Designation: D 1417 – 03a

Standard Test Methods for Rubber Latices—Synthetic[1]

This standard is issued under the fixed designation D 1417; 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 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*~~

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

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2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1076 Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex
D 1331 Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents
D 1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis
D 1418 Practice for Rubber and Rubber Latices—Nomenclature
D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)
D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
D 3314 Test Method for Rubber—Chemical Analysis for Polystyrene Blocks in SBR (Styrene-Butadiene Rubber) and Styrene-Reinforced Latices
D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries
D 6204 Test Methods for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode
E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

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 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.

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

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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*~~;~~ ~~Vol 09.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.

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;

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

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 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:

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

(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 ± 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 D 1331.

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 (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:

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

where:
M = reading of the tensiometer, and
N = a correction factor calculated as follows:

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

where:
M = reading of the tensiometer,
R = radius of the ring, cm, and
P = a constant calculated as follows:

$$P = 0.04534 - (1.679S/R) \tag{5}$$

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*[3]—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± 2°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

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~~*Annual Book*~~

[3] The sole source of supply of Brookfield Viscometers, Models LVF, LVT, RVF, RVF-100, and RVT known to the committee at this time is Brookfield Engineering, Inc., Stoughton, MA 02072. If you are aware of alternative suppliers, please provide this information to ASTM ~~Standards, Vol 15.04.~~ International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

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:

$$\text{RDG.X } 1 = \text{cps} \tag{6}$$

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. Filter the sample through the nylon filter with the aspirator slowly. If drainage stops, use a plastic pipet and gently swirl the liquid in a whirlpool fashion slowly, taking care not to touch the nylon filter on the bottom of the liquid. This usually allows drainage to resume. Rinse the filter gently with squirts of DMW (50 mL). All latex must be rinsed through the filter. Dry the filters in the 70°C blue M oven for 15 min.

NOTE 7—Take care not to lose any floc in transferring from oven to counter to balances. Cool the filter for 15 min in a desiccator. Weigh the filter plus residue to four (4) decimal places. Calculate and report the residue to the nearest third decimal place [Spec:0.010 % maximum].

9.3 *Calculation*:

$$\frac{(\text{Weight of filter \& residue}) - (\text{Weight of filter})}{5} = \%\ \text{Residue} \tag{7}$$

For a 500 gram sample:

$$\frac{(\text{Weight of filter \& residue}) - (\text{Weight of filter})}{5} = \%\ \text{Residue} \tag{8}$$

9.3.1 *Alternate Calculation*:

The residue may also be expressed in terms of parts per million (ppm), as follows:

$$(\textit{Weight of filter \& residue}) - (\textit{Weight of filter})\ x\ 1\,000 = \textit{Residue, in ppm} \tag{9}$$

NOTE 8—When mechanically concentrated latices are tested, use distilled water as the diluent.

10. Determination of Bound Styrene in Contained Polymer

10.1 *Scope*—This test method is designed to measure the bound styrene content of butadiene-styrene copolymers. The test method is not applicable when a high styrene latex (above 50 % bound styrene) is a component of the latex system.

10.2 *Coagulation Procedure*—Use the test method outlined in 11.5.

10.3 *Bound Styrene Determination*—Use the test method described in Test Methods D 1416.

11. Determination of Mooney Viscosity or Rotorless Shear Rheometer Rheological Properteis of Contained Polymer

11.1 *Scope*—This test method is designed to measure the viscosity and rheological properties of the contained polymer of anionic stabilized synthetic rubber latices having a residual monomer content of less than 0.50 % by weight. It is not applicable to latices of polymers containing substitute carboxylic acid groups on the polymer chain, hot polymerized BR (2004), CR, NCR, SCR, or ABR.

11.2 *Summary of Test Method*—The latex is coagulated by the consecutive addition of salt, methanol, and acid solutions while under fast agitation. The resulting crumb is filtered, dried, and massed on a rubber mill. The viscosity of the contained polymer is measured in a shearing disk viscometer.

11.3 *Apparatus*:

11.3.1 *Blendor*[4]—Explosion-proof type.

11.3.2 *Cheesecloth*.

11.3.3 *Drying Tray*, approximately 30 by 20 by 2.5 cm (12 by 8 by 1 in.) with a 1.00 to 1.41 mm mesh plastic screen supported by a coarse screen bottom.

11.3.4 *Draft Oven*, maintained at 125 ± 2°C.

11.3.5 *Laboratory Mill*, as described in Practice D 3182.

11.3.6 *Shearing Disk Viscometer*, as described in Test Methods D 1646, or *Rotorless Shear Rheometer*, as described in Test Method D 6204.

