Standard Test Method for Determination of Dynamic Viscosity and Derived Kinematic Viscosity of Liquids by Oscillating Piston Viscometer[1]

1. Scope

1.1 This test method covers the measurement of dynamic viscosity and derivation of kinematic viscosity of liquids, such as new and in-service lubricating oils, by means of an oscillating piston viscometer.

1.2 This test method is applicable to Newtonian and non-Newtonian liquids; however the precision statement was developed using Newtonian liquids.

1.3 The range of dynamic viscosity covered by this test method is from 0.2 mPa·s to 20 000 mPa·s (which is approximately the kinematic viscosity range of 0.2 mm^2/s to 22 000 mm^2/s for new oils) in the temperature range between –40 to 190°C; however the precision has been determined only for new and used oils in the range of 1.434 mPa·s to 154.4 mPa·s at temperatures of 40 and 100°C (as stated in the precision section).

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:*[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

D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine

D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

2.2 *ISO Standards:*[3]

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

2.3 *NIST Standard:*[4]

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

3. Terminology

3.1 *Definitions:*

3.1.1 *dynamic viscosity (η), n*—the ratio between the applied shear stress and rate of shear of a liquid.

3.1.1.1 *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.

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

3.1.2 *kinematic viscosity (ν), n*—the ratio of the dynamic viscosity (η) to the density (ρ) of a liquid.

3.1.2.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, (ρ). Therefore the kinematic viscosity, (ν), is a measure of the resistance to flow of a liquid under gravity.

3.1.3 *rate of shear (shear rate), n*— in liquid flow, the velocity gradient across the liquid.

3.1.4 *shear stress, n*—the force per unit area in the direction of the flow.

3.1.4.1 *Discussion*—The SI unit for shear stress is the pascal (Pa).

3.1.5 *density (ρ), n*—mass per unit volume.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[4] Available from http://physics.nist.gov/ccu/Uncertainty/index.html.

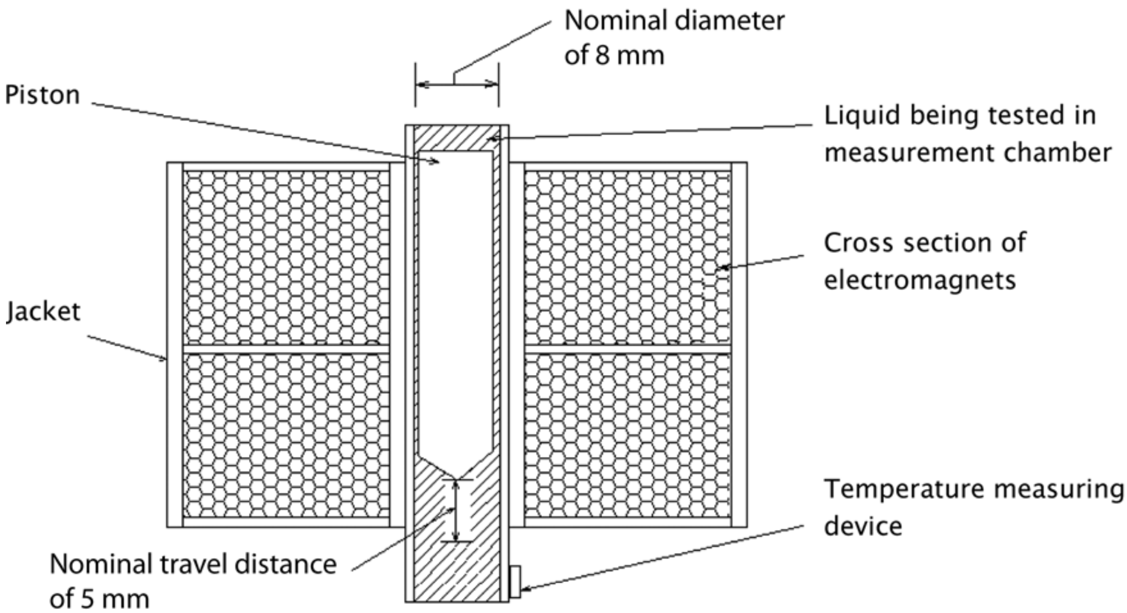

FIG. 1 Cross Sectional View of Measurement Chamber

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oscillating piston viscometer*, *n*—a device that measures the travel time of a piston driven electromagnetically into stationary oscillating motion through a liquid at a controlled force in order to determine the dynamic viscosity of the liquid.

