Designation: ~~D2983 – 09~~ D2983 – 15

Standard Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer[1,2]

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~~ Scope*

1.1 This test method covers the use of Brookfield viscometers of appropriate torque for the determination of the low-shear-rate viscosity of lubricants. The test ~~is~~ may be applied over the viscosity range of ~~500~~500 mPa·s to ~~900 000 mPa·s~~ 900 000 mPa·s within a low temperature range appropriate to the capacity of the viscometer head.[3]

1.2 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 in preparation for viscosity measurement.

1.3 The range of viscosity used to generate the precision data for this test method was from ~~1000 to 900 000 mPa·s.~~ 300 mPa·s to 170 000 mPa·s at test temperatures from –12 °C to –40 °C. The ILS also included viscosities beyond 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~~500 mPa·s to 1700 mPa·s.

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

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

1.5 **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.

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D341 Practice for Viscosity-Temperature Charts for Liquid Petroleum Products

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D5133 Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique

E1 Specification for ASTM Liquid-in-Glass Thermometers

E644 Test Methods for Testing Industrial Resistance Thermometers

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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.

Current edition approved ~~Aug. 1, 2009~~Oct. 15, 2015. Published ~~November 2009~~February 2016. Originally approved in 1971. Last previous edition approved in ~~2004~~2009 as ~~D2983~~D2983 – 09.~~-04a.~~ DOI: ~~10.1520/D2983-09.~~10.1520/D2983-15.

[2] Brookfield viscometer and accessories are a trademark of Brookfield Engineering Laboratories, Inc., 11 Commerce Blvd., Middleboro, MA 02346, www.brookfieldengineering.com.

[3] 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.

[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**

E1137 Specification for Industrial Platinum Resistance Thermometers
E2877 Guide for Digital Contact Thermometers
2.2 *European Procedure:* *ISO Standard:*[5]
CEC L18-A-80 ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:* *Definitions:*

3.1.1 *apparent viscosity—viscosity, n—*dynamic the determined viscosity determined obtained by use of this test method. Apparent viscosity may vary with the spindle speed (shear rate) of the Brookfield viscometer if the lubricant is non-Newtonian. See Appendix X1 for a brief explanation. **D4175**

[5] Available from The Coordinating European Council for the Development of Performance Tests for Fuels, Lubricants and Other Fluids, Madou Plaza, 25th floor, Place Madou 1, B – 1210, Brussels, Belgium, American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org. www.cectests.org.

3.1.1.1 *Discussion—*

Apparent viscosity may vary with the spindle speed (shear rate) of the Brookfield viscometer if the lubricant is non-Newtonian. See Appendix X1 for a brief explanation.

3.1.2 *reference viscosity*—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 *Definitions of Terms Specific to This Standard:*

3.2.1 *bath temperature offset, n*—this is the setpoint adjustment needed during the calibration of the Programmable Liquid Bath, Procedure B, to give acceptable reference oil viscosities.

3.2.2 *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.

3.2.3 *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.

3.2.4 *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.

3.2.5 *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.

3.2.6 *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.

3.2.7 *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.

3.2.8 *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.

3.2.9 *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 16 h and analyzed by a Brookfield 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 verified at that 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 Annex A4 Appendix X4) are of considerable importance to the proper operation of

NOTE 1—Spindle dimensions:
OD = 3.15 mm ± 0.03 mm at bottom;
length = 31.1 mm ± 0.1 mm

