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

1.1 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 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:*[5]

CEC L18-A-80

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *apparent viscosity*—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. 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.

## 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 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 automatic transmission fluids, gear oils, torque and tractor fluids, and industrial and automotive hydraulic 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 acceptance for service. This test method is used in a number of specifications.

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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.

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[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.

[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.

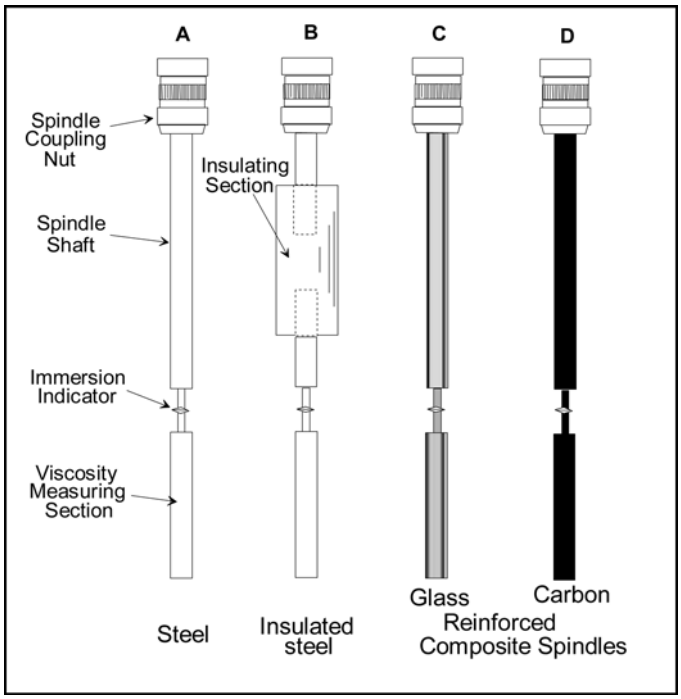
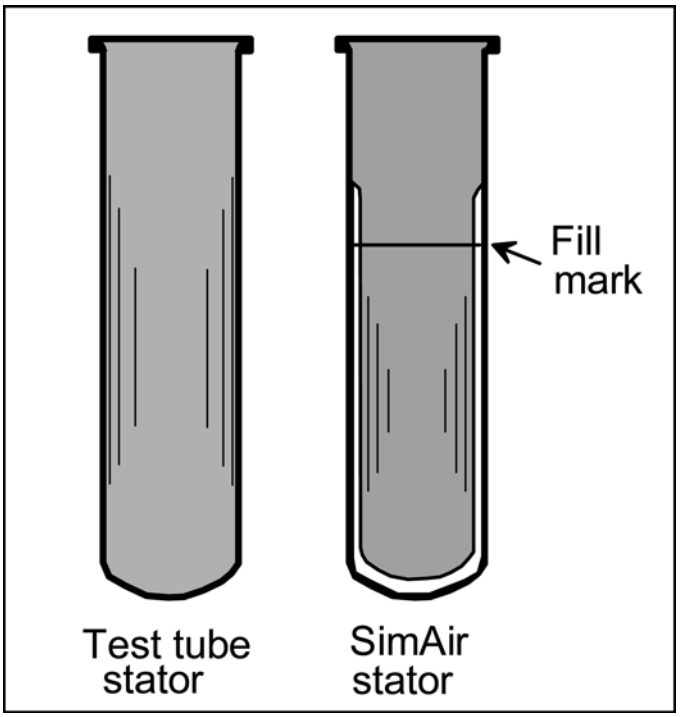



**FIG. 1 Diagram of Four Forms of Spindles Used in this Test Method**




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**

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.

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. 1a, 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)

[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.

