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Standard Test Methods for
Use of Emulsion Polymers in Floor Polishes[1]

1. Scope

1.1 These test methods cover test procedures for emulsion polymers that are used in water-based floor polishes. The term "emulsion polymers" is used primarily to denote those materials produced by regular emulsion polymerization techniques, but may be extended to those polymers that are subsequently emulsified or dispersed after polymerization. Unless otherwise noted, the tests may be used for any polymer or copolymer systems. The methods appear in the following order:

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
E1 Specification for ASTM Liquid-in-Glass Thermometers
E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

E100 Specification for ASTM Hydrometers

3. Significance and Use

3.1 The purpose of this test is not to fully identify and characterize a polymer, but to identify a variety of basic parameters needed to predetermine the usefulness of the polymer in formulations as well as for Quality Control purposes.

3.2 A statement of precision and accuracy is not appropriate in this case.

4. Sampling

4.1 *Outline of Test Method*—Since stratification may occur in emulsion polymers, they must be thoroughly agitated to obtain a homogeneous blend as a representative sample. The procedure required differs with the type of container and facilities available.

4.2 *Sampling from Tank Cars*—Take three samples of at least 1 pt (473 mL) each, one at the center of the tank, another half way between the center and the bottom, and the third half way between the center and the top. Take the top sample first, then the center sample, and the bottom sample last. Use a weighted sampler with a remotely operated, removable top, or other suitable sampling device that will accomplish the same results. Determine the applicable specified or characteristic properties of the samples by the standard procedures or methods. If there is any evidence that stratification has occurred, then thoroughly agitate the contents of the car until samples obtained agree within 1 % of total solids.

4.3 *Sampling from Drums:*

4.3.1 *Blending of Contents*—Blend the emulsion polymer by one of the following methods:

4.3.1.1 *Method A*—If the drum is fitted with a bung and contains 2 % air space, lay it on its side and roll to and fro briskly for not less than 10 min. Then turn the drum upside down for about 15 min and repeat the rolling operation for an additional 10 min. If the drum contains less than 2 % air space, transfer the contents to a larger vessel and thoroughly stir, preferably by means of a perforated steel disk plunger. Stirring for about 10 min will normally suffice.

4.3.1.2 *Method B*—Agitate the contents of the drum by means of a suitable motor-driven stirrer for as long as is

necessary to obtain uniformity. Excessive stirring and unnecessary exposure of the emulsion polymer to air must be avoided. A suitable type of stirrer consists of a collapsible two-bladed stainless steel propeller of 110-mm minimum diameter, when fully opened, mounted on a stainless steel shaft sufficiently long for the propeller to be distant about one quarter the height of the emulsion polymer from the bottom of the drum. Operate stirrer at a minimum speed of 900 r/min. If desired, two propellers may be used on the same shaft, the lower one being near the end of the shaft. The shaft speed shall give a brisk turnover without creating a vortex. The part of the equipment immersed in the emulsion polymer must contain no copper or brass.

4.3.2 *Removal of Sample*—After blending, take the sample without delay. A suitable method is by slowly inserting a clean, dry, glass tube of not more than 15-mm internal diameter and open at both ends, until it reaches the bottom of the container. Then close the upper end of the tube and transfer the contents to a clean, dry sample bottle. Repeat the operation until sufficient emulsion polymer has been obtained.

4.3.3 *Bulk Sample*—Where samples are drawn from several containers, for example 10 % sampling of emulsion polymer in drums, or where samples are taken at different depths, for example from tanks, combine the samples and thoroughly blend by stirring or shaking immediately before taking the final average sample.

5. Total Solids

5.1 *Apparatus*—Tared aluminum dishes with a close-fitting cover, having a diameter of approximately 60 mm and a height of 15 mm.

5.2 *Procedure*—If the temperature of the emulsion polymer is above room temperature, allow it to cool to room temperature. Then weigh two samples of approximately 1 g each to the nearest 1.0 mg in tared aluminum weighing dishes. Dry the samples for 2 h in a convection or forced-draft oven at a temperature of 105 + 2, − 0.5°C. Remove the samples from the oven, cool the container and contents to room temperature in a desiccator, and weigh them to the nearest 0.1 mg. Average the values if they are within 0.1 %. If not, make additional duplicate determinations until a pair of duplicate determinations agree within 0.1 %.

