Standards:
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
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

2.2 ISO Standards:
ISO 5725 Accuracy (Trueness and Precision) of Measurement Methods and Results
ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 Definitions:
3.1.1 density ($\rho$), $n$—mass per unit volume.
3.1.2 dynamic viscosity ($\eta$), $n$—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 to deformation of a liquid under external shear forces.

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.

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*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.


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.

3.1.3 kinematic viscosity \( (\nu) \), \( n \)—the ratio of the dynamic viscosity \( (\eta) \) to the density \( (\rho) \) of a liquid.

3.1.3.1 Discussion—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density \( (\rho) \). Therefore the kinematic viscosity \( (\nu) \) is a measure of the resistance to flow of a liquid under gravity.

4. Summary of Test Method

4.1 A test specimen is introduced into the measuring cells, which are controlled at a specified and known temperature. The measuring cells consist of a horizontal capillary tube with optical sensors and an oscillating U-tube densitometer. The dynamic viscosity is determined from the flow time of the test specimen along the capillary under a constant pressure of compressed air in conjunction with calculations. The density is determined by the oscillation frequency of the U-tube in conjunction with calculations. The kinematic viscosity is calculated by dividing the dynamic viscosity by the density.

5. Significance and Use

5.1 Many petroleum products 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.

5.2 Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and petroleum products and in this test method is used for the calculation from dynamic to kinematic viscosity.

6. Apparatus

6.1 Constant Pressure Viscometer:

6.1.1 Viscosity Measurement—The Constant Pressure viscometer uses the Hagen-Poiseuille principle of capillary flow to determine the viscosity. A length of capillary tube is enclosed horizontally in a thermal block maintained at a constant temperature by thermoelectric coolers/heaters. The test specimen is driven to flow along the tube by a constant and regulated pressure of compressed air. The transit time of the test sample as it flows past an array of optical detectors is measured. (See Fig. 1.) The dynamic viscosity is proportional to the measured transit time.

6.1.1.1 Pressure Control—A pressure generating and regulating device able to maintain an air pressure between 6.89 kPa to 68.9 kPa (1 psi to 10 psi) used to drive a test specimen to flow along a capillary tube.

6.1.2 Density Measurement—Density is measured by a suitable method so to achieve the precision in kinematic viscosity as stated in the tables in Section 12. A U-shaped oscillating sample tube with a system for electronic excitation and frequency counting as described in the manufacturer’s instructions is suitable. However, for this test method, the purpose of the density result is for the calculation from dynamic to kinematic viscosity.

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4 The Constant Pressure viscometer is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative to this patented item to the ASTM International headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

5 The sole source of supply of the apparatus known to the committee at this time is PhasePSL, 11168 Hammersmith Gate, Richmond, BC Canada. 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, which you may attend.
6.1.3 Temperature Control—A thermal block surrounds the viscosity measuring cell so that both are at the same temperature. A thermolectric heating and cooling system (see Fig. 1) ensures temperature stability of the block to be within ±0.01 °C from the set temperature.

6.2 Autosampler, for use in sample introduction process. The autosampler shall be designed to ensure the integrity of the test specimen prior to and during the analysis and be equipped to transfer a representative volume of test specimen into the measuring cells. The autosampler shall transfer the test specimen from the sample vial to the measuring cells of the apparatus without interfering with the integrity of the test specimen. The autosampler may have heating capability as a means to lower the viscosity of the sample for filling the measuring cells.

6.3 Screen, with an aperture of 75 µm, to remove particles from samples that may contain them (see 8.2).

7. Reagents and Materials

7.1 Sample Solvent, completely miscible with the sample.

7.1.1 For samples that are mutually soluble such as light middle distillate test specimen, the use of the same or similar middle distillates as solvent is suitable. If the solvent dries up without residues in an applicable time frame, the use of a separate drying solvent is not required.

