Designation: D5293 – 17ε1

Standard Test Method for Apparent Viscosity of Engine Oils and Base Stocks Between –10 °C and –35 °C Using Cold-Cranking Simulator[1]

This standard is issued under the fixed designation D5293; 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.

ε[1] NOTE—Bold emphasis was added editorially to values in Table 1 in August 2017.

1. Scope*

1.1 This test method covers the laboratory determination of apparent viscosity of engine oils and base stocks by cold cranking simulator (CCS) at temperatures between –10 °C and –35 °C at shear stresses of approximately 50 000 Pa to 100 000 Pa and shear rates of approximately 10^5 to 10^4 s^{-1} for viscosities of approximately 900 mPa·s to 25 000 mPa·s. The range of an instrument is dependent on the instrument model and software version installed. Apparent Cranking Viscosity results by this method are related to engine-cranking characteristics of engine oils.

1.2 A special procedure is provided for measurement of highly viscoelastic oils in manual instruments. See Appendix X2.

1.3 Procedures are provided for both manual and automated determination of the apparent viscosity of engine oils using the cold-cranking simulator.

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

1.5 *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.* Specific warning statements are given in Section 8.

1.6 *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]

D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

D2602 Test Method for Apparent Viscosity of Engine Oils At Low Temperature Using the Cold-Cranking Simulator (Withdrawn 1993)[3]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

2.2 *ISO Standard:*

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *Newtonian oil or fluid, n*—one that exhibits a constant viscosity at all shear rates.

3.1.2 *non-Newtonian oil or fluid, n*—one that exhibits a viscosity that varies with changing shear stress or shear rate.

3.1.3 *viscosity, η, n*—the property of a fluid that determines its internal resistance to flow under stress, expressed by:

$$\eta = \frac{\tau}{\gamma} \quad (1)$$

where:

τ = the stress per unit area, and
γ = the rate of shear.

3.1.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. In the SI, the unit of viscosity is the pascal-second; for practical use, a submultiple

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] 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

TABLE 1 Calibration Oil Sets by Test Temperature

Test Temp	–35 °C	–30 °C	–25 °C	–20 °C	–15 °C	–10 °C
CL080	A	...	...	...	...	...
CL090	**A**	...	...	...	...	...
CL100	A	A	...	...	...	...
CL110	B	**A**	...	...	...	...
CL120	B	A	A	...	...	...
CL130	B	B	**A**	...	...	...
CL140	B	B	A	A	...	...
CL150	B	B	B	**A**	...	...
CL160	B	B	B	A	...	...
CL170	B	B	B	B	A	...
CL190	B	B	B	B	**A**	...
CL200	B	B	B	B	A	A
CL220	C	B	B	B	B	**A**
CL240	C	B	B	B	B	A
CL250	C	B	B	B	B	B
CL260	...	B	B	B	B	B
CL280	...	C	B	B	B	B
CL300	...	C	B	B	B	B
CL320	...	C	C	B	B	B
CL340	...	...	C	B	B	B
CL380	...	...	C	B	B	B
CL420	...	...	...	C	B	B
CL480	...	...	...	C	B	B
CL530	...	...	...	C	C	B
CL600	...	...	...	...	C	C
CL680	...	...	...	...	C	C

Group A	Include at least one **Preferred** (bold) or one Alternate. Nominal Values –35 °C to –25 °C; 800 mPa·s to 1500 mPa·s –20 °C to –10 °C; 800 mPa·s to 1400 mPa·s
Group B	Include at least 3. The selection is to be uniformly distributed over the range. Nominal Values –35 °C to –20 °C; 1000 mPa·s to 15 000 mPa·s –15 °C; 1000 mPa·s to 13 000 mPa·s –10 °C; 1000 mPa·s to 9000 mPa·s
Group C	Include at least one. Nominal Values –35 °C to –20 °C; > 13500 mPa·s –15 °C; >11 500 mPa·s –10 °C; > 9000 mPa·s

(millipascal-second) is more convenient and is customarily used. The millipascal second is 1 cP (centipoise).

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.1.1 *Discussion*—Since many engine oils are non-Newtonian at low temperature, apparent viscosity can vary with shear rate.

3.2.2 *calibration oils, n*—oils with known viscosity and viscosity/temperature functionality that are used to define the calibration relationship between viscosity and cold-cranking simulator rotor speed.

3.2.3 *check oil, n*—a batch of test oil used to monitor measurement performance.

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

3.2.5 *viscoelastic oil, n*—a non-Newtonian oil or fluid that climbs up the rotor shaft during rotation.

4. Summary of Test Method

4.1 An electric motor drives a rotor that is closely fitted inside a stator. The space between the rotor and stator is filled with oil. Test temperature is measured near the stator inner wall and maintained by removing heat with a controlled process to maintain a constant stator temperature during test. The speed of the rotor is calibrated as a function of viscosity. Test oil viscosity is determined from this calibration and the measured rotor speed.

5. Significance and Use

5.1 The CCS apparent viscosity of automotive engine oils correlates with low temperature engine cranking. CCS apparent viscosity is not suitable for predicting low temperature flow

to the engine oil pump and oil distribution system. Engine cranking data were measured by the Coordinating Research Council (CRC) L-49[5] test with reference oils that had viscosities between 600 mPa·s and 8400 mPa·s (cP) at –17.8 °C and between 2000 mPa·s and 20 000 mPa·s (cP) at –28.9 °C. The detailed relationship between this engine cranking data and CCS apparent viscosities is in Appendixes X1 and X2 of the 1967 T edition of Test Method D2602[6] and CRC Report 409.[5] Because the CRC L-49 test is much less precise and standardized than the CCS procedures, CCS apparent viscosity need not accurately predict the engine cranking behavior of an oil in a specific engine. However, the correlation of CCS apparent viscosity with average CRC L-49 engine cranking results is satisfactory.