11.4 *Reagents and Materials*:

11.4.1 *Sodium Chloride Solution* (250 g/dm^3)—Dissolve 250 g of ACS Grade NaCl in 1 dm^3 of demineralized water.

11.4.2 *Methanol Solution* (7.5 g/dm ^{3})—Dissolve 7.5 g of a suitable antioxidant[5] in 1 dm^3 of USP methanol.

11.4.3 *Sulfuric Acid* (1 + 9) —Add 100 cm^3 of concentrated ACS Grade H_2SO_4(sp gr 1.84) to 900 cm^3 of demineralized water.

11.4.4 *Congo Red Indicator Paper* .

11.4.5 Agerite White (KD-64 Harwick) stock dispersion

11.4.6 2.2 % Aluminum Sulfate, dissolve 378 grams in 5 gallons of tap water and agitate vigorously. Some precipitate will remain in the reagent.

11.5 *Procedure*:

11.5.1 *Coagulation Procedure*—If the total solids content of the latex is greater than 30 %, dilute the latex with distilled water to a total solids contents of approximately 30 %. Add to 250 cm^3 of the diluted latex, 50 cm^3 of the NaCl solution and mix thoroughly.

11.5.1.1 Add 250 cm^3 of methanol-anti-oxidant solution while continuing to agitate. Add 10 cm^3 of H_2SO_4(1 + 9) slowly during a period of 2 to 3 min. If the coagulating solution does not turn Congo Red paper from red to blue, add additional H_2SO_4 while stirring until the color does change.

11.5.1.2 Pour the contents of the coagulating cup onto cheesecloth and press as much serum as possible from the mass. Hand separate the mass, wash it thoroughly with demineralized water, and transfer the pieces to a drying tray.

11.5.2 *Drying Procedure*—Dry the crumb in a draft oven at 125 ± 2°C. Drying time will vary with the crumb consistency, polymer ~~Mooney~~ viscosity, and oven conditions and should be determined by experimentation. Drying time shall be determined by reference to a volatile matter versus time plot. The desired drying time shall be taken at a time less than 5 min after the minimum level has been reached. Over drying and under drying will both give incorrect Mooney viscosity values and are to be avoided.

11.5.3 *Determination of Mooney Viscosity or Processability Characteristics*—Mill mass the rubber and determine the Mooney viscosity in accordance with Test Methods D 1646, or processability characteristics in accordance with Test Methods D 6204.

11.5.4 *Special Procedure for Vinylpyridine latex (PSBR)*:

11.5.4.1 *Coagulation of Latex*—Add 2400 ml of warm water (130°F) to the container and start agitation. Add 25 ml of Agerite White Dispersion. Add 200 ml of latex to be tested (or sufficient to provide 75–80 grams of polymer). Increase agitation and slowly add 2 % aluminum sulfate (increase agitation if coagulated polymer starts to form a ball) until coagulation is complete. This can be determined by observing the serum. The serum solution will be relatively clear and contain a few very fine particles when coagulation is complete. Continue agitation for one (1) minute after complete coagulation. Allow to stand for 10 minutes. Seperate the crumb from the serum by filtering through a fine, lint-free cloth.

11.5.4.2 *Drying*—The coagulum during filtration may tend to agglomerate into a mass. Break this mass into very small pieces. Dry the crumb in an air oven at 90°C. When the crumb is dry, it will completely lose its white color and turn brown. This should

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~~*Annual Book of ASTM Standards*, Vol 15.05.~~

[4] A Model FCI-15 Waring Blendor, or equivalent, has been found satisfactory for this purpose.

~~[5] The sole source~~

[5] Antioxidants of ~~supply of Brookfield Viscometers, Models LVF, LVT, RVF, RVF-100, and RVT known to~~ the ~~committee at this time is Brookfield Engineering, Inc., Stoughton, MA 02072. If you are aware~~ bis or polyphenol types such as Ethyl 702, Santowhite Powder, and Wingstay L, and of ~~alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of~~ the ~~responsible technical committee [1], which you may attend.~~ diaryl-*p*-phenylenediamine type such as Wingstay 200, have been found to be equivalent to PBNA.

require 1½ to 2½ hours. Check for dryness after one (1) hour drying time and every 15 minutes thereafter til dry. Remove when dry. To determine if the crumb is dry, pull several pieces apart and, if any white shows, dry for an additional ½ hour. Do this until the crumb is completely dry.

11.5.4.3 *Milling of Crumb*— Using the sample prepared in 10.4.2 above, mass the crumb together into a ball in your hands. Ensure that the dry crumb is at least 60 grams. Set mill temperature at 85±5°F and the distance between rolls at 0.065±0.005 inches. With the speed set at 40 rpm, pass the sample through the mill ten times, doubling it during passes 2 through 9. Do not band the material, or let it stick to the rolls so that it is carried around. It may be necessary to actually stop the mill after each pass in order to make seperate, independent passes. Allow the milled samples to sit for thirty minutes at room temperature before testing.

