

Submitted for recognition as an American National Standard

(R) METHOD OF VISCOSITY TEST FOR AUTOMOTIVE TYPE ADHESIVES, SEALERS, AND DEADENERS

1. **Scope**—This SAE Recommended Practice contains a series of test methods for use in measuring the viscosity of automotive-type adhesives, sealers, and deadeners.

The test methods which are contained in this document are as follows:

- 1.1 Brookfield® Method
- 1.2 Castor-Severs Rheometer or Pressure Flowmeter
- 1.3 Penetrometer
- 1.4 Capillary Rheometer
- 1.5 Plate Rheometers

2. **References**

- 2.1 **Applicable Documents**—The following publications form a part of this specification to the extent specified herein.

- 2.1.1 ANSI/ASME PUBLICATION—Available from ANSI, 11 West 42nd Street, New York, NY 10036-8002.

ANSI/ASME B40.1—Pressure Gauge

- 2.1.2 ASTM PUBLICATIONS—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 5—Needle Penetrometer

ASTM D 217—Cone Penetrometer

ASTM D 1823—Standard Test Method for Apparent Viscosity of Plastics and Organosols at High Shear Rates by Extrusion Viscometer

ASTM D 1824—Standard Test Method for Apparent Viscosity of Plastics and Organosols at Low Shear Rates

ASTM D 2196—Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield®) Viscometer

ASTM D 2240—Durometer

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- ASTM D 3364—Standard Test Method for Flow Rates for Poly(Vinyl Chloride) and Rheologically Unstable Thermoplastics
- ASTM D 3835—Standard Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer
- ASTM D 4440—Standard Practice for Rheological Measurement of Polymer Melts Using Dynamic Mechanical Procedures

3. **Principle of Methods**—The viscosity of a material is a measurement of the shear stress that must be applied before a change of form (shear rate) can be accomplished. For the purpose of this test method, however, viscosity refers to a finished product parameter that is quantified in various ways and used by the automotive industry as a means of identifying and assigning a minimum and maximum value for the application characteristics of a material.

The use of these values should be tempered by the knowledge that the numbers generated are quality control aids and may not fully address the plant-to-plant variations encountered in application systems.

Each piece of equipment is best suited to a particular viscosity and shear rate range, although there are no sharp viscosity demarcations between the various viscometers. Each method has its own idiosyncrasies and limitations and therefore must be evaluated with the particular material being tested. The rheometers (1.4 and 1.5) have the ability to compare results under a range of conditions, and thus determine the sensitivity of the material to shear rate, shear history, and temperature. The following suggestions are intended as general guidelines for use of these viscosity measuring devices. They may be used outside the suggested ranges if desired.

Generally, a Brookfield® viscometer is used for low viscosity materials in the 0.025 to 200 Pa·s (Pa·s = 1000 cP) range with limited shear thinning. The pressure flowmeter is usually used for materials with viscosities up to 80 Pa·s at 50 s⁻¹. Since these materials are highly shear thinning they may exceed 1000 Pa·s at 1 s⁻¹. The penetrometer is used for very high viscosity materials such as putties and highly gelled compounds.

The capillary rheometer is used for materials from 1 to 10 000 Pa·s at shear rates from 10 to 50 000 s⁻¹. The plate rheometers are used for materials from 0.025 to 1000 Pa·s at shear rates from 10⁻⁷ to 200 s⁻¹.

4. **Sample Temperature Conditioning**—Prior to the actual viscosity measurement, the sample to be tested must be conditioned thermally.
- 4.1 **As Received Viscosity**—Unless specified otherwise, as received viscosity shall refer to material stored at "room temperature" and tested at 23 °C ± 1 °C (75 °F ± 2 °F) and 50% ± 5% R.H.
- 4.2 **Aged Viscosity**—Aged viscosity refers to a value obtained by subjecting a sample prior to testing to a specified temperature for 72 h. The sample is then conditioned to 23 °C ± 1 °C and the viscosity is measured. The size of the sample to be tested should be 500 mL (1 pt). Samples smaller than this conditioned at elevated temperatures may give different results.
5. **Sample Mechanical Conditioning**—Prior to the actual viscosity measurement, the sample to be tested must be conditioned mechanically.
- 5.1 **Conditioning Method A—Unagitated**—Unagitated testing refers to the transfer of materials from the shipping container to the testing apparatus with the minimum amount of agitation. This viscosity is supposed to be indicative of a compound in its shipping container prior to use. The tester should be aware that the very act of removing a sample for test can affect the rheology of thixotropic materials and, in so doing, may substantially alter the value measured.

