



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

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 by cold cranking simulator (CCS) at temperatures 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<sup>-1</sup> and viscosities of approximately 500 to 25 000 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.

1.2 A special procedure is provided in Annex A1 for highly viscoelastic oils.

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

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

1.6 *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 8.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D 2602 Test Method for Apparent Viscosity of Engine Oils at Low Temperature Using the Cold-Cranking Simulator<sup>3</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

<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>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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.

## 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:

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

where:

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

$\dot{\gamma}$  = 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.

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

3.2.4 *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. The speed of the rotor is calibrated as a

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



FIG. 1 Cold Cranking Simulator

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>4</sup> test with reference oils that had viscosities between 600 and 8400 mPa·s (cP) at  $-17.8^{\circ}\text{C}$  and between 2000 and 20 000 mPa·s (cP) at  $-28.9^{\circ}\text{C}$ . 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 D 2602<sup>5</sup> and CRC Report 409.<sup>4</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 L-49 engine cranking results is satisfactory.

5.2 The correlation between CCS and apparent viscosity and engine cranking was confirmed at temperatures between  $-1$  and  $-40^{\circ}\text{C}$  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>6</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

1990's engines at temperatures ranging from  $-5$  down to  $-40^{\circ}\text{C}$  with six commercial engine oils (SAE 0W, 5W, 10W, 15W, 20W, and 25W).<sup>7</sup>

## 6. Apparatus

6.1 Two types of apparatus are available for use in this test method: the manual cold-cranking simulator (see 6.2) and the automated CCS (see 6.3 and 6.4).

6.2 *Manual CCS*<sup>8</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  $\pm 0.05^{\circ}\text{C}$  of set point; and a coolant circulator compatible with the temperature control system. See Fig. 1.

6.3 *Automated CCS*<sup>8</sup>, consisting of the CCS 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.

6.4 *Automatic Automated CCS*<sup>8</sup>—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.

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  $10^{\circ}\text{C}$

<sup>4</sup> CRC Report No. 409 "Evaluation of Laboratory Viscometers for Predicting Cranking Characteristics of Engine Oils at  $-0^{\circ}\text{F}$  and  $-20^{\circ}\text{F}$ ," April 1968 available from the Coordinating Research Council, Inc., 219 Perimeter Center Parkway, Atlanta, GA 30346.

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

<sup>6</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>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1442.

<sup>8</sup> 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<sup>1</sup>, which you may attend.

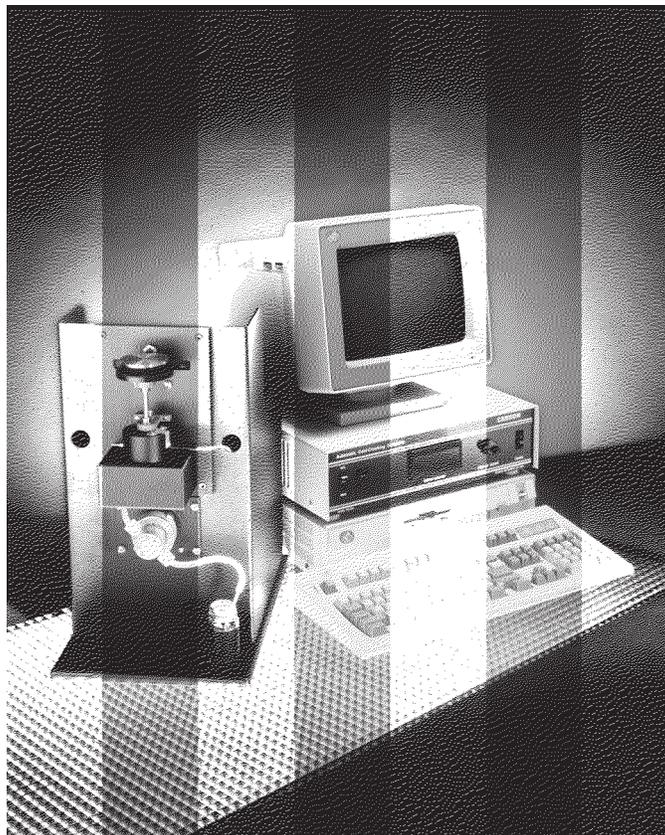


FIG. 2 Automated Cold-Cracking Simulator

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.

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.

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*<sup>8</sup>—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. Reagents and Materials

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

7.2 *Methanol*—(**Warning**—Danger—Flammable. Vapor harmful.)

