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An American National Standard

Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature[1]

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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07.0C on Low Temperature Rheology of Non-Newtonian Fluids.

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1. Scope *

1.1 This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period exceeding 45 h to a final test temperature between −10 and −40°C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s^{-1}.

1.2 This test method is applicable for unused oils, sometimes referred to as fresh oils, designed for both light duty and heavy duty engine applications. It also has been shown to be suitable for used diesel oils. The applicability to petroleum products other than engine oils has not been determined.

1.3 This test method uses the millipascal second (mPa·s) as the unit of viscosity. For information, the equivalent centipoise unit is shown in parentheses.

1.4 *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. Terminology

2.1 *Definitions:*

2.1.1 *apparent viscosity*—the determined viscosity obtained by use of this test method.

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

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

2.1.4 *shear rate*—the velocity gradient in fluid flow. For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner cylinder surface (such as this apparatus, described in 5.1), and ignoring any end effects, the shear rate is given as follows:

$$G_r = \frac{2(\Omega)R_s^2}{R_s^2 - R_r^2} \tag{1}$$

$$= \frac{4(\pi)\,R_s^2}{t\,(R_s^2 - R_r^2)} \tag{2}$$

where:

G_r = shear rate at the surface of the rotor in reciprocal seconds, s^{-1},
Ω = angular velocity, rad/s,
R_s = stator radius, mm,
R_r = rotor radius, mm, and
t = time in seconds for one revolution of the rotor.

For the specific apparatus being described in 5.1.1,

$$G_r = 63/t \tag{3}$$

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

2.1.5 *shear stress*—the motivating force per unit area for fluid flow. For the rotary viscometer being described, the rotor surface is the area under shear or the shear area.

$$T_r = 9.81\, M\, (R_o + R_t) \times 10^{-6} \tag{4}$$

$$S_r = \frac{T_r}{2\,(\pi) R_r^2\, h} \times 10^{9} \tag{5}$$

where:
T_r = torque applied to rotor, N·m,
M = applied mass, g,
R_o = radius of the shaft, mm,
R_t = radius of the string, mm,
S_r = shear stress at the rotor surface, Pa, and
h = height of the rotor, mm.

For the dimensions given in 5.1.1,

$$T_r = 31.7\, M \times 10^{-6} \tag{6}$$

$$S_r = 3.5\, M \tag{7}$$

2.1.6 *viscosity*—the ratio between the applied shear stress and rate of shear, sometimes called the coefficient of dynamic viscosity. This value is thus a measure of the resistance to flow of the liquid. The SI unit of viscosity is the pascal second [Pa·s]. A centipoise (cP) is one millipascal second [mPa·s].

2.2 *Definitions of Terms Specific to This Standard:*

2.2.1 *calibration oils*—those oils that establish the instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined. Calibration oils, which are essentially Newtonian fluids, are available commercially, and have an approximate viscosity of 30 Pa·s (30 000 cP) at −20°C.[2]

2.2.2 *test oil*—any oil for which the apparent viscosity and yield stress are to be determined by this test method.

2.2.3 *unused oil*—an oil which has not been used in an operating engine.

2.2.4 *used oil*—an oil which has been used in an operating engine.

2.2.5 *yield stress*—the shear stress required to initiate flow. For all Newtonian fluids and some non-Newtonian fluids, the yield stress is zero. An engine oil can have a yield stress that is a function of its low-temperature cooling rate, soak time, and temperature. Yield stress measurement by this test method determines only whether the test oil has a yield stress of at least 35 Pa; a yield stress below 35 Pa is considered to be insignificant.

3. Summary of Test Method

3.1 An engine oil sample is held at 80°C and then cooled at a programmed cooling rate to a final test temperature. A low torque is applied to the rotor shaft to measure the yield stress. A higher torque is then applied to determine the apparent viscosity of the sample.

