~~Designation:D 2983–04a~~

~~An American National Standard~~ Designation: D2983 – 09

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 Department of Defense.

1. Scope *

~~1.1This test method covers the use of the Brookfield viscometer and a low-temperature bath for the determination of the low-shear-rate viscosity of lubricants. The test may operate in the viscosity range of 500 to 1000000 mPa·s (cP). The bath-controlled temperature is selected within the range of +5° to –40°C.~~

~~1.2The test method uses the SI unit, milliPascal-second (mPa·s), as the unit of viscosity. (1 cP = 1 mPa·s).~~

~~1.3~~

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 applied over the viscosity range of 500 to 900 000 mPa·s within a low temperature range appropriate to the capacity of the viscometer head.[3]

1.2 The range of viscosity used to generate the precision data for this test method was from 1000 to 900 000 mPa·s. Appendix X4 lists another interlaboratory study that specifically targeted hydraulic fluid ranging from 500 to 1700 mPa·s.

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

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

1.4 *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 ~~Standard Viscosity-Temperature Charts for Liquid Petroleum Products~~ Practice for Viscosity-Temperature Charts for Liquid Petroleum Products

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

2.2 *European Procedure:~~CEC L18-A-80~~*

~~2.3ASTM Adjuncts:~~

~~ADJD6300D2PP, Version 4.43, Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products~~ [5]

CEC L18-A-80

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

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

~~[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.~~

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

~~[3] Available from CEC, Mandou Plaza-25th Floor, B-1210 Brussells, Belgium.~~

[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] Available from ASTM International Headquarters. Order Adjunct No. ADJD6300.~~

[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.

~~[5] The sole source of supply of the Standard Newtonian Brookfield viscosity reference fluids known to the committee at this time is Cannon Instrument Co., Post Office Box 16, State College, PA 16801.~~

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

3.1.2 *reference viscosity*,—the 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.

4. Summary of Test Method

4.1A lubricant fluid sample is preheated, allowed to stabilize at room temperature, and then poured into a glass cell with a special spindle. The glass cell is then placed into a pre-cooled cold cabinet set at a predetermined test temperature between +5 to –40°C for 16 h. Then a viscometer is utilized that rotates the specified spindle within the sample at the speed giving a maximum torque reading on the viscometer. The resulting torque reading is used to calculate the viscosity of the oil.

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 inserted through a special stopper and suspended by a clip. The contained sample is cooled to a predetermined temperature for 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.

5. Significance and Use

5.1 The low-temperature, low-shear-rate viscosity of gear oils, automatic transmission fluids, gear oils, torque and tractor fluids, and industrial and automotive hydraulic oils,oils (see Annex A4,) 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 acceptability. 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 either interpolation orearlier techniques using extrapolation of experimental data. viscometric data obtained at higher temperatures.

NOTE1—Viscosity values obtained by either interpolation or extrapolation are subject to errors caused by gelation or non-Newtonian response to rotor speed, or both. Only in the case of known Newtonian oils is interpolation acceptable for the purpose of calibrating the rotor and glass cell. If such viscosity

[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, www.cectests.org.