7.1.2 For more viscous test specimen, an aromatic solvent such as toluene is suitable.

7.2 Drying Solvent, a volatile solvent miscible with the sample solvent (see 7.1).

7.2.1 n-Pentane is suitable.

7.3 Dry Air, for blowing and drying of the measuring cells.

NOTE 1—If the measuring cell temperature is below or near the dew point temperature of the ambient air, the use of an appropriate desiccator is recommended.

8. Sampling, Test Specimens, and Test Units

8.1 Sampling is defined as all the steps required to obtain an aliquot, and to place the sample into the laboratory test container. The laboratory test container shall be of sufficient volume to mix the sample and obtain a homogeneous sample for analysis.

8.2 Test Specimen—A volume of sample obtained from the laboratory sample and delivered to the measuring cells. The test specimen is obtained as follows:

8.2.1 Mix the sample, if required, to homogenize at room temperature into an open sample vial. If loss of volatile material can occur in an open container, then mixing in closed containers, or at sub-ambient temperatures is recommended.

8.2.2 Deliver the test specimen from a properly mixed laboratory sample to the measuring cells using an autosampler. For waxy or other samples with a high pour point, before delivering the test specimen, heat the laboratory sample to the desired test temperature, which has to be high enough to dissolve the wax crystals.

9. Calibration and Verification

9.1 Use only a calibrated apparatus as described in section 6.1.1 and as shown in Fig. 1. The calibration shall be checked as defined by the lab QA procedures using certified reference standards as described in 9.2. The recommended interval for viscosity and density calibration is once a year as a minimum or when lab QA procedures dictate. For the calibration procedure, follow the instructions of the manufacturer of the apparatus.

9.2 Certified Viscosity and Density Reference Standards—These are for use as confirmatory checks on the procedure in the laboratory. Certified viscosity and density 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. Density standards shall have a certified uncertainty of the density values of 0.0001 g/cm³. The uncertainty of the reference standards shall be stated for each certified value (k = 2; 95% confidence level). See ISO 5725.

9.2.1 Use certified reference standards appropriate to the desired measuring temperatures of viscosity and density measurements for both calibration and verification.

10. Procedure

10.1 Standard Procedure Using an Autosampler:

10.1.1 Set the internal temperature control to the desired measuring temperature.

10.1.2 Set the determinability limits to the values stated in Table 1 for the specific product and test temperature.

10.1.2.1 For products not listed in the precision section, it is the responsibility of the user of this test method to establish reasonable determinability by a series of tests.

10.1.3 Configure the cleaning and drying routines for the autosampler using sample solvent (see 7.1), drying solvent (see 7.2) and drying air (see 7.3) for sufficient cleaning efficiency of the product being tested.

NOTE 2—For specific information on proper configuration, follow the manufacturer’s instructions.

10.1.4 Transfer a minimum of 25 mL of the test specimen into a sample vial. Cap or cover the vial as necessary.

10.1.5 Load sample vial(s) onto vial tray or holder and analyze the test specimens.

10.1.6 Rerun samples which exceed the determinability criteria established for the sample type being analyzed. (See Table 1.)

10.1.6.1 If the two determined values of kinematic viscosity calculated from the flow time measurements exceed the stated determinability figure (see Table 1) for the product, repeat the measurements of flow times until the calculated kinematic viscosity determinations agree with the stated determinability.

NOTE 3—When a sample is run or when the procedure is repeated, the dynamic viscosity and density are determined in calculating the kinematic viscosity.

10.1.7 Press the “Run” or “Start” key. The apparatus measures the transit time of the test specimen through the capillary tube as per 6.1.1 (and Fig. 1) and thereby calculates dynamic viscosity. The density of the test specimen is measured per
6.1.2, and the kinematic viscosity is calculated by dividing the dynamic viscosity by the density.

11. Report of Results

11.1 Report the result, expressed as kinematic viscosity in mm²/s to four significant figures, stating the temperature of the test.

