



# Standard Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex<sup>1</sup>

This standard is issued under the fixed designation D1076; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This specification covers requirements for first grade concentrated natural rubber latex (see [Table 1](#)) of the following categories:

*Category 1*—Centrifuged *Hevea* natural latex preserved with ammonia only or by formaldehyde followed by ammonia.

*Category 2*—Creamed *Hevea* natural latex preserved with ammonia only or by formaldehyde followed by ammonia.

*Category 3*—Centrifuged *Hevea* natural latex preserved with low ammonia with other necessary preservatives.

*Category 4*—Centrifuged, or centrifuged and creamed, guayule latex, or other natural rubber latex, containing less than 200  $\mu\text{g}$  total protein per gram dry weight of latex, with ammonia or other hydroxide, with other necessary preservatives and stabilizers.

*Category 5*—Centrifuged *Hevea* natural latex treated with aluminum hydroxide or by other means, preserved with ammonia only or by formaldehyde followed by ammonia containing less than 0.5 % non-rubber content.

1.2 This specification is not necessarily applicable to latices prepared or preserved by other methods, and shall not be construed as limiting the desirability or usefulness of other categories of latices. It does apply to natural latex sources other than *Hevea brasiliensis* but does not apply to compounded latex concentrates.

1.3 The analytical procedures applicable to the specifications are included and appear in the following order:

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1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- [D1278 Test Methods for Rubber from Natural Sources—Chemical Analysis](#)
- [D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)
- [D5712 Test Method for Analysis of Aqueous Extractable Protein in Latex, Natural Rubber, and Elastomeric Products Using the Modified Lowry Method](#)
- [D6499 Test Method for The Immunological Measurement of Antigenic Protein in Natural Rubber and its Products](#)
- [E70 Test Method for pH of Aqueous Solutions With the Glass Electrode](#)

## 3. General Specification Requirements

3.1 In manufacturing, the material shall be processed in accordance with the best commercial practice and shall be of uniform composition.

3.2 The material shall conform to the chemical and physical requirements prescribed in [Table 1](#).

## 4. Significance and Use

4.1 This specification denotes limits on the 4 categories of latex as defined in the scope and defines the test methods to use for the specified properties. These test methods may be used for production control or for referee purposes.

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.22 on Natural Rubber.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

**TABLE 1 Requirements for Specified Latex Categories**

	Category 1	Category 2	Category 3	Category 4	Category 5
Total solids, min, %	61.3	66.0	61.3	44.0	61.3
Dry rubber content (DRC), <sup>A</sup> min, %	59.8	64.0	59.8	42.0	60.8
Total solids content minus dry rubber content, max, %	2.0	2.0	2.0	2.0	0.5
Protein content (µg/g dw latex)				200 max	
Total protein by <b>D5712</b>				None detectable	
<i>Hevea</i> antigenic protein by <b>D6499</b>					
Total alkalinity calculated as ammonia, as % latex	0.60 min	0.55 min	0.29 max	0.60 min	0.60 min
Or: total alkalinity calculated as KOH, as % latex				0.10 min	
Sludge content, max, %	0.10	0.10	0.10	0.10	0.1
Coagulum content, max, %	0.050	0.050	0.050	0.050	0.05
KOH number, max <sup>B</sup>	0.80	0.80	0.80	0.80	0.8
Mechanical stability, s, min	650	650	650	90	650
Copper content, max, % of total solids	0.0008	0.0008	0.0008	0.0008	0.0008
Manganese content, max, % of total solids	0.0008	0.0008	0.0008	0.0008	0.0008
Color on visual inspection		no pronounced blue or gray <sup>C</sup>			
Odor after neutralization with boric acid		no putrefactive odor			

<sup>A</sup> Dry rubber content by definition and use is the acid coagulable portion of latex after washing and drying.

<sup>B</sup> It is accepted that KOH numbers for boric acid preserved latices will be higher than normal, equivalent to the amount of boric acid in the latex.

<sup>C</sup> Blue or gray color usually denotes iron contamination caused by improper storage in containers.

## 5. Inspection

5.1 Inspection of the material shall be made as agreed upon between the purchaser and the seller as part of the purchase contract.

### SAMPLING AND METHODS OF TESTING

## 6. Preparations for Sampling

### 6.1 Drums:

6.1.1 *Open-Head Drums*—The top shall be removed and the contents stirred with a high-speed stirrer for 10 min.

6.1.2 *Closed-Head Drums*—If the drum has at least 2 % air space, which is 20 mm (0.75 in.) on a standard drum, lay it on its side and roll for not less than 10 min. Up end the drum to its original position and allow to stand for 15 min and then repeat the rolling operation for at least a further 10 min. In the case of drums with less than 2 % air space, all of the latex in the closed-head drum shall be transferred to a larger vessel and mixed with a high-speed stirrer for 10 min.

6.2 *Tank Cars*—Samples shall be taken from the top and bottom of the car. If the total solids in the top and bottom samples agree within 0.5 %, the car shall be considered uniform enough for sampling. If top and bottom samples do not agree within 0.5 %, the contents of the car shall be agitated until samples taken from the top and bottom do agree on total solids within 0.5 %.