11.5.4.4 *Determination of Mooney Viscosity* —After the sample has been prepared as detailed above, run the Mooney test as listed in ASTM D 1646, Paragraphs 9–12.

12. Determination of Mechanical Stability

12.1 *Scope*—This test method covers the determination of the stability of synthetic latices when subjected to highspeed mechanical stirring.

12.2 *Summary of Test Method*—A sample of latex is subjected to mechanical shear by the use of a high-speed stirrer and the extent of instability noted. The amount of coagulum formed after a given time of agitation is considered a measure of latex instability.

12.3 *Apparatus*:

12.3.1 *Tester*[3 ,6]—The test apparatus shall consist of a vertical shaft high-speed stirrer capable of maintaining a speed of 14 000 ± 200 r/min for the duration of the test. The stirrer shaft shall be approximately 6.3 mm (0.25 in.) in diameter at its lower end at the point of attachment of the agitator disk and may taper upward for greater strength. It shall be of sufficient length to reach conveniently to the bottom of the test bottle. The shaft shall run with not more than 0.25 mm (0.010 in.) out of true at the speed specified.

12.3.2 *Agitator Disk*—The agitator itself shall consist of a polished stainless steel disk 36.12 ± 0.03 mm (1.422± 0.001 in.) in diameter and 1.58 ± 0.05 mm (0.062 ± 0.002 in.) in thickness having a threaded stud at its exact center for attachment to the center of the lower end of the stirrer shaft.

12.3.3 *Test Bottle*—The test bottle shall be a flat-bottom cylindrical glass container 57.8 ± 1 mm (2.28 ± 0.04 in.) in inside diameter by approximately 12.7 cm (5 in.) in height, and having a wall thickness of approximately 2.3 mm (0.09 in.).

12.3.4 *Bottle Holder*—The bottle holder shall be so constructed that the bottle may be conveniently lowered and raised to the exact specified position with relation to the shaft and agitator. The position of the test bottle shall be such that the axis of the stirrer shaft is concentric with the axis of the bottle and that the bottom of the agitator disk is 12.7 ± 2 mm (0.5 ± 0.1 in.) from the bottom inside of the bottle.

12.3.5 *Stainless Steel Screen*—No. 180 µm sieve with 0.180 ± 0.009-mm (0.0070 ± 0.0004-in.) opening and 0.131 ± 0.01-mm (0.0052 ± 0.0005-in.) wire diameter. The screen shall be cut to fit into the seat of a pipe union having inside diameter of about 5 cm (nominal 1½-in. pipe size).

12.4 *Procedure*:

12.4.1 If the viscosity of the latex as determined by Section 8 of this test method is greater than 350 mPa·s (cP), dilute the latex with sufficient water so that the viscosity is below 350 mPa·s (cP), except that in no case should the latex solids be reduced by more than 10 % relative.

12.4.2 Strain the latex through a 180-µm sieve and weigh 50 ± 0.5 g of the strained latex into the test bottle. Place the bottle containing the latex in the holder of the stability tester.

12.4.3 Start the tester and agitate for exactly 30 min (or other agreed-upon time) at 1470 ± 21 rad/s (14 000 ± 200 r/min). The time of agitation should be adjusted so that the temperature of the latex does not increase to more than 60°C. Remove the sample immediately to avoid any filming and rinse the agitator shaft and disk with distilled water. Strain the latex through a tared 180-µm screen that has previously been wetted with a 2 % aqueous solution of a surfactant such as potassium oleate. Finally, flush the screen with the same solution and then follow with a rinse of distilled water. Dry the screen and coagulum in a force draft oven at 100 ± 2°C to constant mass (approximately 15 min). Cool and weigh again to ±0.001 g.

12.5 *Calculation*—Calculate the percent coagulum as follows:

$$\text{Coagulum, \%} = [(A - B)/50] \times 100 \tag{10}$$

where:

A = mass of tared screen plus coagulum, and

B = mass of tared screen.

12.5.1 *Alternate Calculation*—The coagulum may also be expressed in terms of parts per million (ppm), as follows:

$$\text{Coagulum, ppm} = [(A - B)/50] \text{ x } 1\,000\,000 \tag{11}$$

~~[6] A Model FCI-15 Waring Blendor, or equivalent, has been found satisfactory for~~

[6] The sole source of supply of the apparatus known to the committee at this ~~purpose.~~ time is Custom Scientific Instruments, Inc., 13 Wing Dr., Whippany, NJ 07981.

