Designation: D2983 – 17

Standard Test Method for
Low-Temperature Viscosity of Automatic Transmission Fluids, Hydraulic Fluids, and Lubricants using a Rotational Viscometer[1]

This standard is issued under the fixed designation D2983; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the use of rotational viscometers with an appropriate torque range and specific spindle for the determination of the low-shear-rate viscosity of automatic transmission fluids, gear oils, hydraulic fluids, and some lubricants. This test method covers the viscosity range of 300 mPa·s to 900 000 mPa·s

1.2 This test method was previously titled "Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer." In the lubricant industry, D2983 test results have often been referred to as "Brookfield[2] Viscosity" which implies a viscosity determined by this method.

1.3 This test method contains three procedures: Procedure A is used when only an air bath is used to cool samples in preparation for viscosity measurement. Procedure B is used when a mechanically refrigerated programmable liquid bath is used to cool samples in preparation for viscosity measurement. Procedure C is used when a mechanically refrigerated constant temperature liquid bath is used to cool samples by means of a simulated air cell (SimAir)[3] Cell in preparation for viscosity measurement.

1.4 The range of viscosity used to generate the precision data for this test method was from 300 mPa·s to 170 000 mPa·s at test temperatures from –12 °C to –40 °C. The ILS also included viscosities greater than 500 000 mPa·s and temperatures down to –55 °C and are included in Appendix X5. Appendix X4 lists another interlaboratory study that specifically targeted hydraulic fluid ranging from 500 mPa·s to 1700 mPa·s.

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

1.5.1 The test method uses the SI unit, milliPascal-second (mPa·s), as the unit of viscosity. (1 cP = 1 mPa·s).

1.6 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.7 *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.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D341 Practice for Viscosity-Temperature Charts for Liquid Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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

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[2] Brookfield viscometer and accessories are a trademark of AMETEK Brookfield, Inc, 11 Commerce Blvd., Middleboro, MA 02346, http://www.brookfieldengineering.com.

[3] SimAir is a trademark of Tannas Co., 4800 James Savage Rd., Midland, MI 48642, http://www.savantgroup.com.

[4] 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.

A Summary of Changes section appears at the end of this standard

2.2 *ISO Standard:*[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, n*—the determined viscosity obtained by use of this test method.   **D4175**

3.1.1.1 *Discussion*—Apparent viscosity may vary with the spindle speed (shear rate) of a rotational viscometer if the fluid is non-Newtonian. See Appendix X1 for a brief explanation.

3.1.2 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.1.2.1 *Discussion*—This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device. These devices are referred to as a "digital thermometer."

3.1.2.2 *Discussion*—PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blank sample, n*—a Newtonian standard reference fluid used to monitor the temperature experienced by the sample in the cold-air cabinet by inserting a thermometric device while placed in the center of the turntable; this fluid shall have a viscosity as low as possible and be changed on a regular basis.

3.2.2 *final test temperature, n*—for the programmable liquid bath is the test temperature at which the liquid bath will be held for the rest of the 16 h after the cooling profile is completed.

3.2.3 *intermediate setpoints, n*—for the programmable liquid bath are the series of setpoints the bath is taken through while the cooling profile is executing. This cooling profile calculated from A2.2 is automatically executed by the software.

3.2.4 *Procedure A*—This test protocol utilizes an air bath for the cooling portion of the test and then requires moving the test cells to either a constant liquid bath or balsa blocks during the viscosity analysis.

3.2.5 *Procedure B*—This test protocol utilizes a programmable liquid bath to cool the samples at a pre-determined rate and then the viscosity analysis is performed in the same bath.

3.2.6 *Procedure C*—This test protocol utilizes a constant liquid bath and Sim-Air cells, which allow the samples to cool at the same rate as the air bath, and be tested within the same constant liquid bath.

3.2.7 *reference viscosity, n*—viscosity of a Newtonian standard reference fluid specified at each of several user-specified temperatures. Reference viscosities of typical standard reference fluids are listed in Appendix X2.

3.2.8 *starting temperature, n*—for the programmable liquid bath is the temperature of the liquid bath at which the samples are loaded into the turn table. This is calculated from A2.2 at zero time. The software provided with the programmable liquid bath automatically calculates this value.

4. Summary of Test Method

4.1 An oleaginous fluid sample is preheated, allowed to stabilize at room temperature, and then poured to a predetermined depth into a glass cell, and an insulated or uninsulated spindle is inserted through a special stopper and suspended by a clip. An alternative sample preparation is to fill a glass cell or stator to the predetermined depth with the oleaginous fluid, an insulated or uninsulated spindle is inserted through a special stopper and suspended by a clip; then this entire sample assembly is preheated and allowed to come to room temperature. A reference fluid with a known viscosity value is also prepared. The contained sample is cooled to a predetermined temperature for 16 h and analyzed by a rotational viscometer and, depending on the viscometer model used, the viscosity of the test fluid is read directly from the viscometer or the resultant torque reading is used to calculate the viscosity of the oil at the temperature chosen. The reference fluid is used to verify the test temperature for accuracy purposes.

5. Significance and Use

5.1 The low-temperature, low-shear-rate viscosity of automatic transmission fluids, gear oils, torque and tractor fluids, and industrial and automotive hydraulic oils (see Appendix X4) are of considerable importance to the proper operation of many mechanical devices. Measurement of the viscometric properties of these oils and fluids at low temperatures is often used to specify their acceptance for service. This test method is used in a number of specifications.

5.2 Initially this test method was developed to determine whether an automatic transmission fluid (ATF) would meet OEM low temperature performance criterion originally defined using a particular model viscometer.[6] The viscosity range covered in the original ATF performance correlation studies was from less than 1000 mPa·s to more than 60 000 mPa·s. The success of the ATF correlation and the development of this test

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[6] Selby, T. W., "Automatic Transmission Fluid Viscosity at Low-Temperatures and Its Effect on Transmission Performance," *Transactions*, Society of Automotive Engineers, Vol. 68, 1960, pp. 457-465.

method has over time been applied to other fluids and lubricants such as gear oils, hydraulic fluids, and so forth.

5.3 This test method describes how to measure apparent viscosity directly without the errors associated with earlier techniques using extrapolation of experimental viscometric data obtained at higher temperatures.

NOTE 1—Low temperature viscosity values obtained by either interpolation or extrapolation of oils may be subject to errors caused by gelation and other forms of non-Newtonian response to spindle speed and torque.

6. Apparatus

6.1 *Rotational Viscometer*[7]—A rotational viscometer with a maximum torque between 0.0670 mN·m and 0.0680 mN·m and capable of sensing a change in torque of less than 0.00067 mN·m. It shall acquire torque data at a rate of at least 60 times per revolution and have a selection of spindle speeds consisting of at least 0.6 r∕min, 1.5 r∕min, 3.0 r∕min, 6.0 r∕min, 12.0 r∕min, 30.0 r∕min, and 60.0 r∕min. Additional spindle speeds of 0.3 r∕min and 120 r∕min are desirable. The viscometer is to be calibrated at least yearly.

6.2 *Viscometer Spindle*—Spindles conforming to the following dimensions (Fig. 1): A ~115 mm, B ~3.15 mm, C = 3.15 mm ± 0.03 mm, D = 31.1 mm ± 0.1 mm and made from stainless steel or a composite material that has a lower thermal conductivity. The narrow middle segment shall be ~9.5 mm in length and ~1.8 mm in diameter. In the center of the middle segment will be a narrow band that is centered in this segment and 35.6 mm ± 0.5 mm from the bottom of the spindle. The insulated spindle shown in Fig. 1 shall have a gap of ~4 mm in the upper segment, which is covered by a material with poor thermal conduction and pinned to both the upper and lower portions of the upper segment. The gap is to be placed at approximately the mid-point of upper segment. A Brookfield

(trademarked) #4B2 conforms to these requirements. The Tannas No. 4 composite spindles[8] must be used in Procedure C. Uninsulated steel spindles (No. 4) shall only be used with the Air Bath Method (Procedure A).

6.2.1 When using an insulated steel spindle, such as Brookfield No. 4B2 spindle, ensure that both steel ends are firmly connected to the insulating section. When a slight twist is given to the two metal sections on either side of the insulating cylinder, they should not move relative to each other.

6.2.2 Periodically (depending on use, but at least every 3 months) inspect spindles for run-out (wobble) when attached to the viscometer. The total run-out of the spindle shall not exceed 1 mm (0 mm ± 0.5 mm).

NOTE 2—It is good laboratory practice to store spindles in a protective manner. Do not leave composite spindles for extended periods in cleaning solvent.

6.3 *Test Stator*—A glass tube of sufficient diameter to have essentially no influence on the rotation of the spindle compared to the viscous drag of the test fluid even at viscosities above 100 000 mPa·s.

6.3.1 *Test Tube Stator*—(See Fig. 2.) A commercially standard test tube of approximately 25 mm ID and 115 mm in length, with a fill line indicating approximately 30 mL.

