Designation: D 6821 – 02

An American National Standard

Standard Test Method for Low Temperature Viscosity of Drive Line Lubricants in a Constant Shear Stress Viscometer[1]

This standard is issued under the fixed designation D 6821; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the viscosity of drive line lubricants (gear oils, automatic transmission fluids, and so forth) with a constant shear stress viscometer at temperatures from –40 to 10°C after a prescribed preheat and controlled cooling to the final test temperature.

1.2 The applicability of this particular test method to petroleum products other than drive line lubricants has not been determined.

1.3 This test method uses the millipascal second (mPa·s) as the unit of viscosity. One millipascal second is equal to one centipoise.

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 2983 Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer[2]

D 3829 Test Method for Predicting the Borderline Pumping Temperature of Engine Oil[3]

D 4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity*—the determined viscosity obtained by the 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.

3.1.4.1 *Discussion*—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:

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

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

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 for one revolution of the rotor, s.

For the specific apparatus being described in 6.1.1,

$$G_r = \frac{33}{t} \tag{3}$$

3.1.5 *shear stress*—the motivating force per unit area for fluid flow.

3.1.5.1 *Discussion*—For the rotary viscometer being described in 6.1, the rotor surface is the area under shear or the shear area. For this test method, end effects are not considered.

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

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

where:

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

For the dimensions given in 6.1.1,

$$T_r = 32\,M \times 10^{-6} \tag{6}$$

$$S_r = 4.5\,M \tag{7}$$

3.1.6 *viscosity*—the ratio between the applied shear stress

[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.

Current edition approved August 10, 2002. Published October 2002.

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

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

and rate of shear, sometimes called the coefficient of dynamic viscosity.

3.1.6.1 *Discussion*—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]. The submultiple unit is millipascal seconds (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 (angular velocity), from which the apparent viscosities of test oils are determined.

3.2.1.1 *Discussion*—Calibration oils, which are essentially Newtonian fluids, are available commercially and are described in 7.1.

3.2.2 *test oil*—any oil for which the apparent viscosity is to be determined by this test method.

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

3.2.3.1 *Discussion*—For Newtonian fluids and some non-Newtonian fluids the yield stress is very small.

4. Summary of Test Method

4.1 A drive line fluid is preheated to 50°C for a specified time and then cooled at a programmed rate (see Table X1.1) to the final test temperature and soaked at the final temperature for a defined period of time. At the completion of the soak time, the viscosity is measured by applying a prescribed torque and measuring rotational speed to determine the apparent viscosity of the sample.

5. Significance and Use

5.1 Viscosity of drive line lubricants at low temperature is critical for both gear lubrication and the circulation of the fluid in automatic transmissions. For gear oils (GOs), the issue is whether the fluid characteristics are such that the oil will flow into the channel dug out by the submerged gears as they begin rotating and re-lubricating them as they continue to rotate. For automatic transmission fluids, torque, and tractor fluids the issue is whether the fluid will flow into a pump and through the distribution system rapidly enough for the device to function.

5.2 The low temperature performance of drive line lubricant flow characteristics was originally evaluated by the channel test. In this test, a pan was filled to a specified depth of approximately 2.5 cm and then cooled to test temperature. The test was performed by scraping a channel through the full depth of the fluid and across the length of the pan after it had soaked at test temperature for a specified time. The time it took the fluid to cover the channel was measured and reported. The channel test was replaced by Test Method D 2983 in 1971.

5.3 The results of this test procedure correlate with the viscometric measurements obtained in Test Method D 2983.[4] The correlation obtained is:

$$V = 0.941 \times V_{D\,2983} \quad (8)$$

where:

V = the apparent viscosity measured by this test method, and

$V_{D\,2983}$ = the apparent viscosity measured by Test Method D 2983.

5.3.1 The equation was obtained by forcing the fit through zero. The coefficient of variation (R^2) for this correlation is 0.9948.

6. Apparatus

6.1 *Mini-Rotary Viscometer*, an apparatus that consists of one or more viscometric cells in a temperature controlled aluminum block. Each cell, when fitted with the specified rotor, becomes a calibrated rotor-stator set. Rotation of the rotor is achieved by an applied load acting through a string wound around the rotor shaft. The top bearing plate is fitted with locking pins for holding the rotors stationary. Time of rotation is measured electronically by a device attached to the timing wheel.

NOTE 1—The rotors for use with this test method can be distinguished from the rotors used for other mini-rotary viscometer test methods by the white rotor and the white band on the upper half of the shaft. The white band provides rotor identification while the rotor is in the cell.