12.6 *Report*—Report the percent coagulum, the total solids content at which the latex was tested, and the duration of stirring, in minutes.

12.7 *Mechanical Stability of Latex (Alternate method)*

12.7.1 *Scope*—This method determines the amount of latex which is coagulated under shearing conditions of the test. A sample of latex is subjected to mechanical shear by the use of a high speed stirrer and the extent of instability is noted. The amount of coagulum formed after a given time of agitation is considered a measure of latex stability.

12.7.2 *Equipment*:

1. 200 mesh screen
2. 80 mesh polyester screen
3. 100 ml graduated cylinder
4. Hamilton Beach Drinkmaster No. 30
5. Plastic seal for the mixer cup (1 qt)
6. Clamp, screw chain
7. Microwave oven

12.7.3 *Procedure*—Cool the latex to 100°F if it is found to be above this temperature. Filter approximately 170 ml of latex through a 200 mesh screen. Pour 150 ml of this latex into the mixer cup. To eliminate air drying, place the plastic seal over the mixer cup and secure with the chain clamp while the test is running. Agitate on the Hamilton Beach mixer for 30 minutes at the "low" setting. Preweigh an 80 mesh polyester screen. At the conclusion of the test, inspect the sample for air dried latex. If any is found, dispose of the sample and rerun, making sure that the seal is tight. Immediately after removing the mixer cup, pour the latex through the preweighed 80 mesh polyester screen. Wash the residue from the cup through the screen using additional wash water. Dry to constant weight using microwave oven. Reweigh the screen containing the dried coagulum and find by difference the amount of coagulum collected. Express the final results in ppm as shown below:

$$\frac{\text{Grams Coagulum}}{150} \times 1{,}000{,}000 = \text{ppm Residue} \tag{12}$$

12.8 *Centrifuge Stability*

12.8.1 *Scope*—This method was designed to readily predict creaming stability of vinylpyridine containing latices. The amount of agglomerated cream is determined after diliting, centrifuging and filtering.

12.8.2 *Equipment*:

1. Centrifuge, Sorvall GLC1 or equivalent, at least 2500 rpm
2. Centrifuge jars, 100 ml with caps
3. Jars, 1 quart, wide mouth, with lids
4. Balance, 3000–gram capacity
5. Balance, Analytical ± 0.1 mg
6. Screen, 325 mesh, to fit residue holders

12.8.3 *Procedure*:

12.8.3.1 Mix the latex well and filter through a paint strainer. Dilute the amount of latex obtained from from the formula listed in 13.4.1 below to 900 grams with deionized water and mix well. Immediately transfer 100 ml of the diluted latex to a 100 ml centrifuge jar. Make certain that the jar is clean and dry. Centrifuge the 100 ml sample at 2200 rpm for 1 hour. After centrifuging, pour the liquid through a tared 325 mesh screen. Rinse the jar several times, pouring each rinse through the filter. Dry the screen in a microwave over for 3 minutes on high and weigh. Record the weight of residue as specified by the calculation listed in 3.4.2 below.

12.8.4 *Calculations*:

$$\text{Grams of latex to dilute} = \frac{900 \times 30}{\text{TSC}} \tag{13}$$

$$\text{Grams of residue} \times 10{,}000 \text{ ppm} = \text{residue in ppm} \tag{14}$$

13. Determination of Polystyrene Reinforcement in Contained Polymer

13.1 *Scope*—This test method is designed to measure the polystyrene content of polystyrene-reinforced SBR latices in the range from 1 to 100 % polystyrene. The test method is not applicable on polymers containing gel, unless it has been proven that gel does not interfere.

13.2 *Procedure*—Determine the polystyrene content in accordance with Test Method D 3314.

14. Determination of Residual Acrylonitrile Content

14.1 *Scope*—This test method measures the residual acrylonitrile content of nitrile rubber latices that have a residual acrylonitrile content of less than 0.2 %.

14.2 *Summary of Test Method*—The latex is distilled and the distillate is collected in methanol. *n*-Dodecyl mercaptan is added to the distillate and the excess is titrated with iodine solution.

14.3 *Apparatus*:

14.3.1 *Distillation Apparatus*, consisting of a 500-cm^3 distillation flask, still head, vertical water-cooled condenser, and 100-cm^3 receiver with glass stopper through which pass a glass tube connected to the condenser and a shorter exit glass tube leading into a 50-cm^3 beaker.

14.3.2 *Volumetric Flask*, 100-cm^3.

14.3.3 *Conical Flask*, at least 250-cm^3.

14.4 *Reagents*:

14.4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

14.4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

14.4.3 *Acetic Acid*, glacial (CH_3COOH).