4. Significance and Use

4.1 When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test, an engine oil is slowly cooled through a temperature range where wax crystallization is known to occur, followed by relatively rapid cooling to the final test temperature. These laboratory test results have predicted as failures the known engine oils that have failed in the field due to the lack of oil pumpability.[3] These documented field failing oils have all consisted of oils normally tested at −25°C. These field failures are believed to be the result of the oil forming a gel structure that results in excessive yield stress or viscosity of the engine oil, or both.

4.2 *Cooling Profiles*:

4.2.1 For oils to be tested at −20°C or colder, Table X1.1 applies. The cooling profile described in Table X1.1 is based on the viscosity properties of the ASTM Pumpability Reference Oils (PRO). This series of oils includes oils with normal low-temperature flow properties and oils that have been associated with low-temperature pumpability problems **(1-5)**.[4] Significance for the −35 and −40°C temperature profiles is based on the data collected from the "Cold Starting and Pumpability Studies in Modern Engines" conducted by ASTM **(6,7)**.

4.2.2 For oils to be tested at −15 or −10°C, Table X1.2 applies. No significance has been determined for this temperature profile because of the absence of appropriate reference oils. Similarly, precision of the test method using this profile for the −10°C test temperature is unknown. The temperature profile of Table X1.2 is derived from the one in Table X1.1 and has been moved up in

[2] The sole source of supply of the apparatus known to the committee at this time is Cannon Instrument Co., P.O. Box 16, State College, PA 16804. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee[1], which you may attend.

[3] Pumpability Reference Oils (PRO) 21 through 29.

[4] The boldface numbers in parentheses refer to the references at the end of this standard.

temperature, relative to Table X1.1, in consideration of the expected higher cloud points of the viscous oils tested at −15 and −10°C.

5. Apparatus

5.1 *Mini-Rotary Viscometer*[2]—An apparatus that consists of one or more viscometric cells in a temperature-controlled aluminum block. Each cell contains a calibrated rotor-stator set. Rotation of the rotor is achieved by an applied load acting through a string wound around the rotor shaft.

5.1.1 The mini-rotary viscometric cell has the following typical dimensions:

Diameter of rotor	17.0 mm
Length of rotor	20.0 mm
Inside diameter of cell	19.0 mm
Radius of shaft	3.18 mm
~~Radius of string~~	~~0.05mm~~
Radius of string	0.1 mm

5.2 *Temperature Control System* —Will regulate the mini-rotary viscometer block temperature in accordance with the temperature limits described in Table X1.1 or Table X1.2.

5.2.1 *Temperature Controller*—The most critical part of this procedure. A description of the requirements that the controller shall meet are included in Appendix X2.

5.2.2 *Temperature Profile*—The temperature profile is fully described in Table X1.1 and Table X1.2.

5.3 *Thermometers*[2]—For measuring the temperature of the block. Two are required, one graduated from at least +70 to 90°C in 1°C subdivisions, the other with a scale from at least −36 to +5°C or −45 to +5°C, in 0.2°C subdivisions. Other thermometric devices of equal accuracy and resolution may be used to calibrate the temperature sensor.

5.4 *Refrigeration Device*—Consisting of a means of maintaining a coolant (such as methanol) to at least 10°C below the lowest test temperature.

5.5 *Circulating System*[2]—That will circulate the liquid coolant to the block as needed. Methanol is a suitable coolant. One should observe toxicity and flammability precautions that apply to the use of methanol. The circulating system shall be capable of maintaining test temperature during the test. If methanol is leaking from the system, discontinue the test and repair the leak. (**Warning—**Methanol is flammable.)

5.6 *Chart Recorder*—To verify that the correct cooling curve is being followed, it is recommended that a chart recorder be used to monitor the block temperature.

6. Reagents and Materials

6.1 *Newtonian Oil*[2]—A low cloud-point of approximately 30 Pa·s (30 000 cP) viscosity at −20°C for calibration of the viscometric cells.

6.2 *Methanol*—Commercial or technical grade of dry methanol is suitable for the cooling bath.

6.3 *Oil Solvent*—Commercial heptanes or similar solvent that evaporates without leaving a residue is suitable. (**Warning—** Flammable.)

6.4 *Acetone*—A technical grade of acetone is suitable provided it does not leave a residue upon evaporation. (**Warning—** Flammable.)

