



# Standard Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer<sup>1</sup>

This standard is issued under the fixed designation D 2983; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

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

## 1. Scope

1.1 This test method describes 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 1 000 000 mPa·s (cP). The bath-controlled temperature is selected within the range of +5° to –40°C.

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

1.3 *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:

D 341 Standard Viscosity-Temperature Charts for Liquid Petroleum Products<sup>2</sup>

### 2.2 European Procedure:

CEC L18-A-80<sup>3</sup>

## 3. Terminology

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

3.1.1 *apparent viscosity*—the 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. See Appendix X1 for a brief explanation.

3.1.2 *reference viscosity*<sup>4</sup>—the 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 A 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.

## 5. Significance and Use

5.1 The low-temperature, low-shear-rate viscosity of gear oils, automatic transmission fluids, torque and tractor fluids, and industrial and automotive hydraulic oils, Annex A4, are of considerable importance to the proper operation of many mechanical devices. Measurement of the viscometric properties of these oils and fluids are often used to specify their acceptability. 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 or extrapolation of experimental data.

NOTE 1—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 versus temperature plots are required, they can be made by the procedure outlined in Annex A1.

## 6. Apparatus

6.1 *Brookfield Viscometer*<sup>5</sup>—Analog Model LVT or more recent digital models (for example, LVDV-II+) are required.

<sup>5</sup> 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. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee<sup>1</sup>, which you may attend.

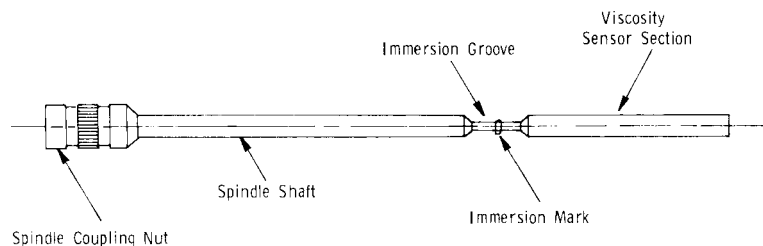
<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>3</sup> Available from CEC, Mandou Plaza-25th Floor, B-1210 Brussels, Belgium.

<sup>4</sup> 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. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee<sup>1</sup>, which you may attend.



NOTE 1—Adapted from drawings of Brookfield Engineering Laboratories.

**FIG. 1 Diagram of No. 4 LV Cylindrical Spindle**

6.2 *Viscometer Spindle*<sup>5</sup>—Uninsulated viscometer No. 4 spindle or insulated No. 4B2 spindle may be used. Periodically inspect for wobble, and for No. 4B2 spindles, ensure firm adhesion of the lower part of the spindle. A number of spindles are needed for multiple determinations. See Fig. 1 for diagram.

6.3 *Spindle Clip*<sup>6</sup>—A thin clip or spacer that supports the spindle at proper immersion depth during cool-down.

6.4 *Test Cell*<sup>6</sup>—A glass test tube 22 to 22.5 mm in inside diameter and 115 ± 5 mm in overall length.

6.5 *Cell Stopper*<sup>6</sup> (Fig. 2)—A cap that fits onto the test cell with a hole large enough for the spindle to turn with sufficient clearance.

6.6 *Insulated Cell Carrier*<sup>6</sup> (Fig. 2)—A balsa wood block with windows that keeps the test cell cold during testing.

NOTE 2—A liquid bath<sup>6</sup>, 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 10).

6.7 *Cold Cabinet*<sup>6</sup>—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, 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.

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.

6.8 *Turntable*<sup>6</sup>—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. For air-bath-style cold cabinets, it is recommended to use IP Brookfield Viscometer Total Immersion Thermometers (listed in 6.9.1) 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.

6.9.1 For liquid-style baths, it is recommended to use the IP Brookfield Viscometer Total Immersion Thermometers IP 94C (-45° to -35°C), IP 95C (-35° to -25°C), IP 96C (-25° to -15°C), IP 97C (-15° to -5°C), and ASTM 63C (-8° to +32°C) to cover this range. ASTM numbers for these thermometers are 122C, 123C, 124C, 125C, respectively. As with the air bath, these must also be calibrated with reference to an RTD in order to get an accurate reading. See also Annex A4. (**Warning**—Store thermometers in an upright position to help maintain calibration. Make certain that there are no separations in the column. Mercury-thallium amalgam used in ASTM 122C (IP 94C) is toxic if the thermometer is broken.)

