Designation: D1986 – 14

Standard Test Method for Determining the Apparent Viscosity of Polyethylene Wax[1]

This standard is issued under the fixed designation D1986; 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.

1. Scope

1.1 This test method describes the determination of the viscosity of polyethylene wax using small-volume, concentric cylinder, rotational viscometer and a temperature bath at 140°C.

1.2 The values stated in SI units are to be regarded as standard. The units in parentheses are provided for user convenience and are not standard.

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:*[2]

E1970 Practice for Statistical Treatment of Thermoanalytical Data

3. Terminology

3.1 *Definitions:*

3.1.1 *polyethylene wax*—polymerized ethylene with a molecular weight 2,000 to 10,000 g/mol and a density of 0.9 to 1.0 g/cm^3. These polymers may be oxidized or copolymerized but should have a melt point lower than 140°C.

3.1.2 *viscosity*—the ratio of shear stress to shear rate. The viscosity of a liquid is a measure of the internal friction of the liquid in motion.

3.1.2.1 *Discussion*—The unit of viscosity is the pascal second (Pa·s). For a Newtonian liquid, the viscosity is constant at all shear rates. For a non-Newtonian liquid, viscosity will vary depending on shear rate.

3.1.3 *apparent viscosity*—the viscosity determined by this test method and expressed in millipascal seconds (mPa·s or centipoises (cP)). (1 cP=1 mPa·s). Its value may vary with the spindle and rotational speed selected because many waxes are non-Newtonian.

4. Significance and Use

4.1 This test method is used to determine a physical property of a wax and may determine the utility of the wax, as well as being a significant quality control test.

5. Apparatus

5.1 *Small-volume, Coaxial Rotational Viscometer*[3]—The essential instrumentation required providing the minimal rotational viscometer analytical capabilities for this method include:

5.1.1 *Drive Motor,* to apply an unidirectional rotational displacement to the specimen at a rate of 0.05 to 6.0 rad/s (0.5 to 60 r/min) constant to ±1 %.

5.1.2 *Force Sensor,* to measure the torque up to 0.6 mN·m developed by the specimen to the rotational displacement of the rotational element.

5.1.3 *Coupling Shaft,* or other means to transmit the rotational displacement from the motor to the specimen.

5.1.4 *Rotational Element, Spindle or Tool,* to fix the specimen between the drive shaft and a stationary position. A coaxial spindle with the dimensions shown in Fig. 1 is suitable for this test method.

5.1.5 *Data Collection Device,* to acquire and display measured or calculated signals from the test. The minimum output signals required for apparent viscosity are torque, rotational speed, temperature and time.

5.1.6 *Stand,* to support, level and adjust the height of the drive motor, shaft and rotational element.

5.1.7 *Auxiliary Instrumentation,* useful in conducting this test method includes:

5.1.7.1 *Data Analysis Capability,* to provide viscosity, stress or other useful parameters derived from measured signals.

5.1.7.2 *Level,* to indicate the vertical plumb of the drive motor and shaft geometry.

[1] This test method is under the jurisdiction of ASTM Committee D21 on Polishes and is the direct responsibility of Subcommittee D21.02 on Raw Materials.

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

[3] Available from most manufacturers of precision laboratory instruments. Many versions have an integral controlled temperature bath and other specified apparatus. The product literature or sales information usually specifies that the instrument is suitable for use in ASTM D1986.

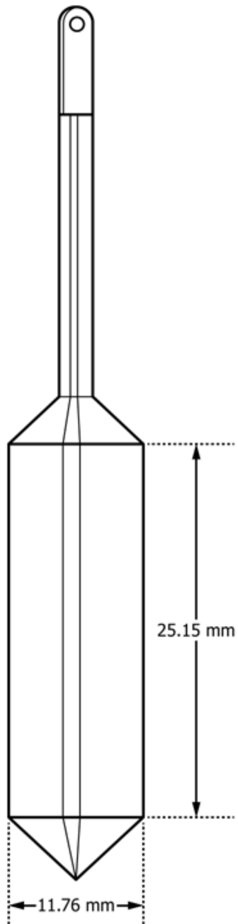

FIG. 1 Coaxial Spindle

5.2 *Temperature Sensor,* to provide an indication of the specimen temperature over the range of 130 to 160°C to within ±0.1°C.

5.3 *Temperature Bath,* to provide a controlled isothermal temperature environment for the specimen.

5.4 *Temperature Controller,* capable of operating the temperature bath at an isothermal temperature over the range of 130 to 160°C, constant to within ±0.5°C.

