Designation: D4741 – 12

StandardTest Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

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

1. Scope*

1.1 This test method[2] covers the laboratory determination of the viscosity of oils at 150°C and $1 \times 10^{6} s^{-1}$ and at 100°C and $1 \times 10^{6} s^{-1}$, 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 1.8 to 5.9 mPa-s (cP) at 150°C and 4.2 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.

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-A90, 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.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

2.2 *Coordinating European Council (CEC) Standard:*[4]

L36-A90 The Measurement of Lubricant Dynamic Viscosity under Conditions of High Shear (Ravenfield)

2.3 *Energy Institute:*[5]

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*—the determined viscosity obtained by this test method.

3.1.2 *density, n*—the mass per unit volume. In the SI, the unit of density is the kg/m^3, but for practical use, a submultiple is more convenient. The g/cm^3 is 10^{-3} kg/m^3 and is customarily used.

3.1.3 *kinematic viscosity, n*—the ratio of the viscosity to the density of a liquid. It is a measure of the resistance of flow of a liquid under gravity. In the SI, the unit of kinematic viscosity is the metre squared per second; for practical use, a submultiple (millimetre squared per second) is more convenient. The centistoke (cSt) is 1 mm^2/s and is often used.

[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.

Current edition approved Nov. 1, 2012. Published February 2013. Originally approved in 1987. Last previous edition approved in 2006 as D4741–06. DOI: 10.1520/D4741-12.

[2] This test method is technically identical to that described in CEC L36–A90 (under the jurisdiction of the CEC Engine Lubricants Technical Committee) and in IP 370.

[3] 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.

[4] Available from CEC Secretariat, Interlynk Administrative Services, Ltd., Lynk House, 17 Peckleton Lane, Desford, Leicestershire, LE9 9JU, United Kingdom.

[5] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

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

3.1.4 *Newtonian oil or fluid, n*—an oil or fluid, which at a given temperature, exhibits a constant viscosity at all shear rates or shear stresses.

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

3.1.6 *shear rate, n*—the velocity gradient in fluid flow. The SI unit for shear rate is the reciprocal second (s^{-1}).

3.1.7 *shear stress, n*—the motivating force per area for fluid flow. The area is the area of shear. In the SI, the unit for shear stress is the Pascal (Pa).

3.1.8 *viscosity, n*—the ratio between the applied shear stress and rate of shear. It is sometimes called the coefficient of dynamic viscosity. This coefficient is a measure of the resistance to flow of the liquid. In the SI, the unit of viscosity is the pascal second (Pa-s); for practical use, a submultiple, millipascal second (mPa-s), is more convenient. The centipoise (cP) is 1 mPa-s and is commonly used.

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 1×10^6 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.

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.[6]

6. Apparatus

6.1 *Tapered-Plug High Shear Rate Viscometer,* Model BE/C (single speed) or BS/C (multi-speed). 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^2/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*[7]—CEC Reference Oils RL 102, RL 103, RL 104, RL 105, RL 106, and RL 107.

7.2 *Non-Newtonian Reference Oil*[7]—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.)

7.4 *Flushing Solvent*— While 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 3µm. If heavy concentration of smaller particles is still visible after filtration through a filter of pore size 3µm, 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

[6] For a comprehensive review, see "The Relationship Between High-Temperature Oil Rheology and Engine Operation," ASTM Data Series Publication 62 (out of print).

[7] Under the jurisdiction of CEC Engine Lubricants Technical Committee. Ravenfield Designs Limited are distributors.

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.

9. Initial Preparation of Apparatus

9.1 These instructions relate to instruments incorporating a computer, in other words, Models BE/C and BS/C. Changes from earlier editions of this test method are those given in 10.1.5, 10.5.1, 10.5.2, 11.1.2, and 11.1.3 and the use of a vacuum extract pipe to ensure constant oil level (see 6.2).

9.2 Set up the apparatus in accordance with the manufacturer's manual. Attach the funnel to the side arm, using the rubber sleeve provided.

NOTE 4—The funnel has a larger bore than stock funnels in order to increase the rate of flow of oil samples.