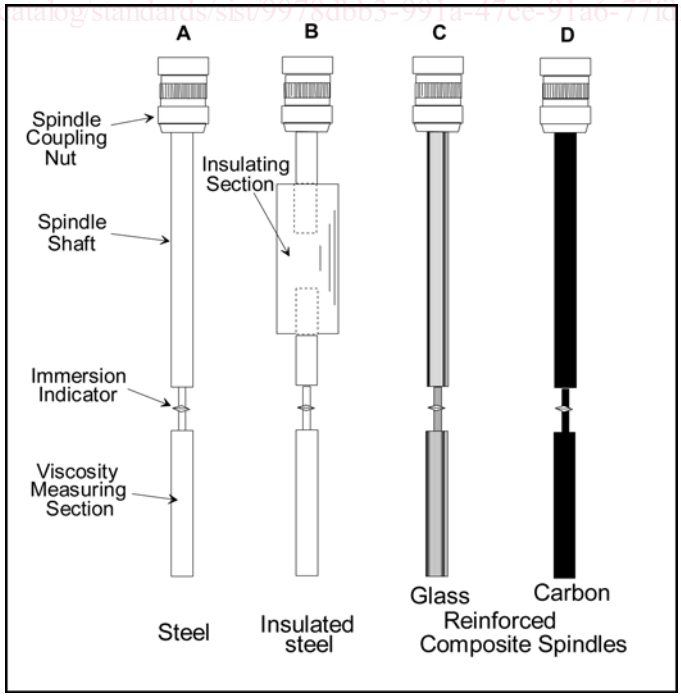


FIG. 1 Diagram of Four Forms of Spindles Used in this Test Method

versus temperature plots are required, they can be made by the procedure outlined in 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]—Analog Model LVT or more recent digital models (for example, LVDV-II+) are required. Make certain that the viscometer is calibrated and in good working order prior to operation.

6.2 *Viscometer Spindle*[6,7]—Uninsulated viscometer No. 4 spindle or insulated No. 4B2 spindle may be used. Periodically (depending on use, but at least every 3 months) inspect for wobble of the spindles. The run-out (wobble) of the spindle must not exceed 1 mm. Contact the manufacturer for measuring details. For No. 4B2 spindles, ensure firm adhesion of the lower part of the spindle. A number of spindles are needed for multiple determinations. See [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.

6.2 *Viscometer Spindle*[2,6]—Non-insulated Brookfield Viscometer No. 4 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 Fig. 1for diagram.

6.3*a, b, c, and d, respectively).*

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 Fig. 1b), ensure that both steel ends are firmly embedded in the insulating section between them (see Fig. 1b). A slight twist given to the two metal sections on either side of the insulating cylinder should not be able to detect movement.

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 Table A3.1).

NOTE 4—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.

6.3.2 *SimAir Stator*[7]—(See Fig. 2.) The stator portion of a special air sealed cell made for this ASTM Method.

NOTE 5—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). 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]—A thin clip or spacer that supports the spindle at proper immersion depth during cool-down.

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

6.5*Cell Stopper*[6,8] (Fig. 2)—A cap that fits onto the test cell with a hole large enough for the spindle to turn with sufficient clearance.—(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.

6.6 *Insulated Cell Carrier*[6,8] (Fig. 2)—A balsa wood block with windows that keeps the test cell cold during testing.

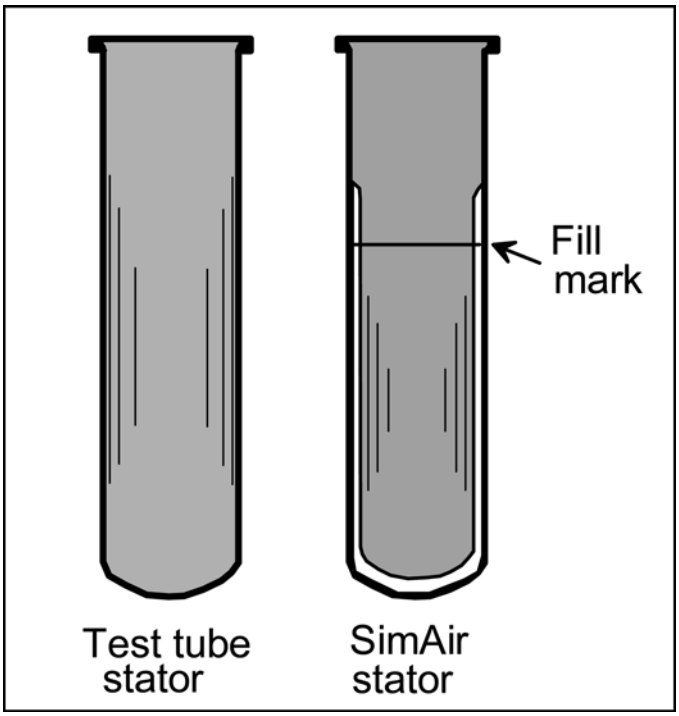
NOTE2—A liquid bath,[6,8] with a viewing window, held at the specified temperature, within 0.1°C, can be used in place of the test cell carrier (see also Note 10Insulated Cell Carrier—(Fig. 4.) A balsa wood carrier block used only with cold-air cabinets that keeps the test cell cold during transfer of 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).

[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, which you may attend.

[7] The sole source of supply of the Brookfield viscometer and accessories known to the committee at this time is Brookfield Engineering Laboratories, Inc., Stoughton, MA 02072.