7. Sampling

7.1 A representative sample of test oil free from suspended solid material and water is necessary to obtain valid viscosity measurements. If the sample in its container is received below the dew-point temperature of the room, allow the sample to warm to room temperature before opening the container.

8. Calibration and Standardization

8.1 Calibrate the temperature sensor while attached to the temperature controller. The sensed temperature shall be verified using a reference thermometer noted in 5.3 at a minimum of three temperatures. Make these temperature measurements at least 5°C apart to establish a calibration curve for this combination of temperature sensor and controller.

NOTE 1—All temperatures in this test method refer to the actual temperature and *not* necessarily the indicated temperature.

8.2 The calibration of each viscometric cell (viscometer constants) can be determined with the viscosity standard and the following procedure at −20°C:

8.2.1 Use steps 9.2-9.2.5.

8.2.2 Program the temperature controller to cool the mini-rotary viscometer block to −20°C within 1 h or less, then start the program.

8.2.3 Allow the oil in the cells to soak at −20 ± 0.2°C for at least 1 h, making small temperature adjustments, if necessary, to maintain the test temperature.

8.2.4 At the end of the soak period, record the temperature reading (test temperature) and remove the cover of the viscometer cell.

8.2.5 Proceed to 9.3.1-9.3.3.

8.2.6 Perform step 9.4.1.

8.2.7 Repeat 8.2.5 and 8.2.6 for each of the remaining cells, taking the cells in order from left to right.

8.2.8 Calculate the viscometer constant for each cell (rotor/stator combination) with the following equation:

$$C = \eta_o/t \quad (8)$$

where:

η_o = viscosity of the standard oil, cP (mPa·s) at −20°C,
C = cell constant with 150 g mass, Pa, and
t = time in seconds for three complete revolutions.

8.2.9 If any cell has a calibration constant more than 10 % higher or lower than the average for the other cells, the fault may be a problem with rotor operation. Examine rotor for damage, and recalibrate the instrument.

8.3 If corrected values for controller temperature and thermometer deviate by more than the tolerance, use X2.2 to assist in determining the fault.

9. Procedure

9.1 Program the temperature controller to control the mini-rotary viscometer block temperature as outlined in Table X1.1 or Table X1.2. The programmed temperature is the temperature in Table X1.1 or Table X1.2 plus the appropriate temperature correction factor determined in 8.1. Table X1.3 lists the nominal times to reach a particular test temperature.

9.2 *Test Sample and Viscometric Cell Preparation*:

9.2.1 Remove the nine rotors from the viscometric cells and ensure that both the cells and rotors are clean. See 9.6 for the cleaning procedure.

9.2.2 Place a 10 ± 1.0 mL oil sample in each cell.

9.2.3 Install the rotors in the proper stators and install the upper pivots.

9.2.4 Place the loop of the 700-mm long string over the crossarm at the top of the rotor shaft and wind all but 200 mm of the length of the string around the shaft. Do not overlap strings. Loop the remaining end of the string over the top bearing cover. Orient the rotor such that an end of the crossarm at the top of the rotor shaft is pointing directly forward. If available, secure crossarm with locking pin. If the rotations are manually timed, it is helpful to color one end of the cross arm.

9.2.4.1 The string may be prewound around the shaft before installation of the rotor in 9.2.3.

9.2.5 Place the housing cover over the viscometric cells to minimize the formation of frost on the cold metal parts exposed to air. In some climates, it may be necessary to flush the cover with a dry gas (for example, dry air or nitrogen) to minimize the frost formation.

9.2.6 Start the programmed temperature profile. The program will warm the oil samples to 80 ± 1°C and maintain at 80 ± 1°C for 2 h to allow solution of any material not in true solution at room temperature.

9.2.7 At the end of the 2-h soak at 80°C, the cooling cycle starts to cool the samples in accordance with the programmed cooling sequence as programmed in 9.1.

9.2.8 At the completion of the temperature profile, the temperature of the block should be within 0.2°C of the desired test temperature when measured by a thermometer other than the temperature controller in the same thermometer well used during calibration. If the block temperature is within this range, proceed with the yield stress and viscosity measurements within 30 min of the completion of the temperature profile (see 9.3).

