



Designation: D4741 – 21

Standard Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer¹

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 U.S. Department of Defense.

1. Scope*

1.1 This test method² 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 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 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.

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² 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.

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:³

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, Liquid Fuels, 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 Coordinating European Council (CEC) Standard:⁴

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

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

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

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*—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.

3.1.3 *kinematic viscosity, n*—the ratio of the dynamic viscosity (η) to the density (ρ) of a liquid at a given temperature.

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

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

3.1.6 *shear rate, n*—velocity gradient perpendicular to the direction of flow.

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

3.1.8 *viscosity, n*—the ratio between the applied shear stress and rate of shear which is sometimes called the coefficient of dynamic viscosity and is a measure of the resistance to flow of the liquid.

3.1.8.1 *Discussion*—It is sometimes called dynamic or absolute viscosity (in contrast to kinematic viscosity, see 3.1.3).

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.

⁵ 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.⁶

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).⁷ 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 mm²/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 $\pm 0.20 \text{ °C}$ 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.

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 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,¹ which you may attend.

7. Materials

7.1 *Newtonian Calibration Oils*⁸—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*⁸—CEC Reference Oil RL 232.

7.3 *Flushing Solvent*—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.

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 Flush out the measurement chamber using flushing solvent

9.7.2 Turn on the heating bath.

9.7.3 Refill the measurement chamber with Non-Newtonian 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 (7.1) to adjust the working gap and for calibration of the apparatus. These calibration oils cover a range from approximately 1.5 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. 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).

NOTE 5—When operating at temperatures other than 100 °C and 150 °C, contact the instrument manufacturer for the appropriate calibration standards.

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 A1 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

⁸ Under the jurisdiction of CEC Engine Lubricants Technical Committee. Cannon Instrument Co. are distributors.

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 s 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 is normally indicative of inadequate flushing of a previous sample. An extra flush before taking a measurement may help to obtain accurate results more quickly.

10.2 Sample Insertion:

10.2.1 Insert oils, whether reference fluids or sample fluids, by means of the funnel mounted on the side arm and withdrawn by the constant level vacuum pipe to waste.

10.2.2 Fill the funnel, then allow to drain into the measurement cell, then refill one or more times, as detailed below.

10.2.3 When inserting an oil of noticeably different viscosity from the previous sample (for example, RL102 following after RL106), use four funnefuls. Otherwise, use two funnefuls. One funneful is approximately 10 mL.

10.2.4 For all repeat measurements, one funneful is adequate.

NOTE 6—The object of inserting oil for a repeat measurement is to ensure that the indicated temperature falls. To ensure this, it is advisable to try not to trap a bubble below the funnel.

10.2.5 A minimum temperature drop before making a measurement shall be not less than 1 °C.

10.3 Set Shear Rate:

10.3.1 It is necessary to adjust the operating gap between the rotor and stator so that the test shear rate shall be 1×10^6 s⁻¹.

10.3.2 Use Reference Oil RL106, and adjust to the correct torque as instructed in the manufacturer's manual.

10.3.3 When, as the temperature passes from 149.9 °C to 150.0 °C or 99.9 °C to 100.0 °C, the torque indicated agrees with the torque calculated at the current depth indicated by the dial gage reading, proceed to 10.4. It is possible that this may be no longer true after adjustments called for by the non-Newtonian reference oil.

NOTE 7—This apparent discrepancy is caused by the existence of offsets in the torque measurement system and metallic contact in the rotor and stator.

10.4 Prepare Calibration Line:

10.4.1 Use six Newtonian oils to prepare a calibration line of viscosity versus torque. This shall be used to prepare a linear regression, which shall meet the requirement detailed in 10.1.5.

10.4.2 Measure RL102 at least twice to obtain a result as detailed in 10.1.11. Then measure oils RL103 to RL107 in order of ascending viscosity, repeating RL106, and prepare a calibration line as described in 10.1.4 and 10.1.5.

10.5 Use the non-Newtonian reference oil. Measure the viscosity of the non-Newtonian reference oil.

10.5.1 The value obtained for the reference oil shall meet the requirements detailed in 10.1.7. If it does not, then make adjustments as detailed in the manufacturer's manual and repeat 10.4 and 10.5.

10.5.2 Testing shall only proceed when a satisfactory correlation coefficient and a satisfactory value for the reference oil have been obtained.

11. Test Operation

11.1 Test Operation:

11.1.1 Inset a sample oil in accordance with 10.2, note the torque reading as detailed in 10.1.9 and repeat at least once as detailed in 10.1.11.

11.1.2 Calculate the viscosity from the linear regression of viscosity on torque obtained in 10.4.

11.1.3 Repeat the measurement of the non-Newtonian reference oil not less often than every six sample results.

12. Report

12.1 Report the following information:

12.1.1 The temperature at which the HTHS viscosity was measured, typically 150 °C or 100 °C and 1×10^6 s⁻¹ in mPa·s.

12.1.2 The shear rate of the viscosity measurement, typically 1×10^6 s⁻¹ in mPa·s.

12.1.3 The HTHS viscosity to two decimal places.

12.1.4 When it is necessary to reduce the number of decimal places in accordance with 12.1.3, this shall be done by rounding to the nearest figure. NOT by truncation. Where the last digit to be rounded is five, the last significant digit shall be rounded up.

13. Test Evaluation

13.1 The evaluation of the test method is performed by observing the correlation coefficient and the reference oil value, and is continuously monitored by use of the reference oil.