6.10 *Blank Sample*—A fluid that is close in chemistry to those being tested for the purpose of determining the temperature experienced within a sample.

## 7. Use of Reference Fluids

7.1 The 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 Annex A3. Although the dial reading limits listed 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.

## 8. Procedure

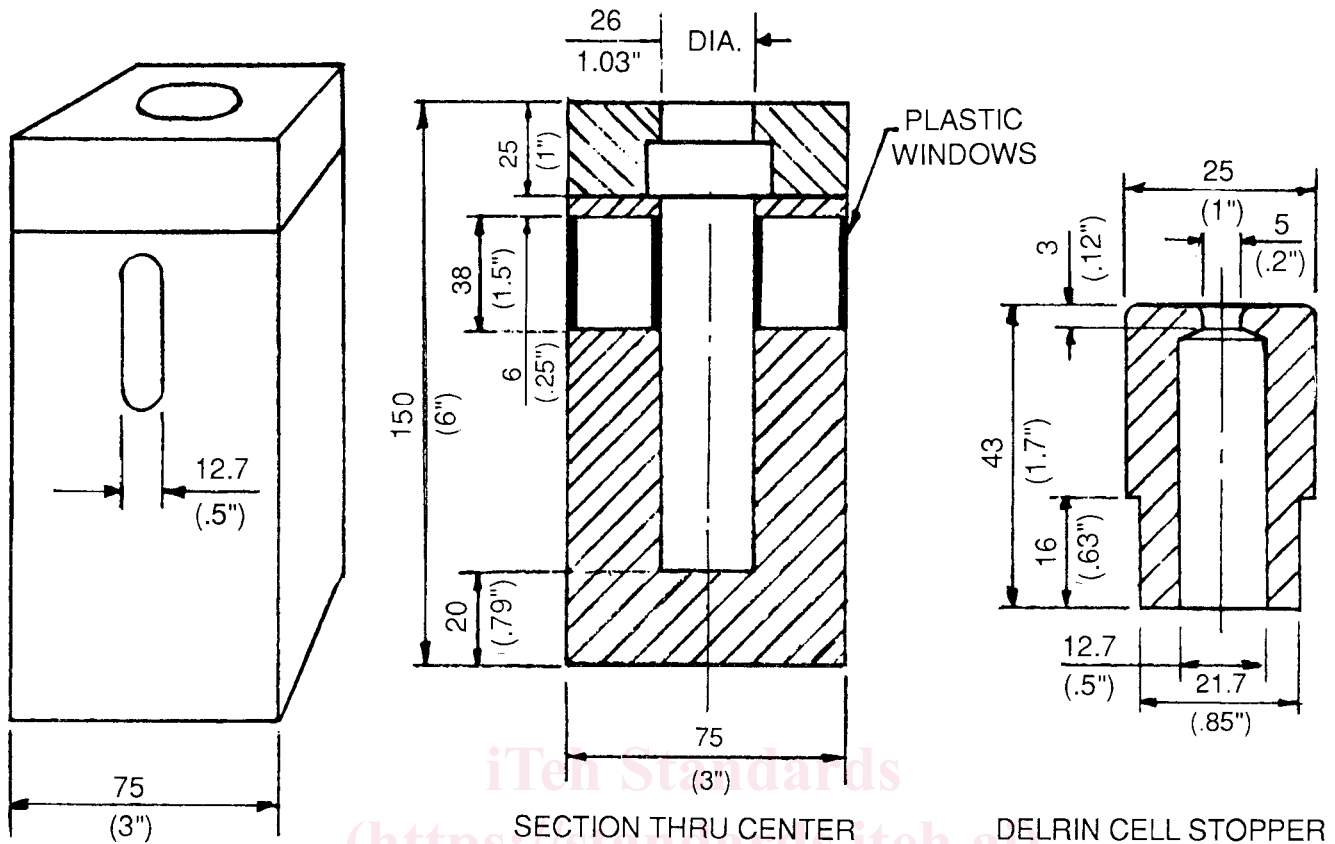
### 8.1 Preparation of the Bath:

8.1.1 Set 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.

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

NOTE 4—If a temperature adjustment is made, it may require at least 1

<sup>6</sup> These items are available from Lawler Manufacturing, Inc., 7 Kilmer Ct., Edison, NJ 08817.



