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

This standard is issued under the fixed designation D 3716; 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 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 1 Specification for ASTM Thermometers[2]

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[3]

E 100 Specification for ASTM Hydrometers[2]

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 rpm. If desired, two propellers may be used on the same shaft, the

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[1] These test methods are under the jurisdiction of ASTM Committee D-21 on Polishes, and are the direct responsibility of Subcommittee D 21.03 on Chemical and Physical Testing.

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[2] *Annual Book of ASTM Standards*, Vol 14.03.

[3] *Annual Book of ASTM Standards*, Vol 15.05.

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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:

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

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 E 70. 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*, Brookfield RV or LV Type.

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 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*—With the strained emulsion polymer at a temperature at 25 ± 2°C, pour the emulsion polymer into a 600-mL beaker. Insert the shielded spindle of the viscometer into the emulsion polymer until the surface of the emulsion polymer is within the notch in the shaft of the spindle. Use a spindle and speed that will give medium scale reading.

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,[4,5] or Goetz Phosphorus Tube with a stopper, 100-mL capacity, small stem.[5,6]

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 rpm.

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.

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

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