NOTE 3—Over time, the fill line may become difficult to see. For liquid baths, this is especially important, as it ensures that the stator is filled to a point where the spindle can be properly positioned and have the sample level below the fluid level of the bath.
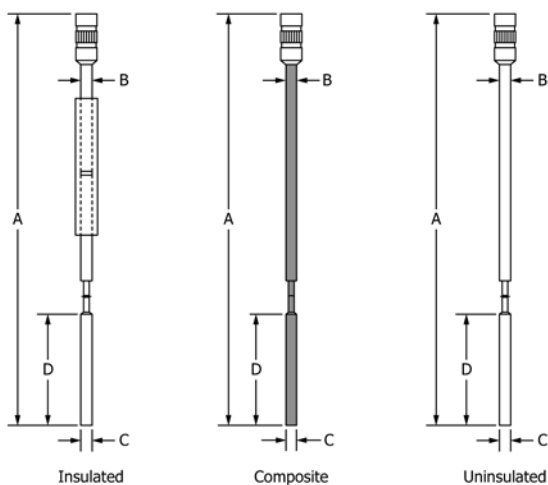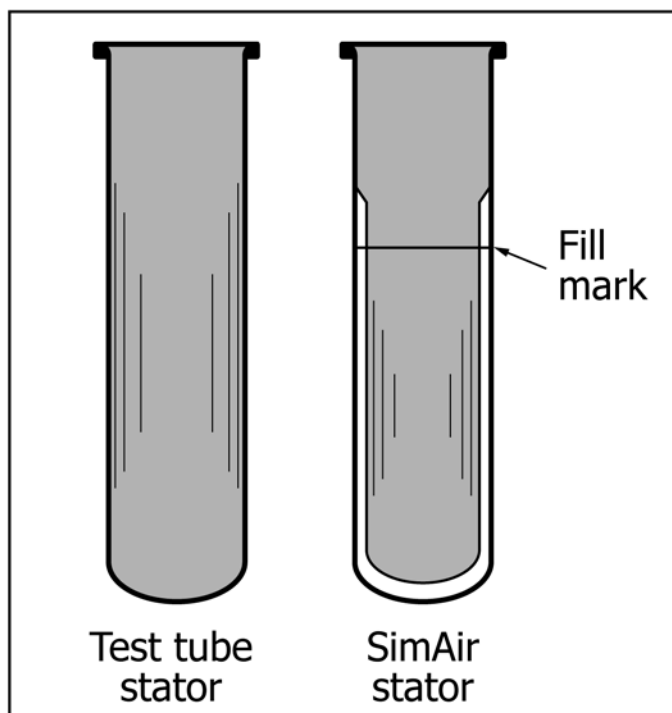
FIG. 1 D2983 Spindles

FIG. 2 Diagram of Two Forms of Stators

6.3.2 *SimAir Stator*[8]—(See Fig. 2.) The stator portion of a special air sealed cell made for this ASTM method. The inside diameter of this stator is 15 mm minimum with a fill mark 49 mm below the top of the stator. This results in a sample volume of approximately 15 mL. The outside diameter of this stator is 25.3 mm maximum.

NOTE 4—This patented cell (which also includes a composite rotor, keyed connecting device for quick spindle engagement, and cell stopper) simulates the air-bath cooling rate when inserted into a constant temperature liquid bath (see 6.8.2). The keyed connector is not essential to the test but makes spindle attachment faster with fewer disturbances of the sample.

6.4 *Cell Stopper*—An insulating cap that fits on and into the test cell with a centered hole large enough for the spindle to turn with sufficient clearance to avoid contact with the walls of the centered hole and of a height above the cell that allows a spindle clip to hold the spindle at the proper height in the test fluid during cooling. Suitable dimensions for the stopper are:

Overall height ~27 mm to ~44 mm
OD Top ~25 mm
OD Bottom ~21.7 mm
ID Bottom ~12.7 mm
ID Top ~9.4 mm
Height from bottom to change to larger OD ~16 mm
Tolerances ~±10 %

6.5 *Spindle Clip*—A clip or spacer that lies on top of the cell stopper or is affixed to the spindle and supports the spindle at proper immersion depth during cool-down.

6.6 *Insulated Cell Carrier*—(Fig. 3.) An insulated container, such as a balsa wood block or similar device, used only with cold-air cabinets to keep the test cell cold during transfer of the test cell from the cold air cabinet to the viscometer and subsequent analysis. Opposing plastic windows in the carrier side walls permit adjustment of the spindle immersion indicator for testing (see 8.4).

6.6.1 When a refrigerated liquid bath is used for final sample soak during the last half hour at analysis temperature, the insulated cell carrier is also used for sample transfer to the liquid bath and immediately returned to the cold cabinet.

6.7 *Cold-Air Cabinets*—Mechanically refrigerated cabinets with an air-circulation device and a turntable and rack for samples. The cold cabinet shall be capable of cooling the sample to any chosen test temperature from +5 °C to –40 °C and holding that temperature within ±0.3 °C. Air circulation and the sample turntable shall be able to be switched off prior to fully opening the bath top.

6.7.1 *Turntable*—This motor-driven device is used only in the cold-air cabinets. A cell rack holding the test cells is set on top of the turntable. The turntable shall rotate at a speed of 3 r∕min to 5 r∕min. This item is often supplied with the cold air cabinet.

6.7.1.1 In the case of the liquid baths, the turntable does not rotate since all samples experience a uniformly stable temperature ensured by the bath medium stirrer.

NOTE 5—To minimize disturbance and loss of cold air, it is recommended that the cabinet has an inner cover with hand-holes for sample insertion in the balsa carrier and removal of the carrier to the point of analysis.

6.8 *Liquid Baths*—Mechanically refrigerated liquid baths are used in three significantly different protocols to gain the same analytical results (see Procedures B and C, respectively, for details).

6.8.1 *Programmable Liquid Baths for Cold-Air Cabinet Cooling Simulation*—Baths capable of closely following the sample cooling in the cold-air bath as outlined in Annex A2.

6.8.1.1 *Glass Caps*—Individual glass covers for each test cell used to cover individual cells when the sample conditioning is in process.

6.8.1.2 *Turntable Cover*—This is an insulated overall cover for the turntable to prevent undue temperature upsets of the samples.

6.8.2 *Constant Temperature Liquid Baths*—Baths used to either condition the sample at the chosen final temperature after cooling in an air cabinet for 15.5 h to that temperature, or as described in Procedure C, used to receive SimAir test cells[3] at any time for analysis 16 h after the individual test sample is immersed in the bath. The liquid bath is set at the final temperature and shall be capable of holding the sample at ±0.1 °C.

NOTE 6—The SimAir cell[3] simulates the cooling curve of the air cabinet, Procedure C.

6.9 *Temperature Measuring Device*—Either a liquid-in-glass thermometer as described in 6.9.1 or a digital contact thermometer (DCT) meeting the requirements described in 6.9.2.

6.9.1 *Liquid-in-Glass Thermometer*—Use an appropriate thermometer from Table 1.
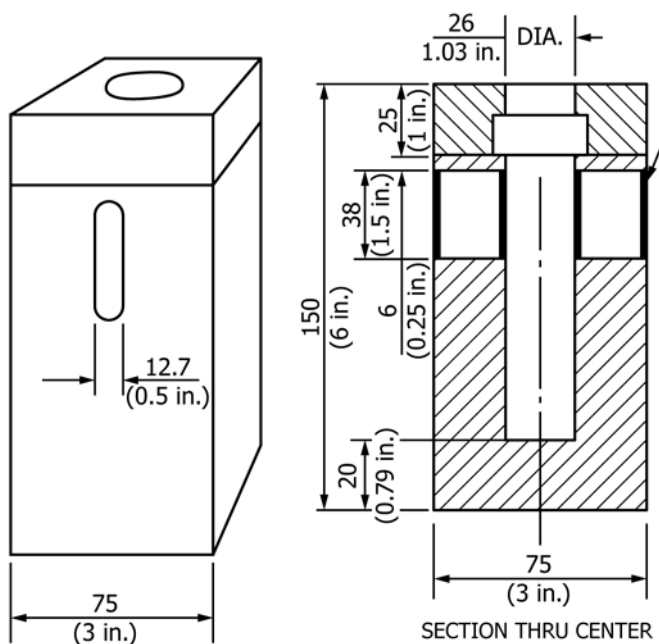
BALSA WOOD CELL CARRIER
FIG. 3 An Example of an Insulated Cell Carrier

TABLE 1 Calibrating Thermometers (see Specification E1)

IP 94C	–45 °C to –35 °C	ASTM 122C
IP 95C	–35 °C to –25 °C	ASTM 123C
IP 96C	–25 °C to –15 °C	ASTM 124C
IP 97C	–15 °C to –5 °C	ASTM 125C

TABLE 2 Chart for Spindle Speed Selection of Generic Factors

Note 1—If determined apparent viscosity is below range indicated for the selected spindle speed, use next higher spindle speed value.

Spindle Speed, r/min	Multiply torque by below number to calculate viscosity at speed selection used	Viscosity Range, mPa·s
0.6	10 000	400 000 to 1 000 000
1.5	4000	200 000 to 400 000
3.0	2000	100 000 to 200 000
6.0	1000	50 000 to 100 000
12.0	500	20 000 to 50 000
30.0	200	9800 to 20 000
60.0	100	1500 to 9800
120.0[A]	50	250 to 1500

[A] 120.0 r/min speed may not be available on some viscometer models.