[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 these itemsthe apparatus known to the committee at this time is Lawler Manufacturing, Inc.,Manufacturing Corporation, 7 Kilmer Ct., Eddison,Court, Edison, NJ 08817, www.lawlercorp.com.



NOTE—SimAir is a trademark of Tannas Co., 4800 James Savage Rd., Midland,MI 48642, www.savantgroup.com.

FIG. 2 Diagram of Two Forms of Stators

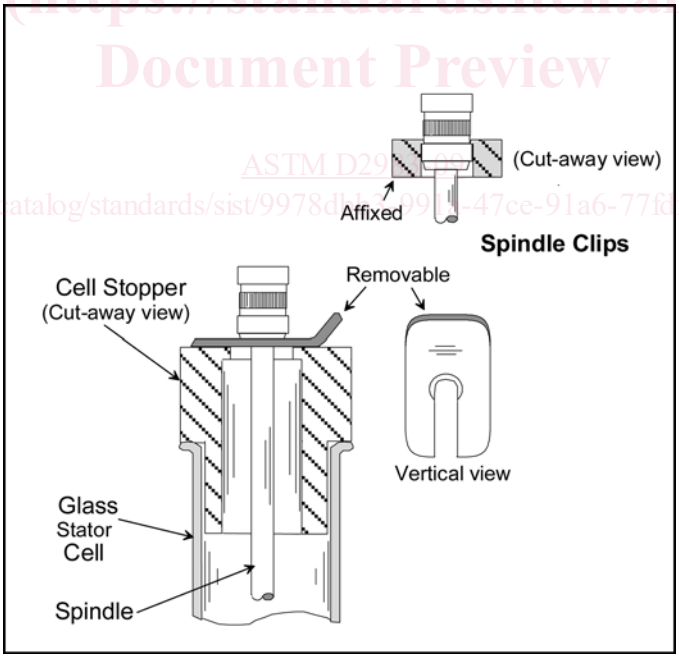


FIG. 3 Cell Stopper, Removable and Affixed

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

6.7 ~~*Cold Cabinet*[6.8]—A top-opening cold cabinet with an air-circulation device may be used (see Note 3). To minimize disturbance, it is recommended that the cabinet has an inner cover with hand-holes for sample insertion and removal. The cold cabinet must cool the sample to a chosen constant test temperature over a range from +5° to –40°C and hold that temperature within ± 0.3°C. The air circulation device and the turntable must be able to be switched off prior to fully opening the bath top. Mechanically refrigerated liquid baths may be used for apparent viscosity determinations. A European procedure, CEC L18-A-80,~~

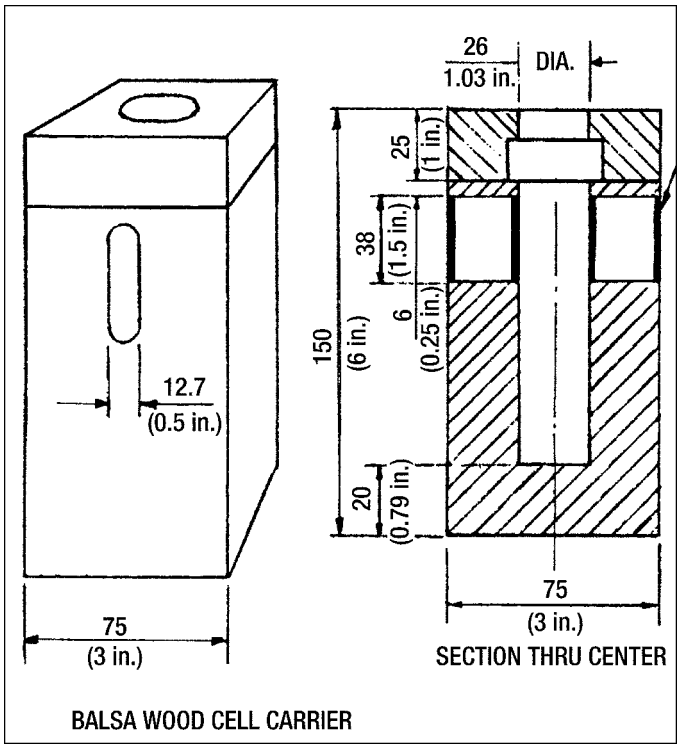


FIG. 4 Balsa Wood Test Cell Carrier

~~describes the use of such baths. A liquid bath can be used for sample conditioning if it can duplicate the sample cooling rates outlined in Annex A2. The main advantage of a liquid bath over an air bath is more precise temperature control and thus more precise apparent viscosity measurement.~~ 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° 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.

