Designation: D5293 – 15

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.

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.

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D2602 Test Method for Apparent Viscosity of Engine Oils At Low Temperature Using the Cold-Cranking Simulator (Withdrawn 1993)[3]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

---

[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.

Current edition approved April 1, 2015. Published May 2015. Originally approved in 1991. Last previous edition approved in 2014 as D5293 – 14. DOI: 10.1520/D5293-15.

[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**

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

[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, Alpharetta, GA 30022.

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

[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.

[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.

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. 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 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 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.

TABLE 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.
^{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.

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 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, use Table 2 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:

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

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 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

TABLE 2 Calibration Oil Sets by Test Temperature

Test Temperature	Group A Include at least one **Preferred** and Alternate[A]	Group B Include at least 3. The selection is to be uniformly distributed over the range.	Group C Include at least one.
–35 °C	CL080, **CL090**, or CL100 (~800 mPa·s to 1500 mPa·s)	CL110, CL120, CL130, CL140, CL150, CL160, CL170, CL190, CL200 (~1000 mPa·s to 15 000 mPa·s)	CL220, CL240, CL250 (> 13 500 mPa·s)
–30 °C	CL100, **CL110**, or CL120 (~800 mPa·s to 1500 mPa·s)	CL130, CL140, CL150, CL160, CL170, CL190, CL200, CL220, CL240, CL250, CL260 (~1000 mPa·s to 15 000 mPa·s)	CL280, CL300, CL320 (> 13 500 mPa·s)
–25 °C	CL120, **CL130**, or CL140 (~800 mPa·s to 1500 mPa·s)	CL150, CL160, CL170, CL190, CL200, CL220, CL240, CL250, CL260, CL280, CL300 (~1000 mPa·s to 15000 mPa·s)	CL320, CL340, CL380 (> 13 500 mPa·s)
–20 °C	CL140, **CL150**, or CL160 (~800 mPa·s to 1400 mPa·s)	CL170, CL190, CL200, CL220, CL240, CL250, CL260, CL280, CL300, CL320, CL340, CL380 (~1000 mPa·s to 15 000 mPa·s)	CL420, CL480, CL530 (> 13 500 mPa·s)
–15 °C	CL170, **CL190**, or CL200 (~800 mPa·s to 1400 mPa·s)	CL220, CL240, CL250, CL260, CL280, CL300, CL320, CL340, CL380, CL420, CL480 (~1000 mPa·s to 13 000 mPa·s)	CL530, CL600, CL680 (> 11 500 mPa·s)
–10 °C	CL200, **CL220**, or CL240 (~800 mPa·s to 1400 mPa·s)	CL250, CL260, CL280, CL300, CL320, CL340, CL380, CL420, CL480, CL530 (~1000 mPa·s to 9000 mPa·s)	CL600, CL680 (> 9000 mPa·s)

[A] It is strongly recommended that the preferred Calibration Oil be used from Group 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 2—When using mini-sample adapter, the instructions in Appendix X3 replace those in 11.1 – 11.3.

NOTE 3—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 4—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 5—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.

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

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:

$$\eta = \frac{B_0}{N} + B_1 + B_2 N \tag{X1.1}$$

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:

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