5.2 The correlation between CCS and apparent viscosity and engine cranking was confirmed at temperatures between –1 °C and –40 °C by work on 17 commercial engine oils (SAE grades 5W, 10W, 15W, and 20W). Both synthetic and mineral oil based products were evaluated. See ASTM STP 621.[7]

5.3 A correlation was established in a low temperature engine performance study between light duty engine startability and CCS measured apparent viscosity. This study used ten 1990s engines at temperatures ranging from –5 °C down to –40 °C with six commercial engine oils (SAE 0W, 5W, 10W, 15W, 20W, and 25W).[8]

5.4 The measurement of the cranking viscosity of base stocks is typically done to determine their suitability for use in engine oil formulations. A significant number of the calibration oils for this method are base stocks that could be used in engine oil formulations.

6. Apparatus

6.1 Two types of apparatus are described for use in this test method: the manual cold-cranking simulator (see Appendix X1) and the automated CCS (see 6.2 and 6.3).

6.2 *Automated CCS,*[9]consisting of a direct current (dc) electric motor that drives a rotor inside a stator; a rotor speed sensor or tachometer that measures rotor speed; a dc ammeter and fine current-control adjust dial; a stator temperature control system that maintains temperature within 0.05 °C of set point; and a heat removal system with a temperature control system, a computer, computer interface, and test sample injection pump.

6.3 *Automatic Automated CCS,*[9]as described in 6.2 with the addition of an automated sample table allowing multiple test samples to be run sequentially under computer control without operator attention.

6.4 *Calibrated Thermistor,*[9]sensor for insertion in a well near the inside surface of the stator to indicate the test temperature.

6.4.1 There must be good thermal contact between the temperature sensor and the thermal well in the stator; clean this thermal well periodically and replace the small drop of high-silver-containing heat transfer medium.

6.5 *Heat Removal System:*

6.5.1 For stators with coolant contact, a refrigerator for the liquid coolant is needed to maintain coolant temperature at least 10 °C below the test temperature. When the coolant temperature is below –30 °C a two-stage refrigeration system is likely needed. The length of the tubing connections between the CCS and the refrigerator should be as short as possible (less than 1 m) and well insulated.

6.5.1.1 *Coolant, Dry Methanol*—If contaminated with water from operating under high humidity conditions, replace it with dry methanol to ensure consistent temperature control.

6.5.2 For thermoelectric cooled stators, the liquid cooling temperature of the water or other appropriate liquid used in the refrigeration system (chiller) should be set to approximately 5 °C in order to maintain the sample test temperature. The coolant should contain 10 % glycol to prevent blocking of the flow path by ice formation.

6.6 *Ultrasonic Bath, Unheated—(optional)*—with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤100 W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating frequencies and power outputs outside this range, however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

7. Reagents and Materials

7.1 *Calibration Oils*—Low-cloud point Newtonian oils shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. The calibration oils shall be traceable to master viscometer procedures described in Test Method D2162. Table 1 shows the sets of possible test oils to be used for each test temperature. Approximate viscosities at certain temperatures are listed in Appendix X5, whereas exact viscosities are supplied with each standard.

8. Hazards

8.1 Observe both toxicity and flammability warnings that apply to the use of methanol or glycol.

[5] CRC Report No. 409 "Evaluation of Laboratory Viscometers for Predicting Cranking Characteristics of Engine Oils at -0°F and -20°F," April 1968 available from the Coordinating Research Council, 5755 North Point Pkwy, Suite 265, Alpharetta, GA 30022.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1402. Contact ASTM Customer Service at service@astm.org.

[7] Stewart, R. M., "Engine Pumpability and Crankability Tests on Commercial "W" Grade Engine Oils Compared to Bench Test Results," *ASTM STP 621* ASTM 1967, 1968. *1969 Annual Book of ASTM Standards* , Part 17 (Also published as SAE Paper 780369 in SAE Publication SP-429.).

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1442. Contact ASTM Customer Service at service@astm.org.

[9] The sole source of supply of the apparatus known to the committee at this time is Cannon Instrument Co., State College, PA 16804. Website: www.cannoninstrument.com. 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.

8.2 If methanol is leaking from the apparatus, repair the leak before continuing the test.

9. Sampling

9.1 To obtain valid results, use an appropriate means of bulk sampling (see Practice D4057) to obtain a representative sample of test oil free from suspended solid material and water. When 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 its container. When the sample contains suspended solid material, use centrifuge to remove particles greater than 5 µm in size and decant off the supernate. Filtering is not recommended. DO NOT shake the sample of test oil. This leads to entrainment of air, and a false viscosity reading.

9.2 For some sample types, such as viscous lube oils that are prone to having entrained air or gas bubbles present in the sample, the use of an ultrasonic bath (see 6.6) without the heater turned on (if so equipped), has been found effective in dissipating bubbles typically within 5 min.

10. Calibration

10.1 On installation of a new instrument or when any part of the viscometric cell or drive component (motor, belt, and so forth) is replaced, set the motor current as described below. Recheck the motor current (as described in 10.3) monthly until the change in motor current in consecutive months is less than 0.005 A and every three months thereafter.

NOTE 1—See Appendix X4 for a flowchart for calibration.

10.2 *Temperature Verification*—Using the temperature verification plugs, verify that the instrument is accurately computing the correct temperature. (Only available on newer model instruments.)