7.3 *Petroleum Naphtha*—(**Warning**—Combustible vapor harmful.)

7.4 *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.

NOTE 4—Blind reference samples are available from the supplier of the calibration oils for checking on the shear rate of the viscometric cell and the overall procedure.

## 8. Hazards

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

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 a filter or

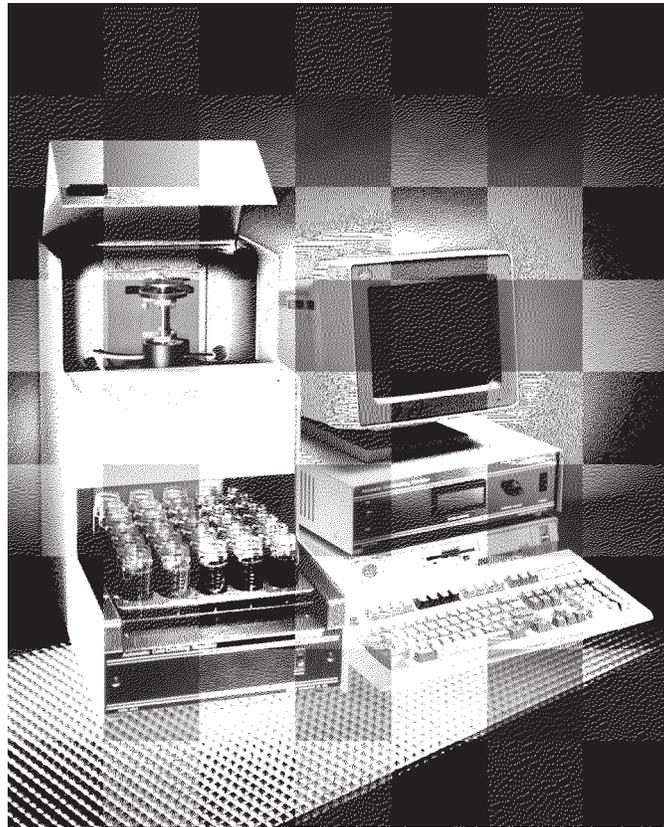


FIG. 3 Automatic Automated Cold-Cranking Simulator

TABLE 1 Calibration Oils

	Calibration Oil						
	Approximate <sup>A</sup> Viscosity in mPa-s at:						
	-5°C	-10°C	-15°C	-20°C	-25°C	-30°C	-35°C
CL-10	...	...	...	...	...	...	1 700
CL-12	...	...	...	...	800	1 600	3 200
CL-14	...	...	...	...	1 600	3 250 <sup>B</sup>	7 000 <sup>C</sup>
CL-16	...	...	...	...	2 500	5 500	11 000
CL-19	...	...	...	1 800	3 500 <sup>B</sup>	7 400 <sup>C</sup>	17 000
CL-22	...	...	1 300	2 500	5 100	11 000	...
CL-25	...	...	1 800	3 500 <sup>B</sup>	7 400 <sup>C</sup>	17 200	...
CL-28	...	1 200	2 500	5 000	9 300	...	...
CL-32	...	1 800	3 500 <sup>B</sup>	7 300 <sup>C</sup>	15 900	...	...
CL-38	...	2 900	5 800 <sup>C</sup>	13 000	...	...	...
CL-48	2300	4 500 <sup>B</sup>	9 500	21 000	...	...	...
CL-60	3700	7 400 <sup>C</sup>	15 600	...	...	...	...
CL-74	6000 <sup>B</sup>	12 000	...	...	...	...	...

<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.

<sup>C</sup> Oil to be used for calibration checks with CCS-4 or 5 software versions 4.x or 6.x.

centrifuge to remove particles greater than 5 µm in size. Do not shake the sample of test oil. This leads to entrainment of air, and a false viscosity reading.

## 10. Calibration

### 10.1 Calibration of Manual CCS:

10.1.1 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 10.1.2) monthly until the change in drive current in consecutive months is less than 0.020 A and every three months thereafter.

10.1.2 *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  $0.240 \pm 0.010$  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  $0.240 \pm 0.010$  units with the 3500 mPa·s reference oil at  $-20^{\circ}\text{C}$ , recalibrate the instrument by either procedure described in 10.1.3.

10.1.3 *Calibration Procedure*—At each test temperature, calibrate with the oils listed for that temperature in Table 1 by using the procedure described in Section 11.