## 7. Sampling

7.1 *Drums*—After preparations for sampling, sample without delay. A suitable method is by slowly inserting a clean, dry glass tube of 10 to 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. Alternatively, a specially constructed metal sampling tube may be used, the bottom of which can be closed

by remote control. No copper or brass shall be used in any part of its construction. At least 10 % of the drums in a shipment shall be sampled.

7.2 *Tank Cars*—Separate samples of at least 475 cm<sup>3</sup> each shall be taken from the top, center, and bottom of the tank car. Take the top sample first, then the center sample, and the bottom sample last. Use a weighted sampler with a remotely operated removable top, or other suitable sampling device that will accomplish the same results. Blend the three samples thoroughly. Each sample shall be poured immediately into a tightly stoppered container. The three samples shall be combined and thoroughly blended into a composite sample. At least 950 cm<sup>3</sup> of this composite sample shall be used for test. One composite sample only is required from each tank car.

## 8. Total Solids

8.1 *Apparatus*—Tared, covered, flat-bottom weighing dish approximately 60 mm (2.5 in.) in diameter, which may be made of glass, tinned metal, or aluminum.

8.2 *Reagent*—Distilled water.

8.3 *Procedure*—Weigh  $2.5 \pm 0.5$  g of the latex to the nearest 1 mg in the tared, covered weighing dish. Remove the cover and distribute the latex over the bottom of the dish over an area of approximately 32 cm<sup>2</sup> (5 in.<sup>2</sup>). This may be facilitated by carefully adding approximately 1 cm<sup>3</sup> of distilled water to the latex and gently swirling the dish. With the dish uncovered, dry the specimen in a vented air oven for 16 h at  $70 \pm 2^\circ\text{C}$  or 2 h at  $100 \pm 2^\circ\text{C}$ . Replace the cover, cool in a desiccator to room temperature, and weigh. Repeat drying and weighing until the mass is constant to 1 mg or less. Tests shall be run in duplicate and shall check within 0.15 %. The average of the two determinations shall be taken as the result.

8.4 *Calculations*—Calculate the percentage of total solids as follows:

$$\text{Total solids, \%} = [(C - A)/(B - A)] \times 100 \quad (1)$$

where:

- $A$  = mass of the weighing dish, g,  
 $B$  = mass of the dish plus the original sample, g, and  
 $C$  = mass of the dish plus the dried sample, g.

## 9. Dry Rubber Content

9.1 *Apparatus*—Porcelain evaporating dish approximately 100 mm in diameter and 50 mm deep.

9.2 *Reagent*—Acetic acid aqueous solution (20 Mg/m<sup>3</sup>).

9.3 Weigh approximately 10 g of the latex to the nearest 1 mg into a porcelain evaporating dish, and add distilled water until the total solids content is approximately 25 %. Add sufficient acetic acid (2 %), while stirring constantly over a 5-min period, to coagulate completely the latex (80 cm<sup>3</sup> should be sufficient). Category 4 guayule latex may also require the addition of up to 20 mL hydrochloric acid (2 %) to complete its coagulation.

9.3.1 Place the dish on a steam bath and leave undisturbed for 15 to 30 min. A clear serum should result, and 30 min is the maximum time allowed. If the serum is milky, the acid was either added too fast or in insufficient amount and the procedure should be repeated until a clear serum results. Pick up coagulated latex particles with the main body of the coagulum. Wash coagulum in running water and pass between rolls. Repeat this process 5 times and reduce the sheet of coagulated rubber to a maximum thickness of 2 mm and dry at 70 ± 2°C in a vented air oven atmosphere. If oxidation occurs, the test may be run with the option of using a drying temperature of 55 ± 2°C, or an antioxidant may be added to the latex before coagulation. If polymer oxidation occurs, the test may be rerun with the option of using a drying temperature of 55 ± 2°C. Cool in a desiccator to room temperature and weigh. Repeat drying and weighing until the mass is constant to 1 mg or less.

9.4 Duplicate samples shall be run and shall check within 0.2 %. The average of the two determinations shall be taken as the result.

9.5 *Calculation*—Calculate the dry rubber content as follows:

$$\text{Dry rubber content, \%} = \frac{\text{mass of dry coagulum}}{\text{mass of sample}} \times 100 \quad (2)$$

## 10. Protein Content

10.1 *Total Protein*—Solubilize latex proteins in 1 % SDS and 50 mM sodium phosphate buffer (final concentration) and then quantify using the modified Lowry test according to Test Method **D5712**.