NOTE ~~3—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—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.

NOTE 7—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 ~~*Turntable*[6.8]—This device contains the cell rack. The turntable should rotate at a speed of 3 to 5 rpm. This item is often supplied with the cold cabinet. The turntable is not required for a liquid bath.~~

~~6.9*Temperature Sensing Devices*—Use certified or other calibrated thermometric devices of equal or greater accuracy that cover the range from +5° to –40°C with 0.1°C (or finer) increments. For air-bath-style cold cabinets, it is recommended to use IP Brookfield Viscometer Total Immersion Thermometers:~~

~~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~~

~~and ASTM 63C (–8° to +32°C)) in conjunction with a calibrated resistance temperature detector (RTD) device. The RTD must be effectively calibrated at 0°C and –40°C. The thermometers can be compared to the RTD in order to get an accurate reading. Store thermometers in an upright position to help maintain calibration. Make certain that there are no separations in the column.~~

~~6.9.1For liquid baths, use certified or other calibrated thermometric devices of equal or greater accuracy that cover the range from +5 to –40°C with 0.1°C (or finer) increments (consult the bath manufacturer for calibration details). As with the air bath, compare these results with an RTD in order to verify an accurate reading. For further verification of the temperature control in air or liquid baths, see Annex A4.~~ Liquid Baths—Mechanically refrigerated liquid baths are used in three significantly different protocols to gain the same analytical results (see 8.5, 8.6, and 8.7 for details). The programmable liquid bath method's precision is in question and currently being investigated by Subcommittee D02.07.

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

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.

6.8.1 *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 8.5) or 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 8.7). The liquid bath is set at the final temperature and shall be capable of holding the sample at ±0.1°C.

NOTE 10—The SimAir cell[7] simulates the cooling curve of the air cabinet (see Annex A2). Samples may be inserted in the bath at any time since the bath temperature remains constant.

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 certified or otherwise calibrated thermometric analog or digital devices that cover the range from +5° 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.

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 chemistry to those being tested for the purpose of determining the temperature experienced within a 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

7.1The use of standard reference fluids, detailed in Annex A3 and Annex A4, was developed to ensure more precise control of the apparent viscosity measurements. Each new spindle should be run with a reference oil prior to testing samples to ensure accurate results. With analog viscometers, the procedure to calculate expected reference fluid dial readings and interpret observed reference fluid dial readings is given in

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 ± 0.03 mm diameter and 38.0 ± 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 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 . Although the dial reading limits listed in and Annex A4.) For potentially increased accuracy, spindles may be calibrated.

TABLE 2 1 StCandardlized Observationg Tihermometers (see Specification E1)

rpm	Maximum Observation Time, (min)	Maximum Spindle Rotations	Record
0.6	5	3	Highest dial reading seen as the scale pointer passes instrument window during observation time.
IP 94C	–45 to –35°C	3	
1.5	3	4.5	
IP 95C	–35 to –25°C	ASTM 123C	
3.0	3	9	
IP 96C	–25 to –15°C	ASTM 124C	
		6.0	2
IP 97C	–15 to –5°C	6.0	2
12.0	–1	12	
30.0	30s	15Observe dial reading atend of 30s. Dothistwice andrecord the higher	
60.0	30s	30	
			reading. reading.C

TABLE 1 2 RPMChart for r/min Selection Cha of Generic Factors

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

Spindle Speed, rpm	Multiply torque by below number to calculate viscosity at speed selection used	Viscosity Range, mPa·s
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	500 to 9800
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.

7.2.1 Use of standard reference fluids and technique for calibration is detailed in Annex A3 are typical of the data received from several extensive round robins, more precise control is both desirable and possible with digital equipment. and Annex A4. This protocol was developed to provide, if desired, an option for more precise determination of the apparent viscosity measurements.

NOTE 12—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 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 13—When spindles are calibrated, it is desirable to mark each spindle with some unique identification.

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 14—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 because of Opening and Closing of the Air Cabinet Lid*:

NOTE 15—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.