14.4.4 *n-Dodecyl Mercaptan, Alcoholic Solution* (1.25 %)—Dissolve 12.5 g of *n*-dodecyl mercaptan ($CH_3(CH_2)_{11}SH$) in 500 cm^3 of 2-propanol dilute to 1000 cm^3.

14.4.5 *Iodine Solution (0.0125 M)*—Transfer 3.2 g of iodine and 15 g of potassium iodide (KI) to an 800-cm^3 beaker, add 30 ml of water, and stir until all solution is complete. Dilute with water to 500 cm^3, and filter through a sintered-glass filter. Wash the filter with about 15 cm^3 of water, transfer the combined filtrate and washing to a 1000-cm^3 volumetric flask, dilute to the mark with water, and mix. Store the solution in a glass-stoppered, amber-glass bottle in a cool place. Standardize with sodium thiosulfate.

14.4.6 *Methanol* (CH_3OH).

14.4.7 *Potassium Hydroxide, Alcoholic Solution (6 %)* —Grind 6 g of potassium hydroxide (KOH) with successive 25-cm^3 portions of 95 % ethanol until dissolution is complete. Dilute to 100 cm^3 with 95 % ethanol. Filter immediately into a dark bottle and keep tightly closed. Let this solution stand overnight before using. When it becomes noticeably discolored, it should be discarded. Storing in a nitrogen atmosphere will preserve the solution considerably.

14.4.8 *2-Propanol* ($(CH_3)_2CHOH$).

14.4.9 *Sodium Carbonate* (Na_2CO_3).

14.4.10 *Sodium Thiosulfate Solution (0.0125 M)*—Dissolve 3.1 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 500 cm^3 of freshly boiled and cooled water, and add 0.1 g of sodium carbonate (Na_2CO_3). Dilute to 1000 cm^3 with freshly boiled and cooled water, and let stand for 24 h. Store the solution in a tightly closed glass bottle. Standardize in accordance with the procedure in Practice E 200.

14.4.11 *Silicone Antifoam Agent*.

14.5 *Procedure*:

14.5.1 Weigh 25.0 ± 0.2 g of latex into the distillation flask and add 100 cm^3 of water and 1 cm^3 of a suitable silicone antifoam agent. Place 25 cm^3 of methanol in the receiver and approximately 10 cm^3 of methanol in the 50-cm^3 beaker. Assemble the distillation apparatus so that the end of the tube connected to the condenser is immersed in the methanol in the receiver and the end of the exit tube connected to the receiver is immersed in the methanol in the 50-cm^3 beaker. (The purpose of the 50-cm^3 beaker is to recover any distillate not collected in the receiver.) Immerse the receiver and 50-cm^3 beaker in ice.

14.5.2 Distill the mixture, adjusting the rate of boiling to control frothing, and collect 50 cm^3 of distillate in the receiver.

14.5.3 Empty the contents of the receiver and 50-cm^3 beaker into the 100-cm^3 volumetric flask. Rinse twice with about 5 cm^3 of methanol by pouring the methanol through the condenser into the receiver and add the washings to the volumetric flask. Dilute to the mark with methanol.

14.5.4 Pipet a 50-cm^3 aliquot of the diluted distillate into the conical flask that contains 25 cm^3 of 2-propanol. Pipet 10 cm^3 of mercaptan solution into the flask. Add 1 cm^3 of alcoholic potassium hydroxide and allow the solution to react for exactly 2 min at 23 ± 2°C. Add 2 cm^3 of glacial acetic acid to stop the reaction. The resulting pH value should be between 4 and 6. Titrate with freshly standardized iodine solution to a yellow color that persists for at least 60 s. Discard the iodine solution remaining in the buret, unless it is required for immediate use.

14.5.5 Run a blank determination, omitting the distillation stage, using 50 cm^3 of 50-50 methanol-water mixture.

14.6 *Expression of Results*—Calculate the residual acrylonitrile content as a percentage by mass of the latex as follows:

$$\frac{A \times (B - C) \times V \times E \times F}{D \times M} \times 100 \tag{15}$$

which simplifies to:

$$\text{Residual acrylonitrile, \%} = \frac{A \times (B - C) \times 0.424}{D} \times 100 \tag{16}$$

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[7] *Reagent Chemicals, American Chemical Society Specificatio~~x~~ns* , Am. Chemi~~d~~cal Soc., Washington, DC. For suggestions on the testing of reagents not listed by the ~~bis or polyphenol types such as Ethyl 702, Santowhite Powder,~~ American Chemical Society, see *Reagent Chemicals and ~~Wingstay L,~~ Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and ~~of~~ the ~~diaryl-*p*-phenylenediamine type such as Wingstay 200, have been found to be equivalent to PBNA.~~ *United States Pharmacopeia*.

where:

A = molarity of the iodine solution,
B = iodine solution used in the blank determination, cm^3,
C = iodine solution used in the sample titration, cm ^{3},
D = aliquot of dilute distillate, cm^3,
E = 2 milliequivalents of iodine per millimole of iodine,
F = 0.05306 of acrylonitrile per millimole,
M = mass of sample, 25 g, and
V = total volume of distillate, 100 cm^3.