10.2.1 Unplug thermistor connector from the back panel and insert blue TVP.

10.2.2 Enter the TVP resistance for the plug inserted in the software screen *Service>CCS Temperature Verification Service*, and record the difference between the two temperature windows.

10.2.3 Repeat with second plug.

10.2.4 The recorded differences should be less that 0.06 °C. If they are greater, contact instrument service.

10.3 *Motor Current*—Use the *Set Motor Current* option in the software with CL250 (3500 mPa·s) calibration oil as the sample. This option will cool then soak the sample at test temperature of –20.0 °C in the same manner as for a test sample. For a recalibration proceed with 10.3.1. If rechecking motor current, proceed with 10.3.2.

10.3.1 To set the rotor speed, 20 s after the drive motor turns on, monitor the speed reading and adjust to 0.240 KRPM ± 0.001 KRPM (displayed as SPEED on the computer monitor) by slowly turning the CURRENT ADJUST DIAL. This should be completed with in 50 s to 75 s after the motor begins to turn. If more time is taken, repeat 10.3.

10.3.2 When rechecking the motor current, note the speed after the motor is on for 55 s to 60 s. If the speed is less than 0.005 KRPM from 0.240 KRPM, note the speed and current before continuing with normal operation. Alternatively, you can readjust speed to 0.240 KRPM and note new current setting. Recalibration is optional *unless* two consecutive adjustments in motor speed have been made in one direction since last calibration. If recalibration is not necessary, proceed with Section 11. Otherwise, proceed with 10.4.

10.3.3 When rechecking the motor current, and the rotor speed is found to differ from 0.240 KRPM by more than 0.005 KRPM, then readjust rotor speed to 0.240 KRPM, and record the current setting. Continue the calibration with 10.4.

10.4 *Calibration Procedure*—At each test temperature, calibrate the instrument with the oils listed for that temperature in Table 1 using the selection criteria below and the measurement procedure described in Section 11.

NOTE 2—Users of CCS 4/5 instruments using DOS based software need to run the set of calibration oils as samples. Users should enter the speed and viscosity data into VISDISK to calculate calibration constants. These new constants would then be entered manually into the calibration data file used by the CCS software. Contact their instrument supplier for assistance.

10.4.1 *Calibration Oil Matrix Requirements*—For each test temperature calibrated, use Table 1 and select a minimum of: one calibration oil from Group A, three from Group B, and one from Group C. The Group B oils are to be selected so that the distribution is uniform across the group. A minimum of ten data sets consisting of temperature, speed, and known viscosity are required for determining the calibration coefficients in 10.5. A calibration oil can be included twice to achieve the required ten data sets, however doing so will reduce the robustness of the calibration. When including a calibration oil a second time, they should be placed so they are not in adjacent measurement positions. For example –35 °C calibration could have CL090, CL120, CL150, CL170, CL190, CL240 followed by another set CL090, CL120, CL150, CL170, CL190, CL240 samples.

10.5 *Calibration Equation*—The computer program regresses the calibration data over the viscosity range at each calibration temperature to fit the following equation:

$$\eta = \frac{B_0}{(r)} + B_1 + B_2 \cdot (r) \qquad (2)$$

where:

η = the apparent viscosity,
B_0, B_1, B_2 = the coefficients of regression, and
r = the rotor speed in KRPM.

10.6 The calibration will meet the following to be valid:

10.6.1 The regression coefficient shown by the software will be 0.99 or greater.

10.6.2 No calibration data that deviates by more than 1.6 % from Certified Reference Viscosity will be included. It is preferable that all deviations be less than 1 %.

10.6.3 For a test temperature, if more than three pairs of data are excluded because of excessive deviation, repeat the calibration. When a full calibration sample set is used on a repeat calibration within the four operating day time span, all data may be included in calculating the coefficients of regression. When choosing to only run the excluded calibration oils, two calibration oils from the retained data set are to be included in this sample set.

10.6.4 At a test temperature, the calibration data should be collected within the shortest period of time which is possible. When the period of time is greater than four operating days between starting and completing the calibration at a given temperature, the operator must rerun one or two of the earliest calibration oils and include the data in the analysis. This is to ensure the instrument is operating in the same domain that it was initially. When it is the practice of the user to routinely add calibration data to the active calibration data set, the four day period does not apply.

10.6.5 A calibration dataset at a test temperature shall contain at least 10 data points distributed over the available viscosity calibration range after discarding any outliers.

11. Procedure for Automated and Automatic Automated CCS Operation

11.1 Place a minimum of 55 mL of the sample to be tested into a 60 mL sample container.

NOTE 3—When using mini-sample adapter, the instructions in Appendix X3 replace those in 11.1 – 11.3.

NOTE 4—When using an automatic sample changer, ensure the sample containers are designed to fit the sample tray and that the injection tube does not reach to the bottom of the container, as this will avoid drawing any sediment into the instrument.

11.2 Enter sample identification and test temperature(s) for the sample.

11.3 For instruments with automatic sample changer, repeat 11.1 and 11.2 until all sample containers are on the tray and entered into the test matrix on the computer.

NOTE 5—It is recommended that a check oil be run with each sample set.

11.4 Start the sample testing following the software instructions. During the sample testing the instrument will cool the sample to near the test temperature and hold it at that temperature for 180 s. After the soak, the rotor will start turning and the rotor speed will be recorded, but only the average speed between 55 s and 60 s will be used to calculate viscosity.

NOTE 6—The new sample will automatically displace the previous test sample in the viscometric cell without the use of solvent. The temperature control and running of the CCS motor will be computer controlled. The rotor speed measurement and viscosity calculation for the test sample are performed and displayed by the computer.