8. Procedure Procedures for Different Cooling Approaches

8.1 *Preparation of the Bath*Preparation of Cold-Air Cabinet Setting Operating Temperature:

8.1.1Set the test temperature of the cold cabinet, monitor the temperature with a blank sample, and allow the bath to stabilize at the desired test temperature. Do not put any test samples in the turntable.

8.1.2After equilibration, check the bath temperature by the thermometer or thermometric device immersed in a blank sample of oil held by the rotating rack.

8.1.1 To set the desired test temperature of the cold-air cabinet:

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.

8.1.1.2 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 temperature.

8.1.1.3 Close the cold-air cabinet, turn on the cooling cycle using the temperature controller and allow the cabinet temperature to come to 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, the indicated temperature shown by the cabinet's temperature controller (which may not completely agree with the blank sample temperature) will be the cabinet temperature set and used for further test runs at this 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 4—If a temperature adjustment is made, it may require at least 1 h for temperature equilibration. Depending on specific bath characteristics, longer times for equilibration may be required after major temperature changes. Do not adjust bath temperature after 4 h into the sample-conditioning period because the apparent viscosity of the sample may be significantly changed.~~

~~8.2 *Preparation of Sample*~~ 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.

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

~~8.2.1 Shake the sample container thoroughly and place about 30 mL into the test cell. It is essential that the appropriate reference fluids be run at the beginning and end of each test series (and results recorded) to indicate the sample temperature change that results from frequent opening of the cold cabinet. 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 rpm data. 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.~~

8.2.1 For analysis of samples in a cold-air cabinet, two samples of each fluid are required (see Note 17 and 9.3). This is not required in liquid baths.

~~NOTE 5—If the apparent viscosity of the sample is unknown, use two samples, one for determination of the rpm and one for determination of apparent viscosity.~~

~~8.2.2 Cover each sample (such as with aluminum foil). Maintain the sample at 50 ± 3°C for 30 ± 5 minutes. If using a liquid bath, then cover each sample with an airtight seal (such as a finger cot).~~ 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 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.

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 6—This preheating step has been proven important in other critical low-temperature ASTM methods and is designed to remove any memory effects that may develop from previous low-temperature exposures or structure formations. Higher temperatures and longer preheating times are being explored.~~

~~8.3 Remove the test cells from the heating source and allow to cool to room temperature (25 ± 5°C) and then remove the covers.~~

~~8.3.1 Cover the test cell (6.4) with the cell stopper (6.5) and use the spindle clip (6.3) to support the spindle (6.2) and lower into the test cell so that the center of the spindle immersion mark is slightly below the liquid surface (to allow sample shrinkage due to cooling). This reduces the amount of disturbance placed on the sample before measurement later in the method. See~~ 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.

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.

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

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~~for diagram.~~) should be slightly below the liquid surface (to allow for contraction of the oil sample upon cooling to the temperature of analysis).

~~NOTE 7—Handle and store the spindles and instrument with care at all times. Check the calibration of each spindle periodically with reference oil (see Section~~ 20—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.

~~8.4 Put the test cells and insulated test cell carriers into the cold cabinet. So as not to restrict airflow within the air chamber, do not put too many cell carriers into the air chamber. This can be checked by contacting the cold cabinet manufacturer.~~

~~8.5Once the last cell has been loaded, let the sample soak for no less than 16 h. Experience has shown that 6 h is a sufficient soak time for automatic transmission fluids at –17.8°C. Since this shorter soak time speeds data production and is used in some automatic transmission fluids specifications, it is the only exception to the 16-h soak time allowed by this test method (see 8.10~~

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).

~~8.6During the soak period, align the viscometer by using the bubble level located on the viscometer.~~

~~8.6.1After turning on the power, zero the viscometer with no spindle attached (some digital models have an auto-zeroing feature).~~

~~8.6.2For digital viscometers select the S64 spindle selection or the setting that corresponds to the No. 4 or No. 4B2 spindles. After selecting the S64 spindle, immediately press the spindle selection key again to store the change. Failure to press the spindle selection key within 2 s will cause the viscometer to retain the last spindle used and therefore may lead to the use of the wrong spindle selection.~~

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 ~~8—Refer to the viscometer manufacturer for more detailed instructions on viscometer care and calibration.~~

~~8.7After the 16-h soak is complete, individually transfer and test the samples as follows (It is essential that the procedure be followed in detail for the proper operation of this test):~~

~~8.7.1Check the level of the viscometer.~~ 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.