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

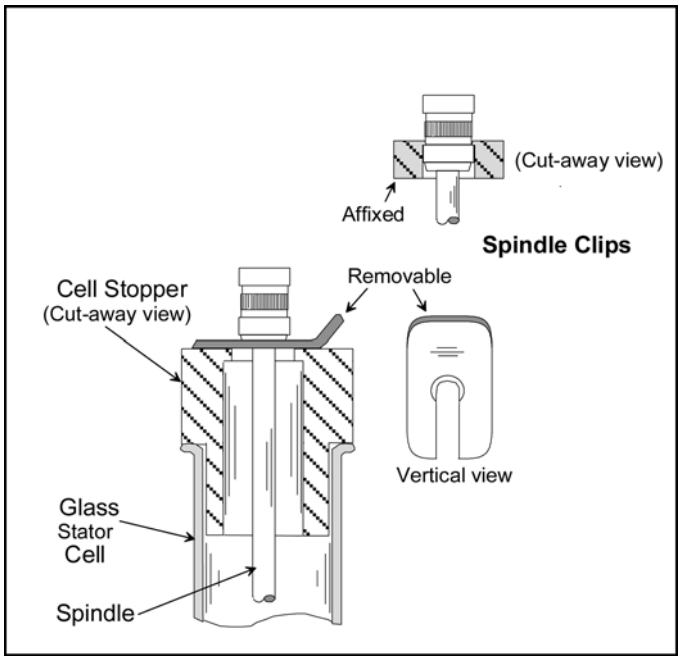



FIG. 3 Cell Stopper, Removable and Affixed

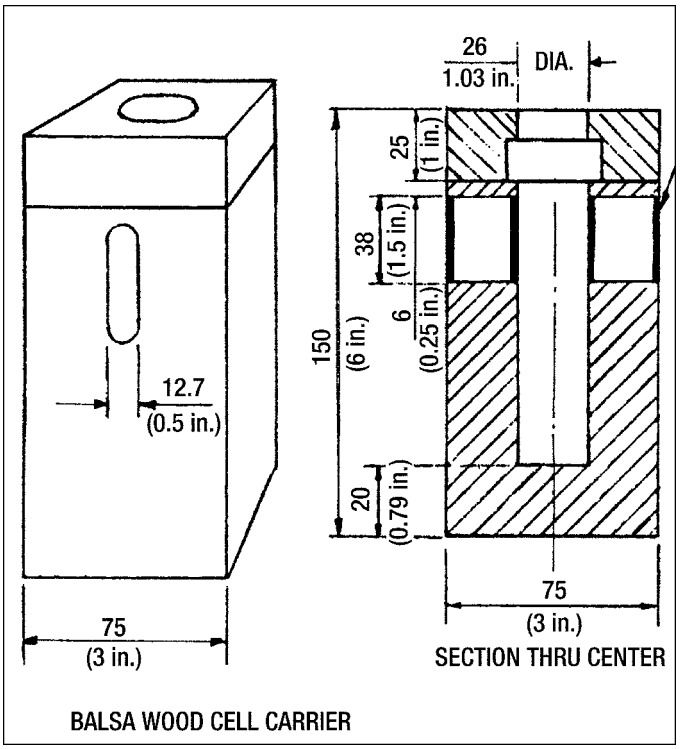



FIG. 4 Balsa Wood Test Cell Carrier

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]—(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*—(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).

6.6.1 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-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.

[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 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 *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

**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 |

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 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 ± 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 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 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.

**TABLE 2 Chart for r/min Selection of Generic Factors**

NOTE—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.

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. Procedures for Different Cooling Approaches

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

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

NOTE 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.

8.3 *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.

8.4 *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 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 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).

8.5.4.2 Again, make certain that the immersion indicator on the rotor is level with the meniscus of the oil. Turn on the viscometer motor and immediately adjust spindle speed to give a torque reading between ~40 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 centiPoise (cP) and percent full scale torque (see 8.4.4.1).

8.5.4.3 For cold-air cabinets, transfer time and adjustment of spindle speed may permit slight sample warming. For this reason, dual samples are cooled with the spindle speed for the second sample set at the optimum speed determined using the first sample (see 8.2.1 and Section 9).

8.5.5 *Determining the Viscosity of the Test Fluid*:

8.5.5.1 When using a calibrated spindle, after the spindle speed has been optimized, note the highest torque reading observed and the rotational speed of the spindle. If using a digital viscometer with a direct viscosity reading, simply read the highest viscosity value. Using these two values and the calibration factor determined for the particular spindle from Annex A3, The following equation gives the test fluid's viscosity:

$$\text{Viscosity} = \text{Factor} \cdot \text{Torque} + \text{r/min}$$

8.5.5.2 When using the standard method to determine viscosity, after the spindle speed has been optimized, note and use the highest torque reading observed and the factor given in Table 2 for the rotational speed used, and apply the following equation. If using a digital viscometer with a direct viscosity reading, simply read the highest viscosity value.