5.2 Conditioning Method B—Moderate Agitation—Moderate agitation of a sample is achieved by stirring a 500 mL sample 50 stirs in 60 s with a 25 x 150 mm (1 x 6 in) steel bladed spatula. This agitation can also be used to assure the uniform 23 °C necessary for testing.

5.3 Conditioning Method C—Grease Working—Grease working refers to subjecting a sample to 150 cycles, unless otherwise specified, in a standard mechanical grease-worker, as outlined in ASTM D 217.

5.4 Conditioning Method D—Pre-shear—Steady shear rheometers including Brookfield® and Plate devices allow the sample to be pre-sheared at a fixed shear rate for a known time before testing. Ten s⁻¹ for 10 min is a suggested condition.

6. Brookfield® Method

6.1 Application—This procedure is used to determine the viscosity of adhesives, sprayable sound deadeners, and thin body sealers. The values obtained are expressed in Pa•s (Pa•s = 1000 cP) and are a measure of the resistance a rotating spindle encounters when immersed in the compound to be tested. Due to secondary flows, this method provides only relative values for shear thinning materials. Refer to ASTM D 1824 and ASTM D 2196 for additional details and reproducibility data.

6.2 Equipment—Commercially available Brookfield® Viscometer (see Figure 1), or equivalent.

6.3 Procedure—Test equipment and sample shall be maintained at 23 °C ± 1 °C.

6.3.1 Set viscometer at specified rpm.

6.3.2 Attach specified spindle.

6.3.3 Lower viscometer into 500 mL of test material so that the groove cut in the spindle is flush with the fluid's level.

6.3.4 Level the viscometer.

6.3.5 Depress the clutch, turn on the viscometer, and release the clutch. Take the reading and record the time after the indicator has stabilized or after a specified time, typically 60 s. (When measuring thixotropic materials at specific times, other than stable readings, it will be necessary to test separate samples in order to obtain reproducible values.)

6.3.6 Use conversion table to convert to centipoise.

6.3.7 Take the average of three readings and report the viscosity, spindle, rpm, time of recording, and the model of the viscometer used.

6.3.8 Repeat the procedure at a second rpm, and note extent of shear thinning. (Note that Brookfield® results are a relative measure for thixotropic or shear thinning materials.)

