Designation: D 4741 – 00

An American National Standard

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

This standard is issued under the fixed designation D 4741; 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.

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:*

D 91 Test Method for Precipitation Number of Lubricating Oils[3]

D 4683 Test Method for Measuring Viscosity at High Shear Rate and High Temperature by Tapered Bearing Simulator[4]

2.2 *Coordinating European Council (CEC) Standard:*[5]

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

2.3 *Institute of Petroleum Standard:*[6]

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.

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

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[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] 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] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

[5] Available from Coordination European Council, Madou Plaza, 25th floor, Place Madou 1, B-1030, Brussels, Belgium.

[6] Available from Institute of Petroleum, 61 New Cavendish St., London WIM 8AR, U.K.

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.[7]

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*[8]—CEC Reference Oils RL 102, RL 103, RL 104, RL 105, RL 106, and RL 107.

7.2 *Non-Newtonian Reference Oil*[8]—CEC Reference Oil RL 174.

7.3 *Washing Solvent*—ASTM precipitation naphtha as specified in Test Method D 91 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 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

[7] For a comprehensive review see "The Relationship Between High-Temperature Oil Rheology and Engine Operation," ASTM Data Series Publication 62. Available from ASTM Headquarters.

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

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 174.

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.[5]

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 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 funnelfuls. Otherwise, use two funnelfuls. One funnelful is approximately 10 mL.

10.2.4 For all repeat measurements, one funnelful is adequate.

NOTE 5—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 $1 \times 10^6 s^{-1}$.

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 6—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 result as the high temperature, high shear viscosity at 150°C or 100°C and $1 \times 10^6 s^{-1}$ in mPa-s.

12.1.2 The value to two decimal places.

12.1.3 When it is necessary to reduce the number of decimal places in accordance with 12.1.2, 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.

14. Precision and Bias

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

1.7 % of the mean at 150°C
1.0 % of the mean at 100°C

14.1.1 This repeatability was established for viscosities from 2.6 to 4.7 mPa-s at 150°C and from 4.9 to 11.8 mPa-s at 100°C and is independent of viscosity within these ranges.

14.2 *Reproducibility*—The difference between two single independent results obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty.

2.6 % of the mean at 150°C
2.4 % of the mean at 100°C

14.2.1 This reproducibility was established for viscosities from 2.6 to 4.7 mPa-s at 150°C and from 4.9 to 11.8 mPa-s at 100°C.

14.3 The precision values in 14.1 and 14.2 for 150°C were obtained by statistical examination of interlaboratory results on 12 non-Newtonian test oils tested in 9 laboratories (432 observations in total). The test oil viscosities were between 2.5 mPa-s and 4.8 mPa-s at 150°C and $1 \times 10^6 s^{-1}$ and viscosity grades were SAE 0W-30, 5W-30, 10W-30, 10W-40, and 15W-50.[9]

14.4 The precision values in 14.1 and 14.2 for 100°C were obtained by statistical examination of interlaboratory results on 9 non-Newtonian test oils in 7 laboratories (126 observations in total). The test oil viscosities were between 4.9 mPa-s and 11.8 mPa-s at 100°C and $1 \times 10^6 s^{-1}$ and viscosity grades were SAE 0W-10, 5W-30, 15W-40, 20W-40, 20W-50, and 25W-30, 30, and 40.[10]

14.5 *Bias*—The bias of this test method is less than 0.06 mPa-s. This is guaranteed by the requirement to verify periodically the value of a reference oil to within these limits.

14.6 *Relative Bias*—Results from this test method were found, by interlaboratory test studies[10,11] to agree with those from Test Method D 4683 at both 100°C and 150°C. They can be expected to give, on average in the long run, the same results for the same oil.

15. Keywords

15.1 dynamic viscosity; high shear viscosity; high temperature; HTHS; rotational viscometer

[9] Supporting data have been filed at ASTM Headquarters. Request RR:D02-1497.

[10] Supporting data have been filed at ASTM Headquarters. Request RR:D02-1496.

[11] Supporting data have been filed at ASTM Headquarters. Request RR:D02-1378.

ANNEXES

(Mandatory Information)

A1. PRECAUTIONARY STATEMENT FOR PRECIPITATION NAPTHA

A1.1 Warning

A1.1.1 Extremely inflammable, harmful if inhaled. Keep away from heat sparks and open flames. Keep container closed, use with adequate ventilation. Avoid build-up of vapors, and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.

A1.1.2 Avoid prolonged breathing of vapor or spray mist.

A1.1.3 Avoid prolonged or repeated skin contact.

A2. DEFINITION OF CORRELATION COEFFICIENT

A2.1 Definition of Correlation Coefficient

A2.1.1 Correlation coefficients have been defined in different ways. The correlation coefficient to be used for this test method is defined by the formula:

$$r = \frac{M\sum_{i=1}^{M} x_i y_i - (\sum_{i=1}^{M} x_i)(\sum_{i=1}^{M} y_i)}{\sqrt{[M\sum_{i=1}^{M} x_i^2 - (\sum_{i=1}^{M} x_i)^2][M\sum_{i=1}^{M} y_i^2 - (\sum_{i=1}^{M} y_i)^2]}} \quad \text{(A2.1)}$$

where:

M = the number of data points, and

x_i and y_i = the observed values of the two variables.

This is the equation used by the computer program built into the instruments covered by this test method.