10.1.1 *Solubilization Method*—Mix latex sample (500 µl) with 450 µl 100 mM sodium phosphate buffer (1:1) into three microfuge tubes for each sample; add 50 µl 20 % SDS into each tube, mix; incubate at 25°C for 2 h with shaking at 200 rpm; spin for 5 min, remove rubber pad; transfer aqueous phase into new tubes and spin again to clarify; divide each sample into 3 × 0.6 mL tubes for each sample (these can be stored at 4°C overnight); prepare bovine serum albumin standards in extraction buffer at 0, 5, 10, 15, 25, 50, 100, 200, 300, and 400 µg/mL; add 60 µl 1.5 mg/mL sodium deoxycholate to samples and standards, mix, stand for 10 min; add

120 µl of 72 % freshly mixed trichloroacetic acid and phosphotungstic acid (1:1) into each sample, mix, incubate for 30 min at RT; spin 15 min at 6000 xg, remove supernatant, air dry pellet; suspend each pellet in 250 µl 0.2 M sodium hydroxide and store at 4°C until assayed; assay within 24 h using the modified Lowry test according to Test Method **D5712**.

10.2 *Hevea Antigenic Protein*—Solubilize latex proteins with 1 % SDS and 50 mM sodium phosphate buffer (final concentration) then quantify using the antigenic protein assay according to Test Method **D6499**.

## 11. Total Alkalinity

11.1 *Apparatus*—Glass electrode pH meter.

11.2 *Reagent*—0.1 mol standard HCl.

11.3 *Preparation of Specimen*—Place approximately 5 g of latex into a glass weighing bottle of approximately 10-cm<sup>3</sup> capacity, having a ground glass cap, and weigh to the nearest 1 mg. Pour the specimen into a beaker containing approximately 300 cm<sup>3</sup> of distilled water, restopper quickly to prevent loss of ammonia, and set aside for reweighing. The specimen mass is equal to the difference between the two weighings. The transfer of the sample to the beaker shall be done in such a way that no latex runs down the outside of the weighing bottle.

11.4 *Recommended Procedure*—Insert the electrodes of a calibrated glass electrode pH meter into the liquid and note the pH. The meter should be calibrated and the pH measurements made in accordance with Test Method **E70**, and the directions given by the manufacturer of the meter. Slowly, and while stirring constantly, add 0.1 mol HCl until a pH of 6.0 is reached. Too rapid addition or inadequate stirring of the sample while the acid is being added may cause local coagulation of the rubber. With samples of unknown alkalinity, it is recommended that the acid be added in 1-cm<sup>3</sup> increments, and a pH reading taken 10 s after each addition. As the pH of 6.0 is approached, smaller increments should be added.

11.5 *Alternative Procedure*—Prepare the sample as described in **11.3**. Add 6 drops of a 0.10 % alcoholic solution of methyl red and titrate with approximately 0.1 mol HCl until the indicator turns pink. The end point occurs before complete coagulation takes place and the color change of the indicator can be detected against the white background of the slightly coagulated latex.

11.6 *Calculation*—Calculate the total alkalinity, reported as NH<sub>3</sub> based on grams of NH<sub>3</sub> per 100 g of latex as follows:

$$\text{Total alkalinity, (as NH}_3\text{) \%} = (1.7 \times M \times n) / W \quad (3)$$

where:

- $M$  = mole of the standard HCl,  
 $n$  = volume of standard HCl required, cm<sup>3</sup>, and  
 $W$  = original mass of the latex, g.

11.6.1 If total alkalinity is wanted as KOH, the following calculation applies to both procedures:

$$\text{Total alkalinity, (as KOH) \%} = (5.61 \times M \times n) / W \quad (4)$$

where:

- $M$  = mole of the standard HCl,

$n$  = volume of standard HCl required, cm<sup>3</sup>, and  
 $W$  = original mass of the latex, g.

11.6.2 If total alkalinity is wanted based on the water phase of the latex, calculate as follows:

$$\text{Total alkalinity, as \% of water} = (1.7 \times M \times n) / W(1 - TS/100) \quad (5)$$

where:

$TS$  = percent total solids,  
 $M$  = mole of the standard HCl,  
 $n$  = volume of standard HCl required, cm<sup>3</sup>, and  
 $W$  = original mass of the latex, g.

11.6.3 If total alkalinity is wanted as KOH based on the water phase of the latex, calculate as follows:

$$\text{Total alkalinity, (as KOH) as \% of water} = (5.61 \times M \times n) / W(1 - TS/100) \quad (6)$$

where:

$TS$  = percent total solids,  
 $M$  = mole of the standard HCl,  
 $n$  = volume of standard HCl required, cm<sup>3</sup>, and  
 $W$  = original mass of the latex, g.

## 12. Viscosity

### 12.1 Apparatus:

12.1.1 *Rotational Viscometer*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this method include:

12.1.1.1 *Drive Motor*, to apply an unidirectional rotational displacement to the specimen a rate of 0.63 to 6.3 rad/s (6 to 60 rpm) constant to within  $\pm 1\%$ .

12.1.1.2 *Coupling Shaft*, or other means to transmit the rotational displacement from the motor to the specimen.

12.1.1.3 *Geometry, Spindle, Tool, or Rotational Element*, to fix the specimen between the drive shaft and a stationary position.

