Designation: D6821 – 17

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 D6821; 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 °C to 10 °C after a prescribed preheat and controlled cooling to the final test temperature. The precision is stated for test temperatures from –40 °C to –26 °C.

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

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 This standard uses the SI based unit of milliPascal second (mPa·s) for viscosity which is equivalent to centiPoise (cP).

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.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2983 Test Method for Low-Temperature Viscosity of Automatic Transmission Fluids, Hydraulic Fluids, and Lubricants using a Rotational Viscometer

D3829 Test Method for Predicting the Borderline Pumping Temperature of Engine Oil

D4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature

D6896 Test Method for Determination of Yield Stress and Apparent Viscosity of Used Engine Oils at Low Temperature

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E644 Test Methods for Testing Industrial Resistance Thermometers

E1137 Specification for Industrial Platinum Resistance Thermometers

E2877 Guide for Digital Contact Thermometers

2.2 *ISO Standards:*[3]

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

ISO Guide 34 General Requirements for the Competence of Reference Material Producers

3. Terminology

3.1 *Definitions:*

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

3.1.2 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.1.2.1 *Discussion*—This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device. These devices are sometimes referred to as "digital thermometers."

3.1.2.2 *Discussion*—PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

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

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

3.1.5 *shear rate, n*—the velocity gradient in fluid flow.

3.1.5.1 *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:

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

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

where:

$\dot{\gamma}$ = 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,

$$\dot{\gamma} = \frac{33}{t} \tag{3}$$

3.1.6 *shear stress, n*—the motivating force per unit area for fluid flow.

3.1.6.1 *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.

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

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

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,
τ = shear stress at the rotor surface, Pa, and
h = height of the rotor face, mm.

For the dimensions given in 6.1.1,

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

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

3.1.7 *viscosity, n*—the ratio between the applied shear stress and rate of shear, sometimes called the coefficient of dynamic viscosity.

3.1.7.1 *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, n*—those oils that establish an instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined.

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

3.2.3 *yield stress, n*—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 D2983 in 1971.

5.3 The results of this test procedure correlate with the viscometric measurements obtained in Test Method D2983.[4] The correlation obtained is:

$$V = 0.941 \times V_{D2983} \tag{8}$$

where:

V = the apparent viscosity measured by this test method, and
V_{D2983} = the apparent viscosity measured by Test Method D2983.

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.

[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.

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.

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

Diameter of rotor	15.00 mm ± 0.08 mm
Length of rotor	20.00 mm ± 0.14 mm
Inside diameter of cell	19.07 mm ± 0.08 mm
Radius of shaft	3.18 mm ± 0.13 mm
Radius of string	0.1 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. One segment will be the weight holder.

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 *Temperature Measuring Device*—Use either a DCT meeting the requirements described in 6.4.1 or liquid-in-glass thermometers described in 6.4.2. A calibrated DCT or calibrated low temperature liquid-in-glass thermometer shall be used as the thermometer for temperature measurement below 25 °C independent of the instrument's temperature control, and shall be located in the thermowell.

NOTE 1—The DCT display device and sensor must be correctly paired. Incorrect pairing will result in temperature measurement errors and possibly irreversible damage to the electronics of the display.

6.4.1 *Digital Contact Thermometer Requirements:*

Criteria	Minimum Requirements
DCT	Guide E2877, Class B
Temperature range	–45 °C to 100 °C
Display resolution	0.1 °C minimum, preferably 0.01 °C
Sensor type	RTD, such as a PRT or thermistor
Sensor, metal sheathed	3 mm O.D. with an sensing element less than 30 mm in length to be used with a thermowell sleeve, 6 mm O.D. × 58 mm long with a ~3 mm hole in center.
Sensor, glass sheathed	6 mm O.D. with a sensing element less than 12 mm in length
Display accuracy	±50 mK (0.05 °C) for combined probe and sensor
Response time	less than or equal to 8 s as defined in Specification E1137
Drift	less than 50 mK (0.05 °C) per year
Calibration Error	less than 50 mK (0.05 °C) over the range of intended use.
Calibration Range	–40 °C to 85 °C
Calibration Data	4 data points evenly distributed over the range of –40 °C to –1 °C and included in calibration report.
Calibration Report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body

NOTE 2—With respect to DCT probe immersion depth, a procedure to determine minimum depth can be found in Guide E2877, Section 5.3, or Test Methods E644, Section 7.