5.5 *Small Volume Container, with Insulating Cap,* with a capacity of 9 to 10 mL to hold the test specimen in the temperature bath during testing.

6. Calibration

6.1 The viscometer is calibrated using Newtonian fluids provided by the manufacturer. No zero adjustment is needed since experience has shown that the zero point will not vary a well maintained instrument. The viscometer instrument and spindles are precision equipment that should be protected from undue shock and mishandling. Physical damage to the instrument will often reveal itself as lack of pointer oscillation evenly about the zero point when the instrument is operated in air.

6.2 The instrument may be further calibrated using standard reference fluids. Suitable fluids are available in nominal viscosities up to 15 Pa·s at 149°C (300°F).[3] A procedure for instrument calibration using standard reference fluids should be available from the instrument manufacturer. Results obtained using standard reference fluids should not deviate from the nominal viscosity by more than 2 %.

6.3 To check the temperature controller and bath, place a quantity of polyethylene wax in the sample container and allow it to melt. Insert the thermal sensor through the insulating cap and fix it at a proper depth, but not in contact with the walls of the container. Adjust the temperature controller to provide the desired test temperature. Rotate the sensor during temperature reading to minimize the effect of thermal gradients in the sample. Continue temperature readings and controller adjustment until minimum deviation from test temperature is obtained. Minimum deviation may vary between laboratories, depending upon the controller, but in no case should exceed ±0.5°C. Repeat this procedure for any test temperature desired within the scope of this test method.

7. Procedure

7.1 *Preparation of Sample*—Place approximately 8 g of sample in the sample chamber. Melt the sample in the temperature bath preheated to 140°C with the insulating cap in place. Heat until the sample is molten and at the test temperature.

NOTE 1—Avoid temperatures excessively above the melt temperature or prolonged heating of the molten sample to minimize thermal and oxidative decomposition.

NOTE 2—If polyethylene wax has a melt point higher than 135°C, the procedure should be run at 150°C.

7.2 *System Alignment and Spindle Insertion*—With the viscometer raised, connect the spindle to the motor shaft. Lower the leveled viscometer and insert the spindle in the sample chamber until the tip of the alignment bracket is 2 mm (1⁄16 in.) above the top of the container and in contact with the back of the locating index. Place the insulating cap on the sample chamber.

7.3 Start the rotation of the spindle at the lowest rotational speed. When temperature of the test specimen is within ±0.1°C of the test temperature, stop the spindle rotation, remove the cap, raise the viscometer and spindle and inspect the liquid level on the spindle shaft. The liquid should extend about 0.6 to 1.3 cm (¼ to ½ in.) up the spindle shaft. If the liquid level varies significantly from this, add or remove sample.

7.4 Lower the spindle into the test specimen and start the spindle rotation of the spindle at the lowest rotational speed setting. Wait for the temperatures to equilibrate within ±0.1°C of the test temperature.

7.5 Once equilibrium is reached, select a rotational speed that will produce a scale reading between 10 and 95 of full scale, and preferably near the midpoint of the scale. Allow the spindle to rotate at this speed for 15 min before engaging the pointer clutch and stopping the motor to record the scale readings. Restart the motor, and allow at least five additional revolutions before taking a reading. Repeat this operation until three consecutive readings differ by no more than 0.5 units

7.6 After 15 min, engage the pointer clutch, stop the rotation of the spindle, and record the scale reading.

7.7 After 1 min, restart the spindle rotation at the same speed.

7.8 Repeat 7.6 and 7.7 until three consecutive readings are obtained which differ by no more than 0.5 %. Record these three values.

8. Calculations

8.1 Convert each of the three readings from 7.8 to viscosity, using the method described in the instrument instruction manual.

8.2 Calculate and report the mean viscosity of the three results of 8.1 (see Practice E1970).

9. Report

9.1 Report the following information with the determined mean apparent viscosity:

9.1.1 A complete description of the viscometer, including manufacturer, model number, spindle used, rotational speed and test temperature.

10. Precision

10.1 *Repeatability*—Duplicate results by the same operator and using the same viscometer shall not be considered suspect unless they differ by more than ±1 %.

10.2 *Reproducibility*—To be determined.

11. Bias

11.1 The procedure in this test method for measuring the apparent viscosity of polyethylene wax has no bias because the value for the viscosity is defined only in terms of this test method.

12. Keywords

12.1 polish; polyethylene wax; viscometer; viscosity; wax