NOTE 1—Each rotational element typically covers a range of about 1.5 decades of viscosity. The geometry is selected so that the measured viscosity is between 10 and 90 % of the range of that geometry.

12.1.1.4 *Force Sensor*, to measure the torque developed by the specimen to the rotational displacement of the geometry.

12.1.1.5 *Guard*, to protect the geometry from mechanical damage.

12.1.1.6 *Temperature Sensor*, to provide an indication of the specimen temperature over the range from 20 to 30°C readable to within  $\pm 0.02^\circ\text{C}$ .

12.1.1.7 *Data Collection Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscosity are torque, rotational speed, temperature and time.

12.1.1.8 *Stand*, to support, level, and adjust the height of the drive motor, shaft and geometry.

12.1.1.9 *Specimen Container*, of approximately 600 mL capacity to contain the test specimen during testing.

NOTE 2—A 600 mL laboratory beaker has been found to be suitable for this purpose.

12.1.1.10 Auxiliary instrumentation considered necessary or useful in conducting this method include:

(1) *Data analysis capability*, to provide viscosity, stress or other useful parameters derived from measured signals.

(2) *Level*, to indicate the vertical plumb of the drive motor, shaft and geometry.

12.1.1.11 *Temperature Bath*, to provide a controlled isothermal temperature environment for the specimen within the applicable temperature range of this method.

(1) *Temperature Controller*, capable of operating the temperature bath at an isothermal temperature over the range of 20 to 30°C constant to within  $\pm 2^\circ\text{C}$ .

12.1.2 *Calibration*—Calibration can readily be made by using viscosity calibration reference materials available from the apparatus supplier.

### 12.2 Preparation of Specimen:

12.2.1 Strain the specimen through a standard 180- $\mu\text{m}$  sieve with  $0.180 \pm 0.009\text{-mm}$  ( $0.0070 \pm 0.0004\text{-in.}$ ) openings and  $0.131 \pm 0.01\text{-mm}$  ( $0.0052 \pm 0.0005\text{-in.}$ ) wire diameter and adjust the latex to  $60 \pm 0.1\%$  total solids.

12.2.2 Condition the specimen at the  $25 \pm 2^\circ\text{C}$  test temperature in a suitable bath for a period of 2 h or until the latex sample is free of air.

### 12.3 Procedure:

12.3.1 Pour a specimen of latex prepared as described in 12.2 slowly down the side of the container, previously cooled to 25°C, preventing incorporation of air.

12.3.2 Remove guard from the viscometer except for #1 and #2 Spindles of the LV and RV spindle sets.

NOTE 3—This is done to reduce later cleaning problems.

12.3.3 Insert the rotational element of the viscometer into the latex until the surface of the latex is within the depth indicator on the shaft.

NOTE 4—Avoid trapping air bubbles on the surface of the geometry by tilting the instrument slightly during immersion of the geometry. Do not hit the geometry against the side of the container while it is attached to the viscometer, as this can damage the shaft alignment. Level the viscometer so the geometry is plumb.

12.3.4 Ensure that the test temperature is between 23 and 25°C.

12.3.5 Initiate the rotation of the geometry at the lowest speed available and allow at least 5 revolutions of the geometry.

12.3.6 Increase the speed until the torque reading produced is nearest the midpoint of the scale. Allow at least 5 revolutions of the geometry.

NOTE 5—In general, the difference in spindles will not affect the results.

12.3.7 Record and report the viscosity (in mPa•s), rotational speed, and rotational element model number.

NOTE 6—mPa•s = cP

12.3.8 Remove the geometry from the test specimen. Clean the geometry with a suitable solvent. Reassemble the guard. Safely dispose of the test specimen.



### 13. Sludge Content

13.1 *Apparatus*—Centrifuge operating at 12 000 m/s<sup>2</sup> with two 50-cm<sup>3</sup> pear-shaped centrifuge tubes held in a centrifuge accessory.

13.2 *Ammonia-Alcohol Mixture*—Prepare as follows:

Ammonium hydroxide (sp gr 0.90), cp	28 cm <sup>3</sup>
Ethyl alcohol, 95 % min purity	946 cm <sup>3</sup>
Water	2810 cm <sup>3</sup>

13.3 *Procedure*—Weigh 45 to 50 g of the latex into each of two 50-cm<sup>3</sup> centrifuge tubes and centrifuge them for 20 min at approximately 240 rad/s (2300 rpm). During this centrifuging place small filter papers over the ends of the tubes, fastening them with rubber bands, to prevent excessive evaporation of the latex and possible formation of a surface skin. As the creaming is considerable, scoop off most of the heavy top layer with a longhandled porcelain spoon before pipeting. Using a pipet with an end opening of about 2 mm draw off the supernatant latex to approximately 10 mm above the top of the sludge. Fill the tubes to the top with the ammonia-alcohol, solution, and balance them in pairs. Recentrifuge for 25 min. Again pipet off the supernatant liquid to approximately 10 mm above the top of the sludge. Repeat this procedure until the supernatant solution is clear after centrifuging. After the final centrifuging, drain the tubes to the 1-cm mark and transfer the residues to tared 200-cm<sup>3</sup> beakers, using some of the ammonia-alcohol mixture as needed. Evaporate on a hot plate to a low level, complete the drying at 70 ± 2°C, and weigh. The masses of the dried residues run in duplicate should agree within 1 mg.