10.1.3.1 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.

10.1.4 *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. 4 for a typical curve. Use the equation in 10.1.4.1 as an alternative method to this graphical method.

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

$$\eta = b_0N + b_1 + b_2N \quad (2)$$

where:

- $\eta$  = viscosity,
- $b_0, b_1, b_2$  = constants determined with a minimum of three calibration oils, and
- $N$  = observed speed indicator reading.

10.1.4.2 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  $b_2$ :

$$\eta N = b_0 + b_1N + b_2N^2 \quad (3)$$

10.1.5 When check runs of a calibration oil do not fall within  $\pm 5\%$  of the values calculated from the calibration curve, recheck the calibration of the temperature sensor or rerun the calibration oils.

NOTE 5—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.

10.2 *Calibration of Automated CCS:*

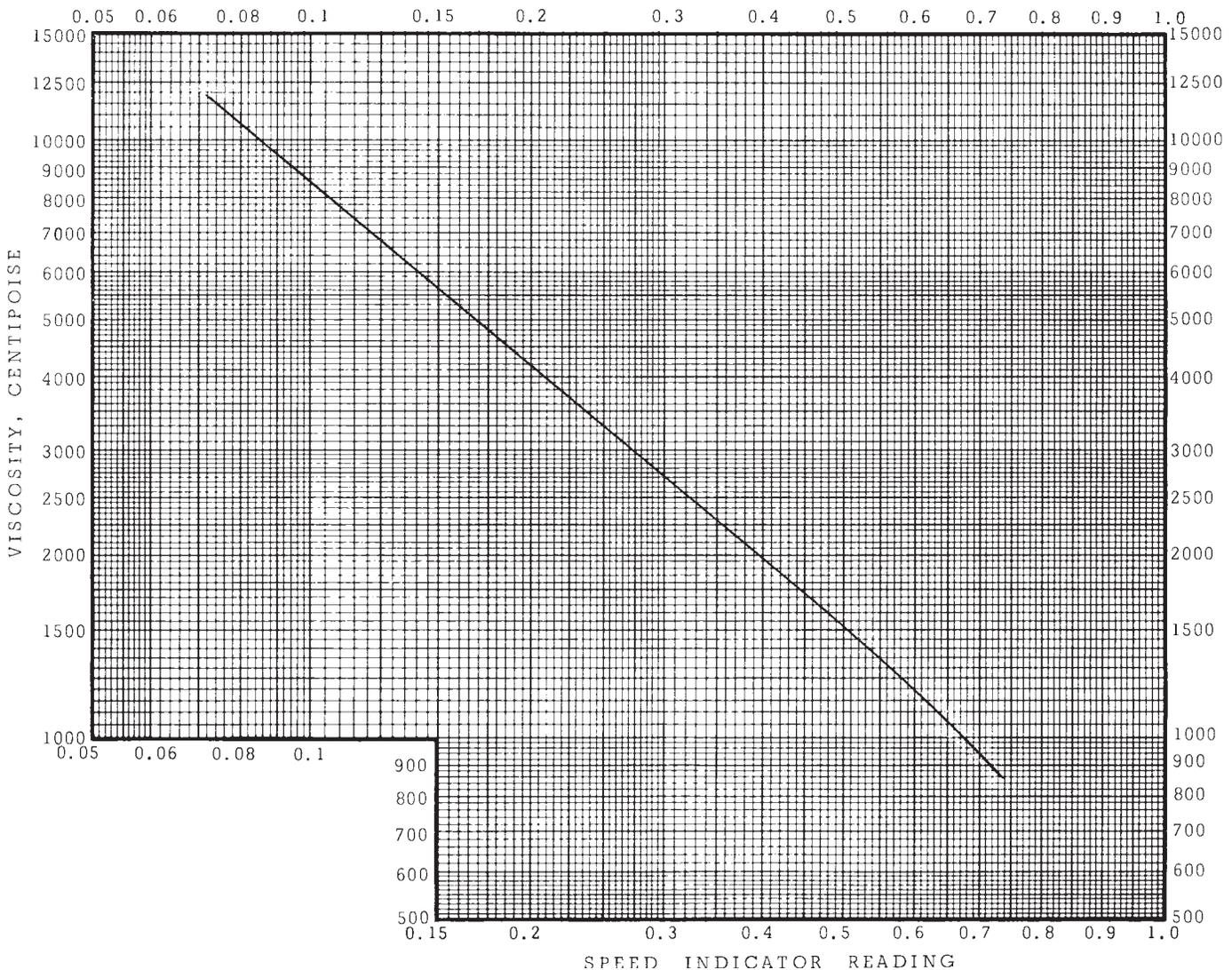


FIG. 4 Linearized Calibration Chart, Cold Cranking Simulator

10.2.1 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, determine the required motor current. Initially, recheck the motor current (as described in 10.2.2) monthly until the change in motor current in consecutive months is less than 0.020 A and every three months thereafter.