11.4.1 When using a check oil and it does not fall within reproducibility of the expected value, the results are considered suspect. If this occurs on two consecutive measurements, then recheck rotor speed with CL 250 at –20 °C. If rotor speed is not within 0.005 KRPM of 0.240 KRPM, then investigate and resolve the cause of the deviation. Recalibration may be necessary.

12. Report

12.1 Report the calculated viscosity and temperature as displayed on the computer monitor or test report.

13. Precision and Bias

13.1 *Precision*[10,11]—The precision of this test method with CCS-4/5 (contact cooling instruments) using version 4.x or higher software and with CCS-2050/2100 (thermoelectrically cooled instruments) using ViscPro CCS software module for 2100 series, as determined by statistical examination of the interlaboratory test over the temperature range from –20 °C to –35 °C and a viscosity range from 2700 mPa·s to 15 000 mPa·s is shown in the table below for each instrument.

	Repeatability	Reproducibility
Constant Cooling Instruments	3.1%	7.3%
Thermoelectrically Cooled Instruments	1.5%	6.0%

13.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of this test method, exceed the values in 13.1 only in one case in twenty.

13.1.2 *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 values in 13.1 only in one case in twenty.

13.2 *Summary of Interlaboratory Study*[10]—The interlaboratory study consisted of thirteen participating laboratories using eleven thermoelectrically cooled instruments and eight contact cooling instruments evaluating twelve engine oils with viscosities ranging from 2700 mPa·s to 15000 mPa·s at test temperatures from –20 °C to –35 °C. All laboratories used instrument software version 4.x or higher for contact cooling instrument or ViscPro CCS software module to measure the apparent viscosity. While no base stocks were included specifically as test samples, the calibration is based on the use of base stocks as calibration oils.

13.3 *Bias*—There is no bias between the apparent viscosity of samples measured using contact cooling instruments and thermoelectrically cooled instruments.

14. Keywords

14.1 apparent viscosity; cold cranking; cranking; engine oils; petroleum and petroleum products; viscosity

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1459. Contact ASTM Customer Service at service@astm.org.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1653. Contact ASTM Customer Service at service@astm.org.

APPENDIXES

(Nonmandatory Information)

X1. PROCEDURE FOR MANUAL CCS OPERATION

X1.1 Apparatus

X1.1.1 *Manual CCS,*[9]consisting of a direct current (dc) electric motor that drives a rotor inside a stator; a rotor speed sensor or tachometer that measures rotor speed; a dc ammeter and fine current-control adjust dial; a stator temperature control system that maintains temperature within ±0.05 °C of set point; and a coolant circulator compatible with the temperature control system.

X1.1.2 *Calibrated Thermistor*—Sensor for insertion in a well near the inside surface of the stator to indicate the test temperature.

X1.1.3 *Refrigeration System*—A refrigerator for the liquid coolant is needed to maintain coolant temperature at least 10 °C below the test temperature. Mechanical refrigeration is preferred, but dry ice systems have been used satisfactorily. The length of the tubing connections between the CCS and the refrigerator should be as short as possible and well insulated.

X1.1.4 There must be good thermal contact between the temperature sensor and the thermal well in the stator; clean this thermal well periodically and replace the small drop of high-silver-containing heat transfer medium. Adjust the temperature of the coolant to the viscometric cell to be at least 10 °C below the test temperature.

X1.1.5 *Coolant, Dry Methanol*—If contaminated with water from operating under high humidity conditions, replace it with dry methanol to ensure consistent temperature control, especially when cooled by dry ice.

X1.1.6 *Optional Methanol Circulator,*[9]This option (for the Manual CCS only) circulates warm methanol through the stator to facilitate sample changes and aid the evaporation of cleaning solvents.

X1.2 Reagents and Materials

X1.2.1 *Acetone*—(**Warning—**Danger. Extremely flammable. Vapors can cause fire.)

X1.2.2 *Methanol*—(**Warning—**Danger. Flammable. Vapor harmful.)

X1.2.3 *Petroleum Naphtha*—(**Warning—**Combustible vapor harmful.)

X1.2.4 *Calibration Oils*—Low-cloud point Newtonian oils of known viscosity and viscosity/temperature functionality. Approximate viscosities at certain temperatures are listed in Table 1, whereas exact viscosities are supplied with each standard.

X1.3 Hazards

X1.3.1 Observe both toxicity and flammability warnings that apply to the use of methanol, acetone, and petroleum naphtha.

X1.3.2 If methanol is leaking from the apparatus, repair the leak before continuing the test.

X1.4 Calibration of Manual CCS

X1.4.1 On start-up of a new instrument or when any part of the viscometric cell or drive component (motor, belt, tachometer-generator, and so forth) is replaced, determine the required motor drive current. Initially, recheck the drive current (as described in X1.4.2) monthly until the change in drive current in consecutive months is less than 0.020 A and every three months thereafter.

X1.4.2 *Drive Current Determination*—Plug the tachometer into the CAL jack, where fitted with a CAL jack. Run the 3500 mPa·s, –20 °C viscosity standard at –20 °C as described in Section 11. When the drive motor is turned on, establish a speed meter reading of 0.240 ± 0.010 by adjustment of the current adjust dial. Keep this current setting constant for all subsequent calibration and test sample runs at all temperatures. When the current setting must be changed to maintain a dial reading of 0.240 ± 0.010 units with the 3500 mPa·s reference oil at –20 °C, recalibrate the instrument by either procedure described in X1.4.3.