13.4 *Calculation*—Calculate the sludge content as follows:

$$\text{Sludge content, \%} = (\text{mass of dried residue/mass of sample}) \times 100 \quad (7)$$

### 14. Coagulum Content

14.1 *Apparatus*—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 40 mm (nominal 1½-in. pipe size).

14.2 *Procedure*—Weigh 200 g of the well-stirred sample and dilute with an equal volume of 5 % alkali soap solution. Sodium or potassium oleates are recommended. Filter this mixture through the 180-μm mesh screen in the steel union and wash the coagulum retained on the screen with a 5 % soap solution. Finally wash the coagulum free of soap with distilled water. Remove the screen from the union and dry at 100 ± 2°C for 30 min. Cool in a desiccator and weigh. Repeat the drying procedure for intervals of 15 min. Cool and weigh until the loss in mass between successive weighings is less than 1 mg. The difference between the original mass of the screen and the mass of the screen plus coagulum retained on it represents the mass of dried coagulum.

14.3 *Calculation*—Calculate the percentage of coagulum as follows:

$$\text{Coagulum content, \%} = (m_1/m_0) \times 100 \quad (8)$$

where:

$m_0$  = mass of test portion, g, and

$m_1$  = mass of coagulum, g.

### 15. KOH Number

15.1 *Apparatus*—Any pH meter dependent on electrometric measurements and a glass electrode-calomel assembly for determining pH may be used. 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 from 8 to 14.

15.2 *Reagents*—The following reagents will be required:

15.2.1 *Formaldehyde Solution (5.0 %), Acid-Free*—Dilute stock USP grade formaldehyde to 5.0 % with distilled water and neutralize with 0.1 mol KOH solution using phenolphthalein as an indicator. Titrate to faint pink color.

15.2.2 *Potassium Hydroxide Solution (0.5 mol), Carbonate-Free*—First prepare a KOH solution of approximately 40 % strength from cp stick KOH and freshly boiled and cooled distilled water. Hold each stick of KOH separately by means of clean stainless steel tongs and rinse it in a stream of distilled water to remove surface carbonate. Drop the washed sticks into a beaker of the distilled water tared on a balance, continuing until sufficient KOH is in the beaker for the 40 % solution. After the 40 % KOH solution has cooled to room temperature, pipet a 10-cm<sup>3</sup> portion into a short 25-cm<sup>3</sup> graduate, add 10 % BaCl<sub>2</sub> solution until no further precipitation takes place upon allowing the BaCO<sub>3</sub> to settle, and then add one more drop of BaCl<sub>2</sub> solution to the clear supernatant liquid. Several 10-cm<sup>3</sup> portions of the 40 % KOH solution may be titrated with the 10 % BaCl<sub>2</sub> solution as needed to determine the exact equivalent amount of BaCl<sub>2</sub>. From these results the proper amount of BaCl<sub>2</sub> solution for the whole 40 % KOH solution is calculated. Add this amount, while stirring, along with the titrated samples if desired. To check the removal of carbonate, dilute a 5-cm<sup>3</sup> sample of the BaCl<sub>2</sub>-treated KOH solution with 10 cm<sup>3</sup> of the distilled water and divide it into two portions. Add one drop of 10 % BaCl<sub>2</sub> solution to one portion and one drop of 10 % K<sub>2</sub>SO<sub>4</sub> solution to the other. A precipitate in the first case shows incomplete precipitation of carbonate. A precipitate with the K<sub>2</sub>SO<sub>4</sub> solution shows an excess of barium. A very slight precipitate in either case can be ignored. If there is an appreciable excess of barium, it may be removed by adding the 10 % K<sub>2</sub>SO<sub>4</sub> solution dropwise, until one drop gives no further precipitation. Any excess of carbonate should likewise be removed by dropwise addition of BaCl<sub>2</sub> solution. Allow the precipitate in the main solution to settle. Then decant the clear KOH solution, dilute to approximately 5 % with freshly boiled and cooled distilled water, and store in wax-lined screw-top bottles. After standardization of this solution against a standard acid or National Bureau of Standards acid potassium phthalate (Standard sample No. 185), dilute it again with the distilled water to 0.5 N and then restandardize for use.

15.3 *Procedure*—Weigh out accurately a specimen of latex containing approximately 50 g of solids into a 400-cm<sup>3</sup> beaker. Adjust the ammonia content to 0.5 % on the water basis by addition of 5 % formaldehyde (1 cm<sup>3</sup> = 0.0189 g NH<sub>3</sub>) while stirring. Calculate the amount of formaldehyde solution for this adjustment as follows:

Formaldehyde solution (5 %),  $\text{cm}^3 = W(100 - TS)$  (9)

(%  $\text{NH}_3$  on water phase - 0.50)/189

where:

$W$  = grams of wet latex sample g, and  
 $TS$  = percentage of total solids.