BALSA WOOD CELL CARRIER

FIG. 2 Cell Carrier and Stopper

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:

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.

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 Seal each sample with an airtight cover such as a finger cot. Precondition the sample to 50 ± 3°C for 30 ± 5 minutes.

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 test cells and allow to cool to room temperature (25° ± 5°C) and remove the airtight seal.

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 Fig. 1 for diagram.

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 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 in too many cell carriers into the air chamber. This can be checked by contacting the cold cabinet manufacturer.

8.5 Once 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).

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

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

8.6.2 For 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.

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

8.7 After 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):

8.7.1 Check the level of the viscometer.

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

8.7.2 Record the temperature of the blank sample.

8.7.3 Turn the turntable rotation and the air blower off.

8.7.4 Allow 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.

NOTE 10—If the laboratory is equipped with a low-temperature liquid bath capable of maintaining test temperature within  $\pm 0.1^\circ\text{C}$  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.

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

8.7.6 Place 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.

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

8.7.8 Look 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.

**TABLE 1 RPM Selection Chart**

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

Spindle Speed, rpm	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	4 000	200 000 to 400 000
3.0	2 000	100 000 to 200 000
6.0	1 000	50 000 to 100 000
12.0	500	20 000 to 50 000
30.0	200	9 800 to 20 000
60.0	100	500 to 9 800

8.7.9 Center 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.

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

8.8.1 Refer to Section 9 for the proper rpm setting. Use the highest viscosity reading after the first 5 s of rotation.

8.8.2 Record viscosity reading (mPa·s), spindle speed (rpm), and test temperature ( $^\circ\text{C}$ ).

8.9 For 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 150 000 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 Table 2 for test times.

8.10 The total test series must be completed within 2 h so that the maximum soak time of 18 h is not exceeded.

8.11 After using the first cell carrier, return it to the bath for reconditioning and use the others in turn if necessary for further testing.

NOTE 12—Frequent opening of the cold box during a long series of runs may cause a temperature rise in the test samples. It is essential to turn the air circulation device off and allow it to come to a complete stop before opening the top. Do not leave the top open unnecessarily. The test series should be run as quickly as possible.

8.12 Upon completion of testing, empty the cells and clean all parts with a suitable hydrocarbon solvent making sure all parts are clean and free of oil.

## 9. RPM Selection

9.1 Because many lubricant fluids are non-Newtonian at low temperatures, the rpm selected for a measurement can strongly influence the resultant viscosity (see Appendix X1). For this reason, Table 1 lists the viscosity range for each rpm.

9.2 If an expected apparent viscosity is known, use the highest rpm corresponding to the known viscosity range. Table 1 must be used in selecting the appropriate rpm. Table 1 also provides the multiplication factor for viscometers without a viscosity readout or if the older analog viscometer is used. Simply multiply the torque display or the dial reading by the

**TABLE 2 Standardized Observation Times**

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.
1.5	3	4.5	
3.0	3	9	
6.0	2	12	Highest dial reading seen during observation time.
12.0	1	12	
30.0	30s	15	Observe dial reading at end of 30 s. Do this twice and record the higher reading.
60.0	30s	30	

number provided for the speed selections used. Make certain that the torque value (or dial reading) is as close to mid-range as possible (that is, 50).

9.3 If the expected viscosity range of the sample is unknown, a first sample must be used to determine the highest rpm that gives an acceptable viscometer reading. This is accomplished by increasing speed in steps from 0.6 to 60 rpm. The second sample is then run at the previously determined speed and only this result is reported.

## 10. Calculation

10.1 For viscometers without direct viscosity readout, calculate the viscosity at the test temperature of the test oil or reference oil as shown in Table 1.

10.2 The shear stress and shear 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 apparent viscosity, the test temperature, and the test rpm. RPM 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, a full report of the Newtonian reference fluid, its reference viscosity, its apparent viscosity, and its test rpm must accompany the test fluid data of 9.3. Reference fluid data are needed to ensure that different laboratories run at the same temperature, shear rate, and viscometric conditions.

