



Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Used Engine Oils at Low Temperature¹

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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 of 43 or 45 h to a final test temperature of -20 or -25°C . The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s^{-1} . This test method is suitable for measurement of viscosities ranging from 4000 mPa·s to $>400\,000\text{ mPa}\cdot\text{s}$, and is suitable for yield stress measurements of 7 Pa to $>350\text{ Pa}$.

1.2 This test method is applicable for used diesel oils. The applicability and precision to other used or unused engine oils or 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. Referenced Documents

2.1 ASTM Standards:

- D 3829 Test Method for Predicting the Borderline Pumping Temperature of Engine Oil²
- D 4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature²
- D 5133 Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique²

3. Terminology

3.1 Definitions:

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

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

3.1.3 *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.

3.1.4 *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 the apparatus described in 6.1), and ignoring any end effects, the shear rate is given as follows:

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

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

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 for one revolution of the rotor, s.

For the specific apparatus described in 6.1,

$$G_r = 63/t \quad (3)$$

3.1.5 *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.

$$T_r = 9.81 M (R_o + R_r) \times 10^{-6} \quad (4)$$

$$S_r = \frac{TT_r}{2(\pi)R_r^2 h} \times 10^9 \quad (5)$$

where:

- T_r = torque applied to rotor, N·m,
- M = applied mass, g,
- R_o = radius of the shaft, mm,
- R_r = 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 6.1.1,

$$T_r = 31.7 M \times 10^{-6} \quad (6)$$

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 *Annual Book of ASTM Standards*, Vol 05.02.

$$S_r = 3.5 M \quad (7)$$

3.1.6 *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.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *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.

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

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

3.2.4 *yield stress*—the shear stress required to initiate flow.

3.2.4.1 *Discussion*—For all Newtonian fluids and some non-Newtonian fluids, the yield stress is zero. An 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 for engine oils.

4. Summary of Test Method

4.1 A used engine oil sample is heated at 80°C and then vigorously agitated. The sample is 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.

5. Significance and Use

5.1 When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test, used 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. As in other low temperature rheological tests such as Test Methods D 3829, D 4684, and D 5133, a preheating condition is required to ensure that all residual waxes are solubilized in the oil prior to the cooldown (that is, remove thermal memory). However, it is also known that highly sooted used diesel engine oils can experience a soot agglomeration phenomenon when heated under quiescent conditions. The current method uses a separate preheat and agitation step to break up any soot agglomeration that may have occurred prior to cooldown. The viscosity of highly sooted diesel engine oils as measured in this test method have been correlated to pressurization times in a motored engine test (1).³

5.2 *Cooling Profiles:*

5.2.1 For oils to be tested at -20°C and -25°C, 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 (2-7).

6. Apparatus

6.1 *Mini-Rotary Viscometer*⁴, 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.

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

	millimetres
Diameter of rotor	17.0
Length of rotor	20.0
Inside diameter of cell	19.0
Radius of shaft	3.18
Radius of string	0.10

6.2 *Weights:*

6.2.1 *Yield Stress*, weight set consists of ten 10 g units with a tolerance of 1 % for each unit.

6.2.2 *Viscosity*, 150 g weight with a 1 % tolerance.

6.3 *Temperature Control System*, that will regulate the mini-rotary viscometer block temperature in accordance with the temperature limits described in Table X1.1.

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

6.3.2 *Temperature Profile*—The temperature profile is fully described in Table X1.1.

6.4 *Thermometers*, for measuring the temperature of the block. Two ranges are required, one graduated from at least +70 to 90°C in 1°C subdivisions, the other with a range 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.

6.5 *Refrigeration Device*, consisting of a means of removing heat from the instrument such that the cell temperature is controlled in accordance with the program described in Table X1.1.

6.6 *Circulating System*, that will circulate the liquid coolant to the instrument as needed. Methanol is a suitable coolant if the circulating coolant is below -10°C. 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.)

6.7 *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.

6.8 *Sample Pre-treatment Oven*, an oven capable of maintaining a temperature of 80 ± 1°C for a minimum of 2 h.

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.

⁴ 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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee ¹, which you may attend.

7. Reagents and Materials

7.1 *Newtonian Oil*, a low cloud-point of approximately 30 Pa·s (30 000 cP) viscosity at -20°C for calibration of the viscometric cells.

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

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

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

8. Sampling

8.1 A representative sample of test oil free from suspended granular 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.