4. Summary of Test Method

4.1 A specimen of sample is placed in the thermally controlled measurement chamber where the piston resides. The piston is driven into oscillatory motion within the measurement chamber by a controlled magnetic field. Once the sample is at the test temperature, as determined by the temperature detector, the piston is propelled repeatedly through the liquid (by the magnetic field). A shear stress (ranging from 5 Pa to 750 Pa) is imposed on the liquid under test due to the piston travel. The dynamic viscosity is determined by measuring the average travel time of the piston. The kinematic viscosity is derived by additionally measuring the ratio between the up and down travel times. This information is then applied to a calibration curve using liquids of known viscosity to calculate the dynamic viscosity and kinematic viscosity of the liquid. See Fig. 1.

5. Significance and Use

5.1 Many petroleum products, as well as non-petroleum materials, are used as lubricants for bearings, gears, compressor cylinders, hydraulic equipment, etc. Proper operation of this equipment depends upon the viscosity of these liquids.

5.2 Oscillating piston viscometers allow viscosity measurement of a broad range of materials including transparent, translucent and opaque liquids. The measurement principle and stainless steel construction makes the Oscillating Piston Viscometer resistant to damage and suitable for portable operations. The measurement itself is automatic and does not require an operator to time the oscillation of the piston. The electromagnetically driven piston mixes the sample while under test. The instrument requires a sample volume of less than 5 mL and typical solvent volume of less than 10 mL which minimizes cleanup effort and waste.

6. Apparatus

6.1 *Oscillating Piston Viscometer:*[5,6]

6.1.1 The oscillating piston viscometer (see Fig. 2) comprises a measurement chamber and calibrated piston capable of measuring the dynamic viscosity within the limits of precision given in Section 16.

6.1.2 *Piston*—Free moving, magnetically driven body within a Oscillating Piston Viscometer which is used for measuring the viscosity of liquids. Individual pistons are sized to measure specific viscosity ranges by varying the sensor annulus. See Table 1 for the selection of the piston according to the viscosity range.

6.1.3 *Measurement Chamber*—Location within Oscillating Piston Viscometer where piston motion (through the liquid under test) occurs due to an imposed electromagnetic field. See Fig. 1.

6.1.4 *Electronics*—Capable of controlling the electromagnetic field to propel and detect the travel time of the piston with a discrimination of 0.01 s or better and uncertainty within ±0.07 %. The travel time is calibrated to be between 0.4 s and 60 s, at a distance of 5 mm.

6.1.5 *Temperature Controlled Jacket*—Sufficient for maintaining measurement chamber temperature within ±0.06°C.

[5] The Oscillating Piston 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.

[6] The sole sources of supply for the apparatus known to the committee at this time is Cambridge Viscosity Inc., 101 Station Landing, Medford, MA 02155 (www.cambridgeviscosity.com). 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.