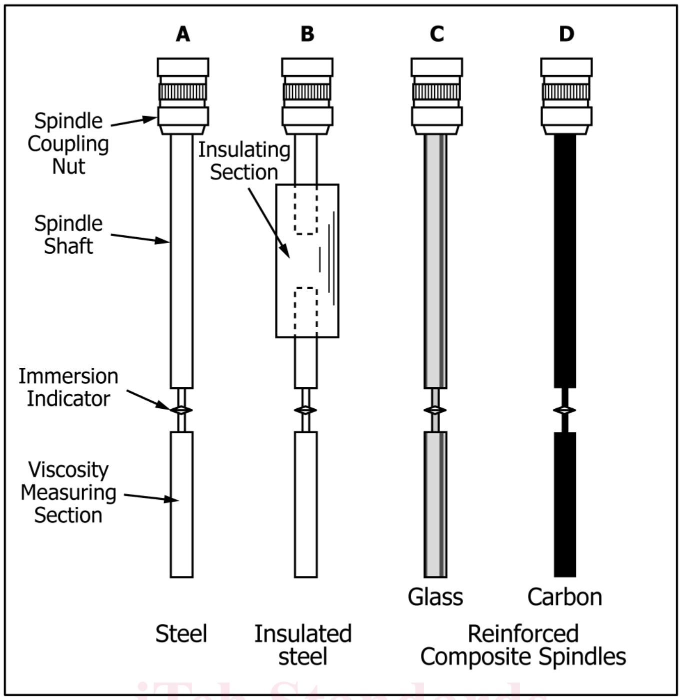


FIG. 1 Diagram of Four Forms of Spindles Used in this Test Method

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 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. ~~Only in the case of known Newtonian oils at the temperature desired is interpolation acceptable for the purpose of calibrating the spindle and glass cell (see Annex A1).~~

6. Apparatus

6.1 *Brookfield Viscometer*[2,6]—Analog Model LVT or more recent digital models (for example, LVDV-II+) are required. It is necessary that the viscometer is in good working order prior to operation and that the viscometer head and spindle is calibrated ~~periodically with a reference fluid.~~yearly.

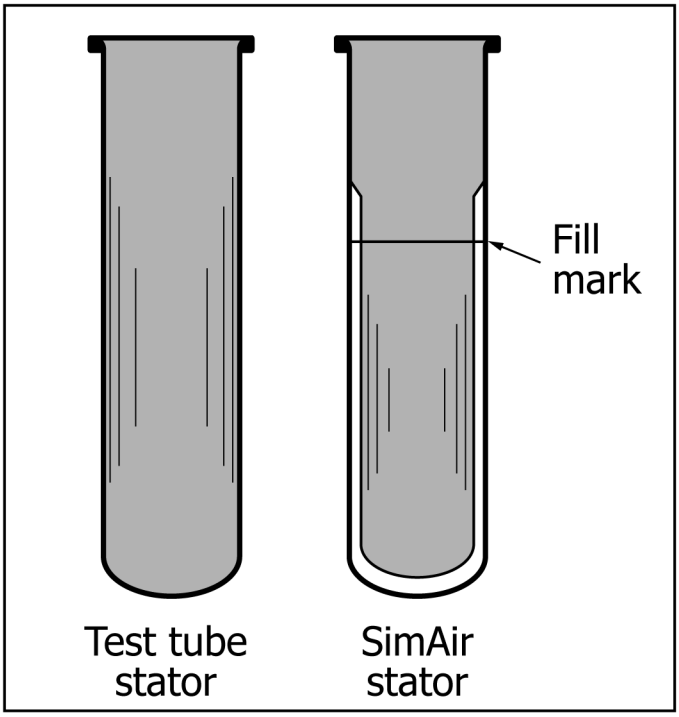
6.2 *Viscometer Spindle*[2,6]—~~Non-insulated~~Insulated Brookfield Viscometer No. ~~4~~4B2 steel spindles (used in air ~~bath), insulated No. 4B2 spindles (air~~ or liquid baths), or Tannas No. 4 glass or carbon composition spindles (air or liquid baths) may be ~~used (see~~ used. Note that the uninsulated spindle shown in Fig. 1~~a, b, c, and d, respectively).~~a may only be used in Air Bath Method (Procedure A).