9.2.8.1 If the final temperature of the block is 0.2 to 0.5°C warmer than the desired temperature, proceed as follows. Set the temperature controller to bring the block temperature to the correct test temperature and then hold at the correct test temperature for 30 min before proceeding. This entire temperature correction should not take longer than 1 h. The data obtained in this way are considered valid test results, otherwise the test is invalid.

9.2.8.2 If the final test temperature is more than 0.2°C cooler or more than 0.5°C warmer than the preselected test temperature, then the test is *NOT VALID* for the preselected temperature. *For Information Only*, the yield stress and viscosity may be measured without further temperature adjustment. These results are characteristic of the actual temperature, not the preselected one.

9.2.9 If the final temperature as noted in 9.2.8 is in error in either direction by more than 0.2°C, see X2.2 before starting another test.

9.2.10 With models CMRV-4 and higher, if the program reports cooling profile out of tolerance, the operation of the instrument shall be thoroughly reviewed for correct operation. With models earlier than CMRV-4, check the logged data for excessive temperature deviation. See X2.2-X2.4.

9.3 *Measurement of the Yield Stress* :

9.3.1 Beginning with the cell farthest to the left of the instrument, follow the procedure below for each cell in turn.

9.3.2 Align the pulley wheel with the rotor shaft for the cell to be tested, such that the string hangs past the front of the housing. Make sure that the weights clear the edge of the bench during testing.

9.3.3 Remove the string from the upper bearing support and carefully place it over the pulley wheel so as not to disturb the test oil. (Do not allow the rotor shaft to turn.)

9.3.4 Follow the instrument model specific instructions:

NOTE 2—For users with CMRV-4 or newer instruments wishing to manually time yield stress and viscosity, follow the instructions in 9.3.4.1 and 9.4.1.1, respectively.

Model CMRV-3 or Earlier

9.3.4.1 *Visually observe* the rotor for movement of the crossarm. (Do not measure yield stress by way of the electronic optics.)

9.3.4.2 For instruments not equipped with locking pins: carefully, so as not to disturb the gel structure, attach a 10-g mass to the string and gently suspend the weight on the string. Proceed to 9.3.4.4.

9.3.4.3 For instruments equipped with locking pins: suspend the 10-g mass on the string, then raise the locking pin.

9.3.4.4 If the end of the crossarm does not move at least 3 mm in 15 s (approximately twice the diameter of the crossarm or 13° of rotation) then record that the sample has yield stress. Proceed to 9.3.4.5. If movement is detected, record weight and proceed to 9.4.

9.3.4.5 If no movement is detected, for instruments without locking pins, hold weight assembly and add 10 g, then proceed with 9.3.4.4. If equipped with locking pins, lower the locking pin to re-engage crossarm. Add 10 g to the weight assembly, raise the locking pin, and proceed with 9.3.4.4.

Model CMRV-4 or Later Model MRV

9.3.4.6 The operator shall follow the on-screen instructions for the addition of weight increments.

9.3.4.7 For instruments with locking pins: suspend 10 g weight cage on string, press the flashing start button, then immediately raise the locking pin and follow the on-screen instructions.

9.3.4.8 If additional weight is requested, capture crossarm in locking pin, then add one additional 10 g weight, and follow the on-screen instructions. Press the flashing start button, then immediately raise the locking pin. Repeat procedure until no additional weight is requested. Proceed to 9.3.4.

9.3.4.9 For instruments without locking pins: carefully suspend and hold the 10 g weight cage on the string without jerking rotor, and follow the on-screen instructions. Press the flashing start button, and immediately release the weight cage.

9.3.4.10 If no movement is detected, then carefully weight the cage. Add next 10 g weight increment as indicated on the computer screen, without pulling on the string, and follow the on-screen instructions. Press the flashing start button, and immediately release weight cage. Repeat procedure until no additional weight is requested. Proceed with 9.3.4.