## 12. Precision and Bias

### 12.1 Statement of Precision:

12.1.1 *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 and correct operation of the test method, exceed the values indicated below only in one case in 20.

$$\text{Repeatability} = 3.79 (X/1000)^{1.7}$$

where:

$X$  = the apparent viscosity, in mPa·s.

12.1.1.1 *For Example Only*—the following shows the repeatability at several viscosities:

20 000 mPa·s: 616 mPa·s	100 000 mPa·s: 9 520 mPa·s
50 000 mPa·s: 2 930 mPa·s	200 000 mPa·s: 30 930 mPa·s

12.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the values indicated below only in one case in 20.

$$\text{Reproducibility} = 11.34 (X/1000)^{1.7}$$

where

$X$  = the apparent viscosity, in mPa·s.

12.1.2.1 *For Example Only*—the following table shows the reproducibility at several viscosities:

20 000 mPa·s: 1 850 mPa·s	100 000 mPa·s
28 560 mPa·s	
50 000 mPa·s: 8 790 mPa·s	200 000 mPa·s
92 790 mPa·s	

12.1.3 No information can be presented on the bias of the procedure in Test Method D 2983 for measuring viscosity because no material having an accepted reference value is available.

### 12.2 General Considerations:

12.2.1 The interlaboratory study for the above precision statement was made on two groups of samples. For viscosities within the range of 500 to 1700 mPa·s, 6 hydraulic oils were evaluated in ten laboratories. For viscosities within the range of 17,000 to 200,000 mPa·s, 8 formulated oils were tested in eleven laboratories.

12.2.2 All laboratories utilized Brookfield viscometers for analysis.

**(Mandatory Information)**
**A1. DETERMINATION OF THE BROOKFIELD VISCOSITY VERSUS TEMPERATURE FUNCTION OF AUTOMOTIVE FLUID LUBRICANTS**

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 versus temperature plots may be useful.

A1.2 Brookfield viscosity versus temperature plots are made by measuring Brookfield viscosity at at least three temperatures and plotting a smooth curve of the logarithm of Brookfield viscosity against temperature. Commonly available semilog paper is suitable for these plots.

A1.3 When Brookfield viscosity versus temperature plots are used for interpolation, the interpolation temperature or viscosity must be within the range of measured data.

A1.4 Special temperature scales may be useful for some applications. Such scales would include the Kelvin instead of the Celsius measurement temperature and the reciprocal of the measurement temperature.

A1.5 The following precautions should be recognized to

avoid misinterpretation of Brookfield viscosity versus temperature plots.

A1.5.1 Extrapolation beyond the range of measured data should be avoided because many automotive fluid lubricants are non-Newtonian at low temperature. The gel structure associated with such non-Newtonian behavior may undergo rapid changes with temperature that are not predicted by simple extrapolation.

A1.5.2 The ASTM viscosity-temperature graphs (Charts D 341) should not be used for extrapolation of Brookfield data or for linear interpolation over a wide temperature range. Again, the non-Newtonian character of many automotive fluid lubricants at low temperature is the reason this test method is not applicable.

A1.5.3 Brookfield viscosities involved in plots that are compared between laboratories must be taken at the same rpm in each laboratory. Because of non-Newtonian behavior, Brookfield viscosity is dependent on rpm. If different rpm measurements are taken in different laboratories, widely different viscosities may be reported.

**A2. TYPICAL SAMPLE COOLING RATES IN BROOKFIELD AIR BATHS**

A2.1 This annex is intended to serve as a guide to Brookfield bath manufacturers. Sample cooling rates in Brookfield baths are considered important because the gel structure of some automotive fluid lubricants is dependent on the rate of cooling. This gel structure influences apparent Brookfield viscosity.

A2.2 The temperature of a sample immersed in a precooled bath should follow the equation:

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

where:

$S$  = sample temperature at observation time

$t$  = elapsed time from start of cooling

$B$  = bath temperature, and

$k$  = cooling constant with units of  $\text{time}^{-1}$ .

Eq A2.1 solves to:

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

where:

$C$  = integration constant, and

$e$  = base Napierian logarithms (2.71828+).

Eq A2.3 may be conveniently plotted as:

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

A2.3 When temperature is in degrees Fahrenheit, a sample in an average air bath 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. Baths that cool samples at rates defined by these limits and meet other method requirements are satisfactory for Brookfield viscometry of automotive fluid lubricants.