~~NOTE 2—All spindles should be calibrated periodically (see Note 4, 7.1, and Annex A3).~~
~~NOTE 3—Use of non-insulated steel spindles can result in low results in liquid baths, particularly at lower temperatures and higher viscosities because of metal heat transfer. It is recommended to use partially or fully insulated spindles such as shown in Fig. 1b, c, and d.~~

6.2.1 When using No. 4B2 ~~spindles (see~~ spindles, ~~Fig. 1b),~~ ensure that both steel ends are firmly embedded in the insulating section between them (see Fig. 1~~b). A~~ a). When a slight twist is given to the two metal sections on either side of the insulating ~~cylinder~~ cylinder, they should not ~~be able to detect movement.~~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 Brookfield viscometer. The total run-out of the spindle shall not exceed ~~1 mm unless the spindle is recalibrated in which case run-out may be considered corrected (see example in~~ 1 mm (0 mm ± 0.5 mm). ~~Table A3.1).~~

[6] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.



NOTE 1—SimAir is a trademark of Tannas Co., 4800 James Savage Rd., Midland,MI 48642, www.savantgroup.com.
NOTE 2—Height of Fill Mark = 36.2 ± 0.1 mm
ID = 15 mm min
OD = 25.3 mm max

FIG. 2 Diagram of Two Forms of Stators

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~~100 000 mPa·s.

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

6.3.2 *SimAir Stator*[7]—(See Fig. 2.) The stator portion of a special air sealed cell made for this ASTM Method. This stator also has a fill line but the sample volume is approximately 15 mL.

NOTE 3—This patented cell[7] (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 ~~8.6~~6.8.2). The keyed connector is not essential to the test but makes spindle attachment faster with fewer disturbances of the sample.

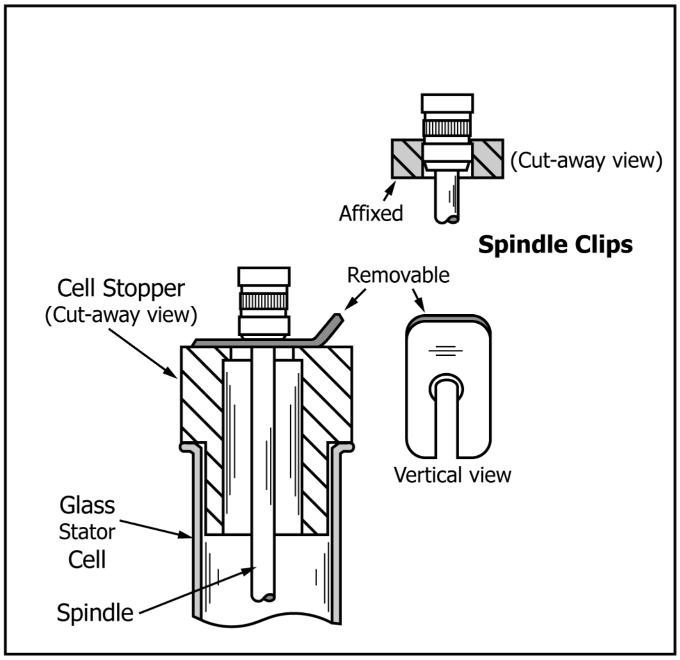
6.4 *Cell Stopper*—(See Fig. 3). An insulating cap that fits on and into the test cell with a centered hole large enough for the spindle to turn with sufficient clearance to avoid contact with the walls of the centered hole and of a height above the cell that allows a spindle clip to hold the spindle at the proper height in the test fluid during cooling.

6.5 *Spindle Clip* [6,8]—(See Fig. 3.) 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. One type of clip is shown in Fig. 3.

6.6 *Insulated Cell Carrier*—(Fig. 4.) A balsa wood carrier block used only with cold-air cabinets that keeps 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 ~~8.5.3.8~~8.4).

6.6.1 When a refrigerated liquid bath is used for final sample soak for the last half hour at analysis ~~temperature (see~~ temperature, ~~8.8),~~ the balsa block is also used for sample transfer to the liquid bath and immediately returned to the cold cabinet.