6.4.1.1 The DCT calibration driftshall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with 6.4.1. With respect to an ice bath, Practice E563 provides guidance on the preparation and use of an ice bath. However, for this use, variance from the specific steps, such as water source, is permitted provided preparation is consistent. The basis for the variance is due to the ice bath reference being used for tracking change in calibration not verification.

NOTE 3—When a DCT's calibration drifts in one direction over several calibration checks, that is, ice point, it may be an indication of deterioration of the DCT.

6.4.2 For liquid-in-glass thermometers, LiG, two are required. One LiG shall be a calibrated 76 mm partial immersion thermometer with a scale from +5 °C to 1 degree lower than the lowest test temperature in 0.2 °C subdivisions. For test temperatures less than –35 °C, use a liquid-in-glass thermometer with at least a scale range of 2 degrees Celsius in 0.2 °C subdivisions. The low temperature LiG thermometer(s) shall have a report of calibration showing the temperature deviation at each calibrated test temperature. The second LiG thermometer shall be a 76 mm partial immersion thermometer graduated from at least +40 °C to 90 °C in 1 °C subdivisions, which is used to verify the preheat temperature.

6.4.2.1 *Calibration Check*—Verify the low temperature thermometer at least annually against a reference thermometer in a constant temperature bath or in an ice bath. The thermometer is to be insert to its immersion depth. If using an ice bath, the ice point reading is to be taken within 60 min after the thermometer has been at test temperature for at least 3 min. If the corrected temperature reading deviates from the reference thermometer or the ice point then repeat this calibration check. If the thermometer deviates from the reference value on two successive checks then a full thermometer recalibration is needed.

6.4.2.2 *Recalibration*—A complete recalibration of the liquid-in-glass thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to liquid-in-glass thermometer's design until the thermometers corrected measured temperature deviates from the reference thermometer or ice point by one scale division, or until five years has elapsed since the last full calibration.

6.5 *Supply of Dry Gas*—A supply of dry filtered gas to minimize moisture condensation on the upper portions of the instrument.

6.5.1 For thermoelectric cooled instruments, which use cell caps, the dry gas supply is connected to the housing cover. The supply of dry gas is discontinued when the cover is removed for the measurement phase of the test.

6.6 *Locking Pin*—A device to keep the rotor from turning prematurely and able to stop the rotor at the nearest half revolution by interaction with the rotor crossbar.

7. Reagents and Materials

7.1 *Low Cloud-point, Newtonian Oil,* a calibration oil of approximately 60 Pa·s viscosity at –25 °C for calibration of the viscometric cells. The calibration oil shall be obtained from suppliers complying with ISO Guide 34 and ISO 17025 with traceability to a national metrology institute (NMI).

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 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 Control Calibration Procedure*—Calibrate the MRV temperature control by comparing the instrument's displayed temperature to a thermometer in the thermowell. The thermometer used shall meet the requirements in 6.4.

9.1.1 Place 10 mL of a typical test fluid and rotors in each cell then place cover on instrument. Cell caps maybe used if available.

9.1.2 Place the thermometer in the thermowell. This thermowell is to be used for all temperature measurements below 25 °C.

NOTE 4—Prior to inserting the thermometer or DCT probe in the thermowell, place several drops (~3) of a heat transfer fluid such as 50/50 water/ethylene glycol mix, CCS reference oil CL100, or a dewaxed low viscosity mineral oil in the thermowell.

9.1.3 Make these temperature measurements at least 5 °C apart and include both –5 °C and the lowest test temperature used. Establish a calibration curve using at least three comparisons of the thermometer and the instrument's temperature control. Make at least two temperature measurements at every calibration temperature with at least 10 min between observations.

9.1.4 Follow the instrument manufacturers instructions for correcting the instrument's measured temperature. Alternatively, establish a correction equation between thermometer and the instrument's measured temperature then adjust each temperature of the cooling program by the offset determined with the correction equation.