9. Calibration and Standardization

9.1 Calibrate the temperature sensor in place while attached to the temperature controller. The sensed temperature shall be verified using a reference thermometer specified in 6.4 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. For instruments using an independent temperature controller, see X2.1 for calibration guidance.

NOTE 1—All temperatures in this test method refer to the actual temperature as measured in the left thermowell and not necessarily the indicated temperature.

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

9.2.1 Use steps 10.2.3-10.6.

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

9.2.3 Allow the oil in the cells to soak at $-20 \pm 0.2^\circ\text{C}$ for at least 1 h, making small temperature control adjustments, if necessary, to maintain the test temperature.

9.2.4 At the end of the soak period, record the temperature reading of the measuring device in the left thermowell (test temperature) and remove the cover of the viscometer cell.

9.2.5 Perform step 10.4.1.

9.2.6 Repeat 9.2.5 for each of the remaining cells, taking the cells in order from left to right.

9.2.7 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 for three complete revolutions, s.

9.2.8 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 instrument.

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

9.4 *Oven*—Check the calibration of the temperature sensing device by appropriate methods. The temperature should be constant at $80 \pm 1^\circ\text{C}$.

10. Procedure

10.1 Select the cooling profile for the desired test temperature. Table X1.2 lists the nominal times to reach a particular test temperature.

10.1.1 Choose the preprogrammed temperature profile. If the profile is not available, enter it using the custom profile part of the software program. The instrument manual provides instructions on adding custom profiles. The entries for a custom program will be found in Table X1.3.

10.1.2 If the instrument temperature is controlled by an external controller, it will need to be programmed to follow the cooling program in Table X1.1 with adjustment for the temperature difference found in 9.1, if any.

10.2 *Test Sample and Viscometric Cell Preparation:*

10.2.1 Using suitable closed container, preheat the samples in an oven to $80 \pm 1^\circ\text{C}$ for 2.25 h. At the end of this time, remove the samples from the oven and allow to cool for 15 min at room temperature.

10.2.2 Agitate each sample using vigorous mechanical or manual shaking for 60 s. Allow the samples to stand for a minimum of 10 min to allow for settling.

10.2.3 Remove the nine rotors from the viscometric cells and ensure that both the cells and rotors are clean. See 10.6 for the cleaning procedure.

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

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

10.2.6 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 crossarm.

10.2.6.1 The string may be prewound around the shaft before installation of the rotor in 10.2.5.

10.2.7 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.

10.2.8 Start the programmed temperature profile.

10.2.9 The cooling cycle starts to cool the samples in accordance with the programmed cooling sequence as programmed in 10.1.

10.2.10 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 10.3).

10.2.10.1 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.

10.2.10.2 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.

10.2.11 If the final temperature as noted in 10.2.10 is in error in either direction by more than 0.2°C, see X2.2 before starting another test.

10.2.12 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.

10.3 *Measurement of the Yield Stress:*

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

10.3.2 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.

10.3.3 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.)

10.3.4 For CMRV-3 and earlier models, follow the instructions in 10.3.5. For CMRV-4 or later models, if using the automatic timing devices, follow the instructions in 10.3.6. If manual timing measurements are used, follow the instructions in 10.3.5.

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

10.3.5.1 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 10.3.5.3.

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

10.3.5.3 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 10.3.5.4. If movement is detected, record weight and proceed to 10.4.

10.3.5.4 If no movement is detected, for instruments without locking pins, hold weight assembly and add 10 g, then proceed with 10.3.5.3. 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 10.3.5.3.

NOTE 2—The total amount of weight available for measurement of yield stress is normally 100 g; if no movement is detected with this weight, yield stress would be recorded as >350 Pa.

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

10.3.6.1 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 on-screen instructions.

10.3.6.2 If additional weight is requested, capture crossarm in locking pin, add one additional 10 g weight, and follow the on-screen instructions. Press the flashing start button, and immediately raise the locking pin. Repeat procedure until no additional weight is requested. Proceed to 10.4.

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

10.3.6.4 If no movement is detected, carefully weight the cage. Add next 10 g weight increment as indicated on computer screen without pulling on string and follow on-screen instructions. Press the flashing start button and immediately release weight cage. Repeat procedure until no additional weight is requested. Proceed with 10.4.

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.

10.4 *Measurement of Apparent Viscosity:*

10.4.1 For CMRV-3 and earlier models follow the instructions in 10.4.2. For CMRV-4 or later models, if using the automatic timing devices, follow the instructions in 10.4.3. If manual timing measurements are used, follow the instructions in 10.4.2.

10.4.2 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.

