

Designation: D5293 –  $08^{\varepsilon 1}$ 

# Standard Test Method for Apparent Viscosity of Engine Oils and Base Stocks Between –5 and –35°C Using Cold-Cranking Simulator<sup>1</sup>

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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

 $\varepsilon^1$  Note—Moved headings in 13.1 and updated Summary of Changes editorially in February 2009.

# 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  $-35^{\circ}$ C at shear stresses of approximately 50 000 to 100 000 Pa and shear rates of approximately  $10^{5}$  to  $10^{4}$  s<sup>-1</sup> 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.

1.4 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:<sup>2</sup>

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<sup>3</sup>
- 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<sup>4</sup>

## 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*,  $\eta$ , *n*—the property of a fluid that determines its internal resistance to flow under stress, expressed by:

$$\eta = \frac{\tau}{\gamma} \tag{1}$$

2-4dc6-9e19-39860265f5ct/astm-d5293-08e where:

 $\tau$  = the stress per unit area, and

 $\gamma$  = 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.

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> 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.

 $<sup>^{3}</sup>$  Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

<sup>&</sup>lt;sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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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<sup>5</sup> 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 D2602<sup>6</sup> and CRC Report 409.<sup>5</sup> 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 -1and  $-40^{\circ}$ 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.<sup>7</sup>

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  $-40^{\circ}$ C with six commercial engine oils (SAE 0W, 5W, 10W, 15W, 20W, and 25W).<sup>8</sup>

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,<sup>9</sup> 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,<sup>9</sup> 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*,<sup>9</sup> 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.

## 06.5 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  $10^{\circ}$ C below the test temperature. When the coolant temperature is below  $-30^{\circ}$ 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.

<sup>&</sup>lt;sup>5</sup> 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.

<sup>&</sup>lt;sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1402.

<sup>&</sup>lt;sup>7</sup> 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.).

<sup>&</sup>lt;sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1442.

<sup>&</sup>lt;sup>9</sup> 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,<sup>1</sup> which you may attend.

## 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  $\mu$ m 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.

## 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  $0.06^{\circ}$ 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  $-20.0^{\circ}$ 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  $0.240 \pm 0.001$  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.

Calibration Oil Approximate <sup>A</sup> Viscosity in rnPa·s at:								
ps://standard	s.iteh_5°ccatalog	/sta_10°Crds/sist/2	22-15°C e0-47	02- <u>20</u> °€0-9e	9-3 <sub>-25°C</sub> 2051	5cf/a <u>_30°c</u> d529	3-08 <u>-</u> 35°C	
CL080							900	
CL090							1200	
CL100 (10)							1700	
CL110						1550	2500	
CL120 (12)					800	1600	3200	
CL130						2900	4850	
CL140 (14)					1600	3250 <sup>B</sup>	7000 <sup>C</sup>	
CL150				1700	2700	4600	8050	
CL160 (16)					2500	5500	11 000	
CL170			1450	2250	3700	6300	11 300	
CL190 (19)				1800	3500 <sup>B</sup>	7400 <sup>C</sup>	17 000	
CL200			1677	2650	4300	7550	13 700	
CL220 (22)			1300	2500	5100	11 000		
CL240			2250	3600	6000	10 700	19 800	
CL250 (25)			1800	3500 <sup>B</sup>	7400 <sup>C</sup>	17 200		
CL260		1750	2700	4400	7500	13 400		
CL280 (28)		1200	2500	5000	9300			
CL300		2400	3750	6100	10 500	19 300		
CL320 (32)		1800	3500 <sup>B</sup>	7300 <sup>C</sup>	15 900			
CL340		2700	4200	7000	12 194			
CL380 (38)		2900	5800 <sup>C</sup>	13 000				
CL420		5200	8500	14 405				
CL480 (48)	2300	4500 <sup>B</sup>	9500	21 000				
CL530		6000	9843	16 881				
CL600 (60)	3700	7400 <sup>C</sup>	15 600					
CL680		9550						
CL740 (47)	6000 <sup>B</sup>	12 000						

TABLE 1 Colliburation Oile

<sup>A</sup> Consult supplier for specific values.

<sup>B</sup> 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.

10.3.2 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-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:

= the apparent viscosity, n

= the coefficients of regression, and  $B_0, B_1, B_2$ 

= 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 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-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.

Test Temperature	Calibration Oil Group A Preferred or Alternate <sup>A</sup>	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

<sup>A</sup> It is strongly recommended that the preferred Calibration Oil be used from Group A.