10.2.2 *Motor Current Determination*—Run the 3500 mPa·s,  $-20^{\circ}\text{C}$  viscosity standard at  $-20^{\circ}\text{C}$  as described in Section 12. When the drive motor is turned on, establish a speed reading of  $0.240 \pm 0.005$  (displayed as SPEED on the computer monitor) 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 speed reading of  $0.240 \pm 0.005$  units with the 3500 mPa·s reference oil at  $-20^{\circ}\text{C}$ , recalibrate the instrument by the procedure described in 10.2.3.

10.2.3 *Calibration Procedure*—At each test temperature, calibrate with the oils listed for that temperature in Table 1 by using the procedure described in Section 12.

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

10.2.4 *Calibration Equation*—The computer program regresses the calibration data over the viscosity range at each calibration temperature as is described in 10.1.4.1. There must be a minimum of four pairs of calibration data (viscosity and speed) for each temperature.

10.2.5 When check runs of a calibration oil do not fall within  $\pm 5\%$  of the values calculated from the stored calibration equation, recheck the calibration of the temperature sensor or rerun the calibration oils.

## 11. Procedure for Manual CCS Operation

NOTE 6—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.

11.1 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 10.1.2, reset the current to the value previously determined in 10.1.2; make the observation and correction after 15 s of running. When the viscosity measurement of the calibration oil differs by more than  $\pm 5\%$  from its certified value, rerun to confirm this observation. When confirmed, recalibrate as in 10.1.3.

NOTE 7—The use of blind reference samples (see Note 4) are recommended for an overall check on all performance, at six month intervals.

11.2 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 11.2.2) 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 Annex A1 for a special procedure for highly viscoelastic test samples.

NOTE 8—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  $\text{mm}^2/\text{s}$  (cSt), warm the sample (not exceeding  $50^{\circ}\text{C}$ ) prior to filling the viscometric cell.

11.2.1 Turn the temperature control and coolant flow on, and allow the stator to cool. To ensure optimum control of temperature, see 6.6.1 and 6.6.1.1. 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 to 60 s for test temperatures down to  $-20^{\circ}\text{C}$  and within 60 to 90 s for test temperatures down to  $-30^{\circ}\text{C}$ ; if not within these limits, replace the cold methanol (see 6.7) 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.

11.2.1.1 If the control temperature is reached more slowly than outlined above, replace the cold methanol (see 6.7), or lower the temperature of the cold methanol (see 6.6).

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

11.2.2 Turn on the rotor drive  $180 \pm 3$  s after the coolant flow is turned on.

11.2.3 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 11.3. Repeat the procedure with a fresh sample starting with 11.2.

11.2.4 Record speed indicator meter reading at  $60 \pm 5$  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.

11.3 Clean the CCS by the following steps:

11.3.1 Circulate warm methanol ( $35$  to  $45^{\circ}\text{C}$ ) around the stator during the time of cleaning. Maintain flow of warm methanol until 11.3.2 has been completed. See 11.3.3 for an alternative procedure.

11.3.2 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.

11.3.3 As an alternative to the use of solvents in 11.3.1 and 11.3.2, 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 11.2.

11.4 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.

## 12. Procedure for Automated and Automatic Automated CCS Operation

12.1 Establish the calibration equation as described in 10.2.

12.2 For automatic automated CCS instruments, place a minimum of 55 mL of the sample to be tested into a 60 mL bottle(s) designed to fit the sample tray. For automated CCS instruments, ensure that a minimum of 55 mL of the sample to be tested is placed in a suitably sized container and insert the injection tube from the instrument into this container; care should be taken to ensure that the injection tube does not reach to the bottom of the container, in order to avoid drawing any sediment into the instrument. Identify the test sample in the computer.

12.2.1 For the automatic automated CCS, place the bottles in the sample tray. Identify the position in the tray and test temperature for each test sample in the computer program. It is recommended that calibration test samples or secondary standards be placed in the sample tray and identified as calibration check samples.

