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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.

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 1×10^{6} s^{-1} using Ravenfield high shear rate tapered-plug viscometer models BE or BS.[3] This test method may readily be adapted to other conditions if required.

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 cP (mPa.s) at 150°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.

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 check oil may be obtained by reference to the chairman of the CEC Surveillance Group for the Ravenfield tapered-plug viscometer method L-36 or to the distributor.

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

1.5 This test method uses the centipoise (cP) as the unit of viscosity. For information, the equivalent SI unit, the millipascal second (mPa.s) is shown in parenthesis.

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]

D 5481 Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer[4]

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

L-36 Test Method for the Measurement of Lubricant Dynamic Viscosity under Conditions of High Shear

2.3 *Institute of Petroleum (IP) 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*—the determined viscosity obtained by this test method.

3.1.2 *density*—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*—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 centistokes (cSt) is 1 mm^2/s and is customarily used.

3.1.4 *Newtonian oil or fluid*—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*—an oil or fluid which exhibits a viscosity that varies with changing shear stress or shear rate.

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

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

[1] This test method is under the jurisdiction of ASTM Committee D-02 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 L-36 (under the jurisdiction of the CEC Engine Lubricants Technical Committee) and in Institute of Petroleum method IP 370.

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

[4] *Annual Book of ASTM Standards*, Vol 05.03.

[5] Available from the CEC, 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. 071636 1004.

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stress is the Pascal (Pa).

3.1.8 *viscosity*—the ratio between the applied shear stress and rate of shear. It is sometimes called the coefficient of dynamic viscosity. This coefficient is thus 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 customarily used.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *non-Newtonian check oil*—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.

3.2.3 *test oil*—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 is thought 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 *Ravenfield Tapered Plug High Shear Viscometer*[3]—Model BE (single speed) or BS (multispeed). The viscometer uses a rotating tapered plug in a matched stator.

6.2 *Calibration Weight*, (supplied with instrument).

6.3 *Wash Bottle*, fitted with metal tip (supplied with instrument).

6.4 *Thermostatically Controlled Heating Bath*, with fluid circulator.

6.5 *Temperature Measuring Instrument*, having a precision not worse than ±0.2°C at 150°C.

6.6 *Vacuum Pump*, with suitable liquid trap.

7. Materials

7.1 *Newtonian Calibration Oils* [8], CEC Reference Oils RL 102-107.

7.2 *Non-Newtonian Check Oil* [8], CEC Reference Oil RL 90.

7.3 *Washing Solvent*, ASTM precipitation naphtha as specified in Test Method D 91 or a solvent of similar volatility.

NOTE 1—**Warning:** Extremely flammable. Vapors may cause flash fire. See Annex A1.

8. Sampling

8.1 Test oils that are visually free from haze and particulates need not be filtered before evaluation. A sample must be free of particles larger than 3 µm. If after filtration through a filter of pore size 3 µm, a heavy concentration of smaller particles is still visible, it is wise at least 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 could be removed.

9. Preparation of Apparatus

9.1 *Insertion of Stator*—For detailed instructions, see Manufacturer's Manual.

9.1.1 Remove the top nylon cover from the stator housing.

9.1.2 Remove the screwed cover.

9.1.3 Place an "O" ring at the bottom of the heating-jacket chamber, making sure it is sitting correctly in the corner of the housing.

9.1.4 Clean the stator thoroughly, preferably using an ultrasonic bath.

9.1.5 Insert the stator into its housing with the thermocouple pocket uppermost and at the left-hand side. Ensure that the stator is sitting squarely in the center of the housing.

9.1.6 Place a second "O" ring above the stator, positioning it in the corner formed between the stator and the housing.

9.1.7 Place the clamping ring (chamfered end down) on top of the "O" ring and replace the screwed cover *as tightly as possible using the fingers only.* Metal-to-metal contact should be achieved both above and below the stator. Failure to do so may result in movement of the stator.

9.1.8 Replace the top nylon cover.

9.1.9 Insert the thermocouple into its pocket. Secure the wire in the clamp provided on the top of the instrument base, ensuring that the thermocouple reaches the bottom of the pocket.