6.9.2 *Digital Contact Thermometer Requirements:*

Parameter	Requirement
DCT	Guide E2877 Class D, or Class A, B, or C
Temperature Range	–60 °C to 60 °C
Display Resolution	0.1 °C minimum
Sensor Type	Platinum Resistance Thermometer (PRT)
Sensor	3 mm o.d. sheath with a sensing element less than 10 mm in length
Minimum Immersion	Not to exceed 40 mm per Practice D7962
Accuracy	±100 mK (±0.1 °C) for combined probe and sensor
Response Time	Less than or equal to 3 s as defined in Specification E1137
Drift	Less than 100 mK (0.1 °C) per year
Calibration Error	Less than 100 mK (0.1 °C) over the range of intended use.
Calibration Range	–60 °C to 20 °C or greater
Calibration Data	4 data points evenly distributed over calibration range with data included in calibration report.
Calibration Report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body.

Note 7—A DCT may not function correctly at low temperatures. Consult manual or manufacturer to determine its temperature limitations.

6.9.2.1 The DCT calibration drift shall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with 6.9.2. See Practice D7962.

Note 8—When a DCT's calibration drifts in one direction over several calibration checks, it may be an indication of deterioration of the DCT.

7. Use of Reference Fluids

7.1 This test method uses metal or composite viscometer spindles described in 6.2 (see Fig. 1). For viscometer heads on which a scale shall be read, these spindles have a table of associated generic conversion factors to permit relatively rapid calculation of the viscosity of an unknown sample. Newer digital viscometers will directly show viscosity and percent full-scale torque using these factors. The generic conversion factors for all spindles are shown in Column 2 of Table 2.

7.2 *Calibration of Spindles*—(See Annex A3 and Annex A4.) For potentially increased accuracy, spindles may be calibrated.

7.2.1 Use of standard reference fluids and technique for calibration is detailed in Annex A3 and Annex A4. This protocol was developed to provide, if desired, an option for more precise determination of the apparent viscosity measurements.

Note 9—Although the generic factors of Table 2 provide acceptable results, somewhat greater precision may be generated by this test method by calibrating spindles. Spindle calibration can also indicate problems with the viscometer that require repair to restore accuracy (see Annex A3).

Note 10—When spindles are calibrated, it is desirable to mark each spindle with some unique identification. Spindle calibration is not valid when used with a different viscometer than that used for the calibration of the spindle.

7.2.2 Concentricity of the relatively thin spindle for this test method strongly affects the resulting apparent viscosity determination. Consequently, it is recommended to calibrate spindles periodically with reference oil, particularly if run-out is observed.

Note 11—Choice of calibration reference oil and the temperature(s) at which it is used is determined by the range of viscosity and temperature required for viscosity determination. Calibration viscosities below 100 000 mPa·s are preferred and easier to use.

7.3 *Specific Use of Reference Oils to Ensure Temperature Control in Cold-Air Cabinets, Procedure A, because of Opening and Closing of the Air Cabinet Lid (Only One Reference Oil Required for Procedures B and C):*

Note 12—Opening and closing of the lid of a cold-air cabinet may influence the control of sample temperature and require more time between sample analyses to permit the cabinet temperature to be reestablished so that this is not a problem.

7.3.1 Fill two stators with the proper amount (see 8.2.1) of the same reference fluid and, when loading the sample rack (see 8.2.1), place these at the beginning and end of the sample set.

7.3.2 If, when the sample set is run, the viscosities shown by these two samples are different by more than the repeatability of the method, the discrepancy should be noted and more time allowed between each sample analyzed in subsequent sets.

7.3.3 *Optional Procedure*—Insert a DCT probe (see 6.9.2) in the reference sample. This procedure was used by some (not *all*) labs running Procedure B during the 2012 round robin study.

Procedure A

8. Procedure A—Cold Air Cabinet

8.1 *Setting the Cold-Air Cabinet Operating Temperature:* There are three different temperatures to consider: the temperature as determined by a blank sample; the cold air cabinet controller temperature; and the temperature as determined from the viscosity result for a reference fluid. Each of these will be discussed below.

8.1.1 *Temperature as Determined by Blank Sample and Associated Cold Air Cabinet Controller Temperature*—With the turntable in proper operating position but turned off, fill a stator to the required depth with the blank sample (3.2.1) and insert an appropriate temperature measuring device, see 6.9.

8.1.1.1 Place the blank sample in the center of the sample rack (turntable) to monitor temperature.

8.1.1.2 Fill a stator to the required depth with the same reference fluid as the blank sample. Place the stator in the first sample position.

8.1.1.3 Close the cold-air cabinet, turn on the cooling cycle using the temperature controller and allow at least 1 h for the cabinet to come to the test temperature as indicated by the blank sample. If it is difficult to read a thermometer, then a precision digital thermometric device can be used.

8.1.1.4 After the cold-air cabinet temperature indicator has been adjusted to reach and hold the desired temperature of the blank sample, record the indicated temperature shown by the cabinet's temperature controller. This temperature may not completely agree with the blank sample temperature.

8.1.1.5 If a cold-air cabinet temperature adjustment is necessary to bring the blank sample to the desired temperature, it is necessary to allow at least an hour or more for temperature equilibration to be re-established depending on the configuration and capacity of the particular cold-air cabinet.

8.1.2 *Temperature as Determined from Viscosity Result for a Reference Fluid:*

8.1.2.1 When setting up the temperature settings or after major maintenance, determine the viscosity of the reference fluid as per the procedure in Annex A4. Use this to determine an estimate of the apparent temperature at which the reference sample was run. If this temperature is different from the required run temperature, adjust the cabinet temperature controller setting to bring the reference fluid viscosity to within 4 % of its reference value. If this temperature is different from the required run temperature by more than 0.3 °C, then check that all components of the system are operating correctly; especially the analog or digital viscometer. If the air bath is operating correctly, all temperatures should be within 0.3 °C of each other.

NOTE 13—If more than one cold-air cabinet temperature is used for the evaluation of the low-temperature properties of oils in this test method, it will be necessary to determine these cabinet temperature settings as well.

8.2 *Preparation of Sample and Immersion in Cold-Air Cabinet:*

8.2.1 Shake the sample container thoroughly and fill the glass stator to the fill mark (see Fig. 2). If the stator does not have a fill mark, fill with appropriate amount of test oil to

permit proper use of the immersion indicator at analysis temperature (approximately 30 mL).

8.2.2 Preheat the test samples in the stator to 50 °C ± 3 °C for 30 min ± 5 min. Protectively cover each sample (such as with aluminum foil or a latex finger cot, etc.) during preheating.

NOTE 14—This preheating step has been proven important in this and other critical low-temperature ASTM test methods. The procedure is designed to remove any memory effects that may develop from previous low-temperature exposures or structure formations.

NOTE 15—Reference fluids do not require pre-conditioning; however, they should be handled in the same manner as the test fluids in all other ways. Annex A4 details the calculation of the apparent run temperature from reference fluid viscosity and r/min data.

8.2.3 It is essential that appropriate reference fluids of the approximate viscosity values be run at the beginning and end of each test series (and results recorded). This will indicate whether there was a change in sample temperature resulting from frequent opening of these cabinets.

8.2.4 If the determined viscosities of these two samples are different by more than the repeatability of the method, the discrepancy should be noted and more time allowed between each sample analyzed in subsequent sets. All samples should be re-run.

8.2.5 Remove the test cells from the pre-heating source and allow them to cool to room temperature and then remove the covers. (Use care in handling the hot stators.)

8.2.6 Place the cell stopper on the stator with the spindle supported by the spindle clip.

8.2.7 The spindle immersion mark (see Fig. 1) should be slightly below the liquid surface (to allow for contraction of the oil sample upon cooling to the temperature of analysis).

NOTE 16—This reduces the amount of sample disturbance before viscosity measurement.

8.2.8 Two samples of each fluid are required.

NOTE 17—There is some susceptibility to sample heating in the process of adjusting the spindle speed for best sensitivity during analysis. For greater accuracy when using cold-air baths and insulated cell carriers, it has become a practice to run two samples of the same fluid; the first to determine best spindle speed, and the second to apply that speed to obtain the viscometric information. Subsequently the second value is chosen.

8.2.9 Place the test cells into the turntable sample rack with a reference fluid sample at the beginning and end of the set of samples. Also place the blank sample (see 3.2.1) in the center position of the rack then place the temperature sensor in it.

8.2.10 Place as many insulated cell carriers (see Fig. 3) within the cold-air cabinet in positions so that they will not unduly restrict airflow around the test samples within the air chamber. Take care to ensure that no insulated cell carrier is placed so it restricts the exit holes for air in the plenum (back wall of air chamber). Close the cabinet lid and turn both the turntable and air blower on.

8.2.11 Cool the samples and insulated cell carriers for 16 h.

8.3 *Using a Liquid Bath for Final Soak and Analysis after Conditioning Samples in an Air-Bath:*

8.3.1 When using a constant temperature liquid bath for the final soak, it is not necessary to use initial and final reference oils as in 8.2.4. Only an initial viscosity value is necessary for

analysis and is not to be used to adjust temperature; but to serve as a guide to know if everything is running accurately in the combined system (that is, temperature, viscometer, spindles, etc.). If the viscosity of the reference oil is not within the precision limits, the test shall be repeated with any necessary mechanical corrections made.