NOTE 3—When the 10-g load is first applied, some oils may show momentary movement of the crossarm. If there is no further movement of the crossarm for 15 s, disregard the initial movement.

9.4 *Measurement of Apparent Viscosity* :

9.4.1 Follow the instrument model specific instructions:

For CMRV-3 or Earlier

9.4.1.1 Attach a 150-g mass to the string and slowly suspend the weight on the string. Start the timer when the crossarm of the rotor shaft points directly forward and continue timing in accordance with the following constraints.

9.4.1.2 If the first half-revolution requires less than 10 s, measure and record the time for the *first three* revolutions.

9.4.1.3 If the first half-revolution requires 10 s or greater, measure and record the time for the first revolution and identify it as the time for one revolution.

9.4.1.4 If the first revolution has not been completed in 60 s, end the measurement. Record the time as greater than 60 s for one revolution. Report that the viscosity is greater than the value calculated in 10.2.

9.4.1.5 If the time for the *first three* revolutions is less than 4 s, record the time as less than 4 s. Report that the viscosity is less than the value calculated in 10.2.

For CMRV-4 and Later

9.4.1.6 Follow the on-screen instructions, press the start button, and slowly suspend the weight on the string. Timing will automatically begin with the first movement. Do not remove weight while viscosity LED on instrument is flashing. The time and viscosity will be displayed. Proceed to 9.5.

9.5 Repeat 9.3-9.4 for each of the remaining cells in order from left to right.

9.6 *Cleaning*:

9.6.1 After all of the cells have been completed, exit the cooling program and turn on the heater to warm the viscometric cells to room temperature or somewhat higher. The temperature shall not exceed 50°C.

9.6.2 Remove the upper rotor pivots and the rotors.

9.6.3 With a vacuum, remove the oil samples and rinse the cells with an oil solvent several times, followed by two washings with acetone. Use a vacuum to remove the solvent from the cells after each rinse and allow the acetone to evaporate to dryness after the final rinse.

9.6.4 Clean the rotors in a similar manner.

10. Calculation of Yield Stress and Apparent Viscosity

10.1 Yield stress is given by the following equation:

$$Ys = 3.5\,M \tag{9}$$

where:
Ys = yield stress, Pa, and
M = applied mass, g.

10.2 The viscosity is given by the following equation when using the cell constant obtained in Eq 8:

$$\eta_a = C\,t\,3/r \tag{10}$$

where:
η_a = apparent viscosity in mPa·s, (cP),
C = cell constant obtained in Eq 8,
t = time for number (*r*) of complete revolutions of the rotor, and
r = number of revolutions timed.

11. Report

11.1 *Apparent Viscosity and Yield Stress*—For unused oils, report the final test temperature and *either* the apparent viscosity *or* the existence of yield stress, but *not* both. For used oils, report both apparent viscosity and yield stress.

11.2 *Yield Stress*—Report as less than the value at which rotation was observed.

11.3 *Apparent Viscosity*—Report as follows:

11.3.1 If the apparent viscosity is less than 5000 mPa·s (cP), then report the apparent viscosity as less than 5000 mPa·s (cP).

11.3.2 If the apparent viscosity is between 5000 and 100 000 mPa·s (cP), then report the apparent viscosity to the nearest 100 mPa·s (cP).

11.3.3 If the apparent viscosity is between 100 000 and 400 000 mPa·s (cP), then report the apparent viscosity to the nearest 1000 mPa·s (cP).

11.3.4 If the apparent viscosity is greater than 400 000 mPa·s (cP), then the apparent viscosity should be reported as greater than 400 000 mPa·s (cP).

11.3.5 When employing software that provides three viscosity values, the first value shall be reported as the apparent viscosity by Test Method D 4684. If desired, report all three values, taking care to also report the sequence of the values. *Never* report a value that is the average of the three measured values.

12. Precision and Bias [5]

12.1 *Precision (Unused Oils)*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

12.1.1 *Yield Stress*—In the case of pass-fail data, no generally accepted method for determining precision is currently available.