[7] SimAir is a trademark of Tannas Co., 4800 James Savage Rd., Midland, MI 48642, www.savantgroup.com.
[8] The sole source of supply of the apparatus known to the committee at this time is Lawler Manufacturing Corporation, 7 Kilmer Court, Edison, NJ 08817, www.lawlercorp.com.



NOTE 1—Dimensions of Removable Cell Stopper:
Overall height = ~43 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 %

FIG. 3 Cell Stopper, Removable and Affixed Spindle Clip

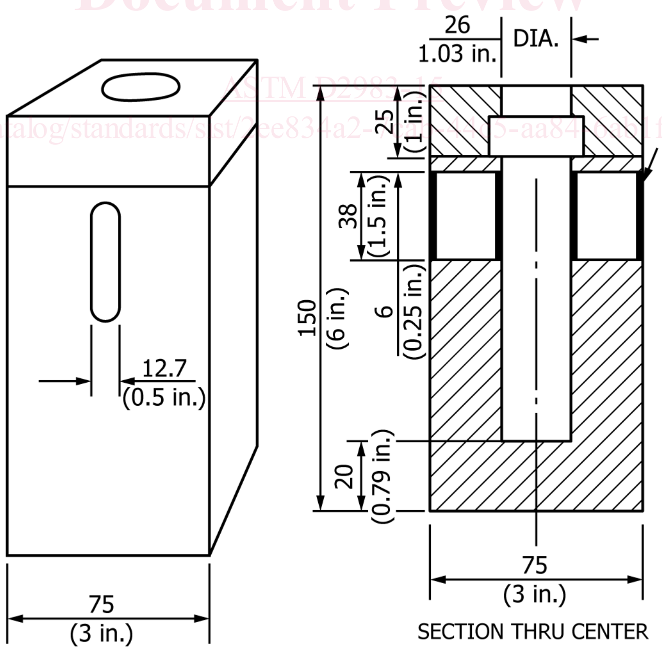


FIG. 4 Balsa Wood Test Cell Carrier

TABLE 1 Calibrating Thermometers (see Specification E1)

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

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

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°~~+5 °C to ~~–40°C~~–40 °C and holding that temperature within ~~±0.3°C.~~±0.3 °C. Air circulation and the sample turntable shall be able to be switched off prior to fully opening the bath top.

~~NOTE 6—Liquid baths are available that can cool at the proper rate and maintain the selected test temperature within 0.1°C of the set point for the 16-h soak period portion of the test. Details on liquid baths can be found in the manufacturer's manual.~~

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 4—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 ~~8.5,~~Procedures ~~8.6,~~B and ~~8.7 for details). The programmable liquid bath method's precision is in question and currently being investigated by Subcommittee D02.07.~~C, respectively, for details).

~~NOTE 8—The main advantage of a liquid bath in comparison to a cold-air cabinet is more precise temperature control, longer permissible time to take a reading, and thus more precise apparent viscosity measurement~~

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.

~~NOTE 9—The turntable should rotate at a speed of 3 to 5 r/min. This item is often supplied with the cold air cabinet.~~

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 (see~~temperature, or ~~8.5) or~~ as described in Procedure C, used to receive SimAir test cells[7] at any time for analysis 16 h after the individual test sample is immersed in the ~~bath (see~~ bath. ~~8.7).~~ The liquid bath is set at the final temperature and shall be capable of holding the sample at ~~±0.1°C.~~±0.1 °C.

NOTE 5—The SimAir cell[7] simulates the cooling curve of the air ~~cabinet (see~~cabinet, Procedure ~~Annex A2). Samples may be inserted in the bath at any time since the bath temperature remains constant.~~C.

~~6.8.2 *Programmed 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. Programmable liquid bath methods precision is currently in question and being investigated by Subcommittee D02.07.~~

6.9 *Temperature Indicating Devices:*

6.9.1 ~~For cold-air cabinets or liquid baths, use~~ Use certified or otherwise calibrated thermometric analog or digital devices that cover the range from ~~+5° to –40°C with 0.1°C~~ +5 °C to –40 °C with 0.1 °C (or finer) increments.