8.3.2 Set liquid bath temperature to that desired for final half-hour soak 2 h before using bath. Make certain that the bath temperature is stable using an appropriate temperature measuring device, see 6.9.

8.3.3 For ensuring proper calibration of the final soak liquid bath, it is recommended it be checked that the reference oil transferred from a properly calibrated air chamber give acceptable viscosity values. If the viscosity values are not acceptable, then the bath temperature should be adjusted and the procedure repeated until acceptable viscosity values are obtained.

8.3.4 Proceed to Section 11 for the setup of the viscometer and selection of spindle speed.

8.4 *Analytical Protocol for Cold-Air Cabinets:*

8.4.1 On completion of the 16 h cold exposure of the samples, check the level of the viscometer to ensure that the drive shaft is vertical (see 11.1) and re-zero (see 11.1.2 to 11.1.3).

8.4.2 Individually transfer and analyze the test samples as follows:

8.4.2.1 Note the cabinet controller temperature. If it is not at the desired temperature as per 8.3.2, adjust the cold-air cabinet. Wait at least 1 h while the cabinet comes to the desired temperature before initiating analysis.

8.4.2.2 Analyze each sample in turn by first turning off the turntable rotation and the air blower. Some cabinets may be designed with a low setting on the blower that can also be used at this time. Different systems may require a different time allowance for shutting off the blower motor and opening the cabinet door. Allow the operator to determine the appropriate time to open the cabinet door.

8.4.2.3 Open the cold-air cabinet and put one temperature-conditioned test cell into a temperature-conditioned insulated cell carrier and remove the now-insulated cell from the cold-air cabinet for analysis. Do not remove more than one sample at a time. Note the temperature of the blank sample; it may not change by more than 0.3 °C when the cabinet is opened.

8.4.2.4 Immediately close the cold-air cabinet lid and restart the turntable and air blower.

8.4.2.5 Transfer the insulated cell carrier and the sample to the viscometer.

8.4.2.6 Place the test cell below the viscometer and align the spindle nut with the viscometer coupling nut. Attach the spindle using a quick attachment device for minimal disturbance of the sample or by screwing the spindle onto the drive shaft thread. Note that this connection is made with a left-handed thread.

8.4.2.7 Remove the spindle clip.

8.4.2.8 Adjust the spindle height by the vertical adjustment knob on the viscometer rack until the spindle immersion indicator (see Fig. 1) is even with the oil level. To facilitate the adjustment of the spindle immersion indicator, place a relatively cool light source, such as a flashlight or diode light,

behind one window of the test cell carrier and observe the spindle position through the other.

NOTE 18—Take care to ensure proper depth of spindle immersion with all samples. Maintenance of proper immersion depth is essential to good reproducibility and repeatability. Data have shown that an immersion variation of as little as 1.2 mm from the immersion mark can produce viscosity errors.

8.4.2.9 Center the spindle in the hole at the top of the cell stopper so that no part of the spindle touches the stopper hole during the measurement process.

8.4.2.10 Proceed to Section 11 for the setup of the viscometer and selection of spindle speed.

Procedure B

9. Procedure B: Programmable Liquid Bath

9.1 *Setting the Programmable Liquid Bath Operating Temperature:*

9.1.1 For the programmable liquid bath, the samples are to be loaded into the turntable at a starting temperature as dictated by Annex A2. The software provided with the programmable liquid bath automatically calculates this starting temperature and displays it as the starting setpoint when the test temperature is entered into the software. When the bath has attained the starting temperature, the samples are loaded and the thermal conditioning program is started (see 9.2.9). The software controls the cooling profile as dictated by Annex A2.

9.1.2 *Procedure for Calibrating the Programmable Liquid Bath:*

9.1.2.1 The calibration of the programmable liquid bath is recommended when the instrument is put into service for the first time or after major maintenance. Three samples of the same reference oil of known viscosity are loaded into the turntable at the starting temperature as discussed in 9.2 to 9.2.9 below. After 1 h the viscosity of the first reference sample is analyzed. If the viscosity values are not in the acceptable range, then the bath set point is suitably adjusted and the next reference sample is analyzed after 1 h. This procedure is repeated until the proper bath temperature offset is obtained. Offset values at various test temperatures may be obtained by this procedure and the value installed in the calibration grid of the software. For future runs, the software automatically finds and applies the pertinent calibrations for the relevant test temperatures.

9.2 *Preparation of Sample and Immersion in a Liquid Programmable Bath:*

9.2.1 Shake the sample container thoroughly and fill the glass stator to the fill mark (see Fig. 2). If the stator does not have a fill mark, fill with appropriate amount of test oil to permit proper use of the immersion indicator (see Fig. 1) at analysis temperature. One reference oil sample must be tested for every run to ensure that test conditions are in control according to the test precision.

NOTE 19—Reference fluids do not require pre-conditioning; however, they should be handled in the same manner as the test fluids in all other ways. Annex A4 details the calculation of the apparent run temperature from reference fluid viscosity and spindle speed data.

9.2.2 Preheat the test samples in the stator to 50 °C ± 3 °C for 30 min ± 5 min. Protectively cover each sample (such as with aluminum foil or a latex finger cot, etc.) during preheating.

NOTE 20—This preheating step has been proven to be important in this and other critical low-temperature ASTM test methods. The procedure is designed to remove any memory effects that may develop from previous low-temperature exposures or structure formations.

9.2.3 Remove the test cells from the pre-heating source and allow them to cool to room temperature and then remove the covers. (Use care in handling the hot stators.)

9.2.4 Place the cell stopper on the stator with the spindle supported by the spindle clip.

9.2.5 The spindle immersion mark (see Fig. 1) should be slightly below the liquid surface (to allow for contraction of the oil sample upon cooling to the temperature of analysis).

NOTE 21—This reduces the amount of sample disturbance before viscosity measurement.
NOTE 22—Handle and store the spindles and instrument with care at all times. For greatest precision and accuracy, check the calibration of each spindle periodically with reference oil. Do not use any damaged or noticeably bent spindles (see 6.2.2).

9.2.6 On the software screen, select the desired test temperature. The starting temperature is calculated and installed as the setpoint which the bath rapidly attains.

9.2.7 Temperature of the bath shall be monitored by a temperature measuring device suitably placed near the test cells (see 6.9).

9.2.8 When the bath has stabilized at the starting temperature, place preheated and assembled sample-containing test cells in the bath. The remaining positions of the turntable should all be occupied by test cells even if empty. This will ensure that the bath medium will be at the proper level with respect to the samples in the test cell. This ensures proper temperature conditioning of samples. Cover the test cells with the glass caps and then with the turntable cover. Start the cooling profile on the software. One reference oil sample must be tested for every run to ensure that test conditions are in control according to the test precision. If the viscosity of the reference oil is not within the precision limits, the test shall be repeated with any necessary corrections made.

9.2.9 The established technique is: After 15 h of cooling, check the bath temperature with a temperature measuring device suitably placed (6.9) near the test cells. If the temperature is not within ±0.1 °C but still within ±0.3 °C, adjust the temperature to the correct temperature and wait 1 h before testing. If the temperature is still not within ±0.3 °C, the temperature shall be corrected and the entire test repeated.

9.2.10 Analyze each test sample 16 h after its immersion according to Section 11.

NOTE 23—When using a constant temperature liquid bath to hold the test sample at temperature, there are no time restraints as in 11.4 on obtaining the torque reading with the exception that the whole series of tests needs to be completed in 2 h so that the maximum sample exposure time of 18 h is not exceeded.
NOTE 24—The following technique was evaluated by the ILS: After 15 h of conditioning, check the temperature of the reference sample using a certified thermometric device with a sensor closely resembling a 4B2 spindle in geometry, or equivalent. If the temperature is not within ±

0.1 °C but still within ± 0.3 °C, adjust the temperature to the correct temperature and wait 1 h before testing. If the temperature is still not within ± 0.1 °C, the temperature shall be corrected and the entire test repeated. The bath temperature will also be noted. This thermometric spindle shaped device can be purchased from Lawler Manufacturing, Inc.

Procedure C

10. Procedure C: SimAir Stators in a Constant Liquid Bath

10.1 *Procedure for Calibrating the Liquid Bath:*

10.1.1 The calibration of the liquid bath is recommended when the instrument is put into service for the first time, after a preventative maintenance or after major maintenance. Set the bath for –40 °C. After the unit has stabilized for 1 h at temperature, carefully measure the temperature using an appropriate temperature measuring device (see 6.9). Adjust the temperature of the bath according to the device. Do this for each temperature where measurements are required and log the offset for each. This will save time during testing and analysis.

10.2 *Preparation of Sample and Immersion in SimAir:*

10.2.1 Shake the sample container thoroughly and fill the glass stator to the fill mark (see Fig. 2). If the stator does not have a fill mark, fill with appropriate amount of test oil to permit proper use of the immersion indicator at analysis temperature (approximately 15 mL). One reference oil sample must be tested for every run to ensure that test conditions are in control according to the test precision.

10.2.2 Set the bath controller to the desired test temperature. The apparent run temperature itself should be within ±0.3 °C of the set test temperature.

NOTE 25—Reference fluids do not require pre-conditioning; however, they should be handled in the same manner as the test fluids in all other ways. Annex A4 details the calculation of the apparent run temperature from reference fluid viscosity and spindle speed data.

