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

This standard is issued under the fixed designation D3716; 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:

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]

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

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[1] These test methods are under the jurisdiction of ASTM Committee D21 on Polishes and are the direct responsibility of Subcommittee D21.03 on Chemical and Physical Testing.

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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.

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 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
Sediment = sedimentation of solid matter

10. Freeze-thaw Stability

10.1 *Scope:*

10.1.1 This test method is intended to indicate the freeze-thaw stability of emulsion polymers used for floor polishes.

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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.

10.1.2 Storage under repeated freeze-thaw conditions is not necessarily indicative of the freeze-thaw stability of an emulsion polymer. Materials stable under these conditions may exhibit instability to prolonged freezing, and this test method is intended to cover both eventualities. Thus, the three-cycle test is meant to be equivalent to repeated freeze-thaw cycles and the longer-term single-cycle test is meant to be equivalent to prolonged freezing.

10.2 *Apparatus:*

10.2.1 *Glass Screw Cap Bottle,* 120-mL, approximately 38 mm in outside diameter, 150 mm high with a 24/400 finish as specified by the Glass Container Manufacturers Institute using a polyethylene-lined screw cap (used in ASTM oven stability tests).

10.2.2 *Freeze Chest,* capable of maintaining the temperature of emulsion samples at − 10± 2°C.

10.3 *Procedure:*

10.3.1 Transfer 100 mL of the emulsion polymer to each of four dry clean test bottles and hand-tighten the screw cap.

10.3.2 Place the test set or series in a freeze chest capable of maintaining − 10 ± 2°C.

10.3.3 Allow two of the samples to remain in the chest for 18 h; remove and allow to thaw and remain at room temperature for 6 h before evaluating. Repeat for two additional cycles or a total of three cycles.

10.3.4 Allow two of the samples to remain in the chest for 72 h; remove and allow to thaw and remain at room temperature for 6 h before making observation. Repeat for two additional cycles.

NOTE 4—It should be noted that emulsion polymers, after freezing, may not perform adequately in floor polishes even though above tests are satisfied.

10.3.5 Observations should be made on the condition of the thawed sample, before and after shaking, after each cycle, noting particularly any increase in particle size, coagulation, separation, or change in viscosity.

11. Specific Gravity

11.1 *Scope*—This method describes a procedure for the determination by means of glass hydrometer of the specific gravity of an emulsion polymer.

11.2 *Definition:*

11.2.1 *specific gravity*—the ratio of weight of a given volume of material to the weight of an equal volume of water. In this method the temperature for determination is 60°F (15°C).

11.3 *Apparatus:*

11.3.1 *Hydrometers,* glass, graduated in Specific Gravity 60/60°F, conforming to Specification E100. For liquids lighter than water, use 98H-62. For liquids heavier than water, use 111H to 120H-62.

11.3.2 *Thermometers,* having a range from − 5 to + 215°F (−21 to + 104°C) and conforming to the requirements for Thermometer 12 F as prescribed in Specification E1.

11.3.3 *Hydrometer Cylinders,* glass, or clear plastic. For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 25 mm than the portion of the hydrometer that is immersed beneath the surface of the sample.

11.4 *Temperature of Test*—The gravity determined by the hydrometer method is most accurate at or near the standard temperature of 60°F (15°C). Use this or any other temperature between 0 and 195°F (−18 and 90°C) for the test, so far as it is consistent with the type of sample and necessary limiting conditions.

11.5 *Procedure:*

11.5.1 Adjust the temperature of the sample to 60°F (15°C). The hydrometer cylinder and thermometer shall be at approximately the same temperature as the sample to be tested.

11.5.2 Pour the sample into the clean hydrometer jar without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower boiling constituents of the more volatile samples. For the more volatile samples, transfer to the hydrometer cylinder by siphoning. Remove any air bubbles formed, after they have collected on the surface of the sample, by touching them with a piece of clean filter paper before inserting the hydrometer. Place the cylinder containing the sample in a vertical position in a location free of air currents. Take precautions to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. During this period the temperature of the surrounding medium should not change by more than 5°F (3°C).

11.5.3 Lower the hydrometer gently into the sample, and when it has settled, depress it about two scale divisions into the liquid and then release it. Keep the rest of the stem dry, as unnecessary liquid on the stem changes the effective weight of the instrument, and so affects the reading obtained. With samples of low viscosity, a slight spin imparted to the instrument on releasing, assists in bringing it to rest, floating freely away from the walls of the hydrometer cylinder. Allow sufficient time for all air bubbles to come to the surface. This is particularly necessary in the case of the more viscous samples.

11.5.4 When the hydrometer has come to rest, floating freely, and the temperature of the sample is constant to 0.2°F (0.1°C), read the hydrometer to the nearest scale division. The correct reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

11.5.5 To make a reading with nontransparent liquids, observe the point on the hydrometer scale to which the sample rises above its main surface, placing the eye slightly above the plane surface of the liquid. This reading requires a correction. Determine this correction for the particular hydrometer in use by observing the height above the main surface of the liquid to which the liquid rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of the sample under test.

11.5.6 Observe the temperature of the sample to the nearest 0.25°F (0.13°C) immediately before and after the observation of the gravity, the liquid in the cylinder being thoroughly but cautiously stirred with the thermometer, the whole of the mercury thread being immersed. Should these temperature readings differ by more than 1°F (0.5°C), repeat the temperature and gravity observations when the temperature of the sample has become more stable. Record the mean of the thermometer reading before and after the final hydrometer reading to the nearest 1°F (0.5°C) as the temperature of the test.

11.6 *Calculation*—When gravities have been observed on opaque liquids by the procedure given in 11.5.5, add the correction for specific gravity to the hydrometer reading observed.

11.7 *Precision*—The following criteria should be used for judging the acceptability of results obtained at temperatures differing from 60°F (15°C) by less than 18°F (10°C):

	Specific Gravity
Repeatability (duplicate results by the same operator)	0.0015
Reproducibility (average of two results in each of two laboratories)	0.0040

11.8 *Bias*—No information can be presented on the bias of the procedure in Test Method D3716 for testing emulsion polymers for use in floor polishes because no material having an accepted reference value is available.

12. Keywords

12.1 apparent viscosity; floor polishes; freeze thaw; polish; polymer emulsions; sediment; specific gravity; storage stability; total solids; viscometer; viscosity

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