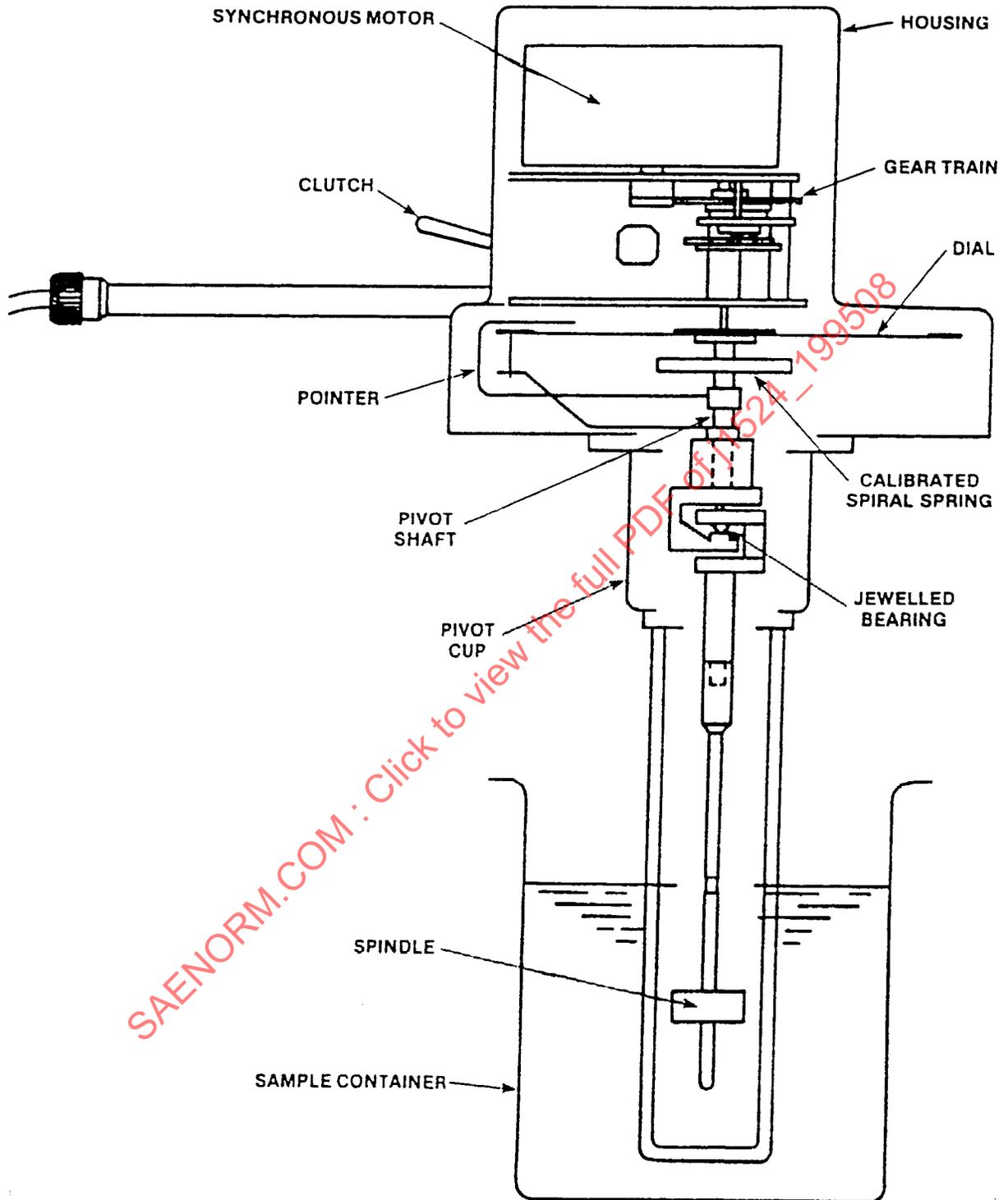


FIGURE 1—BROOKFIELD® VISCOMETER

7. Castor-Severs Method/Pressure Flowmeter Method

7.1 Application—This procedure is used to determine the viscosity of adhesives, sealers, and sprayable sound deadeners. The values obtained are expressed in seconds and are a measurement of the time needed to extrude a specified mass (20 g) through a specified orifice at a specified pressure. These values can be converted to apparent viscosity in centipoise at a specified shear rate using the capillary rheometer procedure as in ASTM D 3835 or ASTM D 1823. If this step is taken, it is recommended to repeat the procedure at three pressures with at least two orifices to obtain a measure of shear thinning and thixotropy.

7.2 Equipment

7.2.1 Rheometer as shown in Figure 2.

7.2.2 Orifices are as follows in Table 1:

TABLE 1—RHEOMETER ORIFICES

No. mm (in)	Diameter mm (in)	Length mm (in)	Typical Use	Limits
1	1.32 (0.052)	13.5 (0.531)	Sprayable	Readings less than 10 s, use Brookfield® Method
2	2.64 (0.104)	13.5 (0.531)	Pumpable	Readings less than 10 s, use orifice 1
3	3.18 (0.125)	51.0 (2.008)	Deadeners	Readings less than 10 s, use orifice 2

Other orifices should be used if the data is to be analyzed by plotting viscosity versus shear rate. A minimum of two orifices is recommended to observe thixotropic behavior. The length should be a minimum of 10 times the diameter. The dimensions should meet the accuracy requirements specified in ASTM D 3835 6.1.3.

7.2.3 Air supply, 700 kPa (100 psi).

7.2.4 Pressure gauge, 700 kPa (100 psi) 1% (ANSI/ASME B40.1-1985 Grade 1A or better).

7.2.5 Pressure regulator and water/oil extractor for air line.

7.2.6 Pipe cleaners for cleaning the orifice.

7.2.7 Stop watch or other timing device calibrated in tenths of seconds.

7.2.8 Balance, double beam type or equivalent, sensitivity to 0.01 g.

7.2.9 Mechanical convection oven capable of maintaining a temperature of ± 1 °C (± 2 °F), or alternate means where temperature conditioning is specified.