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

Diameter of rotor	15.0 mm
Length of rotor	20.0 mm
Inside diameter of cell	19.0 mm
Radius of shaft	3.18 mm
Radius of string	0.10 mm

6.2 *Weight*, for applying mass. Weights are to be in increments of 2.5 g ± 1 %. A minimum of eight weight segments will be needed for the measurements defined in this test method.

6.3 *Temperature Control System*, that will regulate the samples in the cells according to the cooling program described in Table X1.1 and within the tolerances specified in the table.

6.4 *Thermometers*, for measuring the temperature of the block. Two are required, one graduated from at least +40 to 90°C in 1°C subdivisions, and one that has at least a two degree graduated range around the final test temperature in 0.2°C subdivisions. Other thermometric devices of equal or greater accuracy and resolution may be used.

6.5 *Time/Temperature Recording Device*, such as a chart recorder or data logger to verify that the correct cooling of the samples has been accomplished.

7. Reagents and Materials

7.1 *Calibration Oil*, a low cloud-point fluid of approximately 60 000 mPa·s viscosity at −25°C for calibration of the viscometric cells.

7.2 *Oil Solvent*, commercial heptanes or similar solvent for the test fluids that evaporates without leaving a residue. (**Warning**—Flammable.)

7.3 *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 solid material and water is necessary to obtain valid viscosity

[4] SAE Paper 1999–01–3672, "Viscosity of Drive-Line Lubricants by a Special Mini-Rotary Viscometer Technique." Available from Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096-0001.

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

9.1 *Temperature Sensor Calibration*—Calibrate the instrument's temperature sensor by verifying the displayed temperature with the block temperature measured independently in the left thermometer well. The sensed temperature should be verified using a −36 to +5°C reference thermometer with a range of approximately −36 to +5°C in 0.2°C increments, or an independent electronic temperature measurement instrument of equivalent precision at a minimum of three temperatures. Make these temperature measurements at least 5°C apart and equally distributed over the test temperature range to establish a calibration curve for this combination of temperature sensor and controller.

9.2 *Viscometer Cell Calibration*—The calibration cell constant of each viscometric cell (viscometer constants) is to be determined with the viscosity calibration oil.

9.2.1 Use steps 10.2-10.2.5 to place the calibration oil in the cell.

9.2.2 Program the temperature controller to cool the mini-rotary viscometer block to the desired calibration temperature −25°C within 1 h or less, then start the program.

9.2.3 Allow the samples to soak at the test temperature ±0.2°C for at least 1 h. Verify the test temperature by placing a calibrated temperature sensing probe in the left thermometer well.

9.2.4 At the end of the soak period, record the temperature reading (test temperature) and remove cover of the viscometer cell. The temperature made by the independent temperature sensor in the left thermometer well reading should agree with the temperature displayed on the computer screen within 0.1°C.

9.2.5 Measure the time for the first three rotor rotations as described in 10.4.

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:

$$C = \frac{\eta_o}{t} \tag{9}$$

where:

η_o = viscosity of the calibration oil, mPa·s (cP) at −25°C,
C = cell constant for a 20 g mass, Pa, and
t = time for three complete rotor revolutions, s.

10. Procedure

10.1 Program the temperature controller to control the mini-rotary viscometer block temperature in accordance with Table X1.1. The programmed temperature is the temperature in Table X1.1 plus the appropriate temperature correction factor determined in 9.1.

10.2 *Test Sample and Viscometric Cell Preparation*:

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

10.2.2 Place a 10 ± 1 mL oil sample in each cell.

10.2.3 Install the white rotors in their proper stator and install the upper pivots.

NOTE 2—The rotors used with this test method are physically different than those used with other mini-rotary viscometer (MRV) test methods. The rotors are white. To further distinguish them they also have a white band on upper part of the shaft to identify the rotor type while in the instrument. DO NOT use the black rotors for this procedure. The black rotors are used with Test Methods D 3829 and D 4684.

10.2.4 Place the loop of the 700 mm long string over the cross arm at the top of the rotor shaft and wind all but 200 mm of the length around the shaft. Do not overlap the strings. Orient the rotor such that a (red or black) marked end of the cross arm at the top of the shaft is pointing directly forward and lock the rotor with the locking pin. Loop the remaining end of the string over the top bearing cover.

10.2.4.1 The string may be prewound around the shaft before installation of the rotor in step 10.2.3.

10.2.5 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 is desirable to flush the cover with dry air to minimize frost formation.

10.2.6 Start the programmed temperature profile.

10.2.7 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 or electronic temperature measuring device 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. If the temperature measurements do not agree, check accuracy of external temperature sensor. Measuring the ice point is one way to check. If correct, then recalibrate the temperature control system in accordance with 9.1.

10.3 *Measurement of the Yield Stress (Optional)*:

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 shaft of 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.