10.2.3 Preheat the test samples in the stator to 50 °C ± 3 °C for 30 min ± 5 min. Protectively cover each sample (such as with aluminum foil or a latex finger cot, etc.) during preheating.

NOTE 26—This preheating step has been proven to be important in this and other critical low-temperature ASTM test methods. The procedure is designed to remove any memory effects that may develop from previous low-temperature exposures or structure formations.

10.2.4 Remove the test cells from the pre-heating source and allow them to cool to room temperature and then remove the covers. (Use care in handling the hot stators).

10.2.5 Place the cell stopper on the stator with the spindle supported by the spindle clip.

10.2.6 The spindle immersion indicator (see Fig. 1) should be slightly below the liquid surface (to allow for contraction of the oil sample upon cooling to the temperature of analysis).

NOTE 27—This reduces the amount of sample disturbance before viscosity measurement.
NOTE 28—Handle and store the spindles and instrument with care at all times. Do not use any damaged or noticeably bent spindles (see 6.2.2).

10.2.7 Place the test cells into the turntable sample rack in the bath with a reference fluid sample at the beginning of the set of samples.

10.2.8 After 15 h of conditioning, check the bath temperature with an appropriate temperature measuring device (6.9) placed near the test cells. If the temperature is not within ±0.1 °C but still within ±0.3 °C, adjust the temperature to the correct temperature and wait 1 h before testing. If the temperature is still not within ±0.3 °C, the temperature shall be corrected and the entire test repeated.

10.2.9 Analyze each test sample 16 h after its immersion according to Section 11 but not to exceed 18 h. Note that the first sample to test is the reference sample to make sure it is within the test precision. If the viscosity of the reference oil is not within the precision limits, the test shall be repeated with any necessary corrections made.

10.2.10 Calculate viscosity of the test sample as in Section 12.

Note 29—When using a constant temperature liquid bath to hold the test sample at temperature, there are no time restraints as in 11.4 on obtaining the torque reading with the exception that the whole series of tests needs to be completed in 2 h so that the maximum sample exposure time of 18 h is not exceeded.

10.3 Upon completion of testing using any of the four protocols, empty the cells and clean all parts with a suitable hydrocarbon solvent making sure all parts are clean and free of oil.

11. Preparation of the Viscometer (Common to All Bath Procedures)

11.1 Vertically align the viscometer by centering the bubble in the bubble level located on the viscometer.

Note 30—It is important that the viscometer be vertical during measurement, and it is good practice to periodically re-check this level during a set of analyzing test samples

11.1.1 After turning on the power, allow approximately 30 min for the viscometer electronics to warm up, then zero the viscometer with no spindle attached.

11.1.2 For analog viscometers, when making a viscosity reading, use the percent full scale torque reading and multiply this reading by either applying the general approximate factor shown for each speed in Table 2 or, if the spindle is calibrated using Annex A3, for greatest accuracy and precision, use the individual spindle calibration factor so obtained.

11.1.3 For digital viscometers, ensure that the correct spindle speed is selected following the viscometer manufacturer's instructions. **Failure to follow the manufacturer's instructions will likely lead to incorrect measurements.**

11.1.4 The percent torque is used to adjust the spindle speed to an appropriate level for making a viscosity reading with a test or reference fluid.

11.2 *Selection of Spindle Speed and Taking Readings from the Viscometer:*

11.2.1 Again, make certain that the immersion indicator on the spindle is level with the meniscus of the oil.

11.2.2 When the approximate viscosity is known, refer to Table 2 for the expected spindle speed setting that will generate the highest torque reading on the viscometer. This is to be between 20 % and 80 % (see Section 7) for analog instruments and 20 % to 90 % for digital instruments. This table will indicate the proper speed setting if the viscosity is known.

11.2.3 For cold-air cabinets, transfer time and adjustment of the spindle speed may permit slight sample warming. For this reason, if the expected viscosity range of the sample is unknown, a first sample shall be used to determine the highest r/min that gives an acceptable viscometer reading. This is accomplished by increasing speed in steps from 0.6 r/min to 120 r/min. The second sample is then run at the previously determined speed and only this result is reported.

11.2.4 Since fluids may be non-Newtonian at low temperatures, the spindle speed selected for measuring the viscosity of a test fluid can strongly influence the measured viscosity (see Appendix X1). Unless the approximate viscosity is known, it is important to begin at the lowest speed and work up, if possible, to a speed within the range noted in 11.2.2. In these cases, start with a spindle speed set to 0.6 r/min. If the measurements start at a higher speed and are followed by measurements at a lower speed, the result will be invalid.

11.2.5 Turn on the viscometer motor and note the torque level. The torque reading should be between approximately 20 % to 80 % full scale on the viscometer (50 % is optimal). Analog viscometers only read in percent full scale torque while some digital viscometers simultaneously present units of viscosity in milliPascal-second (mPa·s) and percent full scale torque. If the value is below 20 %, increase the speeds until the torque reading is as noted above.

11.3 After the spindle speed has been optimized, record the stabilized torque reading observed between 60 s and 180 s. For analog instruments, utilize the factor given in Table 2 for the rotational speed used and calculate the viscosity using Eq 1. For a digital viscometer with a display that shows the viscosity, simply read the stabilized viscosity and torque between 60 s and 180 s.

Viscosity = (Table 2 factor for spindle speed used)·% Torque (1)

11.4 When using cold-air cabinets, for the greatest precision, testing should be started within 60 s after the sample is removed from the cold-air cabinet.

11.5 Each viscosity measurement shall be completed in 180 s or less once the motor is started and an optimum speed is established. Use the highest viscosity for the recording during the time period for measurement.

Note 31—The opening and closing of the lid on the cold-air cabinet may influence the control of sample temperature and require more time between sample analyses to permit the cabinet temperature to be reestablished so that this is not a problem.

11.5.1 For the cold air cabinet, determine the viscosities of the two reference fluids that bracket the samples. If the result from either fluid is different from the reference viscosity by more than the repeatability of the method, then the entire sample set is considered to be suspect. All other units shall measure the viscosity of the reference compared to the precision of that procedure. If the viscosity of the reference oil is not within the precision limits, the test shall be repeated with any necessary corrections made.

Note 32—Digital viscometers may have the ability to operate at 120 r/min. Analog viscometer units are typically limited to 60 r/min.

Note 33—For measurement of lower viscosity fluids such as hydraulic fluids, it is even more important to calibrate spindles using the protocol of

Appendix X3 as well as to use two samples of the test fluid when using a refrigerated air bath.

11.6 Record viscosity reading (mPa·s) or percent torque reading, spindle speed (r/min), test temperature (°C) and the Procedure used: A, B, or C.

11.7 Upon completion of testing using any of the four protocols, empty the cells and clean all parts with a suitable hydrocarbon solvent making sure all parts are clean and free of oil.

12. Calculation

12.1 Calculate the viscosity at the test temperature of the test oil or reference oil as shown in 11.3.

12.2 If required by the user, the shear stress and rate at the surface of the spindle may be estimated by the procedure in Appendix X3.

13. Report

13.1 A routine report includes the fluid identification, the determined viscosity, the test temperature (°C), the Procedure used (A, B, or C), and the spindle speed. Spindle speed data are needed to ensure that different laboratories use the same shear rates. Data where the reference fluid values are beyond the precision of the method shall not be reported.

13.2 In cases where this test method is used for reference testing between laboratories, a full report including the identity of the Newtonian reference fluid used in bracketing the test fluid, its reference viscosity at the temperature of measurement, its apparent viscosity through measurement by the laboratory, the calibration factor for the spindle (see Annex A3) and the spindle speed at which the test was run shall accompany the test fluid data of Section 9.

Note 34—Reference fluid data are needed to ensure that different laboratories run at the same temperature, shear rate, and viscometric conditions.

14. Precision and Bias[9]

14.1 *Precision*—The precision of this test method using the air bath, programmable liquid, or SimAir is determined by statistical examination of the interlaboratory test results over the temperature range from –12 °C to –40 °C with measurements obtained from 60 s to 180 s and a viscosity range from 300 mPa·s to 170 000 mPa·s, and, based on the following assumptions and conditions:

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[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1821. Contact ASTM Customer Service at service@astm.org.

(1) No separate precision statement is necessary for each procedure.

(2) No separate precision statement is necessary for each ILS test temperature.

(3) Exclusion of 1 % of measurements identified as odd.

(4) Exclusion of samples near/beyond the ILS instrument measurement threshold.

(5) Inclusion of range of results from 60 s to 180 s.

14.1.1 *Repeatability Limit (r)*—The difference between two results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20:

$$r = 0.1348 \cdot X \tag{2}$$

where:

X = average of the two results.

14.1.2 *Reproducibility Limit (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20:

$$R = 0.1814 \cdot X \tag{3}$$

where:

X = average of the two results.

14.2 *General Considerations:*

14.2.1 *Summary of Interlaboratory Study*—The interlaboratory study (ILS 0797) consisted of 16 participating laboratories, 11 samples with viscosities ranging from 300 mPa·s to 170 000 mPa·s at test temperatures from –12 °C to –40 °C. The ILS also included viscosities greater than 500 000 mPa·s and temperatures down to –55 °C and are included in Appendix X5. More specific precision for viscosities within the range of 500 mPa·s to 1700 mPa·s, such as hydraulic fluids, are shown in a separate interlaboratory study depicted in Appendix X4.

