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Standard Test Methods for Rubber Latices—Synthetic¹

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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 D 1418.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Table 1

2. Referenced Documents

2.1 ASTM Standards:

- D 1076 Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex²
- D 1331 Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents³
- D 1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis²
- D 1418 Practice for Rubber and Rubber Latices—Nomenclature²
- D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)²
- D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets²

¹ These test methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.23 on Synthetic Rubbers.

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² Annual Book of ASTM Standards, Vol 09.01.

³ Annual Book of ASTM Standards, Vol 15.04.

TABLE 1 Drying Time for Determination of Total Solids in Latex
(Drying Aid, 1 cm³ 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

D 3314 Test Method for Rubber—Chemical Analysis for Polystyrene Blocks in SBR (Styrene-Butadiene Rubber) and Styrene-Reinforced Latices²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

E 200 Practice for Preparation, Standardization, and Storage of Reagent Solutions for Chemical Analysis⁴

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

⁴ Annual Book of ASTM Standards, Vol 15.05.

D 1417

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 cm³, 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 seconds, 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 %.

NOTE 3—An alternative method for the determination of total solids in synthetic rubber latex is described in Specification D 1076.

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 cm³ each, and distillation apparatus with ground-glass joints.

5.2.2 25-cm³ pipet.

5.2.3 50-cm³ 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 (KBrO₃) and 10.0 g of potassium bromide (KBr) in water and dilute to 1000 cm³. Standardize with 0.1 N sodium thiosulfate (Na₂S₂O₃) solution in the presence of an excess (about 3 g) of potassium iodide (KI) and sulfuric acid (H₂SO₄), (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-cm³ iodine flask. Remove the cover and add 25 cm³ of distilled water to the iodine flask. Add 25 cm³ of synthetic methanol containing 100 ppm of tertiary

D 1417

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 cm³ of distillate in a 250-cm³ iodine flask, rinse the condenser with 20 cm³ 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 cm³ of 0.1 *N* standard KBr-KBrO₃ solution. Cool the solution to 30 °C. Rapidly add 15 cm³ of 18 % H₂SO₄ 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-cm³ 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 cm³ 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 cm³ 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:

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 E 70. 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 ± 1°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 D 1331.

7.3 *Procedure*—Strain approximately 25 cm³ of latex, adjusted to a temperature of 25 ± 0.5°C, into a pan 60 to 65 mm (2.4 to 2.6 in.) in diameter and 18 to 20 mm (0.7 to 0.8 in.) high (Note 4). The surface of the latex must be free of air bubbles, and the surface tension test shall be within 3 to 4 min to avoid “skinning over” of the latex. Clean the tensiometer ring by heating in a bunsen flame. Extreme care must be taken to avoid distortion as the tensiometer ring is handled. Place the pan, containing the latex being tested, beneath the ring on the adjustable platform of the instrument. With the instrument adjusted so that the ring system is in its zero position when the ring is dry and the scale reading is zero, raise the platform until the latex makes contact with the ring. Submerge the ring beneath the surface of the latex. Now slowly lower the platform by means of the platform-adjusting screw and increase the torsion of the wire simultaneously, proportioning these two adjustments so that the torsion arm remains exactly in its zero position. As the film adhering to the ring approaches the breaking point, proceed more slowly with the adjustments to make certain that the moving system is in its zero position when the rupture occurs. The scale reading at which the ring detaches from the latex represents, after proper correction, the surface tension of the latex at the given temperature. Record the average of three consecutive readings, discounting the first reading. The ring is cleaned by flaming between readings and all readings shall agree within 0.5 mN/m (0.5 dynes/cm).

NOTE 4—The pan must be free of an oil film since traces of oil introduce variable results in the surface tension measurement.

7.4 *Calculations*—Calculate the surface tension, in millinewtons/meter (dynes, per centimeter), as follows:

$$\text{Surface tension, mN/m} = M \times N \quad (3)$$

where:

M = reading of the tensiometer, and

N = a correction factor calculated as follows:

$$N = 0.7250 + \sqrt{[(0.0003678 \times M)/R^2] + P} \quad (4)$$

where:

M = reading of the tensiometer,

R = radius of the ring, cm, and

P = a constant calculated as follows:

$$P = 0.04534 - (1.679S/R)$$



where:

S = radius of the wire of the ring, cm, and

R = radius of the ring, cm.

8. Viscosity

8.1 *Scope*—This test method covers the determination of the viscosity of synthetic rubber latices using the Brookfield LV instrument for viscosities up to 2000 mPa·s (2000 cP) and the Brookfield RV instrument for viscosities of above 200 mPa·s (200 cP).

8.2 *Summary of Test Method*—The viscosity is determined by means of a viscometer that measures the torque produced on a specified spindle rotating at constant speed while immersed to a given depth in the latex.

8.3 Apparatus:

8.3.1 Glass Stirring Rod.

8.3.2 Screen—Standard sieve 355 μm .

8.3.3 *Distillation Flask*, three-necked, equipped with a stirrer and vacuum connections.

