



Designation: **D4741–17** ~~D4741~~ – 18

## Standard Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer<sup>1</sup>

This standard is issued under the fixed designation D4741; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

### 1. Scope\*

1.1 This test method<sup>2</sup> covers the laboratory determination of the viscosity of oils at 150 °C and  $1 \times 10^6 \text{ s}^{-1}$  and at 100 °C and  $1 \times 10^6 \text{ s}^{-1}$ , using high shear rate tapered-plug viscometer models BE/C or BS/C.

1.2 Newtonian calibration oils are used to adjust the working gap and for calibration of the apparatus. These calibration oils cover a range from approximately 1.4 mPa·s to 5.9 mPa·s (cP) at 150 °C and 4.2 mPa·s to 18.9 mPa·s (cP) at 100 °C. This test method should not be used for extrapolation to higher viscosities than those of the Newtonian calibration oils used for calibration of the apparatus. If it is so used, the precision statement will no longer apply. The precision has only been determined for the viscosity range 1.48 mPa·s to 5.07 mPa·s at 150 °C and from 4.9 mPa·s to 11.8 mPa·s at 100 °C for the materials listed in the precision section.

1.3 A non-Newtonian reference oil is used to check that the working conditions are correct. The exact viscosity appropriate to each batch of this oil is established by testing on a number of instruments in different laboratories. The agreed value for this reference oil may be obtained from the chairman of the Coordinating European Council (CEC) Surveillance Group for CEC L-36-90, or from the distributor.

1.4 Applicability to products other than engine oils has not been determined in preparing this test method.

1.5 This test method uses the millipascal seconds, mPa·s, as the unit of viscosity. For information, the equivalent cgs unit, centipoise, cP, is shown in parentheses. The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard except those noted below.

1.5.1 *Exception*—This test method uses the SI unit millipascal-second (mPa·s) as the unit of viscosity. (1 cP = 1 mPa·s.)

1.6 *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.*

1.7 *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:*<sup>3</sup>

~~D91 Test Method for Precipitation Number of Lubricating Oils~~

**D4683 Test Method for Measuring Viscosity of New and Used Engine Oils at High Shear Rate and High Temperature by Tapered Bearing Simulator Viscometer at 150 °C**

**D5481 Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer**

**D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants**

<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.

Current edition approved Jan. 1, 2017; June 1, 2018. Published February 2017/July 2018. Originally approved in 1987. Last previous edition approved in 2013/2017 as ~~D4741–13~~ D4741–17. DOI: ~~10.1520/D4741-17~~ 10.1520/D4741-18.

<sup>2</sup> This test method is technically identical to that described in CEC L-36-90 (under the jurisdiction of the CEC Engine Lubricants Technical Committee) and in IP 370.

<sup>3</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.

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

**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 Coordinating European Council (CEC) Standard:<sup>4</sup>**

**CEC L-36-90 The Measurement of Lubricant Dynamic Viscosity under Conditions of High Shear (Ravenfield)**

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<sup>4</sup> Available from Coordinating European Council (CEC), Services provided by Kellen Europe, Avenue Jules Bordet 142 - 1140, Brussels, Belgium, <http://www.cectests.org>.

2.3 *Energy Institute*.<sup>5</sup>

**IP 370 Test Method for the Measurement of Lubricant Dynamic Viscosity Under Conditions of High Shear Using the Ravenfield Viscometer**

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *apparent viscosity, n*—viscosity of a non-Newtonian liquid determined by this test method at a particular shear rate and shear stress.

3.1.2 *density, n*—mass per unit volume of the test liquid at a given temperature.

##### 3.1.2.1 Discussion—

In SI notation, the unit of density is the kilogram per cubic metre. However, for practical use, gram per cubic centimetre is customarily used and is equivalent to  $10^3 \text{ kg/m}^3$ .

3.1.3 *kinematic viscosity, n*—ratio of the viscosity (dynamic, absolute) to the density of the liquid. It is a measure of the resistance to flow of a liquid where the shear stress (force causing flow) is applied by gravity. Kinematic viscosity values are thus affected by both the dynamic viscosity (absolute viscosity) of the liquid and its density.

##### 3.1.3.1 Discussion—

In SI, the unit of kinematic viscosity is the metre squared per second, often conveniently expressed as millimetre squared per second and termed the centiStoke.

3.1.4 *Newtonian oil or liquid, n*—oil or liquid that at a given temperature exhibits a constant viscosity at all shear rates and shear stresses.

3.1.5 *non-Newtonian oil or liquid, n*—oil or liquid that exhibits a viscosity that varies with changing shear stress and shear rate.

3.1.6 *shear rate, n*—velocity gradient in liquid flow in millimetres per second per millimetre (mm/s per mm) resulting from applied shear stress; the System International (SI) unit for shear rate is reciprocal seconds,  $\text{s}^{-1}$ .

3.1.7 *shear stress, n*—force per unit area causing liquid flow over the area where viscous shear is being caused; in SI, the unit of shear stress is the Pascal (Pa).

3.1.8 *viscosity, n*—ratio of applied shear stress and the resulting rate of shear. ~~It is sometimes called dynamic or absolute viscosity (in contrast to kinematic viscosity, see 3.1.3). Viscosity is a measure of the resistance to flow of the liquid at a given temperature.~~

##### 3.1.8.1 Discussion—

~~In SI, the unit of viscosity—It is sometimes called dynamic or absolute viscosity (in contrast to kinematic viscosity, see 3.1.3) is the Pascal-second (Pa·s), often conveniently expressed as milliPascal-second (mPa·s), which has the English system equivalent of the centipoise (cP). Viscosity is a measure of the resistance to flow of the liquid at a given temperature.~~

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration oils, n*—Newtonian oils used to establish the reference framework of viscosity versus torque in this instrument from which the test oil viscosity is determined.

