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An American National Standard

Standard Test Method for ~~Apparent Viscosity of Engine Oils Between–5 and–35°C Using the Cold-Cranking Simulator~~Apparent Viscosity of Engine Oils and Base Stocks Between –5 and –35°C Using Cold-Cranking Simulator[1]

This standard is issued under the fixed designation D 5293; 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 laboratory determination of apparent viscosity of engine oils and base stocks by cold cranking simulator (CCS) at temperatures ~~between–5 and–35°C~~ between –5 and –35°C at shear stresses of approximately 50 000 to 100 000 Pa and shear rates of approximately 10^5 to 10^4 s^{-1} ~~and viscosities of approximately 500 to 25000 mPa·s. The range of an instrument is dependent on the instrument model and software version installed. These results are related to engine-cranking characteristics of engine oils.~~

~~1.2A special procedure is provided in Annex A1 for highly viscoelastic oils.~~ –1 for viscosities of approximately 900 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.

~~1.4A special manual procedure is provided in Annex A1 for highly viscoelastic oils.~~

~~1.5The values stated in SI units are to be regarded as the standard.~~

~~1.6~~

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 7.1, 7.2, 7.3, and Section~~ Specific warning statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

D 2602 Test Method for Apparent Viscosity of Engine Oils at Low Temperature Using the Cold-Cranking Simulator[3]

D 4057 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.

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

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[2] 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] Withdrawn.~~

[3] Withdrawn.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

~~[4] 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, Inc., 219 Perimeter Center Parkway, Atlanta, GA 30346.~~

[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.**

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:

~~$\eta = \tau/\dot{\gamma}$ (1)~~

$\eta = \tau\gamma$

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 (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~~

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.

~~3.2.4~~

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 ~~regulated flow of refrigerated coolant through the stator.~~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~~test with reference oils that had viscosities between 600 and 8400 mPa·s (cP) at–17.8°C and between 2000 and 20000 mPa·s (cP) at–28.9°C. The detailed relation between this engine cranking data and CCS apparent viscosities is in Appendixes X1 and X2 of the 1967 T edition of Test Method D2602[5] and CRC Report 409.[4] 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 L-49 engine cranking results is satisfactory.~~

~~5.2The correlation between CCS and apparent viscosity and engine cranking was confirmed at temperatures between–1 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.~~test with reference oils that had viscosities between 600 and 8400 mPa·s (cP) at –17.8°C and between 2000 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 D 2602[6]

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

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~~[5] Supporting data (Appendixes X1 and X2) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1402.~~

[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, Inc., 219 Perimeter Center Parkway, Atlanta, GA 30346.

~~[6] 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.).~~

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1402.

~~[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1442.~~

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 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

~~6.1Two types of apparatus are available for use in this test method: the manual cold-cranking simulator (see~~

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 the automated CCS (see~~ and 6.3~~and 6.4~~).

6.2 ~~*Manual CCS*~~*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~~0.05°C of set point; and a ~~coolant circulator compatible~~heat removal system with ~~the~~a temperature control ~~system. See Fig. 1.~~ system, a computer, computer interface, and test sample injection pump.

6.3 *Automatic Automated CCS*,~~, consisting of the CCS~~[9] as described in 6.2 ~~, with computer, computer interface, and test sample injection pump. The methanol circulator (see 6.6.1) is not used because the test sample injection displaces the previous test sample. See Fig. 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.

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 D 2162. Approximate viscosities at certain temperatures are listed in Table 1, 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.

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 D 4057) 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.

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

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

~~[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1285.~~

[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, which you may attend.