Add enough distilled water to dilute the latex to about 30 % solids. Place the beaker containing the latex by the pH meter and insert the titration electrodes. Determine the pH and record the results. Add slowly 5  $\text{cm}^3$  of 0.5 mol KOH solution while stirring. After 10 s record the pH. Continue additions of 1- $\text{cm}^3$  increments of 0.5 N KOH solution while stirring, and record the pH after waiting 10 s after each addition.

**15.3.1 End Point Determination**—The end point of the titration is the point of inflection of the curve of pH value versus the volume in  $\text{cm}^3$  of KOH solution. At this point, the slope of the curve, the first differential, reaches a maximum and the second differential is zero. The end point shall be calculated from the second differential on the assumption that this is linear through the 1  $\text{cm}^3$  increment through which it passes from positive to negative. To determine the first differential, tabulate the differences  $\Delta\text{pH}/\Delta\text{cm}^3$  for each 1  $\text{cm}^3$  increment. This can be used as the first differential at the mean of the two values compared for difference. Determine the second differential by the differences of the values of the first differential again using the mean of the two values used for the difference. The first 1  $\text{cm}^3$  addition that shows a decrease in the first differential is the  $\text{cm}^3$  that includes the point of inflection. The point of inflection is where the linear intercept crosses the zero line of the second differential.

**15.3.2 Example of End Point Determination**—The following data is an example of the point of inflection determination. Readings are shown only in the area approaching the inflexion. Points from 6.0 to 12.0  $\text{cm}^3$  would have been taken but are not pertinent to the end point:

KOH Solution, $\text{cm}^3$	pH	First Difference, $\Delta\text{pH}/\Delta\text{cm}^3$	Second Difference, $\Delta(\Delta\text{pH}/\Delta\text{cm}^3)$
13.0	10.47		
13.5	...	0.18	...
14.0	10.65	...	0.03
14.5	...	0.21	...
15.0	10.86	...	0.07
15.5	...	0.28	...
16.0	11.14	...	-0.04
16.5	...	0.24	...
17.0	11.38	...	-0.09
17.5	...	0.15	...
18.0	11.53	...	

By the slope of the line from + 0.07 to - 0.04 the intercept with zero gives a ratio of 7/11 of the distance between 15.0 and 16.0  $\text{cm}^3$  of KOH. The point of inflection is therefore 15 7/11, or 15.64. Proof of the ratio can be done by the geometry of the triangles formed.

**15.4 Calculation**—Calculate the KOH number, expressed as the number of grams of KOH required to neutralize the acids present in 100 g of solids in latex, as follows:

$$\text{KOH No.} = (\text{cm}^3 \text{ KOH} \times M \times 561) / (TS \times \text{mass of sample}) \quad (10)$$

where:

$TS$  = percentage of total solids, and  
 $M$  = mole of standard KOH solution.

## 16. Determination of pH

**16.1 Apparatus**—Any pH meter dependent on electronic measurements, and a glass electrode-calomel assembly for determining pH may be used. 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 from 8 to 14.

**16.2 Procedure**—Calibrate the pH meter in accordance with Test Method E70 and the directions given by the manufacturer of the meter. Take any convenient size of sample and adjust the temperature to a range from  $23 \pm 1^\circ\text{C}$  by mildly agitating the sample-container in a water bath at a suitable temperature. Determine the pH and record both the pH and the temperature of the latex sample.

## 17. Mechanical Stability

**17.1 Scope**—This method covers the determination of the mechanical stability of concentrated natural latices using the high-speed stirring technique.

**17.2 Apparatus:**

**17.2.1 Stirrer**—The stirring apparatus shall consist of a vertical shaft high-speed stirrer capable of maintaining a speed of  $1470 \pm 22 \text{ rad/s}$  ( $14\,000 \pm 200 \text{ rpm}$ ) 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.

**17.2.2 Agitator**—The agitator itself shall consist of a polished stainless steel disk  $20.83 \pm 0.03 \text{ mm}$  ( $0.820 \pm 0.001 \text{ in.}$ ) in diameter and  $1.57 \pm 0.05 \text{ mm}$  ( $0.062 \pm 0.002 \text{ in.}$ ) in thickness, having a threaded stud at its exact center for attachment to the center of the lower end of the stirrer shaft.

**17.2.3 Test Bottle**—The test bottle shall be a flat-bottom cylindrical glass container  $57.8 \pm 1 \text{ mm}$  ( $2.28 \pm 0.04 \text{ in.}$ ) in inside diameter by approximately 127 mm (5 in.) in height, and having a wall thickness of approximately 2.3 mm (0.09 in.).

**17.2.3.1** 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.

**17.3 Conditioning of Latex:**

**17.3.1** The temperature of a sample of latex shall not be artificially lowered during its storage.

**17.3.2** The mechanical stability test shall be carried out within 24 h of first opening of the latex sample. Exposure to air may affect the mechanical stability of the latex.