8.3.4 Beaker, having a capacity of 600 cm^3 .

8.3.5 *Viscometer*⁵—The viscometer shall contain an electric synchronous motor capable of driving at a constant rotational speed a shaft to which spindles of different shapes and dimensions may be attached. It shall contain a scale and a pointer, which will indicate the equilibrium torque developed by the resistance to rotation of a spindle properly immersed in latex. A bubble level shall be incorporated in the motor housing to indicate, with the spindle attached to the motor shaft, when the spindle is vertical. The viscometer must not contain a foot bearing, because of possible interference due to coagulum formation on the bearing.

8.4 Preparation of Sample:

8.4.1 If the latex contains occluded air, remove the air in the following manner before proceeding with the determination of viscosity. After diluting the latex (if viscosity is to be determined at lower designated solids) with distilled water to the approximate desired total solids, pour a sufficient volume of the latex into a three-necked distillation flask equipped with a stirrer and vacuum connections so that at least 500 cm^3 of latex will remain after the removal of the air.

8.4.2 Start the agitator and evacuate the flask to 3.45 to 3.75 kPa (approximately 1 in. Hg) or until the foam rises into the neck of the flask. Break the vacuum. Evacuate several times in this manner to ensure removal of the occluded air.

8.4.3 Strain the deaerated latex through a 355- μm standard screen. Determine the total solids content of the latex and then accurately adjust the solids content of the latex to the designated total solids content by the addition of distilled water. Add the water slowly to the latex, and gently stir the mixture with a glass rod during the dilution.

8.5 Procedure:

8.5.1 With the strained latex at a temperature of $25 \pm 2^\circ\text{C}$, pour the latex into the 600- cm^3 beaker, taking care to avoid air entrapment. Remove guard. This is done to reduce later

cleaning problems. Attach the spindle to the viscometer. Do not hit the spindle against the side of the beaker while it is attached to the viscometer as this can damage the shaft alignment. Lift the shaft slightly and hold firmly with one hand while screwing the spindle on with the other hand. In most cases, spindle No. 1 will give results in the required range. If not, change the spindle. Tilt the instrument slightly while immersing the spindle to avoid trapping air bubbles on the disk surface. Insert the spindle of the viscometer into the latex until the surface of the latex is within the notch in the shaft of the spindle. Alternatively, the spindle may be immersed in the latex in the above manner before attaching it to the viscometer. Level the viscometer. Depress the clutch and turn on the viscometer motor.

8.5.2 Select the speed of rotation of the instrument as follows:

LV instrument: 6.3 rad/s (60 r/min)

RV instrument: 2.1 rad/s (20 r/min)

8.5.3 Measure 200 mL of latex in an 8-oz jar. Connect the guard and spindle #1 to the viscometer. Remember that the spindle has a left-hand thread and must be screwed firmly into place.

NOTE 5—Always lift up on the spindle when attaching it to the viscometer. This is done to avoid damage to the instrument's pivot point and jewel bearing.

Place the 8-oz jar on the platform jack under the viscometer. Raise the jar to the guard and spindle. Hold the jar at an angle to prevent air bubbles from being trapped under the spindle. Adjust the height of the latex to the groove on the spindle. Set the spindle speed to 60 rpm on the left side of the viscometer. When the sample is ready to be analyzed, hold down the toggle switch on the back of the viscometer and turn on the power switch on the right. After the power is on, the toggle switch may be released. Let the viscometer stabilize for approximately 60 s. To obtain the reading, hold down the toggle switch and then turn the power switch off when the red needle stops in the window. The red needle points to the viscosity in units of centipoise (cps). Repeat Steps 7 and 8 at least three times to find an average reading. Report the viscosity to the nearest 1 cps.

8.6 *Calculation*—For spindle No. 1 and 60 r/min:

$$\text{RDG} \times 1 = \text{cps} \quad (6)$$

NOTE 6—If it is desired to better characterize the latex, additional rotational speeds may be used.

9. Coagulum Content

9.1 Apparatus:

9.1.1 Nylon filter cloth, 25 micron.

9.1.2 Beaker, tared.

9.1.3 Demineralized Water (DMW).

9.1.4 Air Oven.

9.1.5 Desiccator.

9.2 *Procedure*—Store clean nylon (25 micron mesh) filter circles (9.0 ± 0.2 cm in diameter) in a desiccator. Weigh the clean filter and record this weight to four decimal places. Place the filter on the porcelain Buchner funnel; set the funnel on a 2000-mL flask. Weigh 500 ± 0.5 g of latex sample into a 1200-mL beaker. Add 500 ± 1 mL of DMW and stir gently.

⁵ Brookfield Viscometers, Models LVE, LVT, RVE, RVE-100, and RVT have been found satisfactory for this purpose and can be obtained from Brookfield Engineering, Inc., Stoughton, MA 02072.