X1.4.3 *Calibration Procedure*—At each test temperature, calibrate with the oils listed for that temperature in Table 1 by using the procedure described in X1.5.

X1.4.3.1 When only a narrow viscosity range of test liquids is to be measured, use a minimum of three calibration oils spanning the narrow viscosity range of the oils to be tested.

X1.4.4 *Preparation of Calibration Curves*—Plot the viscosity of the calibration oils as a function of speed meter readings, and draw a smooth curve. The use of log-log coordinates or special linearized graph paper have been found suitable for this purpose. Take care to get the best fit to the points found; careless use of commercial drawing curves can lead to excessive errors. See Fig. X1.1 for a typical curve. Use the equation in X1.4.4.1 as an alternative method to this graphical method.

X1.4.4.1 *Alternatively Expressing Calibration Results by Equation*—Calibration data over a limited viscosity range are well represented by the following equation:

$$\eta = \frac{B_0}{N} + B_1 + B_2 N \qquad \text{(X1.1)}$$

where:

η = viscosity,
B_0, B_1, B_2 = constants determined with a minimum of three calibration oils, and
N = observed speed indicator reading, in KRPM.

X1.4.4.2 When more than three pairs of data are available, regress these data to the following equation to determine the values of the constants B_0, B_1, and B_2:

$$\eta N = B_0 + (B_1 \cdot N) + (B_2 \cdot N^2) \qquad \text{(X1.2)}$$

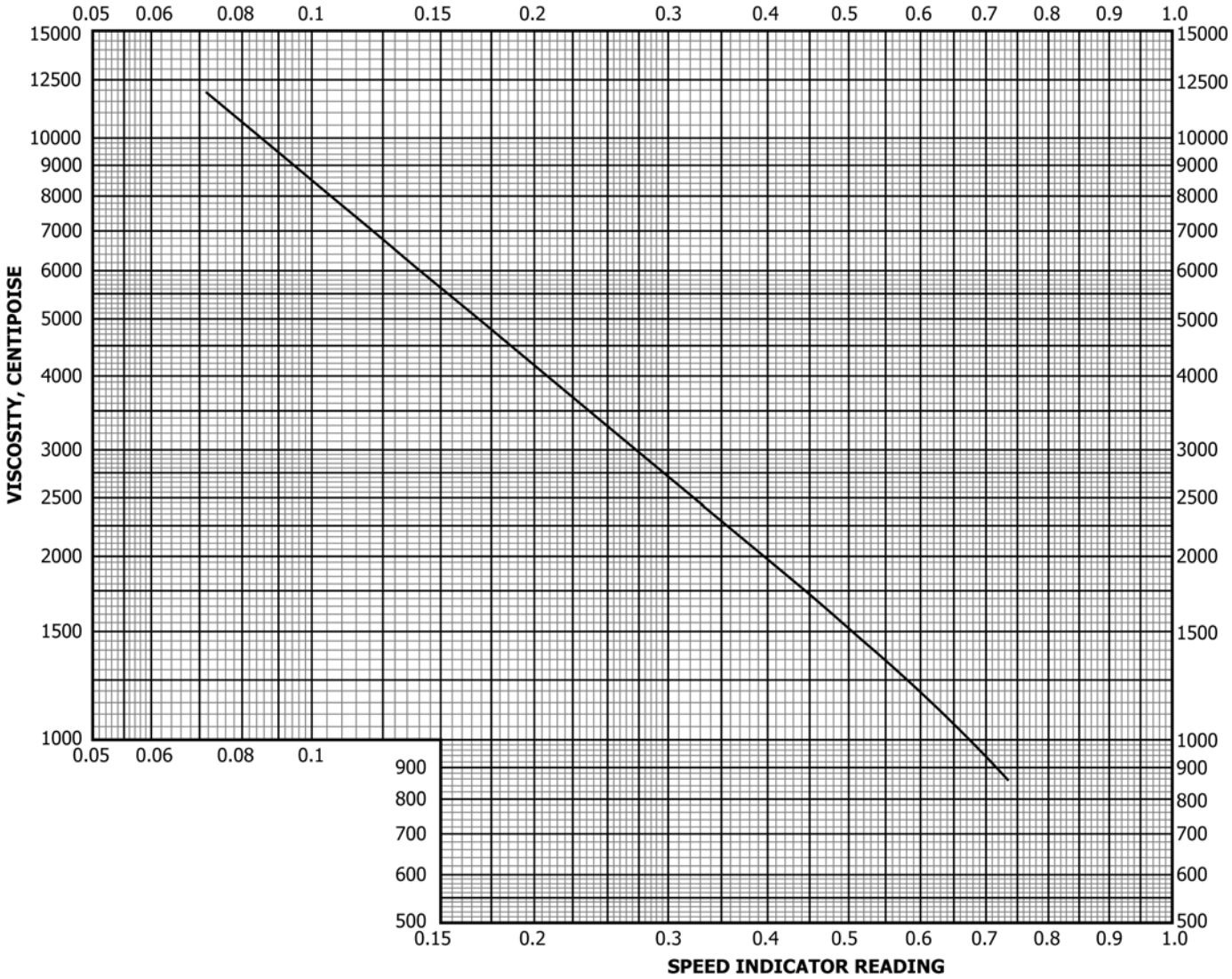

FIG. X1.1 Linearized Calibration Chart, Cold Cranking Simulator

X1.4.5 When check runs of a calibration oil do not fall within ±5 % of the values calculated from the calibration curve, recheck the calibration of the temperature sensor or rerun the calibration oils.