NOTE 5—All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

9.2 *Viscometer Cell Calibration*—The calibration cell constant for each viscometric cell (viscometer constants) is to be determined at –25 °C with the viscosity calibration oil.

9.2.1 Following the steps in 10.1, prepare the cells for calibration using the calibration oil as the sample.

9.2.2 Use either the calibration temperature profile for the instrument or, alternatively, the cooling profile given in Test Method D3829 for a –25 °C test temperature and follow the owner's manual instructions for the instrument to initiate the cooling profile program.

9.2.3 Allow the samples to soak at –25 °C ±0.2 °C for at least 1 h.

9.2.4 Place the thermometer in the thermometer well at least 30 min prior to executing 9.2.6. See Note 4. This thermowell location is to be used for calibration and temperature monitoring during the test procedure.

9.2.5 At the completion of the temperature profile and soak period, check that the test temperature is within ±0.1 °C of the desired calibration temperature with a thermometer.

9.2.6 Perform step10.7.

9.2.7 Repeat 9.2.6 for each of the remaining cells in numerical order.

9.2.8 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 at –25 °C,
C = cell constant for a 20 g mass, mPa, and
t = time for three complete rotor revolutions, s.

10. Procedure

10.1 *Viscometric Cell Preparation:*

10.1.1 If the cells are not clean, clean according to 10.8 cleaning procedure.

10.1.2 Place a 10 mL ± 1 mL oil sample into clean cell.

10.1.2.1 All cells should contain a fluid and rotor; if there are less than a full set of samples to run, fill each of the unused cells with 10 mL of a typical test sample.

10.1.3 Repeat 10.1.2 until all test samples are in their cells.

10.1.4 Place each rotor in its cell then place upper pivot pin in position, including those for any unused cells.

NOTE 6—**Caution:** The rotors used with this test method are physically different than those used with other mini-rotary viscometer (MRV) test methods. The rotors have a white band on upper part of the shaft to identify the rotor type while in the instrument. DO NOT use the larger diameter rotors as they are used with Test Methods D3829, D4684, and D6896. For laboratories using both rotors, ensure that the rotors for this method have a visible identifying mark placed on the shaft to minimize the wrong rotors being used in a test. This could be at the top of the rotor above the crossarm or between the cell caps and below where the string wraps around the shaft.

NOTE 7—Before inserting the rotors in the cells, inspect each rotor to be sure that the shaft is straight, that the rotor surface is smooth and free from dents, scratches, and other imperfections. For rotors with a bearing point at the bottom of the shaft, ensure that the point is sharp and centered on the rotor shaft. If these conditions are not met, repair or replace the rotor.

10.1.5 *Optional*—install a cell cap on all cells, including any unused cells.

10.1.6 For each cell, except any unused ones, place a loop of the nominal 700 mm long string over the crossbar. Hang the string over the timing wheel with a small weight attached such as a large paper clip. Wind the string around the shaft until the end is about 100 mm below the wheel. Do not overlap windings.

NOTE 8—The string may be pre-wound around the shaft before installation of the rotor in step 10.1.4.

10.1.6.1 Engage the locking pin to prevent the rotor from turning.

10.1.6.2 Lay the remaining string over the top of the bearing plate letting it hang over the back of the plate.

10.1.6.3 Repeat 10.1.6 until all cells with samples to be measured are prepared.

NOTE 9—All cells should contain a fluid and rotor; if there are less than a full set of samples to run, fill each of the unused cells with a typical test sample.

10.1.7 Place the housing cover over the viscometric cells.

10.1.8 Connect the dry gas supply to the housing cover, as noted in 6.5. Set the dry gas flow to approximately 1 L/h. Increase or decrease the flow as necessary to minimize frost or moisture condensation around the cells.

10.2 Select the cooling profile for the desired test temperature and follow the instrument instructions to initiate the program.

10.2.1 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 X1.1.

10.3 Place the thermometer in thermowell at least 30 min prior to completion of the cooling profile (see Note 4). The thermowell used must be the same one used during calibration.

