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

This standard is issued under the fixed designation D 1986; 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 covers the determination of the viscosity in centipoise of polyethylene wax using the Brookfield LVT or LVF viscometer and thermosel system.

1.2 *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]

E 1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *polyethylene wax*—polymerized ethylene with a molecular weight 2,000 to 10,000 and a density of 0.9 to 1.0 g/cm^3. These polymers can 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. The units of viscosity is the pascal second. 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. 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 *Brookfield Thermosel System*, with LVT, LVF, or LVD 111 viscometer and SC 4-31 spindle.[3]

6. Calibration

6.1 The viscometer is precalibrated using Newtonian fluids provided by the manufacturer. No zero adjustment is provided since experience has shown that the zero point will not vary due to changes in the spring. The viscometer and spindles are precision equipment and should be kept from undue shock and mishandling. Physical damage to the instrument will often reveal itself as erratic or no oscillation of the pointer when the instrument, with or without the spindle in place, is operated in air. When operating normally, the pointer will be stable and have free oscillation about the zero point in air.

6.2 The instrument may be further calibrated using standard reference fluids. Suitable fluids are available in nominal viscosities up to 15 000 mPa·s at 149°C (300°F).[3] The procedure for instrument calibration using standard reference fluids is not encompassed by this test method. Results obtained using standard reference fluids should not deviate from the nominal viscosity by more than 2 %.

6.3 The temperature controller of the type recommended for this procedure is factory calibrated and has control capability of ±0.5 % of the control point (±1.0°C at 175°C). To further check the controller and further establish controller settings, use the following procedure. Place a sufficient quantity of polyethylene wax in the sample container to permit immersion of the appropriate ASTM thermometer to the proper depth. Do not permit the thermometer bulb to rest on the bottom of the sample container. Suitable thermometers are shown in Specification E 1.

6.4 Insert the thermometer through the insulating cover of the sample container and hold it in place at the point required for proper immersion depth. Adjust the thermal controller to provide the desired test temperature. Rotate the thermometer 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

[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 Brookfield Engineering Laboratories, Inc. 11 Commerce Boulevard, Middleboro, MA 02346.

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laboratories, depending upon the controller, but should in no case exceed ±0.5°C (0.9°F). Repeat this procedure for any test temperature desired within the scope of this procedure.

7. Procedure

7.1 Use spindle number 31 for this test.

7.2 *Preparation of Sample*—Place 8 g or approximately 10 mL of sample in the sample chamber. Melt the sample in the thermo-container preheated and set at 140°C. If polyethylene wax has a melt point higher than 135°C, the procedure should be run at 150°C. Heat for 15 min with insulating cap on sample chamber. Avoid excessive or prolonged heating of the sample to minimize thermal and oxidative effects.

7.3 *System Alignment and Spindle Insertion*—With the viscometer raised, connect the spindle to the coupling nut (note left-hand thread). Lower the leveled viscometer and insert the spindle in the sample chamber until the tips of the alignment bracket is 2 mm (1/16 in.) above the top of the thermo container and in contact with the back of the locating ring. Place insulating cap over the sample chamber.

7.4 Turn on viscometer at the lowest RPM setting. When temperature equilibrium is indicated, turn off viscometer, 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.

7.5 Lower viscometer and spindle and turn it on at the lowest RPM setting and wait for the temperatures to reach equilibrium. After equilibrium is reached, select a spindle speed (RPM), that will produce a scale reading nearest the midpoint of the scale between 10 to 95 unit range. 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.

8. Calculations

8.1 Convert the average of these three readings to millipascal seconds, by multiplying this value by the appropriate factor taken from instrument instruction manual.

9. Report

9.1 Report the following information:

9.1.1 Report the apparent viscosity at a given temperature along with the particulars of the instrument model, the spindle number and rotational speed.

9.1.2 *Conversion*:

$$\text{One pascal second (Pa·s)} = 1000 \text{ cP} \quad (1)$$

$$\text{One millipascal second (mPa·s)} = 1 \text{ cP} \quad (2)$$

10. Precision and Bias

10.1 *Precision*—Duplicate results by the same operator and using the same model (LVT or LVF) viscometer shall not be considered suspect unless they differ by more than ±0.3 non-convert reading (95 % confidence limits for average). (This precision statement will be expanded in the future.)

10.2 *Bias*—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.

11. Keywords

11.1 Brookfield viscometer; polish; polyethylene wax; thermosel; viscosity; wax

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