14.2.2 Both analog and digital Brookfield viscometers with an upper limit of 0.0673 mN·m were used for analysis with both calibrated and uncalibrated spindles.

14.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

15. Keywords

15.1 lubricants; Newtonian oils or liquids; Non-Newtonian oils or fluid

ANNEXES

(Mandatory Information)

A1. USE OF THE D2983 VISCOSITY VERSUS TEMPERATURE RELATIONSHIP INTERPOLATION OF DATA

A1.1 In some cases, a viscosity at a single temperature may not adequately define the low temperature, low-shear-rate behavior of an automotive lubricant fluid. In those cases, viscosity values are often taken at other temperatures and viscosity-temperature plots made.

A1.2 Viscosity-temperature plots are made by plotting the viscosity measured for at least three temperatures on ASTM viscosity-temperature paper (see Practice D341) as a smooth curve.

A1.2.1 Viscosities are generated at the highest spindle speed capable of giving a reading on the analog or digital model viscometer used (see Section 9).

A1.2.2 Comparison of data from analog and digital viscometers should not be made at different spindle speeds.

A1.3 When viscosity versus temperature plots are used to generate interpolated viscosity values, the interpolated viscosity shall be within the temperature range of measured viscosities of the fluid.

A1.3.1 Extrapolation beyond the range of measured viscosity data should be avoided because automotive fluids may form gelated structures unexpectedly at low temperature.

NOTE A1.1—The temperature at which gelation will occur is predictable by the use of Test Method D5133 another low-temperature, low-shear viscosity method that uses a rotational viscometer with a spindle of larger diameter that more closely fits a glass stator.

A1.3.2 When the plotted viscosities are compared between laboratories, the data used shall be at the same spindle speeds that have sufficient sensitivity and at the same temperature.

NOTE A1.2—Because of non-Newtonian behavior, viscosity is dependent on spindle speed. If viscosity measurements are taken at different spindle speeds at different laboratories, considerably different viscosities may result with obvious confusion.

A2. TYPICAL SAMPLE COOLING RATES IN D2983 AIR CABINETS

A2.1 This annex is intended to serve as a guide to refrigerated cabinet manufacturers.

A2.2 Sample cooling rates in these cabinets are considered important because the development of gel structures of some automotive fluids is time dependent and thus dependent on the rate of cooling. This gel structure influences apparent low-temperature, low-shear viscosity.

A2.3 The temperature of a sample immersed in a precooled cabinet should follow the equation:

$$\frac{dS}{dt} = k(S - B) \tag{A2.1}$$

where:
S = sample temperature at the time of observation,
t = elapsed time from start of cooling,
B = cabinet temperature, and
k = cooling constant in units of time^{-1}.
Eq A2.1 solves to:

$$(S - B) = Ce^{kt} \tag{A2.2}$$

where:
C = integration constant, and
e = base Napierian logarithms 2.7182.

Eq A2.2 may be written in a logarithmic form for convenience of plotting as:

$$\ln(S - B) = \ln C + kt \tag{A2.3}$$

A2.4 When temperature is in degrees Fahrenheit, a sample temperature in an average air cabinet can be calculated using the following constants: k is typically –0.08 with a range between –0.12 and –0.040 and ln C is typically between 4.45 and 4.80. Cabinets that cool samples at rates defined by these limits and meet other method requirements are suitable for use.

A2.5 The temperature tolerance for the cooling rate on programmable liquid baths is ±1 °C for temperatures down to –30 °C; for temperatures below –30 °C, the temperature tolerance is ±0.5 °C.

A2.6 See Table A2.1 for bath cooling rate data at –12 °C, –26 °C, –40 °C, and –55 °C.

TABLE A2.1 Bath Cooling Rate Data

Test Temp: −12 °C		Test Temp: −26 °C		Test Temp: −40 °C		Test Temp: −55 °C	
Time (min)	Bath Temp. (°C)	Time (min)	Bath Temp. (°C)	Time (min)	Bath Temp. (°C)	Time (min)	Bath Temp. (°C)
0	11.5	0	6.8	0	2.2	0	−2.7
5	3.7	5	−4.0	5	−11.7	5	−20.0
10	−1.5	10	−11.2	10	−21.0	10	−31.5
15	−4.9	15	−16.1	15	−27.3	15	−39.3
20	−7.3	20	−19.4	20	−31.5	20	−44.4
25	−8.8	25	−21.6	25	−34.5	25	−47.9
30	−9.9	30	−23.0	30	−36.2	30	−50.3
35	−10.6	35	−24.0	35	−37.4	35	−51.8
40	−11.0	40	−24.7	40	−38.3	40	−52.9
45	−11.4	45	−25.1	45	−38.8	45	−53.6
50	−11.6	50	−25.4	50	−39.2	50	−54.0
55	−11.7	55	−25.6	55	−39.5	55	−54.4
60	−11.8	60	−25.7	60	−39.7	60	−54.6
65	−11.9	65	−25.8	65	−39.8	65	−54.7
80	−12.0	70	−25.9	75	−39.9	70	−54.8
		85	−26.0	85	−40.0	75	−54.9
						90	−55.0

A3. CALIBRATION OF SPINDLES WITH A NEWTONIAN REFERENCE FLUID

A3.1 This annex provides the steps for calibrating new or used spindles for determination of D2983 viscosity at low temperatures.

A3.2 Test data have shown that the general factors in Table 2 are acceptable when the spindle is in good condition. However, spindles may sustain some permanent distortion in use. If the run-out becomes excessive (see 6.2.2), the spindle must be discarded or straightened before use.

Note A3.1—Examples of agreement between the general factors of Table 2 and the calibration-derived factors.

Spindles A, B, C in Table A3.1 have acceptable run-out (that is, no more than 1 mm ± 0.5 mm run out). Agreement between the general factors in Column 6 and the calibration-derived factor divided by r/min in Column 10 is good as shown by percent error of each when compared to the known viscosity shown in Column 2 at the temperature in Column 3. Errors range from 0.0 % to −9.4 % for the general factor and from 0.1 % to −2.7 % after calibration.

A3.3 Choice and Use of Reference Fluid for Calibration

A3.3.1 Choose a Newtonian reference oil having a known viscosity-temperature range covering that desired for testing unknown fluid samples.

A3.3.1.1 The chosen Newtonian reference oil will be effective at all temperatures and viscosities over the low temperature range given on the label because of the Newtonian nature of the fluid.

A3.4 Preferably, if available, calibrate the spindle in a well temperature controlled refrigerated liquid bath at a low temperature shown on the label of the calibration fluid.

Note A3.2—Cold-air cabinets may be used but generate less precise calibrations because of warming that occurs during the time it takes to collect the calibration data.

A3.5 Calibration Technique

Note A3.3—It is productive to calibrate a number of spindles at the same time using the same type of calibration fluid.

A3.5.1 Permanently mark the spindles to be calibrated for subsequent identification over their useful life.

A3.5.2 Fill the test cells to the appropriate level with the calibration fluid. No pre-conditioning of the fluid is necessary because of the Newtonian character of the fluid (see Note 20).

A3.5.3 Bring the refrigerated liquid bath or the cold-air cabinet to the desired temperature.

A3.5.4 Allow the calibration test cells to soak at calibration temperature for 2 h in the refrigerated liquid bath. However, it is necessary to have the samples soak overnight in the cold-air cabinet.

A3.5.5 Using the techniques described in this test method, attach the spindle to the viscometer and set the immersion indicator at the surface of the calibration fluid.

A3.5.6 With refrigerated liquid baths, measure the percent full-scale torque at five speeds capable of giving torque responses from 5 % to 90 % of full scale.

Note A3.4—It is necessary to use percent full-scale torque indicated rather than the indicated viscosity since correction of any error in the latter is the purpose of the calibration technique.

A3.5.6.1 When using a cold-air cabinet, measure the percent full-scale torque at three speeds giving torque responses from 10 % to 90 % of full scale. All three values should be taken within a minute of starting the analysis at the particular speed chosen. With the potential for slight warming of the sample, start at the highest speed giving an on-scale torque

reading and reduce the speed to one giving a mid-scale reading and another in the range of 5 % to 20 %.

NOTE A3.5—It is preferable for calibration of the spindle to return the reference oil cell to the air cabinet after each speed and allowing the sample to re-cool and then to re-analyze at the next desired speed.

A3.6 Calculation and Use of Calibration Factor

A3.6.1 Using a spread-sheet computer program such as Excel (or equivalent) or a statistical least squares analysis of the data, obtain the equation of the best straight line through the collected percent torque and spindle speed data as shown in Fig. A3.1.

A3.6.1.1 A plot of torque versus speed should result in the linear relationship shown in Fig. A3.1 and further corroborated by a value above a Coefficient of Determination, R^2, of 0.999 showing high linear interdependence of the two values.

NOTE A3.6—The high value of R^2 indicates the ability of the Brookfield viscometer to give precise results over a broad range of speed and torque.

A3.6.1.2 The graphical analysis of Fig. A3.1 also shows an intercept of 0.0993 % torque. This desirably low value indicates only a desirably small level of mechanical friction in the viscometer head.