7.2.10 Weight per gallon cup, or other means to determine density within 1%.

7.3 Procedure

- 7.3.1 Fill the clean, dry, sealer cup equipped with the specified orifice with the material to be tested. The material should be at $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ unless otherwise specified. Care should be taken to avoid air entrapment and to allow enough room for the plunger.
- 7.3.2 Adjust line pressure to 276 kPa (40 psi), unless otherwise specified. Open air valve and extrude material through orifice until entrapped air is eliminated. Shut air valve. When plotting viscosity versus shear rate using the equations from ASTM D 1823, it is desirable to repeat this procedure at four pressures. The recommended pressures are 69, 276, 483, and 690 kPa (10, 40, 70, and 100 psi).
- 7.3.3 Place a paper on the balance, tare and add specified weight (20 g).
- 7.3.4 Open air valve and start timer when the material touches the paper on the weighing pan.
- 7.3.5 When 20 g of sealer has accumulated on the balance pan, stop the timer, close the air valve, and open the pressure relief valve.
- 7.3.6 Take the average of three readings and report the number of seconds, pressure, and orifice length and diameter.

8. Penetration Method

8.1 Application—This procedure is used to determine the viscosity of heavy-bodied sealers, extrudable and die cut deadeners. In this method, viscosity is a measurement of the depth of penetration by a cone or needle into the material tested and is expressed in 0.1 mm of penetration.

8.2 Equipment

- 8.2.1 Commercially available penetrometer (Figure 3), as described in Table 2.

TABLE 2—PENETRATION INSTRUMENTS

No.	Penetrometer	Typical Use	Limits
1	Cone ASTM D 217	Deadeners	Readings less than 5 mm use Penetrometer No. 2
2	Needle ASTM D 5	Thumbable Materials	Readings less than 5 mm use Durometer hardness ASTM D 2240

- 8.2.2 Stop watch
- 8.2.3 Weights to place on loading bar
- 8.2.4 Penetration instruments (cone ASTM D 217 and needle ASTM D 5).
- 8.2.5 SAMPLE CUP—75 mm diameter by 60 mm depth, or as specified.