12.1.2 *Apparent Viscosity*:

12.1.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, Percent of Mean
−15	4.2
−20	7.3
−25	11.7
−30	9.3
−35	13.2
−40	19.8

12.1.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in 1 case in 20. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Reproducibility, Percent of Mean
−15	8.4
−20	12.1
−25	17.5
−30	18.4
−35	35.8
−40	34.1

12.1.3 The interlaboratory program included nine test oils at the −15°C test temperature with eleven laboratories participating.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR: D02-1212, D02-1249, D02-1277, and D02-1517.

Nine test oils were included at the −20°C test temperature with eleven laboratories participating. The −25°C test temperature included 18 test oils with 14 laboratories participating. Nine test oils were evaluated at −30°C in 13 laboratories. At the −35 and −40°C test temperatures, six test oils were evaluated in twelve laboratories.

12.2 *Precision (Used Diesel Engine Oils)*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

12.2.1 *Yield Stress*:

12.2.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

Test Temperature, °C	Repeatability, Pa
-20	$1.735 \cdot (X + 1)$
-25	$1.014 \cdot (X + 1)$

where:
X = mean value in Pa.

NOTE 4—When no yield stress is detected (movement with 10 g weight), $X = 0$.

12.2.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in 1 case in 20.

Test Temperature, °C	Repeatability, Pa
-20	$2.993 \cdot (X + 1)$
-25	$2.976 \cdot (X + 1)$

where:
X = mean value in Pa.

12.2.2 *Apparent Viscosity*:

12.2.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, Percent of Mean
-20	14.3
-25	10.3

12.2.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in 1 case in 20. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Reproducibility, Percent of Mean
-20	21.1
-25	20.8

12.2.3 The interlaboratory program included nine laboratories and nine test oils at the -20 and -25°C test temperatures. The used oils included end-of-test drain samples from Mack T8, Mack T8E, Cummins M11-EGR and Mack T10 engine tests, with soot loadings (as measured by thermogravimetric analysis) ranging from approximately 5 to 9 % (see RR: D02–1517).[5]

12.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

13. Keywords

13.1 low temperature flow properties; low temperature viscosity; mini-rotary viscometer; pumping viscosity; used diesel engine oil; viscosity; yield stress

APPENDIXES

(Nonmandatory Information)

X1. TEMPERATURE PROFILES FOR TEST TEMPERATURES

X1.1 See Tables X1.1-X1.3.

TABLE X1.1 Temperature Profile for Test Temperatures −20 to −40°C

Segment Time h:min	Segment Temperature[A]				Allowable Temperature Change[B] °C
	Beginning° C		Final °C	Rate of Change °C/h	
nominally 0:20	above 20	to	80		
2:00	80	to	80		±1.0
nominally 0:20	80	to	0		
nominally 0:03	0	to	−3.0		
nominally 0:07	−3.0	to	−4.0	8.5	±0.5
nominally 0:10	−4.0	to	−5.0	6.0	±0.2
6:00	−5.0	to	−8.0	0.5	±0.2
36:00	−8.0	to	−20.0	0.33	±0.2
Hold at this point for − 20°C test temperature.[C]					
2:00	−20.0	to	−25.0	2.5	±0.2
Hold at this point for − 25°C test temperature.[C]					
2:00	−25.0	to	−30.0	2.5	±0.2
Hold at this point for − 30°C test temperature.[C]					
2:00	−30.0	to	−35.0	2.5	±0.2
Hold at this point for − 35°C test temperature.[C]					
2:00	−35.0	to	−40.0	2.5	±0.2
Hold at this point for −40°C test temperature.[C]					

[A] If the dual control loop concept is used, the bath set point temperatures should be 5°C below the corresponding block temperature desired. The maximum bath temperature shall not exceed −5°C.

[B] Holding the temperature variation to less than ±0.1°C improves the precision and reproducibility of your viscosity measurements.

