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Standard Test Methods for Rubber Latices—Synthetic[1]

This standard is issued under the fixed designation D1417; 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 synthetic rubber latices ABR, BR, CR, IIR, IR, NBR, NCR, NIR, PBR, PSBR, SBR, SCR, SIR, synthetic rubber latices having substitute carboxylic acid (COOH) groups on the polymer chain (X), and synthetic rubber latices that are reinforced (Y). Exceptions to the above are noted in the individual test procedures. The test methods include procedures for sampling, and for determining total solids, volatile unsaturates (residual styrene), pH value, surface tension, viscosity, coagulum, bound styrene, Mooney viscosity, mechanical stability, polystyrene reinforcement in contained polymer, and residual acrylonitrile content.

NOTE 1—The nomenclature used in these test methods is in accordance with Practice D1418.

1.2 The values stated in SI units are to be regarded as 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

D1076 Specification for Rubber—Concentrated, Ammonia Stabilized, Creamed, and Centrifuged Natural Latex

D1331 Test Methods for Surface and Interfacial Tension of Solutions of Paints, Solvents, Solutions of Surface-Active Agents, and Related Materials

D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)[3]

D1418 Practice for Rubber and Rubber Latices—Nomenclature

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3314 Test Method for Rubber—Chemical Analysis for Polystyrene Blocks In SBR (Styrene-Butadiene Rubber) and Styrene-Reinforced Latices

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E1970 Practice for Statistical Treatment of Thermoanalytical Data

E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

3. Sampling

3.1 Rubber latex tends to cream on standing. Once stratification has occurred, the latex must be thoroughly agitated to obtain a homogeneous blend as a representative sub-sample. The procedure required differs with the type of container and facilities available.

3.2 *Sub-Sampling from Tank Cars:*

3.2.1 If stratification has occurred, take separate samples about 75 mm (3 in.) from the top surface and about 75 mm from the bottom of the tank. If results from the top and bottom

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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] The last approved version of this historical standard is referenced on www.astm.org.

TABLE 1 Drying Time for Determination of Total Solids in Latex
(Drying Aid, 1 cm^3 of distilled water.)

Type of Latex	Drying Time, min
SBR 2000	45
SBR 2001	45
SBR 2002	45
SBR 2003	45
BR 2004	45
SBR 2005	45
SBR 2006	45
SBR 2076	45
SBR 2100	60
SBR 2101 and X765	45
SBR 2102	60
SBR 2103	60
BR 2104	45
SBR 2105	45
SBR 2106	45
SBR 2107	45
SBR 2108	45
SBR 2109	45
SBR 2110	45
SBR 2111	45
SBR 2112	45
SBR 2113	45
SBR 2114	45

samples do not agree within 1 % total solids, the contents of the car shall be thoroughly agitated until samples obtained do agree within this tolerance.

3.3 *Sub-Sampling from Drums:*

3.3.1 *Blending*—The latex shall be blended by one of the following test methods:

3.3.1.1 *Test Method A*—If the drum is fitted with a bung and contains at least 2 % air space, lay it on its side and roll back and forth 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. If the drum is of the open-head type, remove the end head and thoroughly stir the contents, preferably by means of a perforated steel disk plunger. Stirring for about 10 min will normally suffice.

3.3.1.2 *Test Method B*—Agitate the contents of the drum by means of a suitable motor-driven stirrer for as long as is necessary to disperse the cream. Excessive stirring and unnecessary exposure of the latex to air must be avoided. A suitable type of stirrer consists of a collapsible two-bladed stainless steel propeller of 11 cm 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 latex from the bottom of the drum. The stirrer shall be operated 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 latex must contain no copper or brass.

3.3.2 *Removal of Sub-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 latex has been obtained.

NOTE 2—Alternatively, a specially constructed metal sampling tube may be used, the bottom of which can be closed by remote control. Copper or brass must not be used in any part of its construction.