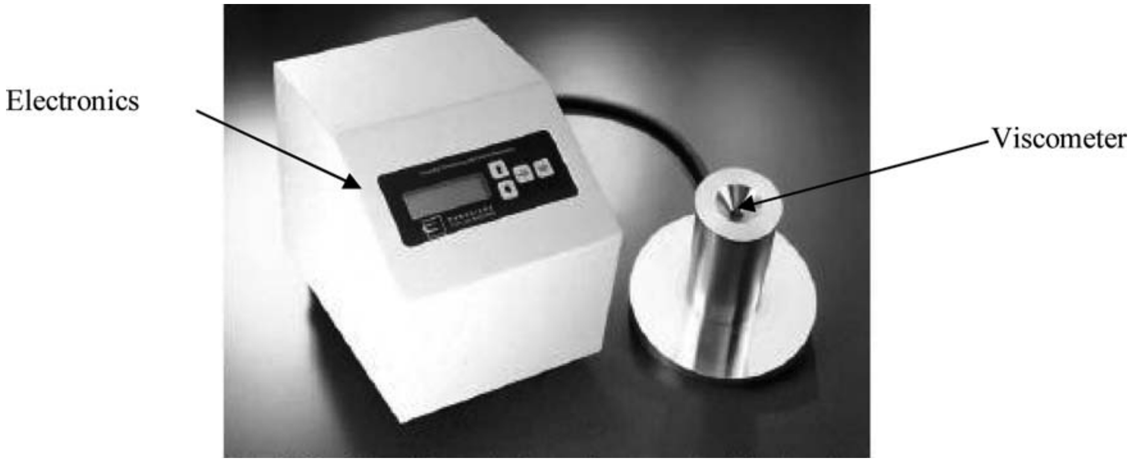

FIG. 2 Viscometer with Electronics

TABLE 1 Viscosity Ranges of Oscillating Viscometer Pistons

Minimum Viscosity (mPa·s)	Maximum Viscosity (mPa·s)	Piston Designation	Nominal Piston Diameter (mm)	Recommended Sample Volume (mL)
0.02	2	SP20	7.87	3.2 - 5
0.25	5	SP50	7.83	3.2 - 5
0.5	10	SP11	7.81	3.2 - 5
1	20	SP21	7.76	3.5 - 5
2.5	50	SP51	7.68	3.5 - 5
5	100	SP12	7.62	3.5 - 5
10	200	SP22	7.54	3.5 - 5
25	500	SP52	7.34	3.5 - 5
50	1000	SP13	7.21	4.0 - 5
100	2000	SP23	6.96	4.0 - 5
250	5000	SP53	6.27	4.0 – 5
500	10000	SP14	6.05	4.0 - 5
1000	20000	SP24	5.72	4.0 - 5

6.1.6 *Temperature Measuring Device*—Industrial platinum resistance thermometer (IPRT) or equivalent sensor with a maximum permissible error of ±0.02°C. It is recommended, that the temperature measuring device be verified with an independent, calibrated temperature probe at the test temperature.

6.2 *Temperature Regulation System*:

6.2.1 Any liquid bath or thermoelectric means for regulating the jacket temperature.

6.2.2 The temperature control must be such that the temperature of the measurement chamber is held within ±0.06°C of the desired measurement temperature.

6.3 *Sample Introduction Mechanism*—A syringe, micropipette, or flow-through adapter for introducing between 3.2 mL and 5 mL, inclusive by pressure, into the measurement chamber.

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. Viscosity standards shall be traceable to master viscometer procedures described in Practice D2162.

7.2 The uncertainty of the certified viscosity reference standard shall be stated for each certified value (k = 2, 95 % confidence). See ISO/EC 17025 or NIST TN 1297.

7.2.1 The certified viscosity reference should have a published viscosity in accordance with Test Method D445 or equivalent means that is close to that of the liquids being tested at the test temperature. For example, if intended measurements are to be made from 5-25 mPa·s at 100°C, then a reference oil viscosity of 15 mPa·s at 100°C would be appropriate.

7.3 Cleaning solvents miscible with the sample and chemically compatible with the wetted viscometer components (such as alcohols, toluene, etc.). These wetted components are typically 316L and 430 Stainless Steel.

7.4 Quality control (QC) liquid similar to 7.1, but with viscosity values internally certified as noted in 12.2.

8. 8. Sampling, Samples, and Test Units

8.1 Ensure that the sample is homogenous. Engine sampling is generally specified in the test method, for example Test Method D5967. When applicable, refer to Practice D4057 (manual) or Practice D4177 (automatic) for proper sampling techniques.

9. Preparation of Apparatus

9.1 Place the viscometer on a stable surface.

9.2 Operate the unit according to the procedure in Section 13.

9.3 Verify calibration accuracy by testing a reference standard or QC liquid at the test temperature. Follow the procedure in Section 13.

10. Calibration and Standardization

10.1 Calibrate according to manufacturer's instructions to obtain a calibration curve (using two test liquids with referenced viscosity values near, but within, the extremes of the piston range being used).

10.2 Certified Viscosity Standards may be used as confirmatory checks on the procedure in the laboratory. This procedure is outlined in Section 13. If the dynamic viscosity result, at the calibration test point, does not agree with the certified value within the limits of precision in Section 16, each step in the procedure should be rechecked, as well as the temperature measuring device and viscometer calibration, to locate the source of error. If the source is not detected, consult the manufacturer.