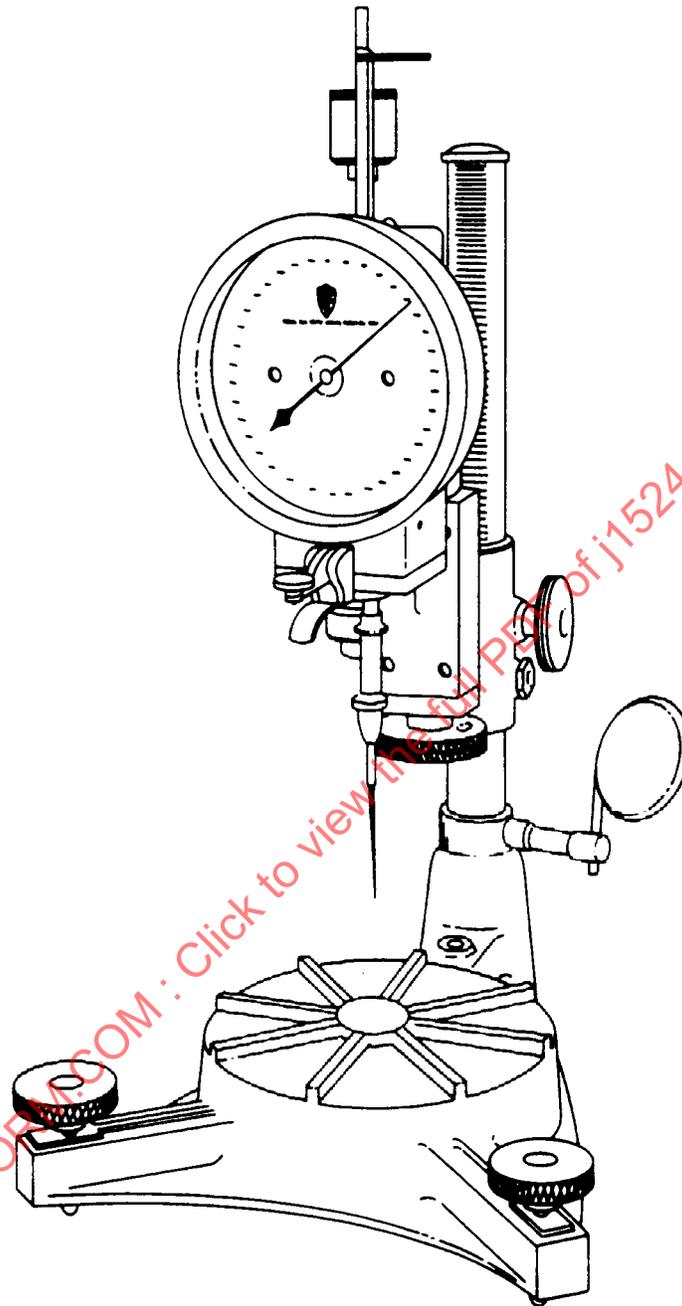


FIGURE 3—PENETROMETER

8.3 Procedure—Test material at $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

8.3.1 Level penetrometer.

8.3.2 Insert specified penetrating instrument into chuck.

8.3.3 Set dial reading to zero.

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- 8.3.4 Add weight to loading bar, as needed, to achieve the specified moving load. Use a 150 g total weight, including rod and added weights, unless otherwise specified.
- 8.3.5 Place the sample to be tested on the center of base plate.
- 8.3.6 Adjust the zeroed height of the penetrometer so that the point of the penetrating instrument is exactly in contact with the surface of the material to be tested.
- 8.3.7 Release test rod by depressing the trigger and keep in this position for 5 s unless otherwise specified.
- 8.3.8 At the end of the specified "drop" period, release the trigger.
- 8.3.9 Gently push the depth gage rod down as far as it will go and record the depth of penetration, units are in 0.1 mm.
- 8.3.10 Take the average of three readings and report the depth of penetration, moving load, and penetration time.

9. Capillary Rheometer Method

9.1 Application—This method is used to determine the viscosity versus shear rate characteristics of adhesives, sealers, and deadeners per ASTM D 3835 (ISO DIS11443). The basic test determines apparent viscosity versus apparent shear rate over a typical shear rate range of 100 to 50 000 s⁻¹. Various corrections are outlined in ASTM D 3835. The piston in barrel arrangement outlined in ASTM D 3835 is preferred for reaching high-shear rate, but the apparatus in Figure 2 and ASTM D 1823 is acceptable as a moderate shear rate screening tool (10 to 100 s⁻¹). Round Robin results are reported in ASTM D 1823.

9.2 Equipment

- 9.2.1 Capillary Rheometer per ASTM D 3835 (ISO DIS11443) and associated orifices (preferred).
- 9.2.2 Castor Severs rheometer as in Figure 2 is an alternate with limited shear rate range. This rheometer will require a balance, a timer, and a means to determine density within 1%, and thus volumetric flow rate. This will follow ASTM D 1823.
- 9.2.3 A set of orifices chosen for the material as outlined in the ASTM specification. If a Bagley plot is not generated, then a length to diameter ratio of at least 10 is desired to minimize the effect of entrance pressure losses. Diameters from 1 to 4 mm (0.050 to 0.150 in) and lengths from 13.5 to 100 mm (0.5 to 4 in) are well suited to the pressures available in the Castor Severs rheometer.

9.3 Procedure—Follow ASTM D 3835 (ISO DIS11443), this is a summary.

- 9.3.1 Clean and inspect barrel and capillary.
- 9.3.2 Charge the barrel with material with minimum air pockets.
- 9.3.3 Place the piston in the barrel, extrude a small quantity, and preheat for at least 6 min.
- 9.3.4 Start extrusion. Verify that steady state is reached. Record force (or pressure) and data necessary to calculate volumetric flowrate Q.
- 9.3.5 Repeat to check for flow stability. Report degree of repeatability observed. Repeat at desired shear rates. Refill barrel and re-equilibrate as necessary.

9.3.6 Repeat with at least one other orifice. Differences between curves from different orifices are due to change in shear history or slip at the wall.

9.3.7 Discharge any remaining material and clean the apparatus.

9.4 Reporting—Capillary results are typically reported on a viscosity versus shear rate plot or a shear stress versus shear rate plot as in Appendix A. The equations to derive this are in ASTM D 3835 and D 1823 and are summarized as follows in Equations 1 to 3. Plot individual data points so that repeatability is apparent on the graph, rather than averaging results. If the Castor-Severs type rheometer (8.2.2) is substituted, this should be noted.

$$\text{Shear Stress, Pa} = Pr/2L \text{ (at the wall)} \quad (\text{Eq. 1})$$

$$\text{Shear Rate, s}^{-1} = 4Q/\pi r^3 \text{ (Apparent shear rate, uncorrected)} \quad (\text{Eq. 2})$$

$$\text{Viscosity, Pa} \cdot \text{s} = P\pi r^4/8LQ \text{ (Apparent viscosity, uncorrected)} \quad (\text{Eq. 3})$$

where:

P = Barrel pressure, Pa

r = Capillary radius, m

L = Capillary length, m

Q = Volumetric flow rate, m³/s = Mass flow rate (kg/s)/density (kg/m³)

It is also important to record observations made during the test as to repeatability and stability of the extrusion. If stability is a concern, ASTM D 3364 can provide guidance in detecting and controlling the level of instability. When requested, it is desirable to perform the Rabinowitsch Shear Rate correction outline in ASTM D 3835 section 11.8 and the Bagley entrance correction outlined in ASTM section 11.7 and Appendix A. Various curve fits to the data may be requested, and a goodness of fit, which often relates to the flow instability of the material, should be reported.

10. Plate Rheometer Method

10.1 Application—This procedure is used to determine the viscosity versus shear rate characteristics of adhesives, sealers, and deadeners. This test uses instrumentation available for dynamic mechanical measurements on polymer melts as outlined in ASTM D 4440. However, these materials are generally not linear viscoelastic, so processing viscosity is not generally the same as complex viscosity. Therefore, it is typically desirable to use these instruments in steady shear mode and report apparent viscosity.

10.2 Equipment—A variety of equipment is commercially available for this type of testing. Both controlled stress and controlled rate units are available, and each have advantages in some measurements. The majority of units are rotational, but sliding plate rheometers are also available. The rotational units utilize both cone and plate and parallel plate geometries. While the cone and plate subjects all the material to a constant shear rate, the parallel plate geometry is typically preferred due to the interference of particles present in the formulation. The parallel plate geometry is required for temperature sweeps to allow compensation for tool expansion.

10.3 Procedure—Follow ASTM D 4440 where applicable. This is a summary.

10.3.1 Equilibrate the test fixture at the desired test temperature.

10.3.2 Touch the plates together and zero the gap indicator.

10.3.3 Open the tool and apply an adequate amount of material to the test fixture. Minimize bubbles as much as possible.

- 10.3.4 Close the test fixture to an appropriate gap, 1 to 3 mm is recommended. Remove excess material. Allow the sample to equilibrate to the desired initial temperature.
- 10.3.5 Program the desired test.
- A thixotropic loop is recommended as a qualitative measure of the effect of time and shear history. Note that this test can be dependent on sample preparation.
 - A steady shear (or stress) sweep where the sample is allowed to equilibrate at each shear condition is also recommended.
 - A temperature sweep may be desired. Before performing a temperature sweep, monitor viscosity versus time at the shear conditions that the sweep will be performed to insure that shear history does not affect results. If viscosity changes with time, separate tests will have to be conducted at each temperature. Note that only the controlled rate instruments will allow viscosity versus temperature to be plotted at constant shear rate. A constant shear stress temperature sweep will combine the change in viscosity due to temperature and shear thinning as the shear rate increases.
- 10.3.6 Conduct the desired test. Watch the sample and stop the test if the sample slips away from one plate or separates at the edges. Edge separation limits the maximum valid shear rate. Edge separation is indicated if torque drops at higher shear rates. For an equilibrium sweep (10.3.5b), results obtained prior to separation may be reported. For a thixotropic loop, the test must be repeated with a lower maximum shear rate value. In this test even a small edge separation will appear as hysteresis in the material.
- 10.3.7 A new sample should be loaded for each test as these materials are sensitive to shear history.
- 10.3.8 Remove the sample and clean the test fixture.
- 10.4 Reporting**—Rheometer tests are typically reported as a plot of viscosity versus shear rate or shear stress versus shear rate as in Appendix A. Include the type of instrument, test geometry, gap setting, and temperature. An overlay of the thixotropic loop and equilibrium sweep data is desired to demonstrate the degree of sensitivity to shear history. Various curve fits may be applied as requested.
- 11. Notes**
- 11.1 Marginal Indicia**—The change bar (|) located in the left margin is for the convenience of the user in locating areas where technical revisions have been made to the previous issue of the report. An (R) symbol to the left of the document title indicates a complete revision of the report.
- 11.2 Key Words**—Viscosity, sealer, adhesive, rheology

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APPENDIX A

A.1 Sample plots for the reporting of viscosity versus shear rate data are attached. Shear rate is plotted as the abscissa (x axis) (Figure A1), and either viscosity or shear stress is plotted as the ordinate (y axis) (Figure A2). The viscosity plot is more common, but the shear stress plot allows extrapolation to determine the yield stress. Log/Log plots are preferred because the values typically vary by more than one order of magnitude and changes in the lower values will not be obvious on a linear plot.