The results of duplicate determinations shall agree within 0.005 % absolute.

15. Particle Size Determination

15.1 *Apparatus*:

15.1.1 *Malvern Autosizer*:

15.2 *Procedure*:

15.2.1 Turn on Malvern Autosizer 2c for warm-up. Warm up for two minutes. Select option nine from starting menu program on computer. Make sure temperature on Malvern is 25°C or ±0.2. Adjust if needed. Make sure beam size on Malvern is set to small. Select F2 for sample description; follow screen instructions. Toggle F5 key for results printout option.

15.3 *Preparation of Sample*:

15.3.1 Using a 5-mL syringe, filter 20 mL DMW into a clean 50-mL beaker using a corning disposable sterile syringe filter. Also with the syringe, filter DMW into a four-sided cuvette to ⅔ full capacity. Using a disposable pipette, add one drop of latex to the 50-mL beaker. Swirl beaker until solution is uniform. Using a disposable pipette, transfer three drops of solution into the cuvette. Cover top with a small piece of parafilm and shake for a few seconds. Remove parafilm and insert a plastic cuvette top snugly. Place sample in Branson 1200 ultrasonic cleaner and sonicate for 30 s. Remove sample and dry outside of cuvette with a Kimwipe. Place cuvette into autosizer machine and close door (beam size—SMALL).

15.4 *Analysis of Sample*:

15.4.1 Adjust counts/1000 using the dial on top of the autosizer. Adjust to approximately 100. Sample level should be near "ideal." If not, adjust very gently with beam dial (near 150) or dilute the latex sample. If printout is desired, make sure printer ribbon is even with top of page. Printer: Power, ready, and on-line lights should be illuminated. F4 key starts sample measurement. Printout starts after about 4.5 min. Printing lasts for approximately 1 min. The *Z* average mean value at the end of the printout is recorded as particle size. Space key returns user to menu screen. *E* exits program. Turn off Malvern instrument.

16. Bound Monomers in Vinylpyridine Latex

16.1 *Scope*—This test method covers the range of vinylpyridine, styrene and butadiene normally encountered with vinylpyridine containing latices. It is not applicable to other synthetic latices. This method utilizes the infrared absorption bands at 1490, 1470 and 910 cm –1 wavenumbers arising from C = C stretching modes in styrene and vinylpyridine and CH=CH2 bending of the vinyl groups of polybutadiene segments, respectively. Because only absorption ratios are used in the calculations, the need to determine film thickness is eliminated.

16.2 *Apparatus*:

16.2.1 Double beam infrared spectrophotometer, Model 283 or equivalent.

16.2.2 KRS-5 crystals - 25 × 4 mm

16.2.3 Crystal holder

16.2.4 Applicator sticks

16.3 *Procedure*:

16.3.1 Place a drop or two of latex on a KRS-5 crystal. Spread the latex evenly over the entire crystal as quickly as possible with an applicator stick. Allow to dry under a heat lamp for 3–5 minutes. The film thickness should be such that all absorbance values remain on scale using an expansion factor of 1 to 1.5x. Use the following instrument parameters: scan time = 60 minutes, response setting = 2, slit setting = 7, absorbance mode, gain = full counterclockwise. Scan over the wavelength regions 1550–1450 cm^{-1} and 950–850 cm^{-1}. Clean the crystals with Tetrahydrofuran solvent. Repeat the procedure two more times, so that triplicate analyses are obtained.

16.4 *Calibration*:

16.4.1 Obtain styrene-butadiene-vinylpyridine latices where the bound monomers are accurately known. At least two different levels of vinylpyridine are needed. The following relationships are used for the calibrations:

$$\frac{C_{styrene}}{C_{VP}} = a + b\frac{A_{1490}}{A_{1470}} \tag{17}$$

$$\frac{C_{styrene}}{C_{BD}} = g + h\frac{A_{1490}}{A_{910}} \tag{18}$$

$$C_{styrene} + C_{VP} + C_{BD} = 100 \tag{19}$$

where:
Cx = wt. % values for the given monomer
Ax = absorbance at wavelength X

Constants a, b, g and h are then determined from a series of standard latices containing different known levels of the monomers. Concentration values for vinylpyridine can also be obtained by determining total nitrogen content using the Kjeldahl or Dumas procedures (preferably the latter). However, to use the total nitrogen methods requires that no other nitrogen containing compounds be present.