9.1.10 Insert the spill-prevention ring.

9.2 *Setting Zero*:

9.2.1 Mechanical zero is determined by the balance of two springs and is not adjustable.

9.2.2 Transducer zero is set by the manufacturer and only requires readjustment if the internal mechanism is disassembled.

9.2.3 Electrical zero is set as follows:

9.2.3.1 Remove the rotor coupling from the motor shaft. (Not necessary for Model BS.)

9.2.3.2 Before setting electrical Zero and Span (see 9.3), the apparatus must be switched on (with the motor switched off) for a period of 30 min to achieve a stable condition. *The heating bath must not be switched on at any time unless a stator is correctly installed to avoid damage to the rotor/stator.*

9.2.3.3 Switch the motor on and allow about 10 s for the brake to release and the motor cage to stabilize. (Not necessary for Model BS.)

9.2.3.4 The indicator shows a value varying only one or two

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

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

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digits up or down. The control labelled "Z" may now be used to set the indicator, such that it swings symmetrically about 000.

NOTE 2—Even if the zero appears to have drifted, it must not be reset with the rotor inserted into the stator (Model BE only).

9.3 *Setting Full-Scale Deflection (Span)*:

9.3.1 Mechanical setting is not possible and is determined by the characteristics of the low-temperature-coefficient springs only.

9.3.2 Electrical full-scale deflection is set by applying a known torque and adjusting the digital read-out to match this value. This adjustment shall be carried out after setting electrical zero (see 9.2.3).

9.3.2.1 Detach the top fiberglass cover from the instrument, after removing the securing screws.

9.3.2.2 Attach about 800 to 1000 mm of strong cotton thread by a loop to the peg on the machined calibration drum at the top of the swinging frame. About 300 mm from the loop, tie another length of cotton thread about 300 mm long and fasten this to an adjustable support (for example, a ring stand). Attach the calibration weight (supplied) to the remaining free end and adjust the assembly as shown in Annex A2.

9.3.2.3 By means of the control labelled "S" on the panel, set the indicator reading to the applied torque in gram centimetres (calibration weight g × 7 cm drum radius). This adjustment must be carried out with the motor running on Model BE and stationary on Model BS.

9.3.3 After setting full-scale deflection, check the zero setting. It may be necessary to repeat both settings due to a small amount of interaction. Both controls retain their setting and regular readjustment is not necessary.

9.4 *Assembly and Installation of Rotor*:

9.4.1 Fit the rotor to the cleaned universal joint and secure it using the set screw, noting that the numbered end of the rotor is the top.

9.4.2 Fit the rotor coupling sleeve similarly.

9.4.3 Thoroughly clean this assembly, preferably using an ultrasonic bath.

9.4.4 Screw the rotor coupling sleeve on to the motor shaft and lock it tightly in position.

9.4.5 Using any clean calibration oil, lubricate the rotor. Lower the rotor into the stator until the top of the rotor is approximately flush with the top of the stator. At this stage lower it no further.

9.5 *Setting of Shear Rate*:

9.5.1 For calibration oil RL 106 calculate the torque to be expected at a shear rate of 1×10^6 s $^{-1}$ using the constants from the calibration certificate supplied with each rotor/stator pair. Make the initial assumption that the active rotor length is equal to "h." (See Annex A3 for the relevant equations.)

9.5.2 With the system at test temperature, carefully adjust the vertical position of the rotor using the handwheel until the top of the rotor is flush with the top of the stator. This may conveniently be done by feeling for a "step" with the end of an Allen key.

9.5.3 Mount the dial gage on its arm and adjust the stop pillar and dial until the gage reads zero.

9.5.4 Lower the rotor using the handwheel for eight complete revolutions of the dial gage. Raise the ball stop until it is supporting the upper system. Turn the handwheel so that the lifting screw is well clear.

9.5.5 Using the washing solvent, flush out the stator several times, turning the rotor gently with the fingers. Apply the solvent to the top of the rotor and remove by suction at the side arm.