10.4.2.1 If the first half-revolution requires less than 10 s, measure and record the time for the first three revolutions, and proceed to 10.5.

10.4.2.2 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; then proceed to 10.5.

10.4.2.3 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, then proceed to 10.5, reporting that the viscosity is greater than the value calculated in 11.2.

10.4.2.4 If the time for the first three revolutions is less than 4 s, record the time as less than 4 s, then proceed to 10.5, reporting that the viscosity is less than the value calculated in 11.2.

10.4.3 Follow on-screen instructions, press start button and slowly suspend the weight on the string. Timing will automatically begin with first movement. Do not remove weight while viscosity LED on instrument is flashing. Once the time and viscosity are displayed or the viscosity LED stops flashing, proceed to 10.5.

10.5 Repeat 10.3 and 10.4 for each of the remaining cells in order from left to right.

10.6 *Cleaning:*

10.6.1 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.

10.6.2 Remove the upper rotor pivots and the rotors.

10.6.3 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.

10.6.4 Clean the rotors in a similar manner.

11. Calculation of Yield Stress and Apparent Viscosity

11.1 Yield stress is given by the following equation:

$$Y_s = 3.5 M \quad (9)$$

where:

Y_s = yield stress, Pa, and

M = applied mass, g.

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

$$\eta_a = C t 3/r \quad (10)$$

where:

η_a = apparent viscosity, 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.

12. Report

12.1 *Apparent Viscosity and Yield Stress*—For used oils, report the final test temperature and both apparent viscosity and yield stress.

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

12.3 *Apparent Viscosity*—Report as follows:

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

12.3.2 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).

12.3.3 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).

12.3.4 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).

12.3.5 When employing software that provides three viscosity values, the first value shall be reported as the apparent

viscosity by this test method, D 6896. If desired, report all three values, exercising care to also report the sequence of the values. Never report a value that is the average of the three measured values.

13. Precision and Bias ⁵

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

13.1.1 *Yield Stress:*

13.1.1.1 *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 one case in twenty:

Test Temperature, °C	Repeatability, Pa
-20	0.543·(X+1)
-25	0.504·(X+1)

where:

X = mean value, Pa.

13.1.1.2 *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 one case in twenty.

Test Temperature, °C	Repeatability, Pa
-20	0.926·(X+1)
-25	0.773·(X+1)

where:

X = mean value, Pa.

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

13.1.2 *Apparent Viscosity:*

13.1.2.1 *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 one case in twenty. The repeatability as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, % of Mean
-20	0.0879·X
-25	0.0616·X

13.1.2.2 *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 one case in twenty. The reproducibility as a percent of the mean apparent viscosity is shown as follows:

Test Temperature, °C	Repeatability, % of Mean
-20	0.186·X
-25	0.209·X

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1442.

13.1.3 The interlaboratory program included six laboratories and seven test oils at the -20°C 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 %.⁶

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1517.

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

14. Keywords

14.1 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 -25°C

Segment Time, h:min	Segment Temperature ^A			Allowable Temperature Change, ^B °C
	Beginning, °C	Final, °C	Rate of Change, °C/h	
nominally 0:02	above 20	to 25.0		
nominally 0:21	25.0	to 1.5		
nominally 0:17	1.5	to 0.0		
nominally 0:03	0.0	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				

^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 Nominal Elapsed Time to Test Temperature

Test Temperature, °C	Nominal Elapsed Time, h
-20	43
-25	45

TABLE X1.3 Custom Profile Values

Elapsed Time, hh:mm	Test Temperature, of -20.0°C	Test Temperature, of -25.0°C	Temperature Tolerance, °C
00:02	25.0	25.0	...
00:23	15.0	15.0	...
00:40	0.0	0.0	...
00:43	-3.0	-3.0	...
00:50	-4.0	-4.0	0.5
01:00	-5.0	-5.0	0.2
07:00	-8.0	-8.0	0.2
43:00	-20.0	-20.0	0.2
45:00		-25.0	0.2

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 1/8-in. (3.2-mm) diameter temperature probe can be installed directly into the 1/8-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.

X2.1.1 When using a temperature controller which is separate from the instrument, the operator will need to make an adjustment in its program to accommodate any differences found in 9.1 between the temperature as measured in the left thermowell and the temperature observed at the temperature control point. Typically, there would be a nearly constant offset that may increase as the temperature decreases.

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 Determine if the coolant is flowing and if there is 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 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. Verify that the cooling rates during the test are in conformance with those in Table X1.1 for temperatures below -4°C.

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