**17.4 Procedure:**

**17.4.1** For Categories 1 and 2, dilute the latex (approximately 100 g) to exactly  $55.0 \pm 0.2 \%$  total solids with aqueous ammonia solution (1.6 %  $\text{NH}_3$ ). For Category 3, dilute the latex to exactly  $55.0 \pm 0.2 \%$  total solids with aqueous ammonia solution (0.6 %  $\text{NH}_3$ ). For Category 4 guayule latex,

dilute the latex to exactly  $43.0 \pm 0.2$  % total solids with aqueous ammonia solution (0.6 %  $\text{NH}_3$ ).

17.4.2 Without delay warm the diluted latex with gentle stirring to 36 to 37°C.

17.4.3 Immediately strain the diluted and warmed latex through a 180- $\mu\text{m}$  stainless steel sieve with  $0.180 \pm 0.009$ -mm ( $0.0070 \pm 0.0004$ -in.) opening and  $0.131 \pm 0.013$ -mm ( $0.0052 \pm 0.0005$ -in.) wire diameter and weigh  $80.0 \pm 0.5$  g of the strained latex into the test bottle. Check that the temperature of the latex is  $35 \pm 1^\circ\text{C}$ .

17.4.4 Place the test bottle in position for stirring. The position of the test bottle shall be such that the axis of the stirrer is concentric with the axis of the stirrer shaft which is concentric with the axis of the bottle, and that the bottom of the agitator disk is  $12.7 \pm 2.5$  mm ( $0.5 \pm 0.1$  in.) from the bottom inside of the bottle.

17.4.5 Stir the latex at  $14\,000 \pm 200$  rpm until the end point is reached.

NOTE 7—The approach of the end point is usually indicated by a drop of the meniscus of the latex, loss of turbulence, and change in sound of the stirring action.

17.4.5.1 The end point is determined by frequently dipping a glass rod into the latex and drawing it once lightly over the palm of the hand. The first appearance of small pieces of coagulated rubber in the film so deposited is the end of the test. This end point should be confirmed by the presence of an increased amount of coagulated rubber in a film deposited after 15 s additional agitation.

NOTE 8—The end point may be further verified by straining the latex through the 180- $\mu\text{m}$  stainless steel screen described above.

17.4.5.2 The end point in this case is the presence of approximately 1 % of coagulum based on total solids.

17.4.6 *Expression of Results*—The mechanical stability value for a latex is expressed as the number of seconds elapsed from the start of the test to the end point. Duplicate tests should check within 5 %.

## 18. Copper and Manganese

18.1 Copper and manganese shall be determined in accordance with Test Methods **D1278**.

## DENSITY TEST

### 19. Scope

19.1 Density determinations are used to calculate the mass of a measured volume of latex in locations where it is not possible to weigh directly. For such purposes it is essential that the density be determined on a latex sample containing the same amount of air as the latex contained when the volume was measured. Before sampling, latex is therefore allowed to stand for a minimum period of 24 h to ensure the removal of air bubbles. Two methods are described. The first, a direct method, is designated as the referee method. The second, an indirect method, may be desirable under certain conditions. The two methods are as follows:

19.1.1 *Method A, Direct Method: Referee Method*—In Method A the density measurement is made at the same temperature as the volume measurement or a correction is applied.

19.1.2 *Method B, Indirect Method: Alternative Method*—Method B has an advantage over Method A in that it can be executed more readily by technician. It also has the advantage of easier elimination of air bubbles. This method should not be used for referee purposes.

## 20. Definition

20.1 *Density*—The mass divided by the volume at a stated temperature. The units must be stated.

## 21. Conversion of Units

21.1 The density of latex is determined in units of megagrams per cubic metre. Conversion to density at other temperatures or to totally different units may be made as follows:

21.1.1 To convert the density determined at one temperature to the density at any other temperature, calculate as follows:

$$D = A + B - C \quad (11)$$

where:

- A = density measured at temperature of test,
- B = correction value from Table X1.1 for temperature of test and dry rubber content of latex,
- C = correction value from Table X1.1 for temperature of bulk of latex and dry rubber content of latex, and
- D = density of latex.

21.1.2 To convert the metric density units of megagrams per cubic metre to pounds mass per U.S. gallon, multiply the former by the factor 8.345.

## METHOD A—DIRECT METHOD REFEREE METHOD

### 22. Summary of Method

22.1 The density of the latex is measured in a special type specific gravity bottle, desirably, at the same temperature as that of the latex in the container when the total volume of latex was measured.

### 23. Apparatus

23.1 *Density Bottle*, 50-cm<sup>3</sup> capacity, having a ground-glass stopper perforated by a capillary and a ground-glass cap.

23.2 *Constant-Temperature Bath*, adjustable within 0.2°C to any temperature at which the volume of latex is measured in the tank car or other vessel.

23.3 *Balance*, to weigh accurately to the nearest 0.001 g.