TABLE 1 Calibration Oils

	Calibration Oil Approximate[A] Viscosity in mPa·s at:						
	–5°C	–10°C	–15°C	–20°C	–25°C	–30°C	–35°C
~~CL 10~~	...	...	...	...	...	...	~~1 700~~
CL080	...	...	...	...	...	...	900
~~CL 12~~	...	...	...	...	~~800~~	~~1 600~~	~~3 200~~
CL090	...	...	...	...	...	...	1200
~~CL 14~~	...	...	...	...	~~1 600~~	...	~~1700~~
CL100 (10)	...	...	...	...	...	...	1700
CL110	...	...	...	...	...	1550	2500
CL120 (12)	...	...	...	...	800	1600	3200
CL130	...	...	...	...	...	2900	4850
~~CL140 (14)~~	...	...	...	...	~~1600~~	~~250[B]~~	~~7 000[C]~~
CL140 (14)	...	...	...	...	1600	3250[B]	7000[C]
~~CL 16~~	...	...	...	~~1700~~	~~2700~~	~~4600~~	~~8050~~
CL150	...	...	...	1700	2700	4600	8050
~~CL160 (16)~~	...	~~2 500~~	~~5 500~~	...	~~2500~~	~~5500~~	~~11 000~~
CL160 (16)	...	...	...	...	2500	5500	11 000
~~CL 19~~	...	...	~~1450~~	~~2250~~	~~3700~~	~~6300~~	~~11 300~~
CL170	...	...	1450	2250	3700	6300	11 300
~~CL190 (19)~~	...	~~1 800~~	~~3...~~	~~1800~~	~~500[B]~~	~~7 400[C]~~	~~17 000~~
CL190 (19)	...	...	...	1800	3500[B]	7400[C]	17 000
~~CL 22~~	...	...	~~1 300~~	~~2 500~~	~~5 100~~	~~11 000~~	~~13 700~~
CL200	...	...	1677	2650	4300	7550	13 700
CL220 (22)	...	...	1300	2500	5100	11 000	...
~~CL 25~~	...	...	~~1 800~~	~~3600~~	~~6000~~	~~10 700~~	~~19 800~~
CL240	...	...	2250	3600	6000	10 700	19 800
~~CL250 (25)~~	...	...	~~1800~~	~~3500[B]~~	~~7 400[C]~~	~~17 200~~	...
CL250 (25)	...	...	1800	3500[B]	7400[C]	17 200	...
~~CL 28~~	...	~~1 200~~	~~2 500~~	~~5 000~~	~~9 300~~	~~13 400~~	...
CL260	...	1750	2700	4400	7500	13 400	...
	...	~~1200~~	~~2500~~	~~5000~~	~~9300~~	...	...
CL280 (28)	...	1200	2500	5000	9300	...	...
~~CL 32~~	...	~~1 800~~	~~3750~~	~~6100~~	~~10 500~~	~~19 300~~	...
CL300	...	2400	3750	6100	10 500	19 300	...
~~CL320 (32)~~	...	~~1800~~	~~3500[B]~~	~~7 300[C]~~	~~15 900~~	...	...
CL320 (32)	...	1800	3500[B]	7300[C]	15 900	...	...
~~CL 38~~	...	~~2 900~~	~~54200~~	~~7000~~	~~12 194~~	...	...
CL340	...	2700	4200	7000	12 194	...	...
~~CL380 (38)~~	...	~~2900~~	~~5800[C]~~	~~13 000~~	...	...	...
CL380 (38)	...	2900	5800[C]	13 000	...	...	...
~~CL 48~~	...	~~5200~~	~~8500~~	~~14 405~~	...	...	...
CL420	...	5200	8500	14 405	...	...	...
~~CL480 (48)~~	~~2300~~	~~4 500[B]~~	~~9 500~~	~~21 000~~	...	...	...
CL480 (48)	2300	4500[B]	9500	21 000	...	...	...
~~CL 60~~	...	~~6000~~	~~9843~~	~~16 881~~	...	...	...
CL530	...	6000	9843	16 881	...	...	...
~~CL600 (60)~~	~~3700~~	~~7 400[C]~~	~~15 600~~	...	...	...	...
CL600 (60)	3700	7400[C]	15 600	...	...	...	...
~~CL 74~~	...	~~9550~~	...	...	...	...	...
CL680	...	9550	...	...	...	...	...
~~CL740 (47)~~	~~6000[B]~~	~~12 000~~	...	...	...	...	...
CL740 (47)	6000[B]	12 000	...	...	...	...	...

[A] Consult supplier for specific values.
[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 used for calibration checks with CCS-4 or 5 software versions 4.x or 6.x.

10. Calibration

10.1 On start-up 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.

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.