6.9.2 ~~For the cold-air cabinets, it is recommended to use~~ IP Brookfield Viscometer Calibrating Thermometers shown in Table 1 or their ASTM liquid-column counterparts. A digital contact thermometer (DCT) with a probe may be used conforming to the requirements of Specification E1137.

6.9.2.1 Calibrate the PRT system (probe and readout) in accordance with Specification E644. Corrections shall be applied to ensure accurate measurements within ± 0.1 °C.

6.9.2.2 Precision of the DCT should be measured to 0.1 °C.

6.9.2.3 The calibration range of said DCT should be from 25 °C to –60 °C, with a minimum of four (4) data points used.

6.9.2.4 The DCT shall be calibrated by an ISO 17025 calibration laboratory with temperature calibration in their scope, and should be traceable to a national metrology lab.

TABLE 2 Chart for r/min Selection of Generic Factors

NOTE 1—If determined apparent viscosity is below range indicated for r/min, use next higher r/min.

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 models of the Brookfield viscometer.

6.9.2.5 Calibration of the DCT should be done with the entire probe sheath immersed.

~~6.10 *Test Cell*[6,8]—A glass test tube 22 to 22.5 mm in inside diameter and 115 ± 5 mm in overall length.~~

~~6.11 *Blank Sample*—A fluid that is close in viscous behavior and response to temperature to those samples being tested. It is used for the purpose of monitoring the temperature experienced by the sample in the cold-air cabinet by inserting a thermometric device. The viscosity is used for temperature adjustments, only to know if a shift has occurred due to opening and closing of the air bath lid.~~

~~NOTE 11—This technique is desirable for assurance of proper analysis temperature in cold-air cabinets but is sometimes practiced in liquid baths as well as an additional assurance of proper temperature control of the test samples.~~

7. Use of Reference Fluids

7.1 This test method uses metal or composite viscometer spindles (see Fig. 1) whose viscosity-measuring surface in contact with the test fluid is a cylinder of ~~3.17~~3.15 mm ± 0.03 mm diameter and ~~38.0~~31.1 mm ± 0.1 mm long (equivalent to Brookfield #4 spindle). 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~~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 6—Although the generic factors of Table 2 provide acceptable results, somewhat greater precision may be generated by this test method by calibrating ~~spindles, particularly after some period of use during which the spindle may have developed run-out greater than permissible (see~~ spindles. ~~6.2.2). Calibration can permit such a spindle to be returned to service.~~ Spindle calibration can also indicate problems with the viscometer that require repair to restore accuracy (see Annex A3).

NOTE 7—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 8—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~~ Cabinets, Procedure A, because of Opening and Closing of the Air Cabinet ~~Lid:~~Lid (Only One Reference Oil Required for Procedures B and C):*

NOTE 9—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.

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*—Obtain a thermometric sensor shaped like a 4B2 spindle and use it to monitor the temperature of the reference sample. This procedure was used by some (not *all*) labs running Procedure B during the 2012 round robin study.

Procedure A

8. ~~Procedures for Different Cooling Approaches~~Procedure A—Cold Air Cabinet

8.1 ~~*Preparation of*~~*Setting the Cold-Air Cabinet* ~~*Setting*~~ *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*—~~To set the desired test temperature~~ With the turntable in proper operating position but turned off, fill a stator to the required depth with the blank sample (3.2.2~~of the cold-air cabinet:~~) and insert a thermometric device capable of being read to ±0.1 °C.