8.4 *Preparation of the Brookfield Viscometer*:

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

NOTE ~~9—It is very important that the viscometer be level during measurement.~~

~~8.7.2Record the temperature of the blank sample.~~

~~8.7.3Turn the turntable rotation and the air blower off.~~

~~8.7.4Allow the air blower and the turntable to come to a complete stop then open the cold cabinet and put one temperature-conditioned test cell into a temperature-conditioned insulated cell carrier and remove from the cold cabinet.~~ 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).

8.4.3 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 Table 2 or, if the spindle is calibrated using Annex A3 for greatest accuracy and precision, use the individual spindle calibration factor so obtained.

8.4.4 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 Fig. 1. After selection, immediately press the spindle selection key again to store the change. (**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).

8.5 *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.

8.5.2 On completion of the 16-h cold exposure of the samples, check the level of the viscometer to assure that the drive shaft is vertical (see 8.4) and re-zero (see 8.4.2).

8.5.3 Individually transfer and analyze the test samples as follows:

8.5.3.1 Note the temperature of the blank sample. If it is not at the temperature desired ±0.3°C, adjust the cold-air cabinet to produce the desired temperature. Wait at least 1 h while the blank sample comes to the desired temperature before initiating analysis.

8.5.3.2 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.

8.5.3.3 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.

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

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

8.5.3.6 Place the test cell below the viscometer and align the spindle nut with the viscometer coupling nut and 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.

8.5.3.7 Remove the spindle clip.

8.5.3.8 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 10—If the laboratory is equipped with a low-temperature liquid bath capable of maintaining test temperature within ±0.1°C and on which the Brookfield viscometer can be conveniently mounted, a cell may be removed from the cold cabinet after 15.5 h and placed in the liquid bath at test temperature for 30 min. The apparent viscosity can then be measured directly on the sample in the cell in the liquid bath without haste and without fear that the sample will warm up as it does in the cell carrier. An insulated spindle is needed if this procedure is used.~~

~~8.7.5Close the cold cabinet lid, immediately restart the turntable and air blower and transfer the insulated cell carrier with the sample to the viscometer.~~

~~8.7.6Place the test cell carrier with test cell below the viscometer and align the spindle nut with the viscometer coupling nut, attach the spindle, and remove the spindle clip being sure to minimize the disturbance of the sample with the spindle.~~

~~8.7.7Select the viscometer display mode to read either as percent of scale or directly as mPa·s.~~

~~8.7.8Look through the windows on the test cell carrier and adjust the assembly until the oil level is even with the immersion mark on the spindle shaft. In order to facilitate the adjustment of the spindle, place a cool light source, such as a flashlight, behind one window of the test cell carrier. Great care must be taken to ensure proper spindle immersion with all samples. Maintenance of proper immersion depth is essential to good reproducibility and repeatability.~~

~~NOTE 11—Data show that an immersion variation of as little as 1.2 mm from the immersion mark can produce viscosity errors.~~

~~8.7.9Center the spindle in the hole at the top of the cell stopper, making certain that no part of the spindle touches the stopper hole during the measurement process.~~

~~8.8Turn on the viscometer motor and take readings from the digital viscometer as follows:~~

~~8.8.1Refer to Section~~ 24—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.

8.5.3.9 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.

8.5.4 *Take Readings from the Viscometer*:

8.5.4.1 Refer to Table 2 for the expected r/min setting that will generate the highest torque reading on the Brookfield Viscometer head (see Section 9~~for the proper rpm setting. Use the highest viscosity reading after the first 5 s of rotation.~~

~~8.8.2Record viscosity reading (mPa·s), spindle speed (rpm), and test temperature (°C).~~

~~8.9For the best precision results, testing should be started within 30 s after the sample is removed from the cold cabinet. The measurement shall be complete in no longer than 60 s (or 90 s for samples with viscosities higher than 150000 mPa·s). Take two readings and record the higher of the two (see Table 1 for speed/viscosity selections). If using a digital viscometer, monitor the reading during the entire measurement and record the highest value. The urgency in this measurement is required to minimize sample temperature increase and erroneously low viscosities. If using an analog dial viscometer, see~~).