10.4 At the completion of the cooling profile, check the time-temperature plot for the run to ensure that the time-temperature profile is within tolerance and that the test temperature as measured in the thermowell is within ±0.2 °C of the final test temperature. Both of these checks may be done automatically by the control software incorporated in some instruments. Final test temperature is to be verified independently from the instrument's temperature control using a thermometer that has been in the thermowell for at least 30 min. See Note 4. If the final test temperature deviates by more than 0.1 °C from the set point on two consecutive runs, the instrument's temperature control must be recalibrated according to 9.1.

10.5 If the temperature profile is within tolerance, proceed with measurements. If not, then abort the test and recalibrate the instrument's temperature control as in 9.1.

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

10.6.1 Immediately prior to starting measurements, take the cell housing cover off the instrument.

10.6.2 *Yield Stress Determination*—Starting with the cell farthest to the left while facing the instrument, use the following procedure for each cell in turn, bypassing the unused cells.

10.6.3 Align the pulley wheel with the shaft of the cell to be tested.

10.6.4 Hang the string over the timing wheel such that the string hangs past the front of the housing. Make sure that the weights clear the edge of the bench during testing.

10.6.5 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.)

10.6.6 Carefully suspend the 2.5 g weight holder from the string.

10.6.7 For instruments with automatic timing, start timing and then release the locking pin. For manual timing, start timing immediately after the locking pin is disengaged.

10.6.8 Observe whether the end of the crossbar moves more than 3 mm in 15 s. (This 3 mm is approximately twice the diameter of the crossbar.) An alternative procedure is the use of a marked rotation of the timing wheel equivalent to a crossbar movement of 3 mm.

10.6.9 Electronic or timing wheel motion-sensing devices, which are available on some instruments, are suitable alternatives to direct observation.

10.6.10 If rotor movement of more than 3 mm in 15 s is observed in 10.6.8, record the total mass, remove weights from the end of the string, and proceed to 10.7.

10.6.11 If a rotor movement of less than 3 mm in 15 s is observed in 10.6.8, stop timing and lift the weight holder so it is not supported by the string, then add an additional 2.5 g weight segment to weight holder.

NOTE 10—As additional weight segments are added to the weight holder, it is necessary to suspend the holder with the additional weights from the string and restart timing without the use of the locking pin for the remainder of the yield stress assessment. When using software available for some instruments, ensure that the mass applied is the mass requested by the program.

10.6.12 Carefully and gently, suspend the weight holder with the additional weights from the string and start timing.

10.6.13 Repeat steps 10.6.8 through 10.6.12 until the accumulated weights causes rotation of the rotor. At this point, remove all the weights from the string.

10.6.14 If no rotation is observed with a total of 20 g, record that the yield stress is >90 Pa, and proceed with 10.7.

10.7 *Measurement of Apparent Viscosity:*

10.7.1 Gently suspend the 20 g mass from the string. (Weight holder plus 7 weight segments.)

10.7.2 If the applied mass of 20 g moves the rotor, then as soon as the cross-arm clears the locking pin, reengage the locking pin. Allow rotation to continue until the cross-arm contacts the locking pin causing rotation to stop. If no appreciable rotation occurs, terminate the test and proceed to 10.7.7.

10.7.3 When using instruments capable of timing rotation automatically, initiate viscosity measurement by starting timing, then release the locking pin. When timing manually, start timing immediately after the locking pin is disengaged.

10.7.4 Stop the timer after three revolutions of the rotor from point of release. When the time for one revolution is greater than 60 s, time only one revolution.

NOTE 11—The timing of three revolutions may be done automatically

10.7.5 After completing three revolutions (one revolution if the time is greater than 60 s), remove mass from string.

10.7.6 Record both the time and the number of revolutions timed.

10.7.7 If no rotation occurs with the application of a 20 g mass, record the result for that sample as being "Too Viscous To Measure" (TVTM).

10.7.8 Repeat 10.6 (optional) and 10.7 for each of the remaining cells in order from left to right.

10.8 *Cleaning:*

10.8.1 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.

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

10.8.3 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.

10.8.4 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,
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[5]

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

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1595.

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

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D6821 – 14) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised 3.1.2.