~~8.1.1.1 With the turntable in proper operating position but turned off, fill a stator to the required depth with the blank sample (see 6.9) and insert a thermometric device capable of being read to ±0.1°C.~~

8.1.1.1 Place the blank sample in the center of the sample rack ~~to monitor the cooling rate of the oil samples and, particularly, the final cold-air cabinet~~ (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 ~~temperature~~ to come to the test temperature ~~equilibrium~~ as indicated by the blank sample. ~~It may be helpful to periodically note the cabinet temperature as the sample cools.~~ 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 (which~~ controller. This temperature may not completely agree with the blank sample ~~temperature) will be the cabinet temperature set and used for further test runs at this temperature.~~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. ~~Do not adjust bath temperature while running samples.~~

~~NOTE 16—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.~~

8.1.2 *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 the repeatability of the method, 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 10—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 or Liquid Bath:~~ Cabinet:*

8.2.1 ~~For analysis of samples in~~ Shake the sample container thoroughly and fill the glass stator to the fill mark (see Fig. 2~~a cold-air cabinet, two samples of each fluid are required~~ (see). If the stator does not have a fill mark, fill with appropriate amount of test oil to permit proper ~~Note 17 and~~use of 9.3~~). This is not required in liquid baths.~~the immersion indicator at analysis temperature (approximately 30 mL).

~~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 balsa carrier blocks, it has become a practice to run two samples of the same fluid first to determine best spindle speed and the second to apply that speed to obtain the viscometric information. Subsequently the second value is chosen (see 9.3).~~

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

~~8.2.2.1 When using cold-air cabinets, 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) to indicate any sample temperature change resulting from frequent opening of these cabinets (see 7.3). The sample viscosity is not intended to be used as a guide to adjust bath temperature, only to make certain that bath temperature did not drift over the course of the testing. The change in apparent run temperature (from run to run) may not exceed 0.4°C. The apparent run temperature itself should be within ±0.3°C of the set test temperature.~~

~~NOTE 18—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.~~

NOTE 11—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 12—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 ~~For lower viscosity fluids preheat the test samples in the stator to 50 ± 3°C for 30 ± 5 min. Protectively cover each sample (such as with aluminum foil or a latex finger cot, etc.) during preheating.~~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) to indicate any sample temperature change resulting from frequent opening of these cabinets.

8.2.4 ~~For higher viscosity fluids preheat the test samples in the stator to 90 ± 3°C for 30 ± 5 min.~~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.

~~NOTE 19—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. Higher temperatures and longer preheating times may be necessary for higher viscosity oils.~~

8.2.5 Remove the test cells from the pre-heating source and allow them to cool to room temperature ~~(25 ± 5°C)~~ and then remove the covers. (Use care in handling the hot stators.)

8.2.6 Place the cell stopper (Fig. 3) on the stator with the spindle supported by the spindle clip as shown in Fig. 3.

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

~~NOTE 21—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 (see Section 7). Do not use any damaged or noticeably bent spindles.~~

8.2.8 Two samples of each fluid are required.

NOTE 14—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 balsa carrier blocks, 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 and the blank temperature-indicating oil in the center of the rack.

8.2.10 Place as many balsa carriers (see Fig. 4) within the cold-air cabinet in positions that will not unduly restrict airflow around the test samples within the air chamber. Take care to ensure that no balsa wood carrier is placed restricting 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 balsa blocks for 16 h.

~~8.3 *Placement and Handling of Samples and Supporting Equipment for Cooling and Analysis:*~~

~~8.3.1 *Cold-Air Cabinet:*~~

~~8.3.1.1 When using a cold-air cabinet, place the test cells into the turntable sample rack with a reference fluid sample at the beginning and end of the set of samples (see 7.3) and the blank temperature-indicating oil in the center of the rack (see 8.1.1.2).~~

~~8.3.1.2 Place as many balsa carriers (see Fig. 4) within the cold-air cabinet in positions that will not unduly restrict airflow around the test samples within the air chamber. Close the cabinet lid and turn both the turntable and air blower on.~~

~~8.3.1.3 Cool the samples and balsa blocks for 16 h.~~

~~8.3.2 *Using Cold-Air Cabinet with Liquid Bath Final Soak:*~~

~~8.3.2.1 When the final soak and analysis of test samples is to be done by transfer to a liquid bath, bring the liquid bath to the desired temperature equal to that of the blank test oil (see 8.1.1.2 and 8.3.1.1) in the cold-air cabinet at least 2 h before analysis is to begin.~~