5.3 *Calculation*—Calculate the percent of total solids as follows:

$$\text{Total solids, \%} = \frac{\text{weight of dry solids}}{\text{weight of sample}} \times 100 \qquad (1)$$

5.4 *Report*—Specify whether the oven used is a convection or a forced-draft type.

6. pH Value

6.1 *Apparatus*—Any pH electrometer and a glass electrode—calomel cell assembly may be used as described in Test Method E70. A flowing calomel electrode has been found particularly suited for the pH range of the latex being tested.

6.2 *Standard Solution,* having a pH of 10, or a standard solution having a pH approximately the same as that of the emulsion polymer to be tested.

6.3 *Procedure*—Before making a determination, take care that the instrument is properly standardized at frequent intervals with a standard solution (see 6.2) and that the electrodes are clean. Permit the polymer emulsion to come to equilibrium with the glass electrode before taking the final reading.

6.4 *Report*—Report the pH value for the polymer emulsion.

7. Apparent Viscosity

7.1 *Apparatus:*

7.1.1 *Sieve, U.S. No. 40 (425-µm).*

7.1.2 *Distillation Flask, Three-Necked,* equipped with stirring and vacuum connections.

7.1.3 *Viscometer, Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this test method include:

7.1.3.1 *Drive Motor,* to apply a unidirectional rotational displacement to the specimen of 0.5 revolutions per minute (r/min) to 60 r/min constant to within ±1 %.

7.1.3.2 *Force Sensor,* to measure the torque developed by the specimen to within ±1 % full scale.

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

7.1.3.4 *Geometry, Spindle, Tool or Rotational Element,* to fix the specimen between the drive shaft and a stationary position.

NOTE 1—Each geometry typically covers a range of 1.5 decades of viscosity. The geometry is selected so that the measured viscosity is between 10 and 90 % of the range of the geometry.

7.1.3.5 *Guard,* to protect the geometry from mechanical damage.

7.1.3.6 *Temperature Sensor,* to provide an indication of the specimen temperature over the temperature range from 15°C to 30°C measurable to within ±0.1°C.

7.1.3.7 *Data Collection Device,* to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscometry are torque, rotational speed, temperature, and time.

7.1.3.8 *Stand,* to support, level and adjust the height of the drive motor, shaft and geometry.

7.1.3.9 *Specimen Container,* with a capacity of 600 mL to contain the test specimen during testing.

NOTE 2—A 600-mL labortory beaker has been found suitable for this service.

7.1.3.10 Auxiliary instrumentation considered necessary or useful in conducting this test method includes:

(1) Data analysis capability to provide viscosity, stress, shear rate or other useful parameters derived from measured signals.

(2) Level to indicate the vertical plumb of the drive motor, shaft and geometry.

7.2 *Preparation of Sample*—Adjust to the desired solids content with distilled water. Bring the pH to the desired point with ammonia. Take a sufficient volume of sample so that at least 500 mL of diluted emulsion polymer will be obtained. Strain the diluted emulsion polymer through the No. 40 (425-µm) sieve. After straining, again gently stir the emulsion

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polymer for approximately 20 s. If the emulsion polymer contains excessive amounts of occluded air, remove the air in the following manner before proceeding with the determination of viscosity: Into a three-necked distillation flask equipped with a stirrer and vacuum connections, pour a sufficient volume of diluted emulsion polymer so that at least 500 mL of emulsion polymer will remain after removal of the air. Start the agitator and evacuate the flask to a vacuum of 26 to 28 mm Hg (3.5 to 3.7 kPa), or until the foam rises to the neck of the flask. Break the vacuum. Evacuate several times in this manner to ensure removal of the occluded air. When required, the same method may be used on emulsion polymer as received, without laboratory removal of occluded air.

7.3 *Procedure:*

7.3.1 Ensure that the strained emulsion polymer is at a temperature at 25 ± 2°C.

7.3.2 Pour the emulsion polymer into the container.

7.3.3 Insert the guarded geometry of the viscometer into the emulsion polymer until the surface of the emulsion polymer is within the measurement indicator on the shaft.