11. Sample Conditioning

11.1 Shake all new and used oil samples using the following procedure.

11.1.1 Ensure cap is tight on the container.

11.1.2 Shake vigorously by hand for 30 s. Wait 10 s, or longer if needed, for air bubbles to dissipate.

11.1.3 A specimen of the sample shall be taken by pipette, pouring or pumping. Suspected nonhomogeneous samples must be conveyed for analysis promptly following the shaking and dissipation procedure of step 11.1.2.

12. Quality Control/Quality Assurance (QC/QA)

12.1 Confirm proper performance of the instrument and the test procedure by analyzing reference oil as QC sample.

12.2 If suitable reference oil is not available, prepare a QC sample by replicate analyses of a batch of oil sample. Then statistically analyze the data to assign a mean value and uncertainty limit to the sample.

12.3 When QC/QA protocols are already established in the testing facility, these may be used to confirm the reliability of the test result.

12.4 When there is no QC/QA protocol established in the testing facility, guidance may be obtained from Practice D6792.

13. Procedure

13.1 Verify or set the temperature control settings, as tested with the control standard, so the viscometer temperature reads the desired set point ±0.06°C while the piston is in motion.

13.2 Remove the piston and clean the specimen from the measurement chamber as described in the viscometer manual.

13.3 Load the measurement chamber with sample using the volume listed in Table 1 related to the piston size being used for the viscosity range anticipated. To minimize contamination, and if sample volume allows, pre-wet the chamber and piston with the sample material and dry wipe with a lint free cloth.

13.4 Load the measurement chamber with a clean piston. The piston size should be selected such that the measured viscosity is between the minimum and maximum viscosity values listed in Table 1. If the reported result is outside of this range, the measurement shall be repeated using the appropriate piston size.

13.5 Start the reporting software in accordance with manufacturer's instructions, which in turn will:

13.5.1 Allow the sample to equilibrate in the measurement chamber for at least 2 min while the piston is oscillating.

13.5.2 Ensure that temperature stability is within the criterion set in 6.2.

13.5.3 Measure the upward and downward piston travel times for each cycle and compute viscosity until the standard deviation as percent of mean, over the previous 20 dynamic viscosity computations, is less than 0.6%.

13.6 Record the average result from the last 20 computations.

14. Calculation and Interpretation

14.1 The calculation of dynamic viscosity and kinematic viscosity are computed and displayed automatically by the apparatus.

15. Report

15.1 Dynamic Viscosity result in mPa·s to three significant figures, as displayed by the apparatus.

15.2 Kinematic Viscosity result in mm^2/s, to three significant figures, as displayed by the apparatus.

15.3 Temperature in degrees Celsius, to the second decimal place, as displayed by the apparatus.

16. Precision and Bias [7]

16.1 The precision and bias of this test method as determined by the statistical examination of test results is as follows:

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

	Percent of Mean
Dynamic viscosity	2.9
Kinematic viscosity	4.2

NOTE 1—This provisional repeatability was obtained from a sample set of ten used and ten fresh oils measured by seven laboratories at 40 and 100°C. Certified Reference Standards were included among the samples set.

16.1.2 *Reproducibility*—The reproducibility of this test method is being determined and will be available on or before December 14, 2009.

16.2 *Bias*—The average results for the standard liquid used to develop the repeatability statement agreed with the reference value within statistical error (see Table 2).

17. Keywords

17.1 dynamic viscosity; kinematic viscosity; oscillating piston; oscillating piston viscometer; viscosity

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1657.

TABLE 2 Measurement Bias

	Viscosity in mP•s							
ILS Average Value in mP•s	1.420	3.077	4.693	9.661	15.36	85.19	155.9	
Certified Reference Value	1.434	3.098	4.643	9.671	15.12	86.05	154.4	
	Viscosity in mm^2/s							
ILS Average Value in mm^2/s	1.856	3.869	5.747	11.71	18.16	18.88	99.76	184.8
Certified Reference Value	1.853	3.901	5.706	11.83	18.00	18.05	100.8	180.0