16.5 *Calculation*:

16.5.1 After the four constants listed above (a, b, g and h) are determined, the three equations listed in 5.4.1 above can then be solved simultaneously. A computer program can be set up to perform these calculations.

17. Residual Monomers

17.1 *Scope*—This method describes the operation of the Perkin Elmer gas chromatograph. This instrument is used to test for residual monomers in the latex, as well as the purity of the raw material. The use of the sample encapsulating unit is described. The method can be applied to any liquid sample and many solis samples provided the proper columns and conditions are used. This preferred method is Test Method D 4026 – Method B. This method may not give the same results as the wet method.

17.2 *Equipment*:

1. Perkin Elmer gas chromatograph, Model 3920 or Sigma 2, with FID
2. Column P&E 10 % apiezon L on 80/100 chromosorb W, or Supelco 10 % SP 2100 and 80/100 Supelcoport.
3. Capsule sampler - Model MS-41
4. Capsules, capsule holder
5. Filter paper, No. 5
6. Micropipetter, 1 µl - Fisher No. 21-170-10
7. Micropipettes, for Micropipetter above, 1 µl, Fisher No. 21-170-11
8. Absorbent tissues, Kimwipes, or equivalent
9. Cork bore, No. 1
10. Integrator, Model 2, Spectraphysics
11. Syringe, Pressure-Lok, 2 ml
12. Pressure bottle, 8 ounce with Teflon lines septum cap
13. Analytical balance

17.3 *Reagents*:

1. Gases:	–Helium, the regulator outlet pressure should be set at 70 psig. –Hydrogen, the regulator outlet pressure should be set at 20 psig. – Plant air, the regulator outlet pressure should be set at 35 psig on No.1 and 2 (Model 3920) and No. 3 (Sigma 2) instruments. All gases must be of prepure quality.
2. Volatile Standard, 99.9 % purity	a. Butadiene b. 4-Vinylcylcohexane c. Ethlybenzene d. Styrene e. Vinylpyridine f. Other monomers and solvents as requested
3. Control latex with known level of styrene.	
4. Solvent, carbon disulfide (CS_2), chromatographic grade.	
5. Methyl alcohol, chromatographic grade	

17.4 *Procedure*:

17.4.1 *Capsulating the Sample*:

17.4.1.1 Using forceps, transfer a capsule to a 10 capsule magazine. The capsule must fit securely. Use new plastic magazine inserts if the capsule is not held securely. Using a No. 1 cork bore, cut out a piece of filter paper and insert in the bottom of the capsule. Place the capsule magazine in the sealing unit with the capsule opposite the cutting blade. Loosening the knurled knob on the micropipettor, carefully insert a micropipette and tighten the knurled knob securely. Depress the plunger and insert the pipette into the sample. With the pipette still in the sample, release the plunger. The sample will be in the micropipette. Remove the pipette from the sample. Wipe the outside of the pipette with a tissue. Discharge it into the capsule, touching the filter paper. Quickly seal the capsule by pulling down on the crimping lever. The sealed capsule in the magazine is then transferred to the capsule holder using the capsule loading unit.

17.4.2 *Injection of the Sample*:

17.4.2.1 *For Model 3920:* Remove the capsule holder from the hot injector assembly and immediately align the capsule holder in the loading unit with capsule in the magazine. Press down on the capsule holder firmly until the capsule is seated in the capsule holder. Be certain to remove any used capsule from the holder and instrument. Remove the capsule holder from the loading unit.

Check for the conditions desired, and wait for the green light to come on. The green light must be on before inserting the sample. Gently and slowly insert the capsule holder containing the sample capsule, just short of puncturing the capsule. The capsule should be in the injection port to be heated. After 15 seconds, insert the capsule holder all the way into the instrument to puncture the capsule. The forepart of the capsule holder will be almost flush with the front of the instrument. Immediately after injecting the sample, press the start button on the integrator. Do not press the light on the chromatograph. After one minute, remove the capsule holder from the instrument. While the instrument is cooling, reinsert the capsule holder to keep it hot. When the door opens at the end of the testing cycle, press the button again to stop the integrator. Turn off the chart. When the instrument has cooled to the initial temperature, another sample can be injected. The integrator prints out the elution time and the peak area, and the ppm of each component of interest.