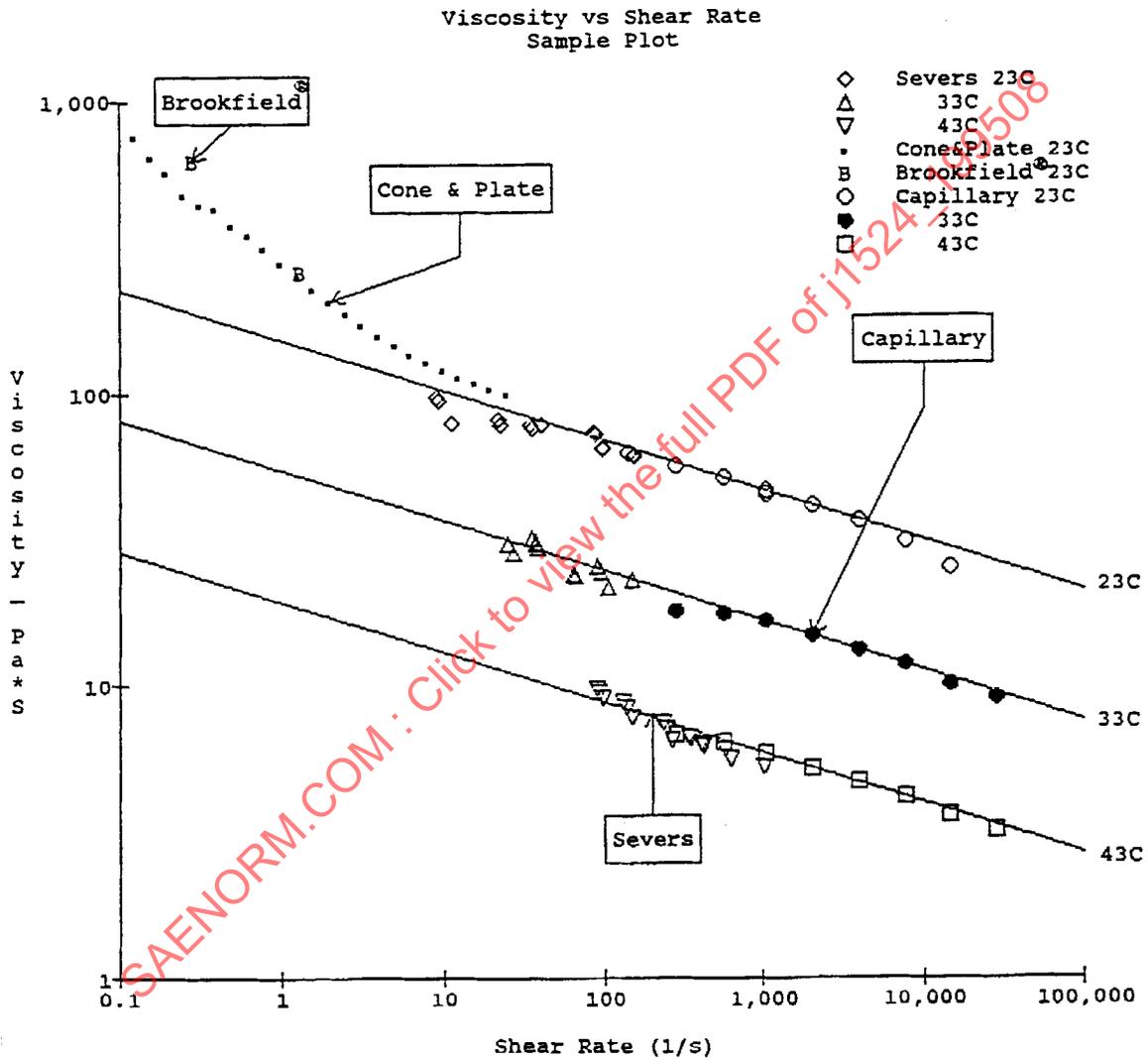


FIGURE A1—VISCOSITY VERSUS SHEAR RATE SAMPLE PLOT