12. Precision and Bias

12.1 Determinability, (d)—Quantitative measure of the variability associated with the same operator in a given laboratory, obtaining successive determined values using the same apparatus for a series of operations leading to a single result. It is defined as that difference between two such single determined values as would be exceeded in the long run, in one case in twenty. (See Table 1.)

12.2 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. (See Table 2.)

12.3 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 only in one case in twenty. (See Table 3.)

Note 4—This test method covers the measurement of dynamic viscosity and density for the purpose of derivation of kinematic viscosity. Therefore determinability, repeatability and reproducibility are only stated for kinematic viscosity.

Table 1 Determinability

<table>
<thead>
<tr>
<th>Kinematic viscosity</th>
<th>40 °C</th>
<th>100 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Determinability (d)</td>
<td>0.0025y (0.25%)</td>
<td>0.0025y (0.25%)</td>
</tr>
</tbody>
</table>

where: y is the average of determined values being compared

Note 5—Repeatability and reproducibility were determined based on ILS study comprised of the following samples:

Distillate samples included kerosene, Regular Sulphur Diesel (RSD), Ultra Low Sulphur Diesel (ULSD), Biodiesel B100 (sor derived), Biodiesel B100 (tallow derived), Biodiesel B10 blend and Biodiesel B20 blend with viscosities between 2.06 and 4.50 mm²/s at 40 °C. Distillate sample included Biodiesel B100 at 1.09 mm²/s at 100 °C. Base Oils included synthetic and semi-synthetic base oils with viscosities between 12.0 and 476.0 mm²/s at 40 °C, and between 2.90 and 32.0 mm²/s at 100 °C.

Formulated Oils include automatic transmission fluids, hydraulic fluids, motor oils, gear oils, polymers in base oil and additives in base oil with viscosities between 28.0 and 472.0 mm²/s at 40 °C, and between 6.50 and 107.0 mm²/s at 100 °C.

Six (6) laboratories participated in the interlaboratory studies at 40 °C, and eight (8) laboratories participated at 100 °C.

The interlaboratory studies were performed according to Practices D6300. Supporting data has been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1797.6

12.4 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.

12.5 Relative Bias—Degree of Agreement between results by Test Method D7945 and Test Method D445 results obtained on the same materials have been assessed in accordance with procedures outlined in Practice D6708. The findings are: No bias-correction considered in Practice D6708 can further improve the agreement between results from Test Method D7945 and Test Method D445 for the material types and property ranges studied (reference Research Report RR:D02-1797). Sample-specific bias, as defined in Practice D6708, was observed for some samples.

12.5.1 Between Methods Reproducibility (RXY)—Differences between results from Test Method D7945 and Test Method D445, for the sample types and property ranges studied, are expected to exceed the following between methods reproducibility (RXY), as defined in Practice D6708, about 5 % of the time.

\[
\text{Kinematic Viscosity at 40 °C: between methods (R}_{XY}\right) = (0.93 \text{ R}_Y^{2.0} + 0.93) \\
\text{Kinematic Viscosity at 100 °C: between methods (R}_{XY}\right) = (0.58 \text{ R}_Y^{2.0} + 0.58)
\]

Note 6—As a consequence of sample-specific biases, RXY may exceed the reproducibility for Test Method D7945 (Rx), or reproducibility for Test Method D445 (Ry), or both. Users intending to use Test Method D7945 as a predictor of Test Method D445 are advised to assess the required degree of prediction agreement relative to the estimated RXY to determine the fitness-for-use of the prediction.

13. Keywords

13.1 constant pressure viscometer; density; kinematic viscosity; viscosity

6 Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1797. Contact ASTM Customer Service at service@astm.org.
SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D7945 – 14) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Added pressure control range to 6.1.1.1.
(2) Added text on calibration to 9.2.1.
(3) Added text on running the samples for additional clarity in 10.1.7.

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