~~8.3.2.2 At the end of 15.5 h of cooling in the cold-air cabinet, using the cold balsa carriers to quickly transfer all samples to the liquid bath for another half-hour soak. Be sure to re-locate the three or four balsa carriers to allow them to regain the cold-air cabinets final temperature.~~

~~8.3.3 *Programmed Liquid Bath:*~~

~~8.3.3.1 When using a programmed liquid bath, place the preheated samples in the test cells into their respective positions in the bath at room temperature. (See 8.6 for programmed cooling liquid baths.)~~

~~8.3.3.2 Temperature of the bath shall be monitored by a separate analog or digital thermometric device accurate to ±0.1°C near the test cells (see 6.9).~~

~~8.3.4 *Constant Temperature Liquid Bath with SimAir*[7] Cells:~~

~~8.3.4.1 When using a constant temperature liquid bath with the SimAir[7] stators, bring the bath to the desired temperature and make sure it is stable ±0.1°C. Temperature of the bath shall be monitored by a separate analog or digital thermometric device accurate to ±0.1°C near the test cells (see 6.9).~~

~~8.3.4.2 Insert samples at any time for analysis 16 h after insertion.~~

~~NOTE 22—Insertion of SimAir[7] stators in the liquid bath may be done at any time. However, to avoid disturbing temperature control, it is best not to insert any of samples simultaneously while Brookfield analyses are being conducted.~~

8.3 ~~*Preparation of the Brookfield Viscometer:*~~ *Using a Liquid Bath for Final Soak and Analysis after Conditioning Samples in an Air-Bath:*

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

~~NOTE 23—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.~~

~~8.4.2 After turning on the power, zero the viscometer with no spindle attached.~~

~~8.4.2.1 Use the auto-zeroing feature available on digital Brookfield models (see the Owner's Manual).~~

8.3.2 ~~For analog Brookfield 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~~Set liquid bath temperature to that desired for final half-hour soak ~~Table 2 or, if the spindle is calibrated~~ 2 h before using ~~Annex A3 for greatest accuracy and precision, use the individual spindle calibration factor so obtained.~~bath. Make certain that the bath temperature is stable and the precision thermometer is reading the proper value.

8.3.3 For ~~Brookfield digital viscometers, select the spindle setting (S64), which is also the correct setting for the #4, #4B2 and the composite spindles shown in~~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 ~~Fig. 1. After selection, immediately press the spindle selection key again to store the change. (~~viscosity values are obtained.~~**Warning—**~~Failure to press the spindle selection key within 2 s will cause the viscometer to retain the last spindle used and may therefore lead to the use of the wrong spindle selection.)~~

~~8.4.4.1 The information panel on digital viscometers will read in both centiPoise (cP) units of viscosity and in percent full scale torque. Percent full scale is used in calibrating the spindle (see Annex A3) and in adjusting the correct speed for making a viscosity reading with a test or reference oil (see 9.1).~~

8.3.4 Proceed to Section 11 for the setup of the viscometer and selection of r/min.

8.4 *Analytical Protocol for Cold-Air Cabinets:*

~~8.5.1 After test samples have been placed in the turntable rack with the bath at the desired temperature (8.3.1.1), start the timer for 16 h.~~

8.4.1 On completion of the ~~16 h~~16 h cold exposure of the samples, check the level of the viscometer to ~~assure~~ensure that the drive shaft is vertical (see ~~8.4~~11.1) and re-zero (see ~~8.4.2~~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 ~~temperature of the blank sample.~~ cabinet controller temperature. If it is not at the desired temperature ~~desired ±0.3°C,~~as per 8.3.2, adjust the cold-air ~~cabinet to produce the desired temperature.~~ cabinet. Wait at least ~~1 h~~ 1 h while the ~~blank sample~~ 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 and allowing them to come to a complete stop before opening the cold-air cabinet.~~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 from shutting of 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 and attach~~ 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 Look through the windows of the balsa carrier and 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 15—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~~ 1.2 mm from the immersion mark can produce viscosity errors.