NOTE 3—Solvent should be applied using a wash bottle fitted with a metal tip. (**Warning**—Extremely flammable. Vapors may cause flash fire. See Annex A1.)

9.5.6 Using the following oil flush procedures, introduce calibration oil RL 106 into the measuring system until the rotor is just covered.

NOTE 4—The oil flush procedure, using the oil level pipe and running the motor continuously, is the preferred method for introducing and changing test oil. If quantities of test oil are limited, the solvent flush procedure may be used. The solvent flush procedure is described in Appendix X2. The precision and bias statements in Section 13 do not apply to the solvent flush procedure.

9.5.7 Mount the suction pipe on the top surface of the instrument with its tip about 4 mm above the top of the stator and about 2 mm away from the rotor drive coupling. The pipe may be bent readily if the height is initially incorrect.

9.5.8 Switch on the suction pump.

9.5.9 Place about 5 to 10 mL of washing solvent into the funnel. This is sufficent to ensure that some is drawn-off by the suction pipe. The use of washing solvent at this stage is to ensure that the rotor/stator is subjected to a periodic wash to minimize buildup of deposits.

9.5.10 Fill the funnel with the first oil sample.

9.5.11 For XBE and BE models, switch on the motor using the motor switch on the front panel. The start-up brake will be heard to operate and release after a few seconds. For XBS and BS models, select 3000 r/min, 5 s (RAMP), 99 h (PAUSE), 1 (STEP), then switch the motor on.

9.5.12 Wait until the oil has fallen to the neck of the funnel. Refill the funnel with a second portion of the oil sample.

9.5.13 Wait until the oil has fallen to the neck of the funnel.

9.5.14 Switch off the suction pump.

NOTE 5—Since the motor is running all the time, the sample in the measuring section is very thoroughly removed. Two full funnels are sufficient to remove previous oils when viscosities are close. If a high vicosity oil (for example, RL 107) is to be followed by one of low viscosity (for example, RL 102), repeat 9.5.12 to 9.5.13 using a third portion of the oil sample.

9.5.15 Switch off the motor.

9.5.16 Lower the ball stop slowly, gently turning the rotor by hand until a definite resistance to rotation is felt. This increased resistance is usually only felt around part of a revolution. Complete lock-up of the rotor shall be avoided. Record the dial gage reading.

9.5.17 Raise the ball stop for two complete revolutions of the dial gage.

9.5.18 Switch on the motor. The starting brake will be heard to release after a few seconds (Model BE).

9.5.18.1 Alternatively, for Model BS, select 3000 r/min, 5 s (RAMP), 99 h (PAUSE), 1 STEP. Switch motor on.

9.5.19 Observe the torque reading. After a few initial

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swings, it will be observed first to read an increased torque, as cold oil is drawn in, then drift steadily downwards as the sample heats up.

9.5.20 Allow the motor to run for 30 to 60 s. During this period the temperature will rise due to viscous heating and the torque reading will fall. Record the torque reading at an appropriate temperature during this period. (An appropriate temperature for this purpose is any temperature in the range 145°C to 155°C which fulfills the rate of rise requirements specified in 9.6.3.)

9.5.21 Switch off the motor.

9.5.22 Change the oil for a fresh sample using the oil flush procedure (see 9.5.8 to 9.5.14).

9.5.23 Again introduce calibration oil RL 106, switch on the motor for several seconds, remove the oil by the side arm and recharge.

9.5.24 Repeat operations 9.5.18 to 9.5.20. The same reading should be obtained plus or minus two digits.

9.5.25 Restart the motor and lower the ball stop in small increments, observing the torque. Allow the motor to run for about 5 min at each stage. The rotor should be lowered in this way until the torque calculated at 9.5.1 is achieved at the desired temperature. Halt the lowering process if the required torque is not achieved by the time the dial gage has approached to within 0.03 mm of the figure noted at 9.5.16 and consult the manufacturer's handbook.