NOTE A3.7—If the calibration has been properly done, a large value for the intercept (an intercept value of 2 or higher) suggests a problem with the viscometer and a likely need for inspection and perhaps cleaning or repair.

A3.6.2 Dividing the known viscosity of the calibration reference oil by the slope of the best line through the data (by linear regression analysis) provides the calibration factor:

Calibration Factor = Viscosity of the Calibration Oil ÷ Slope of Regression Line

NOTE A3.8—Example: A calibration oil with a known viscosity of 20 800 mPa·s is tested. The data are plotted on axes of percent full-scale torque versus rotational speed, and the slope of the regression line is determined to be 3.2127. The calibration factor is then calculated to be 20 800 ÷ 3.2127 = 6474.

A3.6.3 The calibration factor determined in this way is applicable at all temperatures and viscosities covered in this test method (between –18 °C and –55 °C, and viscosities between 300 mPa·s and 1 180 000 mPa·s).

A3.6.4 To calculate the viscosity of an unknown test fluid using the spindle's calibration factor, apply the following equation:

Viscosity = Calibration Factor · % Full-Scale Torque ÷Spindle speed, r/min

NOTE A3.9—Example: Viscosity = 6474 · 36.4 ÷ 12.0 = 19 600 mPa·s

A3.6.5 Another use of a calibrated spindle is in measuring a fluid of known viscosity to determine if the temperature of the cold-air cabinet or refrigerated liquid bath is correct.

NOTE A3.10—It is recommended that spindles be calibrated periodically and the values recorded to determine any significant changes in either the individual spindles, in the bath control, or in the viscometer with the passage of time.

TABLE A3.1 Spindle Calibration to Improve Accuracy and Recover Spindle Use

| Spindle Number	Known Viscosity, mPa·s	Analysis Temp., °C	Spindle Speed	% Full-Scale Torque	General Factor at r/min Used	Using Generic Factor, mPa·s	% Error	Calibration Factor	Calibration Factor, r/min	Using Calibration Factor, mPa·s	% Error
1	2	3	4	5	6	7	8	9	10	11	12
A	20 794	−40	12	38.2	500	19 200	−7.7	6472	539	20 603	−0.9
B			12	40.9		20 600	−0.9	6018	502	20 525	−1.3
C			12	41.5		20 800	0.0	6019	502	20 805	0.1
A	40 439	−20	6	38.3	1000	38 200	−5.5	6472	1079	41 098	1.6
B			6	39.6		40 000	−1.1	6018	1003	40 085	−0.9
C			6	40.7		40 800	0.9	6019	1003	41 020	1.4
A	12 916	−10	12	23.3	500	11 700	−9.4	6472	539	12 567	−2.7
B			12	25.7		13 000	0.7	6018	502	13 040	1.0
C			12	26.4		13 300	3.0	6019	502	13 231	2.4
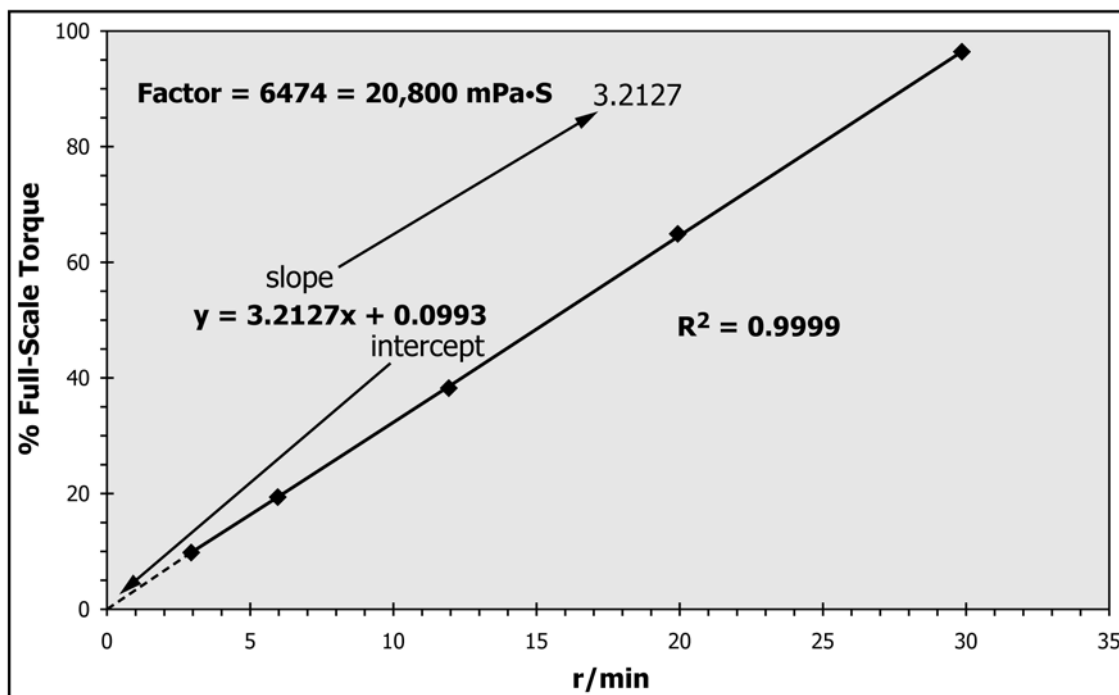
FIG. A3.1 Calibration of Spindle with Newtonian Oil of 20 800 mPa·s Viscosity at Calibration Temperature

A4. ESTIMATION OF APPARENT RUN TEMPERATURE FROM OBSERVED D2983 VISCOSITY OF A NEWTONIAN REFERENCE FLUID

A4.1 This annex provides a way to estimate the apparent temperature at which the reference sample was run. Although the most probable cause of significant deviation between the set test temperature and calculated apparent run temperature is error in the temperature control and monitoring system, errors in spindle immersion depth and viscometer malfunction can also cause noticeable deviations. If viscometer function and spindle immersion depth are satisfactory, then the calculated deviation between set and apparent run temperature is a measure of the size of the temperature control and monitoring error.

A4.2 Known Calculation Constants

A4.2.1 The viscosity-temperature function of the standard reference fluid is listed on the label.

A4.2.2 Spindle calibration factors are in Section 8.

A4.2.3 The percent Full-Scale Torque dial reading and Spindle Speed in r/min for the standard reference fluid are observed.

A4.2.4 The set test temperature is a defined test condition.

A4.3 Calculations

A4.3.1 Determine the D2983 viscosity of the test fluid:

Viscosity = observed dial reading × Calibration factor

A4.3.2 Using two reference fluid viscosities at two temperatures near the set test temperature, calculate Z for each reference fluid viscosity with Eq. A1.2 in the appendix of Test Method D341.

A4.3.3 Determine constants A and B from Eq. A1.1 in the appendix of Test Method D341, using calculated values of Z and the temperature to which each value of Z corresponds.

A4.3.4 Use the following form of Eq. A1.1 in the appendix of Test Method D341 to calculate T, the apparent run temperature in °C.

$$T = 10^{\left(\frac{A - \log\left(\log\ Z\right)}{B}\right)} - 273.15 \qquad (A4.1)$$

A4.3.5 Calculate $T_{deviation} = T -$ set test temperature.

A4.4 Example

A4.4.1 Set temperature, −34.4 °C
Observed dial reading at 12 r/min, 49.5
Reference fluid viscosity at −28.9 °C, 11 360
Reference fluid viscosity at −34.4 °C, 28 580
Spindle factor at 12 r/min, 500
D2983 viscosity = 49.5 × 500 = 24 750
from the appendix charts of Test Method D341,

Eq. A1.2 Z (−34.4 °C)   = 28 580.7
Eq. A1.2 Z (−28.9 °C)   = 11 360.7
Eq. A1.1 A = 10.48607
Eq. A1.1 B = 4.13682
Eq. A1.2 Z observed   = 24 750.7

T = (antilog (10.48607 − log log 24 750.7)/4.13682)
− 273.15
T = −33.58 °C

$T_{deviation}$ = 0.86 °C

A4.5 *Interpretation*—The 0.86 °C temperature deviation from set point is more than twice the allowed 0.3 °C bath temperature variation. Probable temperature control or measurement error is indicated. However, possible severe spindle immersion depth error or viscometer malfunction should also be checked. Data from samples run in this test series should not be reported.

APPENDIXES

(Nonmandatory Information)

X1. LOW-TEMPERATURE, LOW-SHEAR-RATE BEHAVIOR OF NON-NEWTONIAN AND NEWTONIAN FLUIDS IN D2983 VISCOMETRY

X1.1 This appendix illustrates why D2983 viscosity is often a function of viscometer spindle speed. At low temperature, many mineral oil-based lubricants develop shear-rate-sensitive wax or wax-polymer gels. Ideally, this gel appears to have a finite rigidity or strength which is reflected in these measurements as the apparent stress (dial reading) needed before the spindle begins to rotate.

X1.2 The Newtonian fluid in Fig. X1.1 has no yield stress and the dial reading is directly proportional to the spindle r/min. Its D2983 viscosity is proportional to the slope (dial reading/spindle speed). This slope does not vary with spindle speed.