Viscosity = (Table 2 Factor for r/min used) · Torque

8.5.5.3 When using cold-air cabinets, for the greatest precision, testing should be started within 30 s after the sample is removed from the cold-air cabinet. All bath measurements shall be complete in no longer than 60 s once the motor is started and optimum speed established (or 90 s for samples with viscosities higher than 150 000 mPa·s). For digital viscometer use the highest viscosity for the recording during the time period for measurement. Take two readings and record the higher of the two (see Table 2 for speed/viscosity selections).

NOTE 25—Only digital Brookfield Viscometers have the ability to operate at 120 r/min. Analog units are limited to 60 r/min.

NOTE 26—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.

8.5.5.4 Record viscosity reading (mPa·s), spindle speed (r/min), and test temperature (°C).

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

8.5.6.1 When using a constant temperature liquid bath, it is not necessary to use initial and final reference oils as in 8.2.2.1. 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.

8.5.6.2 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 the precision thermometer is reading the proper value.

8.5.6.3 Position Brookfield Viscometer head for analysis, level head and set zero (see 8.4.1).

8.5.6.4 When 15.5 h of test sample exposure in the cold-air cabinet has elapsed and the cabinet and liquid baths are at the same temperature, transfer test samples in pre-chilled balsa carriers (see 8.3.1.2) and insert into the liquid bath for analysis after 0.5 h soak. Balsa carriers shall be immediately returned to the cold-air cabinet for re-chilling and systematic rotation in use.

8.5.6.5 Place the test cell below the viscometer and attach the spindle to the viscometer drive shaft using either the quick connect device (Fig. 3) if it is available or screwing the spindle onto the drive shaft thread. Note that this latter connection is made with a left handed thread.

8.5.6.6 Remove the spindle clip being sure to minimize the disturbance of the sample with the spindle.

8.5.6.7 Bring the spindle immersion indicator to the surface of the test fluid (see Note 26 for caution in this step).

8.5.6.8 Make sure the spindle is centered and not touching the sides of the cell stopper hole.

8.5.6.9 Follow 8.5.4 for determining the viscosity of the test oil.

NOTE 27—When using a constant temperature liquid bath to hold the test sample at temperature, there are no time restraints as in 8.5.4.3 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.

8.6 *Analytical Procedure for Programmed Liquid Baths*:

8.6.1 Program ramping program on temperature controller to obtain the cooling profile detailed in Annex A2.

8.6.2 Place preheated and assembled sample-containing test cells in bath and initiate program.

8.6.3 After 15 h of soak, check the temperature of the bath with the certified thermometer 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 it is not within ±0.3°C, the temperature shall be corrected and the entire tested repeated.

8.6.4 After 16 h, analyze samples using techniques previously presented under 8.5.4 and calculate viscosity using 8.5.5.

8.7 *Analytical Procedure for Constant Temperature Liquid Baths using SimAir[7] Stators*:

8.7.1 Follow 8.2.

8.7.2 After 15 h of soak, check the temperature of the bath with the certified thermometer (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 it is not within ±0.3°C, the temperature shall be corrected and the entire test repeated.

8.7.3 Analyze each test sample 16 h after its immersion according to 8.5.4.

8.7.4 Calculate viscosity of the test sample as in 8.5.5.

8.8 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.

## 9. Selection of r/min

9.1 Since lubricants and fluids may be non-Newtonian at low temperatures, the spindle speed selected for measuring the viscosity of a test fluid can strongly influence the resultant viscosity (see Appendix X1). For this reason, it is best for precision to obtain a test fluid's viscosity at the highest speed that will permit a torque value to be obtained. However, when the sample is unknown, it is important to begin at a lower speed and work up to the speed that will permit a torque value above 20 %.

9.2 If an expected apparent viscosity is known, use the highest spindle speed corresponding to the known viscosity range. Use Table 2 in selecting the appropriate r/min.

9.2.1 If this spindle speed is not high enough to obtain a percent full scale torque reading over 20 %, select the next higher speed.

9.3 If a cold-air cabinet is used for sample analysis and 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 to 100 r/min. The second sample is then run at the previously determined speed and only this result is reported.

## 10. Calculation

10.1 Calculate the viscosity at the test temperature of the test oil or reference oil as shown in 8.5.4 and 8.5.5.

10.2 The shear stress and rate at the surface of the Brookfield spindle may be estimated by the procedure in Appendix X3.

## 11. Report

11.1 A routine report includes the fluid identification, the determined viscosity, the test temperature, and the spindle speed. Spindle speed data are needed to ensure that different laboratories use the same shear rates.

11.2 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 28—Reference fluid data are needed to ensure that different laboratories run at the same temperature, shear rate, and viscometric conditions.