NOTE X1.1—A separate curve or equation is intended for each temperature. However, if the calibration data at two or more temperatures fit a single curve or equation without a bias, a single curve or equation may be used for these temperatures.

X1.5 Procedure for Manual CCS Operation

NOTE X1.2—Ensure that the cooling bath is stirred during the operation of the instrument. Failure to do so will permit large gradients in temperature to exist in the cooling bath. These large gradients will affect the sample temperature and reduce the precision of your viscosity measurements.

X1.5.1 Establish the calibration equation or curve (see Section 10). Before any series of determinations, run a minimum of one calibration oil as an overall check on the apparatus and calibration at each temperature of interest. When the drive current for the oil to be used for the calibration check (see footnote B of Table 1) differs by more than 0.005 A (ampere) from that determined in X1.4.2, reset the current to the value previously determined in X1.4.2; make the observation and correction after 15 s of running. When the viscosity measurement of the calibration oil differs by more than ±5 % from its certified value, rerun to confirm this observation. When confirmed, recalibrate as in X1.4.3.

NOTE X1.3—The use of a check oil or similar reference is recommended for an overall check on all performance, at frequent intervals (at least monthly).

X1.5.2 Insert test sample from a dropping pipet (eye dropper) into the filling tube. Be certain the test sample fills the gap between the rotor and stator with an excess of liquid above the rotor to fill the cup completely. Turn the rotor by hand to ensure complete wetting of the surface of the stator and rotor while the test sample flows between the rotor and stator. Fill the filling tube fully and insert a rubber stopper in the end of the tube; for viscoelastic samples this stopper will have to be pressed tightly while the motor is turned on (see X1.5.2.2) to prevent the sample from forcing the stopper out of the tube and allowing

the sample to become depleted in the shear area of the viscometric cell. See Appendix X2 for a special procedure for highly viscoelastic test samples.

NOTE X1.4—The viscosity of some oils can be high enough at room temperature to impede flow into the annulus between the rotor and stator. For oils whose kinematic viscosity at ambient temperature exceeds 100 mm^2/s (cSt), warm the sample (not exceeding 50 °C) prior to filling the viscometric cell.

X1.5.2.1 Turn the temperature control and coolant flow on, and allow the stator to cool. To ensure optimum control of temperature, see X1.1.3 and X1.1.4. Record the time at which the coolant flow is turned on (use a stopwatch or other means of counting by seconds). Attain control temperature within 30 s to 60 s for test temperatures down to –20 °C and within 60 s to 90 s for test temperatures down to –30 °C; if not within these limits, replace the cold methanol (see X1.1.5) or adjust the temperature of the cold methanol. A null reading on the temperature indicator meter and the cyclic controlling of coolant flow indicate that test temperature is reached. Adjust the null meter reset knob so that the null meter reads slightly to the left of zero, such that when the rotor drive is turned on the test temperature will be established with only minimal further temperature adjustment.

(1) If the control temperature is reached more slowly than outlined above, replace the cold methanol (see X1.1.5), or lower the temperature of the cold methanol (see X1.1.5).

(2) If the control temperature is reached more rapidly than outlined above, raise the temperature of the cold methanol in order to obtain satisfactory control.

X1.5.2.2 Turn on the rotor drive 180 s ± 3 s after the coolant flow is turned on.

X1.5.2.3 With the tachometer plugged into the CAL jack, record the speed meter reading immediately after turning on the motor switch. If the indicator rises and then drops rapidly to a position at least 5 % less than the highest reading, there is possible presence of residual solvent in the shear area. This abnormal digital speed meter change or analog meter needle deflection can also occur as a result of poor temperature control (as indicated on the temperature meter) that is most frequently caused by poor thermal contact between the stator thermal well and the thermistor. Terminate the run. Remove the sample and clean as described in X1.5.3. Repeat the procedure with a fresh sample starting with X1.5.2.

X1.5.2.4 Record speed indicator meter reading at 60 s ± 5 s from rotor startup, estimating the meter reading to the nearest 1/10 of the smallest meter division for the analog meter, when the digital meter is not being used. Turn off rotor drive and coolant flow.

X1.5.3 Clean the CCS by the following steps:

X1.5.3.1 Circulate warm methanol (35 °C to 45 °C) around the stator during the time of cleaning. Maintain flow of warm methanol until X1.5.3.2 has been completed. See X1.5.3.3 for an alternative procedure.

X1.5.3.2 Wash the assembly with petroleum naphtha and finally with acetone (with due care for the flammability of these solvents), using the vacuum to dry the assembly. Turn the rotor several revolutions by hand during final drying with vacuum to ensure that the gap between rotor and stator is clean and dry.

X1.5.3.3 As an alternative to the use of solvents in X1.5.3.1 and X1.5.3.2, inject an excess of 30 mL of the next sample to flush the previous sample and fill the cell with the new sample as in X1.5.2.

X1.5.4 Leave the final sample of a series of runs in the instrument. This will prevent damage if the instrument is accidentally turned on. This final sample can also be used as the sample for the first run after a shutdown period. This allows the electronic components and motor to come up to temperature by operation with a sample already in place. Do not record speed indicator data from this sample upon starting a new set of runs.

X1.6 Manual CCS Report

X1.6.1 Calculate the apparent viscosity of the test sample in mPa·s from the graph referenced in X1.4.4 or Eq X1.1 in X1.4.4.1.

X1.6.2 Report the value determined in X1.6.1 to the nearest 10 mPa·s and the test temperature.