[C] The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

TABLE X1.2 Temperature Profile for Test Temperatures −10 and −15°C

Segment Time h:min	Segment Temperature[A] Beginning °C		Final °C	Rate of Change °C/h	Allowable Temperature Change[B] °C
nominally 0:20	above 20	to	80		
2:00	80	to	80		±1.0
nominally 0:20	80	to	10		
nominally 0:03	10	to	7.0		
nominally 0:07	7.0	to	6.0	8.5	±0.5
nominally 0:10	6.0	to	5.0	6.0	±0.2
6:00	5.0	to	2.0	0.5	±0.2
36:00	2.0	to	−10.0	0.33	±0.2
Hold at this point for − 10°C test temperature.[C]					
2:00	−10.0		−15.0	2.5	±0.2
Hold at this point for − 15°C test temperature.[C]					

[A] If the dual control loop concept is used, the bath set point temperatures should be 5°C below the corresponding block temperature desired. The maximum bath temperature shall not exceed −5°C.

[B] Holding the temperature variation to less than ±0.1°C improves the precision and reproducibility of your viscosity measurements.

[C] The measurement of yield stress and apparent viscosity are to be made within 30 min of reaching the test temperature.

TABLE X1.3 Nominal Elapsed Time to Test Temperature

Test Temperature, °C	Nominal Elapsed Time, h
−10	45
−15	47
−20	45
−25	47
−30	49
−35	51
−40	53

X2. SUPPORTING OPERATIONAL INFORMATION

X2.1 *Temperature Controller* is the most critical part of this procedure. For systems using a liquid media to control cell temperature, the temperature control system can be a single-loop programmable controller to control the block temperature. A process controller that has a proportional band with integral reset and derivative rate control, sometimes referred to as a PID controller, is suitable for controlling the temperature. This programmable controller has one control loop and one temperature sensor that provides the appropriate information to the controller to hold the temperature at the programmed set temperature. It has an internal clock that controls the execution of the program. The controller shall be connected so that only heat is supplied to the block during the first 2 h and 20 min of the temperature profile described in Table X1.1 or Table X1.2 or the heat shall be applied uniformly across all viscometer cells. For systems using a liquid media to control cell temperature, the temperature control during the remaining portion of the temperature profile shall be obtained by controlling the coolant flow. This control system shall have a minimum temperature sensitivity of 0.1°C and be able to change the temperature at a prescribed rate. When the control system's proportional band, integral (reset), and derivative (rate) parameters are optimized, the temperature excursions above and below the profile shall be no greater than 0.2°C at a temperature below -5°C. The temperature sensor can be a platinum resistance thermal detector, a thermistor, or a thermocouple. A platinum resistance thermal detector or thermistor sensor is preferred. A ⅛-in. (3.2-mm) diameter temperature probe can be installed directly into the ⅛-in. diameter well located at the back of the block between cells Nos. 4 and 6. Alternatively, the temperature sensor can be inserted into one of the thermometer wells.

NOTE X2.1—The sensor is placed in the same unit that is being controlled. The sensor should be placed in the block if the supply of coolant is being controlled. Alternatively, the sensor would be placed in the bath if the bath temperature was being controlled. Do not try to control the block temperature by sensing the block temperature and controlling the refrigeration system.

NOTE X2.2—An internally delayed start for the controller is a desirable feature since this will allow starting the temperature profile unattended.

X2.2 If the final temperature is in error in either direction by more than 0.2°C, do the following before starting another analysis:

X2.2.1 Check the thermometer calibration. For liquid in glass thermometers, check the ice point. An error in the ice point usually indicates air in the thermometer bulb or in the column of liquid.

X2.2.2 Check temperature sensor of the temperature controller for accuracy, in accordance with 8.1.

X2.2.3 Is the coolant flowing? Is there adequate coolant in the reservoir?

X2.2.4 For cold sources operating below -20°C, replace methanol if wet, as indicated by ice crystals in the top of the cold source

reservoir. Cold methanol absorbs water, and as it absorbs water, its cooling capacity decreases. In high humidity areas, it may be necessary to change the methanol once a month. Other heat transfer can be used, but it should be similar to methanol in viscosity and heat capacity at the bath temperature.

X2.2.5 Is the bath refrigeration working properly?

X2.2.6 If manually programmed or using a custom profile, examine the temperature profile program for an error and make the appropriate corrections.