7.3.4 Initiate the rotation of the geometry at the lowest speed available for 1 min.

7.3.5 Increase the geometry speed to that required to produce a reading nearest the midpoint of the scale.

7.3.6 Stop the rotation of the geometry and wait for 1 min.

7.3.7 Restart the rotational of the geometry and allow at least 5 revolutions of the geometry.

NOTE 3—For routine measurement of quality control samples where a specific viscosity range is to be expected and rotational geometry and speed settings are pre-defined through method validation, steps 7.3.4 through 7.3.6 can be skipped.

7.3.8 Measure and report the observed viscosity, the rotational speed and the geometry type used.

7.3.9 Stop the rotation of the geometry. Elevate the geometry out of the test specimen. Clean the geometry with a suitable solvent. Safely dispose of the test specimen.

8. Sediment

8.1 *Scope*—This test method covers the determination of the percent sediment content of emulsion polymers.

8.2 *Apparatus and Material:*

8.2.1 *International Oil Testing Centrifuge,* Model DE.

8.2.2 *Centrifuge Tubes (Graduated)*—Goetz Pear-Shaped, 100-mL capacity, large stem,[3,4] or Goetz Phosphorus Tube with a stopper, 100-mL capacity, small stem.[4,5]

8.2.3 *Bromophenol Blue Indicator Solution (0.1 %).*

8.3 *Procedure:*

8.3.1 Fill a Goetz Phosphorus Tube (small stem) with 100 mL of polymer emulsion inverting the tube to fill the stem completely.

8.3.2 Place the sample tube in the centrifuge and place a tube filled with 100 mL of water on the opposite side for instrument balance.

8.3.3 Spin for 30 min at 1750 r/min.

8.3.4 *Alternative Method*—Dilute the polymer emulsion 50/50 with water; then centrifuge for 30 min.

8.4 *Calculations:*

8.4.1 *Undiluted Samples*—Read the sediment level in the tube stem and express directly as percent sediment content.

8.4.2 *Diluted Samples*—Read the sediment level in the tube stem and multiply by the dilution factor (50/50 = 2, 25/75 = 4, etc.); express the result as percent sediment content.

9. Storage Stability

9.1 *Scope:*

9.1.1 This test method is intended to indicate storage stability of emulsion polymers.

9.1.2 Storage for 30 days at 125°F (52°C), while not a guarantee of one year's stability at room temperature, is believed to be a more reliable indicator of storage stability than similar tests of shorter duration.

9.2 *Apparatus:*

9.2.1 *Glass Bottles,* 120-mL, round, narrow-mouth, approximately 120 mm high and 45 mm in diameter with polyethylene-lined screw caps.

9.2.2 *Oven,* capable of maintaining the temperature of the emulsion samples at 125 ± 2°F (52°C).

9.3 *Samples:*

9.3.1 The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself.

9.3.2 Test samples shall be prepared in duplicate for each material in question.

9.4 *Procedure:*

9.4.1 Transfer 100 mL of polymer emulsion to a clean, dry test bottle and hand tighten the screw cap.

9.4.2 Place the test set or series inverted in a drying oven capable of maintaining the temperature of the emulsions at 125 ± 2°F (52°C).

9.4.3 Observe the samples daily for the first 14 days and twice weekly (that is, Monday and Thursday) for the remainder of the 30-day storage period. Samples should be examined as rapidly as possible with a minimum of agitation and returned to the oven. Do not loosen cap.

9.4.4 At the time of each observation, record the following on a suitable data sheet:

9.4.4.1 Date,

9.4.4.2 Number of days in test, and

9.4.4.3 Condition of the emulsion using the following symbols:

OK	=	no signs of instability
SV	=	slightly viscous
V	=	viscous
Cr	=	creamed
G	=	gelled
Sep	=	other types of liquid phase separation

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[3] The sole source of supply of Fisher catalog no. 5-622 known to the committee at this time is Fisher Scientific Co., Pittsburgh, PA.

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

[5] The sole source of supply of Fisher catalog no. 5-624 known to the committee at this time is Fisher Scientific Co., Pittsburgh, PA.