X1.7 Precision and Bias

X1.7.1 *Precision*[12]—The precision of this test method with CCS-2B (manual) as determined by the statistical examination of the interlaboratory test results over the temperature range from –5 °C to –30 °C and viscosity range from 1560 mPa·s to 10 200 mPa·s is as follows:

X1.7.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of this test method, exceed the following values only in one case in twenty:

$$\text{Repeatability} = 5.4\ \%\ \text{of their mean} \qquad (X1.3)$$

X1.7.1.2 *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:

$$\text{Reproducibility} = 8.9\ \%\ \text{of their mean} \qquad (X1.4)$$

[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1285. Contact ASTM Customer Service at service@astm.org.

X2. SPECIAL PROCEDURE FOR TESTING HIGHLY VISCOELASTIC OILS USING THE MANUAL CCS INSTRUMENT

X2.1 Test samples can exhibit different behavior at low temperature in the CCS, thereby requiring procedural variations. Some highly viscoelastic samples will spiral toward the rotor shaft when the rotor drive is started. If the sample climbs from the shear zone, the rotor speed will increase noticeably. The use of the rubber stopper in the fill tube (see X1.5.2) normally will ensure that the procedure in Section 11 will be satisfactory; however, very highly viscoelastic test samples can require this special procedure. The procedure in X2.2 – X2.7 is used for both viscoelastic and non-viscoelastic samples. There are more manipulations in shorter time periods required in X2.5 than in X1.5.2. Calibration oils must be run by the same procedure as the test samples since the calibration curves can differ slightly.

X2.2 Insert test sample from a dropping pipet into the filling tube filling the gap between the rotor and stator, with a slight excess to cover the rotor with about 1 mm of liquid. Turn the rotor by hand to ensure complete wetting of the surfaces of the stator and rotor while the last portion of this sample is flowing up past the rotor sides.

X2.3 Turn the temperature control and coolant flow on, and allow the stator to cool. Control temperature should be reached within 30 s to 60 s for test temperatures down to –20 °C and within 60 s to 90 s for test temperatures down to –30 °C. To ensure optimum control of temperature, the valve settings on the coolant circulator are set for control of coolant with a low-viscosity test sample in the viscometric cell and the simulator motor turned on; the temperature of the coolant to the viscometric cell is approximately 10 °C below the test temperature. There must be good thermal contact with the temperature sensor in the thermal well in the stator. This thermal well should be cleaned periodically (see X1.1.4).

X2.4 The null meter reset knob should be set slightly lower than the test temperature, such that when the rotor drive is turned on the test temperature will be established without further temperature adjustment.

X2.5 Start a timer when test temperature is reached (as indicated by the temperature indicator meter and the cyclic controlling of coolant flow). At 10 s ± 2 s after starting the timer, add additional sample directly into the cup, thus filling the cup completely.

X2.6 Turn on rotor drive at 30 s ± 2 s after start of timer.

X2.7 Record speed indicator meter reading at 10 s ± 2 s from rotor startup, estimating the meter reading to the nearest 0.001 unit. Turn off rotor drive and coolant flow.

X2.8 Clean the CCS by the procedure in X1.5.3 – X1.5.3.3.

X2.9 The precision of the measurement of the apparent viscosity of highly viscoelastic engine oils has not been determined and can be expected to be somewhat poorer from that determined in X1.7.1 – X1.7.1.2.

X3. PROCEDURE FOR MINI-SAMPLE ADAPTER

X3.1 Apparatus

X3.1.1 Mini-sample adapter kit consists of:

(1) Quick disconnect fitting with internal shutoff.

(2) Female Luer lock to quick disconnect fitting.

(3) 10 mL glass syringe with Luer lock.

NOTE X3.1—A mini-sample adapter kit containing all the necessary components is available from the instrument manufacturer.

X3.2 Summary of Procedure

X3.2.1 The Mini-sample test procedure bypasses the automatic sample injection cycle by manually injecting the sample into the stator block from a 10–mL syringe when the software calls for sample injection.

X3.3 Procedure

X3.3.1 With instrument ready to begin testing, enter sample identification and test temperature for sample.

X3.3.2 Fill a clean, dry syringe with 10 mL ± 0.5 mL of sample.

X3.3.3 Connect syringe to quick disconnect fitting on CCS stator block.

X3.3.4 Initiate sample testing by pressing, "Enter."

X3.3.5 When the software calls for the sample to be injected, begin a phased injection by pressing on syringe plunger to push approximately 2 mL into stator every 20 s until syringe is empty. Do not disconnect syringe when empty.

X3.3.6 Instrument software will automatically complete sample testing.

X3.3.7 When this sample's testing is complete, then disconnect syringe.

X3.3.8 If Mini-sample testing is complete, reconnect quick disconnect to pump outlet, then return to Section 12.

X3.3.9 If using the Mini-sample adapter again, then repeat X3.3.1 to X3.3.7.

NOTE X3.2—Detailed instructions are also available from instrument manufacturer.

X4. FLOWCHART FOR CALIBRATION

X4.1 The flowchart in Fig. X4.1 follows the steps in Section 10, Calibration. The steps are meant to give the user an understanding of the potential pathways while following the steps from 10.1 to Section 11.