3.3.3 *Sample:*

3.3.3.1 Where sub-samples are drawn from several containers, for example, 10 % sampling of latex in drums, or where taken at different depths, for example from tanks, the sub-samples shall be combined and thoroughly blended by stirring or shaking immediately before the final sample is taken.

4. Total Solids

4.1 *Apparatus:*

4.1.1 Tared, covered, all-metal ointment boxes, having a capacity of approximately 50 to 60 cm^3, a minimum diameter of 38 mm (1.5 in.), and a maximum height of 23 mm (0.9 in.). The disposable aluminum liner for the metal ointment box may be used.

4.2 *Procedure:*

4.2.1 *Method A*—Accurately weigh a clean, dry aluminum foil dish. Record the weight. Measure 2 mL of sample and put in the aluminum foil dish. Reweigh the dish and record the weight. Put the aluminum foil dish in a 170°C oven; heat for 15 min. Remove the aluminum foil dish from the oven and let cool in a desiccator. Reweigh the aluminum foil dish. Record the weight. Calculations: A = weight of dish empty; B = weight of dish + sample; C = weight of dish + residue;

$$\frac{(C - A)(100)}{(B - A)} = \%\ \text{Total Solids} \quad (1)$$

Report the total solids to the nearest 0.1 %.

4.2.2 *Method B*—The percent solids is determined by measuring gravimetrically the solids remaining after volatilizing off the liquid portion.

4.2.2.1 *Instrument Conditions*—The following instrument conditions are based on work done with a Denver IR-100:

Oven Temperature—145°C
Slope—0.05 %/min
Program—No. 1
A1 pans—Fisher #01-913-356
Fiber paper—Fisher #01-913-318

Place a glass fiber filter circle on an aluminum pan. Open the lid of the IR-100 and center the pan (with filter) between the four prongs of the“ X”-shaped holder. Close the lid and press the START button. When display reads “tare pan,” press the TARE button. Raise the lid and place about 3.0 g of latex in a circle on the filter paper. Again close the lid. Wait 10 s, then press the START button. The instrument will automatically run for about 4 min then shut off. Read the percent (%) solids directly from the printout paper or from the display screen. Report the value to the nearest 0.1 %.

4.2.3 *Method C*—Total solids is determined using a CEM microwave oven. This unit can yield accurate results in a few minutes. For detailed operating instructions, please refer to the operator’s manual.

4.2.3.1 *Equipment:*

(1) Moisture/Solid Analyzer (microwave oven).

(2) Glass fiber sample pads.
(3) 5 mL Disposable Pipet.

4.2.3.2 *Procedure*—Put the pads on the balance. Tare the weight of the pads. Apply the latex sample to the pads by using the pipet. Spread the latex in a zig-zag pattern. Use between 1.0 to 5.0 g of latex. Keep sample application as uniform as possible to maximize reproducibility. Press "Start" button and read the percent solids from the display. Record the result to one decimal place.

NOTE 3—An alternative method for the determination of total solids in synthetic rubber latex is described in Specification D1076.

5. Volatile Unsaturates (Residual Styrene)

5.1 *Scope*—This test method measures the residual styrene of SBR synthetic rubber latices. It is not applicable to other synthetic rubber latices.

5.2 *Apparatus:*

5.2.1 Iodine flasks having capacities of 250 cm^3 each, and distillation apparatus with ground-glass joints.

5.2.2 25-cm^3 pipet.

5.2.3 50-cm^3 buret.

5.3 *Reagents:*

5.3.1 *Synthetic Methanol,* containing 100 ppm of *p*-tertiary butyl catechol.

5.3.2 *Standard Potassium Bromide-Potassium Bromate Solution* (0.1 *N*)—Dissolve 2.784 g of potassium bromate ($KBrO_3$) and 10.0 g of potassium bromide (KBr) in water and dilute to 1000 cm^3. Standardize with 0.1 *N* sodium thiosulfate ($Na_2S_2O_3$) solution in the presence of an excess (about 3 g) of potassium iodide (KI) and sulfuric acid (H_2SO_4), (18 %).