10.3.4 Carefully attach a 2.5 g mass to the string.

10.3.5 Raise the locking pin. If the cross arm moves more than 3 mm in 15 s, let it rotate until it clears the cross arm and then lower the locking pin. (Three millimetres is approximately twice the diameter of the cross arm.) Let the rotor continue to rotate until the cross arm rests against the locking pin. Remove the weight from the string and proceed to 10.4.

10.3.6 If there is no movement of the cross arm, lower the locking pin to secure the cross arm.

10.3.7 Increase the weight on the string by one mass increment (2.5 g) and repeat 10.3.5.

10.4 *Measurement of Apparent Viscosity*:

10.4.1 Attach a 20 g mass to the string and suspend the weight on the string. Start the timer, then release the locking

pin. Continue the timing until one of the following constraints is met.

10.4.1.1 If the first half-revolution requires less than 60 s, measure and record the time for the first three revolutions.

10.4.1.2 If the first half-revolution is longer than 60 s, measure and record the time for the first revolution and identify it as the time for one revolution.

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

10.6 *Cleaning*:

10.6.1 After all the cells have been completed, turn off the cooling program. Start the warm up program to raise the viscometer cells to not more than 50°C.

10.6.2 Once the viscometer has warmed up, remove the upper rotor pivots and rotors.

10.6.3 With vacuum, remove the oil samples. Rinse the cells with an oil solvent several times using the vacuum to remove each rinse from the cells. Additional rinses with acetone can be done to remove any traces of solvent or water, again using the vacuum to remove the rinse from the cells. Allow the solvent to evaporate before beginning a new test.

10.6.4 Clean the rotors in a similar manner.

11. Calculations

11.1 *Yield Stress (Optional)*:

11.1.1 Calculate the yield stress from the following equation:

$$S_{ri} = 4.5\,M \tag{10}$$

where:

S_{ri} = yield stress, Pa, and
M = mass applied to initiate rotation, g.

11.1.2 Report the yield stress as being less than the result rounded to the nearest Pa.

11.2 *Apparent Viscosity*:

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

$$\eta_a = C\,t\,3/r \tag{11}$$

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.

12. Report

12.1 Report the final test temperature, apparent viscosity, and yield stress, if measured.

13. Precision and Bias

13.1 *Precision*:

13.1.1 *Yield Stress*—A determination of precision for the measurement of yield stress has not been made at this time.

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 material would in the long run, in the normal and correct operation of the test method exceed 10.3 % of the mean only in one case in twenty.

13.1.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test materials would, in the long run, exceed 17.2 % of the mean only in one case in twenty.

13.2 The interlaboratory program included nine test oils at the −26°C test temperature and nine oils at the −40°C test temperature. The slate of oils contained commercial gear oils, hydraulic oils, automatic transmission fluids, and nominally Newtonian viscosity reference oils. The oils were tested in six laboratories at −26°C and five laboratories at −40°C. The separate analysis of each temperature yielded essentially the same precision as the combined analysis.

13.3 *Bias*—There is no accepted reference material suitable for determining the bias of this test method; no statement on bias is being made.

14. Keywords

14.1 apparent viscosity; low-temperature flow properties; low-temperature viscosity; mini-rotary viscometer; viscosity; yield stress

APPENDIX

(Nonmandatory Information)

X1. MINI-ROTARY VISCOMETER PROFILE PROGRAM

X1.1 See Table X1.1 for viscometer profile program for specific test temperatures.

TABLE X1.1 Mini-Rotary Viscometer Profile Program for Specific Test Temperatures

Final Temperature, °C	-12	-26	-35	-40	Temperature
Elapsed Time, min	Cell Temperature, °C				Tolerance
Start	Ambient				
30	50.0	50.0	50.0	50.0	. . .
90	50.0	50.0	50.0	50.0	1
96	26.4	21.0	17.6	15.7	. . .
102	11.7	3.1	−2.5	−5.5	. . .
108	2.7	−8.0	−14.9	−18.7	. . .
114	−2.9	−14.9	−22.5	-26.8	. . .
122	−6.4	−19.1	−27.3	−31.8	0.5
130	−8.5	−21.7	−30.2	−34.9	0.5
138	−9.8	−23.4	−32.0	−36.9	0.5
146	−10.7	−24.4	−33.2	−38.1	0.5
154	−11.2	−25.0	−33.9	−38.8	0.3
162	−11.5	−25.4	−34.3	−39.3	0.2
210	−12.0	−26.0	−35.0	−40.0	0.2
240	−12.0	−26.0	−35.0	−40.0	0.2
270	−12.0	−26.0	−35.0	−40.0	0.2
300	−12.0	−26.0	−35.0	−40.0	0.2
330	−12.0	−26.0	−35.0	−40.0	0.2
1050	−12.0	−26.0	−35.0	−40.0	0.2