NOTE 6—A new or reconditioned rotor/stator assembly requires running in and 9.5.25 must not be rushed. Once a rotor/stator pair has been conditioned in this way, it is possible to advance to a known figure of dial reading and thus achieve the correct torque rapidly.

9.5.26 Having achieved the desired torque reading, it is necessary to recalculate the value using the active length of the rotor to take account of the fact that the active length may be less than "h." The active length is determined using the dial gage reading and the dimensions from the calibration certificate; the necessary equations are given in Annex A3. Fine adjustment results in a position where the torque value is valid for the true active length of the rotor.

9.5.27 Check the calibration with a fresh sample.

9.6 *Temperature Setting*:

9.6.1 Initially, set the bath temperature 1°C to 2°C above the test temperature.

9.6.2 Insert a new sample of oil, that causes the indicated temperature to fall.

9.6.3 Switch on the motor; the temperature will be observed to rise, initially fairly rapidly, then at a lower rate. The target rate of temperature increase is 0.1°C in not less than 4 s as the measurement temperature is passed through.

9.6.4 Adjust the bath temperature, initially in 0.5°C increments, then smaller as required, until the condition at 9.6.3 is achieved.

NOTE 7—The rate of shear heating depends upon the viscosity of the test oil, hence some adjustment of the bath temperature will be required when testing oils of widely differing viscosity. With experience, the correct bath temperature may be readily set.

9.7 *Calibration Graph*:

9.7.1 Introduce the next oil using the oil flush procedure (see 9.5.8 to 9.5.14).

9.7.2 If the two determinations of the torque agree within 1 %, the average (denoted here as a result) should be used in the plot of torque against viscosity in 9.7.4. If this agreement is not obtained, repeat operation 9.7.1 until any two determinations within 1 % of each other are obtained. Calculate the average of these two determinations. Usually no more than three determinations are required.

9.7.3 Repeat operations 9.7.1 and 9.7.2 with the other five calibration oils.

9.7.4 Plot torque result against viscosity for the six calibration oils, on as large a scale as convenient. This plot must be a straight line, ideally passing through the origin. Alternatively a regression analysis may be used to determine the relationship between torque result and viscosity. Regression analysis shows that correlation coefficients greater than 0.999 are achievable. Any intercept greater than +10 g·cm suggests that metal-to-metal contact may be occurring, in which case cease testing immediately and consult the manufacturer's handbook.

9.8 *Severity Check:* A non-Newtonian check oil is used to eliminate day-to-day variations in the severity of the test and also to minimize interlaboratory variation, thereby improving reproducibility.

9.8.1 Introduce the non-Newtonian check oil into the viscometer on completion of 9.7 and hence determine the torque value for this oil, at a shear rate of 10^6 s^{-1} and 150°C, by following operations 9.7.1 and 9.7.2.

9.8.2 Determine the viscosity from the calibration graph or regression equation. The value should be within the accepted limits given for the batch of check oil in use.

9.8.3 Record the dial gage reading.

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10. Procedure

10.1 Switch on the heating bath, circulating pump and viscometer electronic unit at least 3 h before carrying out any determinations.

10.2 Lower the rotor into the stator until the frame contacts the ball-stop. Continue winding the handwheel until the lifting mechanism is definitely disengaged.

10.3 Check that the dial gage reading is that obtained at 9.8.3.

10.4 Carry out the severity check as in 9.8.

10.5 Introduce the test oil as detailed in 9.7.1.

10.6 Remove the sample by the side arm and charge with test oil.

10.7 Carry out operations 9.7.1 and 9.7.2 to obtain a torque result, which is the average of two determinations that agree within 1 %.

NOTE 8—When the viscometer is to be left non-operational for more than a few minutes, it is recommended that the rotor be raised slightly by means of the handwheel. To avoid damage to the equipment, the rotor *must* always be raised when the heating is switched off.

11. Calculation

11.1 Using the torque result calculated in 10.7, read the viscosity value from the calibration graph. Alternatively, calculate the viscosity from the regression of viscosity on torque obtained with the calibration oils. A test report sheet is attached as Appendix X1.