5.3.3 *Sulfuric Acid Solution* (18 %).

5.3.4 *Potassium Iodide Solution* (10 %).

5.3.5 *Standard Sodium Thiosulfate Solution* (0.1 *N*).

5.3.6 *Starch Indicator Solution.*

5.4 *Procedure:*

5.4.1 Weigh approximately 25 g of the latex to the nearest 0.1 g in a tared, covered, 250-cm^3 iodine flask. Remove the cover and add 25 cm^3 of distilled water to the iodine flask. Add 25 cm^3 of synthetic methanol containing 100 ppm of *p*-tertiary butyl catechol. Be sure to add the materials in the following order:

1. Latex.
2. Distilled water.
3. Methanol containing *p*-tertiary butyl catechol.

5.4.2 Connect the iodine flask to the distillation apparatus with ground-glass joints and distill the mixture. Collect the first 25 cm^3 of distillate in a 250-cm^3 iodine flask, rinse the condenser with 20 cm^3 of methanol containing 100 ppm of *p*-tertiary butyl catechol, and add the rinsings to the recovery flask.

5.4.3 From a buret add 20 cm^3 of 0.1 *N* standard KBr-$KBrO_3$ solution. Cool the solution to 30 C. Rapidly add 15 cm^3 of 18 % H_2SO_4 solution, stopper the flask, shake it, and add distilled water to the funnel lips as a vapor seal. Allow the bottle to stand for 60 s. If no yellow color remains, add successive 10-cm^3 portions of the bromide-bromate solution until a slight yellow color persists for 60 s after the addition. Make the additions by drawing the standard solution from the buret into the funnel lip and lifting the stopper so that the solution enters the flask around the stopper. Wash the funnel lip with distilled water in the same manner and seal with water. After 60 s has elapsed since the final bromide-bromate addition, add 10 cm^3 of 10 % KI solution to the funnel lip, and lift the stopper to allow the solution to enter the flask around the stopper. Shake the bottle and contents and titrate the liberated iodine with 0.1 *N* standard sodium thiosulfate solution to a faint yellow color. Add 1 cm^3 of starch indicator solution and continue the titration with sodium thiosulfate solution until the solution is clear.

5.4.3.1 For a blank determination, repeat the procedure using distilled water instead of latex.

5.5 *Calculation*—Calculate the percentage of volatile unsaturates (residual styrene) as follows:

$$\text{Volatile unsaturates (residual styrene), \%} = \{[(D \times E) - (F \times G)] \times 0.0521 \times 100/M\} - H \quad (2)$$

where:

D = cubic centimetres of standard bromide-bromate solution used,
E = normality of the bromide-bromate solution,
F = cubic centimetres of standard thiosulfate solution used for the titration,
G = normality of the thiosulfate solution,
H = blank determination, %, and
M = mass of latex used, g.

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 this use. The glass electrode shall be of the type applicable for a pH range of from 2 to 14.

6.2 *Standard Solution*—Use a standard solution having a pH of 10 or a standard solution having a pH approximately the same as that of the latex 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 latex to come to equilibrium with the glass electrode before taking the final reading. After the pH determination has been made, clean the electrodes thoroughly and immerse them in distilled water. Report the pH value for the latex at a temperature of 25 ± 2°C.

7. Surface Tension

7.1 The surface tension of styrene-butadiene rubber latex shall be determined on the total solids of 40 ± 1 %. If the viscosity is below 200 mPa·s (200 cP) on No. 1 spindle at 20 r/min, the latex can be tested with solids as received with little loss in accuracy.

7.2 *Apparatus*—Use a du Nouy tensiometer, carefully calibrated as described in Test Methods D1331.

7.3 *Procedure*—Strain approximately 25 cm^3 of latex, adjusted to a temperature of 25 ± 2°C, into a pan 60 to 65 mm