## 12. Precision and Bias [9]

12.1 *Statement of Precision*:

12.1.1 *Precision*—The precision of this test method using the air bath as determined by statistical examination of the interlaboratory test over the temperature range from –18 to –40°C and a viscosity range from 1000 to 900 000 mPa·s is as follows:

12.1.2 *Repeatability*—The difference between two results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of this test method, exceed the following values only in one case in twenty:

Repeatability = 3.4 % of the average of the two results

12.1.3 *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, exceed the following values only in one case in twenty:

Reproducibility = 20.6 % of the average of the two results

12.1.4 *Precision*—The precision of this test method using the SimAir[7] Liquid Bath as determined by statistical examination of the interlaboratory test over the temperature range from –18 to –40°C and a viscosity range from 1000 to 900000 mPa·s is as follows:

12.1.4.1 *Repeatability*—The difference between two results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of this test method, exceed the following values only in one case in twenty:

Repeatability = 11.0 % of the average of the two results

12.1.4.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty:

Reproducibility = 28.5 % of the average of the two results

12.1.4.3 *Bias*—There is no bias between the apparent viscosity of samples measured using instruments with different cooling systems.

12.2 *General Considerations*:

12.2.1 *Summary of Interlaboratory Study*—The interlaboratory study consisted of twelve participating laboratories, seven samples with viscosities ranging from 1000 to 900 000 mPa·s at test temperatures from –18 to –40°C. The precision for the liquid programmable baths were in question and are being investigated by Subcommittee D02.07. The precision for viscosities within the range of 500 to 1700 mPa·s, such as hydraulic fluids, are shown in a separate interlaboratory study depicted in Appendix X4.

12.2.2 Both analog and digital Brookfield viscometers were used for analysis with both calibrated and uncalibrated spindles.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1673.

# ANNEXES

**(Mandatory Information)**

## A1. USE OF THE BROOKFIELD VISCOSITY VERSUS TEMPERATURE RELATIONSHIP INTERPOLATION OF DATA

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

A1.2 Brookfield viscosity-temperature plots are made by measuring Brookfield viscosity for at least three temperatures and plotting a smooth curve on ASTM viscosity-temperature paper (see Test Method D341).

A1.2.1 Brookfield 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 Brookfield viscometers should not be made at different spindle speeds.

A1.3 When Brookfield 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 Brookfield method using a spindle of larger diameter and a more closely fitting glass stator.

A1.3.2 Brookfield viscosities involved in plots compared between laboratories shall be taken at the same high enough r/min for sensitivity at whatever temperature is involved in each laboratory.

NOTE A1.2—Because of non-Newtonian behavior, Brookfield viscosity is dependent on r/min. 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 BROOKFIELD AIR CABINETS

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

A2.2 Sample cooling rates in Brookfield 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 Brookfield viscosity.

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

$$dS/dt = k(S - B) \quad \text{(A2.1)}$$

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:

$$(S - B) = Ce^{Kt} \quad \text{(A2.2)}$$

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

Eq A2.3 may be conveniently plotted as:

$$\ln \frac{(S - B)}{A} = \ln\ C + kt \quad \text{(A2.3)}$$

A2.4 When temperature is in degrees Fahrenheit, a sample in an average air cabinet cools with k values that may range between –0.12 and –0.040, averaging –0.08. C represents the sample-bath temperature difference at zero soak time. For the tests run, ln C ranged between 4.45 and 4.80. Cabinets that cool samples at rates defined by these limits and meet other method requirements are satisfactory for Brookfield viscometry of automotive fluid lubricants.

## 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 Brookfield viscosity at low temperatures. Such calibration can restore the use of otherwise unacceptable spindles (see 7.2).

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 where run-out becomes excessive (see 6.2.2). In such case, use of somewhat bent spindles can be restored by calibration.