X1.3 The illustrative dial reading-spindle speed function of the non-Newtonian oil Fig. X1.1 has a finite dial reading when extrapolated to 0 r/min. This 0 r/min extrapolated dial reading is the apparent yield stress. Because of the apparent yield stress, the viscosity of the non-Newtonian fluid is a function of spindle speed as follows:

Case	Spindle, r/min	Dial Reading	Slope	Apparent Viscosity (mPa·s)
A	12	36	3	18 000
B	30	60	2	12 000

X1.4 For a non-Newtonian fluid, the strong dependence of viscosity on spindle speed is a result of the definition of the Brookfield slope. This slope is *always* calculated from a line drawn from the origin (the 0 dial reading/0 spindle speed point) to the observed dial reading/set ppm point. When an apparent yield stress exists, this slope is much greater at low spindle speed than at high spindle speed.

X1.5 Because of the large effect of apparent yield stress on viscosity, it is imperative that fluid lubricants of the same viscosity classification be compared at the same spindle speed.

X1.6 Ideally, apparent yield stress can be subtracted from dial readings to give a constant dial reading/spindle speed slope. This slope can be used with an appropriate calibration constant to give a "flow" viscosity, which may be useful for correlation with some low-temperature performance data.
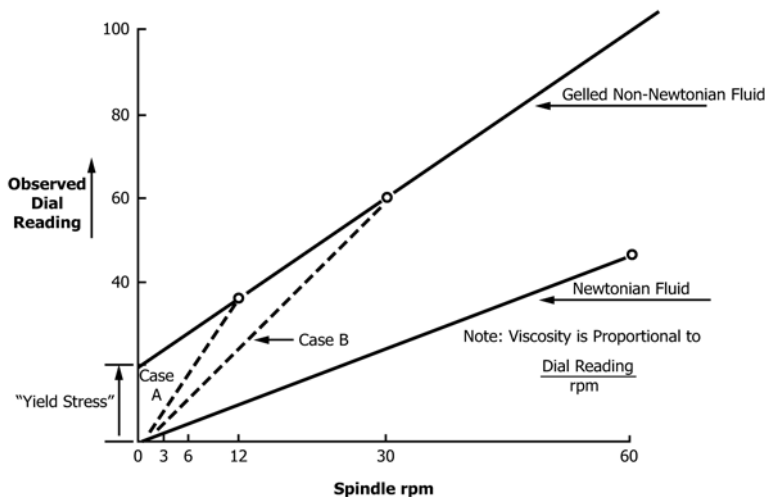

FIG. X1.1 Diagram of Viscometer Dial Reading Versus Spindle Speed

X1.7 In practice, the dial reading/spindle speed functions may not be completely linear. Shear degradation of gel structure or alignment of flow units, or both, may make the dial reading/spindle speed function slightly concave toward the spindle speed axis. Because long measurement times are often needed for a complete dial reading/spindle speed determination, sample heating may also cause some curvature.

X2. TYPICAL REFERENCE FLUID VISCOSITIES

X2.1 The viscosity-temperature function of each standard reference fluid is listed on its bottle by the supplier. The following table lists typical viscosity values:

Reference Fluid	Temperature, °C	Typical Viscosity, mPa·s	Maximum Viscosity Change Due to 0.3°C, mPa·s
N27B	−28.9	5 300	245
	−34.4	12 750	701
	−40.0	36 940	2 324

Reference Fluid	Temperature, °C	Typical Viscosity, mPa·s	Maximum Viscosity Change Due to 0.3°C, mPa·s
N115B	−6.7	5 970	254
	−12.2	13 360	591
	−17.8	32 310	1 589
	−23.3	81 460	4 823
	−28.9	253 700	16 972

X3. SHEAR STRESS AND SHEAR RATE FORMULAS FOR D2983 SPINDLES

X3.1 *Shear Stress (or Yield Stress):*

$$\tau = 1.253 \times M \qquad (X3.1)$$

where:

τ = shear stress, Pa,
M = dial reading, and,
1.253 = constant determined from spindle dimensions and the viscometer's spring constant.

X3.2 *Shear Rate* (at the wall of LV-4 spindle in a 22.25 mm inside diameter test cell):

$$\dot{\gamma} = 0.2156 \times \text{spindle speed} \qquad (X3.2)$$

where:

$\dot{\gamma}$ = shear rate, s^{-1},
spindle speed = rotational speed, r/min, and
0.2156 = constant dependent on spindle radius and test cell internal diameter.

NOTE X3.1—Equations are derived from AMTEK Brookfield literature. AMTEK Brookfield should be consulted for more detailed derivations.[2]

X4. DETERMINATION OF THE VISCOSITY OF HYDRAULIC OILS

X4.1 This appendix provides information regarding the precision of this test method for determining the apparent viscosity of hydraulic oils. Six hydraulic oils, covering a temperature range of –10 °C, –15 °C and –20 °C and a viscosity range of approximately 500 mPa·s to 1900 mPa·s were analyzed by ten laboratories. The results of the 1993 interlaboratory cooperative test program are available from ASTM Headquarters.[10]

X4.2 Test Method D2983–87 was used in this study with the following changes to 10.3:

X4.2.1 Samples were conditioned at 80 °C ± 3 °C for 60 min ± 5 min and allowed to cool at room temperature for a minimum of 60 min prior to transferring to the cooling bath.

X4.2.2 The appropriate reference fluids were run at the beginning and end of each set of samples to ensure the sample temperature change due to the opening and closing of the cold cabinet was not greater than 0.4 °C.

X4.2.3 An attempt was made to complete the testing within 30 s after the sample was removed from the cold cabinet; the testing was completed within no longer than 60 s.

X4.2.4 The test series was completed within 1 h so that a maximum soak time did not exceed 17 h for any sample.

X4.3 Precision

NOTE X4.1—The poor precision of this hydraulic method is directly related to the size of the spindle used (No. 4 spindle used in the Test Method D2983 hydraulic round robin). The reason is that the viscosity of these fluids is much lower than what the original test method was designed to handle. These lower viscosities cause the torque readings from the viscometer to be in a less accurate zone. Further work is being performed with a No. 3 cylindrical rotor to develop increased torque suitable for a more accurate zone for the viscometer.

X4.3.1 The precision of this test method as determined by ADJD6300 (formerly known as ASTM D2PP program)[11] is set forth below. It was determined with samples varying from 500 mPa·s to 1700 mPa·s, and is valid within this range of viscosities. Precision will be subject to increasing uncertainties as measured viscosities depart from this range.

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[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1486. Contact ASTM Customer Service at service@astm.org.

[11] ADJD6300 has been withdrawn and is no longer available from ASTM International.

X4.3.2 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and in the normal an correct operation of the test method, exceed the following value only in one case in 20.

Repeatability = 44 mPa·s

X4.3.3 *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, and in normal and correct operation of the test method, exceed the following value only in one case in 20.

Reproducibility = 141 mPa·s

X5. DETERMINATION OF THE VISCOSITY OF OILS AT –55 °C

X5.1 This appendix provides information regarding the precision of this test method for determining the apparent viscosity of oils at –55 °C. Four oils were analyzed at –55 °C covering a viscosity range of approximately 84 000 mPa·s to 1 180 000 mPa·s were analyzed by ten laboratories. The results of the 2012 interlaboratory cooperative test program are available from ASTM Headquarters as Research Report RR:D02-1821.[9]

X5.2 Test Method D2983 was used in this study.

X5.3 Viscosity was recorded at 60 s and 180 s after the sample was removed from the cold cabinet; there was no significant difference in the test repeatability between these two time intervals. As a result, one precision statement for this temperature will be given and shall cover time ranging from 60 s to 180 s after removing the sample from the bath.

X5.4 Three testing procedures were evaluated by this study (cold air bath, programmable liquid bath, Sim-Air bath); a single precision statement applicable to all three methods is provided below.

X5.5 Example of Precision

X5.5.1 *Repeatability*—The difference between two successive results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, each with a viscosity in the range from 84 000 mPa·s to 1 180 000 mPa·s, obtained at a temperature of –55 °C and measurements taken between 60 s and 180 s that would, in the long run and in the normal an correct operation of the test method, exceed the following value only in one case in 20:

$$r = 0.2328 \cdot X \qquad (X5.1)$$

where:

X = the average of two values.

X5.5.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material each with a viscosity in the range from 84 000 mPa·s to 1 180 000 mPa·s obtained at a temperature of –55 °C and measurements taken between 60 s and 180 s that would, in the long run and in the normal an correct operation of the test method, exceed the following value only in one case in 20:

$$R = 0.4322 \cdot X \qquad (X5.2)$$

where:

X = the average of two values.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D2983 – 16) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised subsections 3.1.2, 6.2, and 6.9.2.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D2983 – 15) that may impact the use of this standard. (Approved Dec. 15, 2016.)

(1) Revised Section 6, including Fig. 1, to define the rotational viscometer in terms of its capabilities.
(2) Revised the title, Scope section, and Significance and Use section to replace "Brookfield" with "rotational viscometer"; removed "Brookfield" throughout standard except in Apparatus and Precision, as it was the only instrument used in the interlaboratory study.

(3) Revised subsection 6.9 regarding temperature devices; added "digital contact thermometer" to Terminology.
(4) Replaced "r/min" with "spindle speed" throughout the document, when appropriate.
(5) Revised Sections 10 and 11.