X2.3 The simplest way to check a liquid in glass thermometer calibration is to check its ice point. Other calibration sources are available for both liquid in glass and electronic temperature sensor and are appropriate if they are sufficiently accurate.

X2.4 The software for controlling temperature creates a temperature log during the test. Inspect the recorded cooling profile temperature data for temperature deviations greater than those permitted in Table X1.1 or Table X1.2. Verify that the cooling rates during the test are in conformance with those in Table X1.1 for temperatures below -4°C or in Table X1.2 for temperatures below 6°C. Verify that the preheat at 80°C lasted for a minimum of 2 h.

REFERENCES

(**1**) Stambaugh, R. L., and O'Mara, J. H., "Low Temperature Flow Properties of Engine Oils," SAE Paper No. 821247 or 820509.
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(**3**) Stewart, R. M., Shaub, H., Smith, M. F., Jr., and Selby, T. W., "Summary of ASTM Activities on Low Temperature Engine Oil Pumpability," SAE Paper No. 821206.
(**4**) Smith, M. F., Jr., "Better Prediction of Engine Oil Pumpability Through a More Effective MRV Cooling Cycle," SAE Paper No. 831714.
(**5**) Henderson, K. O., Manning, R. E., May, C. J., and Rhodes, R. B., "New Mini-Rotary Viscometer Temperature Profiles That Predict Engine Oil Pumpability," SAE Paper No. 850443.
(**6**) ASTM Research Report RR: D02-1442, "Cold Starting and Pumpability Studies in Modern Engines," ASTM International, W. Conshohocken, PA, 1999 (order #COLDSTART).
(**7**) Shaub, Harold, Editor, "Oil Flow Studies at Low Temperature in Modern Engines," ASTM STP 1388, ASTM International, W. Conshohocken, PA, 2000.

SUMMARY OF CHANGES

Committee D02 has identified the principal changes to this standard since its last issue (D 4684-02) that may impact its use.

(*1*) Paragraph 1.2—Added reference to used diesel engine oils in the Scope.
(*2*) Paragraphs 2.2.3 and 2.2.4—Added terminology for used and unused oils.
(*3*) Paragraph 4.2.1—Added reference to recent ASTM pumpability work.
(*4*) Paragraph 5.2—Moved the detailed description on temperature control to Appendix X2.
(*5*) Paragraph 8.1—Simplified instructions on temperature sensor calibration.
(*6*) Paragraph 8.2.9—Added a cautionary statement on individual cell calibration constants.
(*7*) Paragraph 8.3—Changed to make consistent with modifications to Paragraphs 8.1 and 5.2. (Also deleted Paragraph 8.4 for this reason.)
(*8*) Paragraph 9.2.4—Modified to reference instruments with locking pins.
(*9*) Paragraph 9.2.5—Clarified wording on flush gas.
(*10*) Paragraph 9.2.6—Modified wording to make it consistent with instrument operation.
(*11*) Paragraph 9.2.9—Modified wording to make it consistent with changes to Paragraph 5.2.
(*12*) Paragraph 9.2.10—Changed to make consistent with modifications to Paragraph 5.2; clarified to encompass different instrument models.
(*13*) Paragraph 9.3.2—Added "rotor" to "shaft" for clarity.
(*14*) Paragraphs 9.3.4, 9.4.1, and subsections—Modified to address differences between instrument models, including those with or without locking pins.
(*15*) Paragraph 9.6.1—Modified wording to more accurately reflect instrument operation.
(*16*) Section 10—Added yield stress calculation.
(*17*) Section 11 and subsections—Changed to cover different reporting criteria for used and unused oils.
(*18*) Section 12 and subsections—Changed to differentiate between unused and used engine oils precision statements (used engine oil precision statements taken) Changed "Radius of String" from RR: D02-1517).
(*19*) Section 13—Added "used diesel engine oil" 0.05 mm to Keywords.
(*20*) References—Added two references covering recent relevant publications on engine pumpability, cold starting, and MRV measurement of used diesel engine oils. 0.1 mm in 5.1.1.