NOTE A3.1—Examples of agreement between the general factors of Table 2 and the calibration-derived factors with good spindles and correction of unacceptable spindles through calibration are shown in Table A3.1:

Spindles A, C, D in Table A3.1 have acceptable run-out. Agreement between the general factors in Column 6 and the calibration-derived factor

**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[A] | | | 12 | 67.1[A] | | 34 200 | 64.5 | 3711 | 309 | 20 749 | –0.2 |
| C | | | 12 | 40.9 | | 20 600 | –0.9 | 6018 | 502 | 20 525 | –1.3 |
| D | | | 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[A] | | | 6 | 67.3[A] | | 68 900 | 70.4 | 3711 | 618 | 41 374 | 2.3 |
| C | | | 6 | 39.6 | | 40 000 | –1.1 | 6018 | 1003 | 40 085 | –0.9 |
| D | | | 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[A] | | | 12 | 41.6 | | 21 600 | 67.2 | 3711 | 309 | 12 864 | –0.4 |
| C | | | 12 | 25.7 | | 13 000 | 0.7 | 6018 | 502 | 13 040 | 1.0 |
| D | | | 12 | 26.4 | | 13 300 | 3.0 | 6019 | 502 | 13 231 | 2.4 |

[A] Spindle with considerable run-out (wobble).

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 Column3. Errors range from 0.0 to -9.4 % for the general factor and from 0.1 to -2.7 % after calibration.

Spindle B was known to have a total run-out of 5 mm. In this case, using the general factor with Spindle B gave errors from 64.5 to 70.4 % when the actual viscosity value was compared to the value obtained using the general factor. However, when this spindle was calibrated, the error fell to a range of -0.2 to 2.3 %, an acceptable range resulting in recovery of the spindle's accuracy.

### 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. This limitation can be overcome to some degree (see Note A3.4).

### 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 19).

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

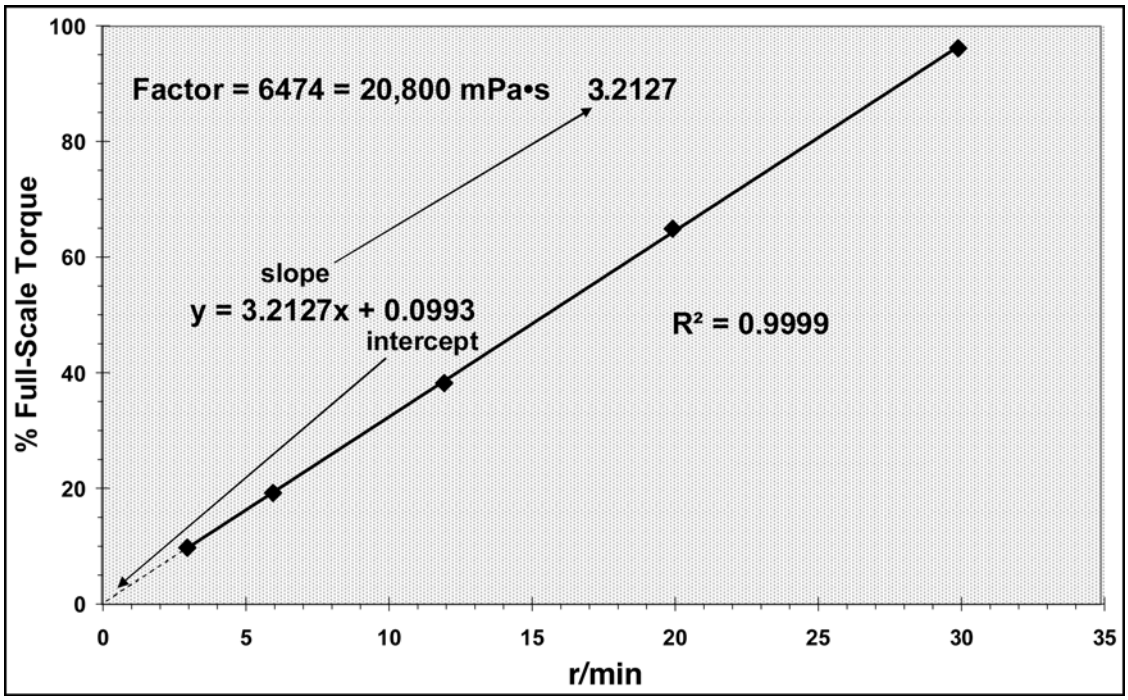



**FIG. A3.1 Calibration of Spindle with Newtonian Oil of 20 800 mPa·s Viscosity at Calibration Temperature**

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—From the information in Fig. A3.1, the Calibration Factor is shown as 6474.

A3.6.3 The Calibration Factor determined in this way is applicable at all temperatures and viscosities.

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 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.

A3.7 A spindle calibration factor is most accurate when used on the Brookfield instrument upon which the calibration value was generated.

NOTE A3.10—If a spindle is to be used on more than one Brookfield viscometer it is good practice to calibrate it on any Brookfield viscometers on which it may be used.

NOTE A3.11—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.