17.4.2.2 *For Model Sigma 2:* Set instrument for conditions desired, and wait for the oven ready light to come on. This light and the start button must be lit before injecting the sample. Gently and slowly insert the capsule holder containing the sample into the instrument, just short of puncturing the capsule. The capsule should be in the injection port section to be heated. After 15 seconds, insert the capsule holder all the way into the instrument to puncture the capsule. The forepart of the capsule holder will be almost flush with the front of the instrument. Immediately after injecting the sample, press the start button on the instrument. This starts the instrument and the computer. At the end of the cycle, the oven will cool and the results will be printed out. When the instrument has cooled to the initial temperature, another sample can be injected.

17.4.3 *Preparation of Standards for Instrument Standardization*:

17.4.3.1 *Liquid Monomers (Styrene, VP, etc):* Place 100 ml of carbon bisulfide in the pressure bottle and seal tightly. Transfer approximately 2 ml of monomer into a Pressure-Lok syringe and close the valve. Expel any air. Make certain the syringe does not leak. Using an absorbent tissue, clean the outside of the syringe and weigh. Carefully inject the required amount of monomer into the pressure bottle below the liquid surface and mix. Withdraw the syringe and re-weigh. Follow the above procedure for each addition of monomer. The syringe may have to be refilled. Any excess monomer should be expelled into a solvent waste safety can for proper disposal and the syringe should be rinsed copiously with methyl alcohol and allowed to air dry.

17.4.3.2 *Butadiene (Bd):* Obtain a small sample of butadiene (liquid) and place it on dry ice. Place a 2 ml Pressure-Lok syringe in the freezer and allow it to come to freezer temperature. Place 100 ml of carbon disulfide in the pressure bottle(s) to which other monomers have been added. Store in the freezer and allow to come to temperature. Transfer the Bd to the syringe and close the valve. Using an absorbent tissue, clean the outside of the syringe, place in an insulator tube and weigh. Carefully inject the required amount of monomer into the pressure bottle below the liquid surface and mix. Withdraw the syringe, place in an insulator tube and re-weigh. The difference is the amount of Bd injected. Prepare standards at about 0.01, 0.04, 0.1, 0.3 and 0.7 grams per 100 ml of solvent. Any excess monomer in the syringe should be vented to the outside air. The pressure bottle of Bd should also be vented to the outside air after standards have been tested. The samples are ready to be injected into the Gas Chromatograph.

18. Precision and Bias

18.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

18.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include these test methods.

18.3 A Type 1 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. Information on the number of laboratories and the materials used is given in Table 2.

18.4 The results of the precision calculations for pooled repeatability and reproducibility are given in Table 2 for a broad series of test parameters.

18.5 The precision of these test methods may be expressed in the format of the following statements which use an “appropriate value” of *r*, *R*, (*r*), or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 2 closest to the mean level under consideration at any given time for any given test, for any given material in routine testing operations.

18.6 *Repeatability*—The repeatability, *r*, of these test methods has been established as the appropriate value tabulated in Table 2. Two single test results, obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *r* (for any given must be considered to have come from different or nonidentical sample populations.

18.7 Repeatability and reproducibility expressed as a percent of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

18.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for these test methods, since the value (of the test property) is exclusively defined by the test methods. Bias therefore cannot be determined.

19. Keywords

19.1 latex; rubber latices; synthetic

TABLE 2 Type 1 Precision for Synthetic Rubber Latices—Various Tests

NOTE—Midpoint of range used for (*r*) and (*R*) calculations:

Sr = Repeatability standard deviation, in measurement units,
r = Repeatability, in measurement units,
(r) = Repeatability, (relative) percent,
SR = Reproducibility standard deviation, in measurement units,
R = Reproducibility, in measurement units, and
(R) = Reproducibility, (relative) percent.

Test Method	Property Range Tested	Within Laboratory			Between Laboratories			Number of Property Levels	Number of Participating Laboratories
		Sr	*r*	(*r*)	*SR*	*R*	(*R*)		
Total solids, %	29.40 to 67.3	0.06	0.17	0.35	0.08	0.23	0.47	4	4
Volatile unsaturates, %	0.012 to 0.12	0.007	0.020	30.3	0.011	0.031	47.0	4	4
pH value	9.52 to 10.50	0.05	0.142	1.4	0.11	0.311	3.1	4	5
Surface tension, Mn/M	31.8 to 51.2	0.30	0.85	2.1	0.80	2.26	5.5	4	3
Viscosity, mPa·s,	6.0 to 40.3	0.40	1.13	4.9	1.60	4.53	19.6	3	4
Coagulum content mass (weight), %	0.000 to 0.0320	0.00050	0.0014	9.4	0.0053	0.015	99.0	4	3
Mooney viscosity,	52.6 to 192.0	3.4	9.62	7.9	4.7	13.3	10.9	4	4
Mechanical stability, %	0.005 to 0.220	0.011	0.031	27.6	0.027	0.0764	67.9	4	3

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