3.2.2 *non-Newtonian check oil, n*—non-Newtonian oil used to check that the gap or distance between the rotor and stator will produce the desired operating shear rate of  $1 \times 10^6 \text{ s}^{-1}$ .

##### 3.2.2.1 Discussion—

Check oil is an acceptable name for non-Newtonian reference oil.

3.2.3 *test oil, n*—any oil for which apparent viscosity is to be determined.

### 4. Summary of Test Method

4.1 The lubricant under test fills the annulus between a close-fitting rotor and stator. The rotor and stator have a slight, matching taper to allow adjustment of the gap and hence the shear rate. The rotor is spun at a known speed, and the lubricant viscosity is determined from measurements of the reaction torque by reference to a curve prepared using Newtonian calibration oils.

<sup>5</sup> Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., <http://www.energyinst.org>.

## 5. Significance and Use

5.1 Viscosity measured under the conditions of this test method is considered to be representative of that at the temperatures and shear rates but not the pressures in the journal bearings of internal combustion engines under operating conditions.

5.2 The relevance of these conditions to the measurement of engine-oil viscosity has been discussed in many publications.<sup>6</sup>

5.3 The high temperature high shear (HTHS) viscosity at this shear rate can be measured at other temperatures using this apparatus. This is achieved by the use of a different range of Newtonian calibration fluids. . The precision has not been studied for any temperature or viscosity range not noted in the precision section.

## 6. Apparatus

6.1 *Tapered-Plug High Shear Rate Viscometer*, Model BE/C (single speed) or BS/C (multi-speed).<sup>7</sup> The viscometer uses a rotating tapered plug in a matched stator.

NOTE 1—Model BE/C has a restricted torque range and may not be capable of measuring higher viscosities at 100 °C.

6.2 *Vacuum Extract Pipe*, to ensure constant oil level. The extract pipe is supplied with all current models.

6.3 *Calibration Weight* (supplied with instrument).

6.4 *Thermostatically Controlled Heating Bath*, with fluid circulator. For acceptable temperature control and recovery time, the temperature difference between the bath and measurement head should be targeted at 4 °C and shall not exceed 8 °C. This temperature difference is influenced by the nature and rate of flow of the circulating fluid; the length and bore of the heating pipes; and the viscosity of the bath fluid.

NOTE 2—Bath oil with kinematic viscosity not greater than 10 mm<sup>2</sup>/s at 150 °C is recommended.

6.5 A means of measuring temperature is not necessary for current instruments since a precision temperature sensor is now built-in. For older instruments still in the field, a device with a precision not worse than ±0.20 °C is necessary.

6.6 The use of an ultrasonic cleaner is recommended.

6.7 The manufacturer offers a package incorporating all the above and including the necessary calibration oils, reference oils, and bath oil.

6.8 *Vacuum Pump*, with suitable liquid trap.

## 7. Materials

7.1 *Newtonian Calibration Oils*<sup>8</sup>—CEC Reference Oils RL 102, RL 103, RL 104, RL 105, RL 106, and RL 107. Cannon Certified Viscosity Reference Standard HT22 (nominal viscosity of 1.5 mPa·s at 150 °C).

7.2 *Non-Newtonian Reference Oil*<sup>8</sup>—CEC Reference Oil RL 232.

7.3 *Washing Solvent*—ASTM precipitation naphtha as specified in Test Method **D91** or a suitable replacement solvent. (**WARNING**—Extremely flammable. Vapors may cause flash fire. See **Annex A1**.)

7.3 *Flushing Solvent*—~~White~~ White mineral spirit or Stoddard solvent.

## 8. Sampling

8.1 Test oils that are visually free from haze and particulates need not be filtered before evaluation. A sample shall be free of particles larger than 3 µm. If heavy concentration of smaller particles is still visible after filtration through a filter of pore size 3 µm, it is recommended to reduce their concentration by further filtration. This will reduce the possibility of the particles wedging in the measurement gap and so causing erosion of the rotor/stator or erroneous readings. Do not filter formulated oils through pore sizes below 1 µm because certain lubricant additives may be removed.

8.2 Used oils may also be tested in these instruments, though no precision statement is available for these materials.

8.2.1 Filter used oils through a suitable filter such as Whatman GF/C fibreglass filter. The process of filtration is greatly accelerated by either warming or applying pressure. Procedures shall be such that all risk of particulate contamination is avoided.

NOTE 3—Suggestions have been made that the process of filtration may itself cause a change of viscosity by the removal of particles. No doubt if there is a very heavy concentration of particles greater than 3 µm, this will be so. It is not expected or intended that this test method will be used for such oils. Evidence to date is that filtration of used oils from normal engines in normal periods of use is acceptable. It is, however, advisable to use pressure filtration rather than vacuum filtration so that volatile components will not be removed. No precision statement is available for used oils.

<sup>6</sup> For a comprehensive review, see “The Relationship Between High-Temperature Oil Rheology and Engine Operation,” ASTM Data Series Publication 62 (out of print).

<sup>7</sup> The sole source of supply of the apparatus known to the committee at this time is Cannon Instrument Co., State College, PA 16803, <http://www.cannoninstrument.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,<sup>1</sup> which you may attend.

<sup>8</sup> Under the jurisdiction of CEC Engine Lubricants Technical Committee. ~~Ravenfield Designs Limited~~ Cannon Instrument Co. are distributors.