~~6.4*Automatic Automated CCS*[8]—The CCS described in 6.3 with the addition of an automated sample table allowing up to 30 test samples to be run sequentially under computer control without operator attention. See Fig. 3.~~

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 ± 0.001 KRPM (displayed as SPEED on the computer monitor) by slowly turning the CURRENT ADJUST DIAL. This should be completed with in 50 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 55–60 s. If the speed is less than 0.005 KRPM from 0.240, 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 it is found to be greater than 0.005 KRPM from 0.240, readjust rotor speed to 0.240 KRPM, and record 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 ~~1—In some CCS instruments, the refrigeration may be achieved using solid state thermoelectric modules.~~

~~6.5*Calibrated Thermistor*—Sensor for insertion in a well near the inside surface of the stator to indicate the test temperature.~~

~~6.6*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.~~ 1—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, using Table 2, select an oil from Group A, at least 3 oils from Group B and at least 1 oil from Group C. The selections from Group B will be evenly distributed over the set of calibration oils. The set of oils selected will be sufficient to provide 10 data sets consisting of temperature, speed and known viscosity for establishing the calibration equation in 10.5. A calibration oil can be included twice to achieve the required 10 data sets. The calibration data set shall have a minimum of 10 data sets for the temperature being calibrated that are evenly distributed over the viscosity range of the calibration oils. When including a calibration oil a second time, it is preferable to not place the samples in adjacent positions for the series. For example –35°C calibration could have CL080, CL100, CL120, CL140, CL160, CL190 followed by another set CL080, CL100, CL120, CL140, CL160, CL190 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:

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

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 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

TABLE 2 Calibration Oil Sets by Test Temperature

Test Temperature	Calibration Oil Group A Preferred or Alternate[A]	Calibration Oil Group B Use at least 3 of this group evenly distributed	Calibration oil Group C Use at least one of this group
–35°C	CL080 or CL090	CL090, CL100, CL110, CL120, CL130, CL140, CL150, CL160, CL170, CL200	CL190, CL220, CL240
–30°C	CL100 or CL110	CL110, CL120, CL130, CL140, CL150, CL160, CL170, CL190, CL200, CL220, CL260	CL250, CL280, CL300
–25°C	CL120 or CL130	CL130, CL140, CL150, CL160, CL170, CL190, CL200, CL220, CL250, CL260, CL280, CL300	CL320, CL340, CL380
–20°C	CL140 or CL150	CL150, CL160, CL170, CL190, CL200, CL220, CL250, CL260, CL280, CL300, CL320, CL340, CL380, CL420	CL480, CL530
–15°C	CL190 or CL170	CL170, CL200, CL220, CL240, CL250, CL260, CL280, CL300, CL340, CL380, CL420, CL480, CL530	CL600
–10°C	CL250 or CL260	CL260, CL280, CL300, CL340, CL380, CL420, CL480, CL530, CL600, CL680	CL740

[A] It is strongly recommended that the preferred Calibration Oil be used from Group A.

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 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 bottle(s).

NOTE 2—Thermoelectric cooling may be used instead of mechanical refrigeration or use of dry ice, and so forth.

6.6.1 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. Adjust the temperature of the coolant to the viscometric cell to be at least 10°C below the test temperature. 2—When using an automatic sample changer, ensure the bottles 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 bottles are on the tray and entered into the test matrix on the computer.

NOTE 3—If a thermoelectric cooling system is used in the instrument, 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.

6.6.1.1 To ensure optimum control of temperature using the dry-ice system, 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.

6.7 *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.

6.8 *Optional Methanol Circulator*[8]—This option (for the Manual CCS only) circulates warm methanol through the stator to facilitate sample changes and aid the evaporation of cleaning solvents.

7. 3—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 and 60 s will be used to calculate viscosity.

NOTE 4—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, investigate and resolve the cause of the deviation.

11.4.2 If current drifts by more than 0.005 A during normal operation, recheck rotor speed with CL 250 at –20°C.

12. Report

12.1 Report the calculated viscosity and temperature as displayed on the computer monitor or test report. The value displayed is rounded to the nearest 10 mPa·s.

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 to –35°C and a viscosity range from 2700 to 15 000 mPa·s is shown in the table below for each instrument.

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

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1459.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1438.D02-1653.