12.2.2 When check runs of the calibration test samples or secondary standards do not fall within  $\pm 5\%$  of the expected value, the results are considered suspect.

12.3 Start the run using the computer program. 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.

12.3.1 Heating and solvent cleaning of the viscometric cell are not necessary to allow injection of the next sample.

## 13. Report

13.1 *Manual CCS Report:*

13.1.1 Calculate the apparent viscosity of the test sample in mPa·s from the graph referenced in 10.1.4 or Eq 2 in 10.1.4.1.

13.1.2 Report the value determined in 13.1.1 to the nearest 10 mPa(s) and the test temperature.

13.2 *Automated CCS Report:*

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

## 14. Precision and Bias

14.1 *Precision*<sup>9</sup>—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  $-5$  to  $-30^{\circ}\text{C}$  and viscosity range from 1560 to 10 200 mPa·s is as follows:

14.1.1 *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.

$$\text{repeatability} = 5.4\% \text{ of their mean} \quad (4)$$

14.1.2 *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:

$$\text{reproducibility} = 8.9\% \text{ of their mean} \quad (5)$$

14.2 *Precision*<sup>10</sup>—The precision of this test method with CCS-4/5 (automatic) as determined by statistical examination of the interlaboratory test over the temperature range from  $-10$  to  $-35^{\circ}\text{C}$  and a viscosity range from 2800 to 18 000 mPa·s, using version 4.x or higher software, is as follows:

14.2.1 *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.

$$\text{repeatability} = 2.6\% \text{ of their mean} \quad (6)$$

14.2.2 *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:

$$\text{reproducibility} = 7.3\% \text{ of their mean} \quad (7)$$

14.2.3 *Summary of Interlaboratory Study*<sup>10</sup>—The interlaboratory study consisted of nine participating laboratories evaluating ten engine oils with viscosities ranging from 2800 to 18 000 mPa(s) at test temperatures from  $-10$  to  $-35^{\circ}\text{C}$ . All laboratories used the CCS 4/5 with version 4.x or higher instrument software to measure the apparent viscosity.

14.3 *Bias*—The procedure in this test method for measuring the apparent viscosity of engine oils at low temperatures has no bias because the apparent viscosity of engine oils at low temperature is defined only in terms of this test method.

14.4 *Automated Precision*—The automated CCS and automatic automated CCS viscometers have been found not to have

<sup>9</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1285.

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

a bias from the manual CCS2 viscometers, and the precision is within the precision of the manual apparatus.<sup>11</sup>

<sup>11</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1438.

## 15. Keywords

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

## ANNEX

### (Mandatory Information)

#### A1. SPECIAL PROCEDURE FOR TESTING HIGHLY VISCOELASTIC OILS USING THE MANUAL CCS INSTRUMENT

A1.1 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 11.2) 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 A1.2-A1.7 is used for both viscoelastic and non-viscoelastic samples. There are more manipulations in shorter time periods required in A1.5 than in 11.2. Calibration oils must be run by the same procedure as the test samples since the calibration curves can differ slightly.

A1.2 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.

A1.3 Turn the temperature control and coolant flow on, and allow the stator to cool. Control temperature should be reached within 30 to 60 s for test temperatures down to  $-20^{\circ}\text{C}$  and within 60 to 90 s for test temperatures down to  $-30^{\circ}\text{C}$ . 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  $10^{\circ}\text{C}$  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 6.6.1).

A1.4 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.

A1.5 Start a timer when test temperature is reached (as indicated by the temperature indicator meter and the cyclic controlling of coolant flow). At  $10 \pm 2$  s after starting the timer, add additional sample directly into the cup, thus filling the cup completely.

A1.6 Turn on rotor drive at  $30 \pm 2$  s after start of timer.

A1.7 Record speed indicator meter reading at  $10 \pm 2$  s from rotor startup, estimating the meter reading to the nearest 0.001 unit. Turn off rotor drive and coolant flow.

A1.8 Clean the CCS by the procedure in 11.3-11.3.3.

A1.9 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 14.1-14.3.

## SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 5293-02) that may impact the use of this standard.

- (1) Added Note 1 and Note 2 allowing the use of solid state thermoelectric coolant systems.
- (2) Added Note 3 clarifying at what temperature the water

refrigeration system is set when a thermoelectric cooling system is used.

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