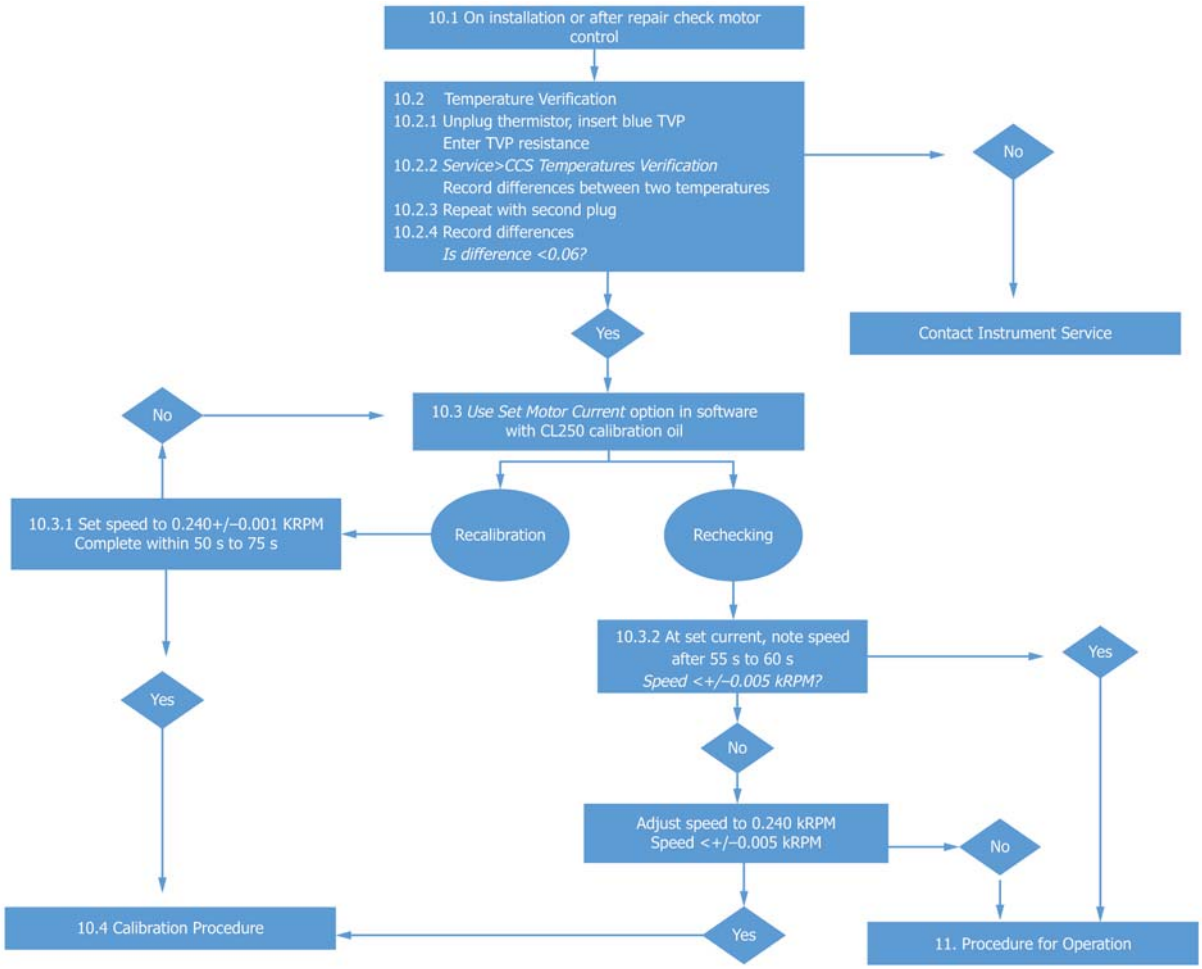

FIG. X4.1 Flowchart for Calibration

X5. CALIBRATION OILS

X5.1 See Table X5.1.

TABLE X5.1 Calibration Oils

	Approximate[A] Viscosity in mPa·s at:					
Reference ID	–10 °C	–15 °C	–20 °C	–25 °C	–30 °C	–35 °C
CL080	...	...	...	...	...	850
CL090	...	...	...	...	...	1150
CL100 (10)	...	...	...	...	875	1450
CL110	...	...	...	...	1025	1675
CL120 (12)	...	...	...	755	1225	2050
CL130	...	...	...	975	1550	2600
CL140 (14)	...	...	800	1250	2075	3550
CL150	...	...	950	1525	2500	4310
CL160 (16)	...	...	1200	1900	3200[B]	5575
CL170	...	850	1325	2175	3650	6425[C]
CL190 (19)	...	1075	1675	2750	4675	8375
CL200	875	1325	2125	3500[B]	6025	10 925
CL220 (22)	1025	1600	2550	4225	7375[C]	13 550
CL240	1225	1900	3050	5175	9100	16 925
CL250 (25)	1375	2175	3500[B]	6000	10 650	20 000
CL260	1675	2650	4300	7300[C]	13 050	...
CL280 (28)	2025	3200[B]	5275	9075	16 500	...
CL300	2425	3875	6475	11 250	20 650	...
CL320 (32)	3000	4850	8150[C]	14 325	...	...
CL340	3475	5650	9575	16 975	...	...
CL380 (38)	4175	6800[C]	11 600	20 800	...	...
CL420	4950[B]	8175	14 025	...	...	...
CL480 (48)	6000	10 050	17 425	...	...	...
CL530	7500[C]	12 525	22 025	...	...	...
CL600 (60)	9300	15 725	...	...	...	...
CL680	11 300	19 350	...	...	...	...

[A] Consult supplier for specific values. 3.x or 5.x.
[B] Oil to be used for calibration checks with CCS 2B or CCS 4 or 5 with software version 3.x or 5.x.
[C] Oil to be used for calibration checks with CCS-4 or 5 software versions 4.x or 6.x.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D5293 – 15) that may impact the use of this standard. (Approved July 1, 2017.)

(1) Completely revised Table 1; added new Appendix X4; moved former Table 1 to new Appendix X5 and deleted former Table 2.