## A4. ESTIMATION OF APPARENT RUN TEMPERATURE FROM OBSERVED BROOKFIELD 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 Brookfield calibration factors are in Section 8.

A4.2.3 The dial reading and 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 Brookfield viscosity:

Brookfield viscosity = observed dial reading
× Brookfield calibration factor

A4.3.2 Determine constants *A* and *B* from Eq A2.2, Eq A2.3, in the appendix of Test Method D341. Use two reference fluid viscosities at two temperatures near the set test temperature.

A4.3.3 Calculate *Z* from Brookfield viscosity and Eq A2.3 in the appendix of Test Method D341.

A4.3.4 Use the following form of Eq A2.2, in the appendix of Test Method D341 to calculate *T*, the apparent run temperature in °F.

$$T = (\text{antilog}\ (A - \log\log Z)/B) - 460 \qquad (A4.1)$$

A4.3.5 Calculate $T = T -$ set test temperature.

### A4.4 Example Set temperature, –30°F (–34.4°C)

Observed dial reading at 12 r/min, 49.5
Reference fluid viscosity at –20°F (–28.9°C), 11 360
Reference fluid viscosity at –30°F (–34.4°C), 28 580
Brookfield factor at 12 r/min, 500
Brookfield viscosity = 49.5 × 500 = 24 750

from the appendix charts of Test Method D341,

Eq A2.3 $Z$ (–30°F) = 28 580.7
Eq A2.3 $Z$ (–20°F) = 11 360.7
Eq A2.2 $A$ = 11.44162
Eq A2.2 $B$ = 4.09827
Eq A2.3 $Z$ observed = 24 750.7

$T$ = (antilog (11.44162 – log log 24 750.7)/4.09827) – 460
$T$ = 28.52°F or –33.62°C
$T$ = 1.48°F or 0.78°C

A4.5 *Interpretation*—The 0.78°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 BROOKFIELD VISCOMETRY

X1.1 This appendix illustrates why Brookfield viscosity is often a function of viscometer r/min. 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 Brookfield 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 Brookfield viscosity is proportional to the slope (dial reading/r/min). This slope does not vary with r/min.

X1.3 The illustrative dial reading-r/min 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 r/min as follows:

| Case | Spindle, r/min | Dial Reading | Slope | Brookfield 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 r/min 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 r/min point) to the observed dial reading/set ppm point. When an apparent yield stress exists, this slope is much greater at low r/min than at high.

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

X1.6 Ideally, apparent yield stress can be subtracted from dial readings to give a constant dial reading/r/min 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.

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

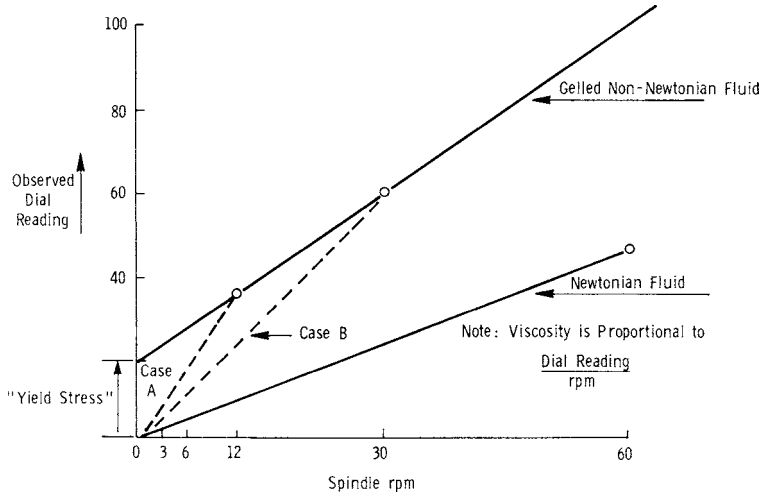



**FIG. X1.1 Diagram of Brookfield Dial Reading Versus r/min**

## 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 |
| 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 BROOKFIELD LV VISCOMETERS WITH LV-4 SPINDLES

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

$$T = 1.253 \times M \quad \text{(X3.1)}$$

where:
$T$ = 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):

$$S = 0.2156 \times \text{r/min} \quad \text{(X3.2)}$$

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

NOTE X3.1—Equations are derived from Brookfield Engineering Laboratories, Inc., literature. Brookfield Engineering Laboratories 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 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 ± 3°C for 60 ± 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.

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1486.

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 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.

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

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