9.3 It is recommended that the instrument is NOT mounted in a fume cupboard since this draws in dirt particles. Local extraction over the heating bath is all that is necessary since the manufacturer's bath is practically sealed.

9.4 When setting up the apparatus, a torque calibration shall be performed following the instructions in the manufacturer's manual.

9.5 The instrument is supplied by the manufacturer with all other functions already calibrated and set up. It is recommended that these other initial settings be accepted until sufficient familiarity is obtained with the use of the apparatus. When it is desired to modify the initial settings, full instructions will be found in the manufacturer's manual.

9.6 It is advisable to gain access to the list of calibration oils held in the memory of the instrument in order to be familiar with its contents and to check that it is in accordance with the standards actually supplied.

9.7 *Preparation of Apparatus on All Other Occasions:*

9.7.1 Turn on the heating bath.

9.7.2 Flush out the measurement chamber using washing solvent.

9.7.3 Refill the measurement chamber with Reference Oil RL 232.

9.7.4 Leave for not less than half an hour for temperature to stabilise.

9.7.4.1 If the bath does not reach correct temperature in this time, then either extend this period or, preferably, address the problem of why heating is slow.

10. Procedure

10.1 *Outline of Method:*

10.1.1 The lubricant under test fills the annulus between a close-fitting rotor and stator. The rotor and stator have a gradual matching taper to allow adjustment of the gap and hence the shear rate. Spin the rotor at a known speed and determine the lubricant viscosity from measurements of the reaction torque by reference to a line prepared using Newtonian calibration oils.

10.1.2 Use Newtonian calibration oils to adjust the working gap and for calibration of the apparatus. These calibration oils cover a range from approximately 1.8 to 5.9 mPa-s (cP) at 150°C and 4.2 to 18.9 mPa-s (cP) at 100°C. The test method should not be used for extrapolation to higher or lower viscosities than those of the Newtonian calibration oils used for calibration of the apparatus (see 1.1).

10.1.3 Use a non-Newtonian reference oil to check that the working conditions are correct. The agreed value for this reference oil may be obtained from the Chair of CEC Surveillance Group SL-036 on Method L-36, or from the distributor.[4]

10.1.4 Use six Newtonian calibration oils to prepare a torque versus viscosity calibration. Perform a linear regression to obtain a measure of the fit of the calibration result to a true straight line and of the intercept of torque offset on the zero viscosity line.

10.1.5 The correlation coefficient is defined in Annex A2 and shall be calculated to five decimal places and shall be not less than 0.99970. The torque offset is a useful indication of the quality of a rotor and stator and its state of running-in. Torque offset may be used as a laboratory quality control parameter.

10.1.6 When a satisfactory correlation coefficient has been obtained, measure the non-Newtonian reference oil. This oil shall also be used after every three to six test measurements to maintain a continuous check on the correct functioning of the instrument.

10.1.7 The initial measured value for reference oil shall be equal to its value as stated by the manufacturer within ±0.04 mPa-s at 150°C and within ±0.06 mPa-s at 100°C. Subsequent measured values for reference oil shall be equal to its value as stated by the manufacturer within ±0.06 mPa-s, providing it is not in the *opposite* direction from the initial deviation from nominal.

10.1.8 If at any point the check oil measured value falls outside the acceptable limits, discard all test oil values determined since the last successful check oil value and remeasure, following an acceptable check oil determination.

10.1.9 Take readings at the point of transition from 149.9°C to 150.0°C or 99.9°C to 100.0°C. This is accomplished automatically in the Model BS/C and manually in other models. The rate of rise of temperature shall not be faster than 0.1°C in 4 s (0.025°C per s) when operated manually. In automatic operation, the rate of rise may be allowed to increase to 0.07°C per s.

10.1.10 No *maximum* limit is specified on how long this rise from 149.9°C to 150.0°C or 99.9°C to 100.0°C may take, but it is suggested that delays of more than 8 or 10 s may make the test method unduly cumbersome to operate. A variation of this period from measurement to measurement will reduce the precision of the test method.

10.1.11 Take at least two measurements to yield a result. If the difference between successive measurements is greater than 1 %, then take a third or even fourth reading. Such a deviation