23.4 *Conical Flasks*, two, of at least 200-cm<sup>3</sup> capacity, each fitted with a rubber stopper, a short glass inlet tube with a rubber bulb at the external end, and a glass outlet tube reaching nearly to the bottom of the flask.

### 24. Procedure

24.1 Adjust the temperature of the constant-temperature bath to the desired temperature. Stir the sample of latex gently without introducing air bubbles. Fill one of the conical flasks with a suitable amount of the latex and place in the bath. In similar manner partially fill the second conical flask with freshly boiled distilled water and place in the bath. Weigh the

clean and dry density bottle to the nearest 0.001 g and immerse up to its neck in the bath with the glass stopper in place but not the cap. Allow the density bottle and the latex and water in the two conical flasks to come to the temperature of the constant-temperature bath. This will require a minimum of 20 min. Blow a few cubic centimetres of latex from the conical flask and discard. Blow sufficient latex from the conical flask into the density bottle to fill it completely. Put the stopper in place and immediately wipe the top surface clean, taking care not to remove any latex from the capillary tube. Remove the bottle from the bath and place the ground-glass cap on immediately. Dry the outside with the minimum of handling and weigh the bottle containing latex to the nearest 0.001 g.

## 25. Calibration of Density Bottle

25.1 Empty the density bottle and wash free from latex with distilled water. Immerse the bottle again in the bath as before. Fill the bottle with distilled water by blowing from the second conical flask and allow to stand for 5 min. While still immersed in the bath, empty it and refill completely by the same procedure. Put the stopper in place and immediately wipe the top surface dry, taking care not to remove any water from the capillary tube. Remove the bottle from the bath and place the ground-glass cap on immediately. Dry the outside with the minimum of handling and weigh the bottle to the nearest 0.001 g. The results of duplicate tests should agree to within 0.001 mg/cm<sup>3</sup> or equivalent tolerance if other units are used.

## 26. Calculation

26.1 Calculate the density of the latex as follows:

$$D = (M_L \times D_w) / M_w \quad (12)$$

where:

- $D$  = density of the latex at the temperature of the constant-temperature bath, mg/cm<sup>3</sup>,
- $M_L$  = mass of latex in the density, bottle, g
- $M_w$  = mass of water in the density bottle, g, and
- $D_w$  = density of water at the bath temperature, mg/m<sup>3</sup>.

## METHOD B—INDIRECT METHOD ALTERNATIVE METHOD

## 27. Summary of Method

27.1 The density of the latex is measured at some convenient laboratory temperature by weighing a known amount of latex and a known amount of distilled water in a flask of known volume. From this measurement and from known expansivities of the latex, the density can be determined at other temperatures, for example, the temperature of the tank car or other container at the time of measuring the volume of the latex.

## 28. Apparatus

28.1 *Volumetric Flask*, 100-cm<sup>3</sup>, having a ground-glass stopper.

28.2 *Analytical Balance*, capable of weighing to the nearest 1 mg.

28.3 *Thermometer*, capable of measuring laboratory temperatures to the nearest 0.2°C.

## 29. Calibration of Volumetric Flask

29.1 Weigh a clean and dry 100-cm<sup>3</sup> volumetric flask to the nearest 1 mg. Fill the flask with distilled water at laboratory temperature to a mark placed high up on the stem of the flask just below the glass stopper. This high mark on the stem diminishes the wall area inside the stem which must be free from adhering latex in the density determination. Weigh the flask plus the water to the nearest 1 mg. Record this mass and also the temperature of the water in the flask. For this temperature,  $t$ , calculate the volume of the flask to the mark as follows:

$$V = (B_t - A) / d_t \quad (13)$$

where:

- $V$  = volume in cubic centimetre of the flask at laboratory temperature,
- $t$  = temperature of the water in the flask,
- $B_t$  = mass of the flask plus the water at temperature  $t$ ,
- $A$  = mass of the empty flask, and
- $d_t$  = density of the distilled water in mg/cm<sup>3</sup> at temperature  $t$ .

NOTE 9—Sample Calculation:

$B_t$ (25.0°C)	156.001 g
$A$	52.997
Mass of water (25.0°C)	103.004 g
Density of water (25.0°C)	0.99707 Mg/m <sup>3</sup>

$$V = 103.004 / 0.99707 = 103.307 \text{ cm}^3,$$

where  $V$  is the volume of the flask to the calibrated mark at laboratory temperature.

## 30. Procedure

30.1 Weigh the clean, dry, calibrated flask to the nearest 1 mg. Introduce latex into the flask until the flask is approximately half full. Stopper the flask and weigh again to the nearest 1 mg. Remove the stopper and add distilled water to the calibrated mark. During the addition of this water swirl the flask at times, so as to free any air bubbles which may be present in the latex. With the liquid level at the mark, stopper the flask and weigh again to the nearest 1 mg. Mix the contents well, and measure the temperature. Report the density measurement for the latex at 25°C.

## 31. Calculations

31.1 Calculate the density of the latex as follows:

$$D_t = (B - A) / [V - (C - B) / d_t] \quad (14)$$

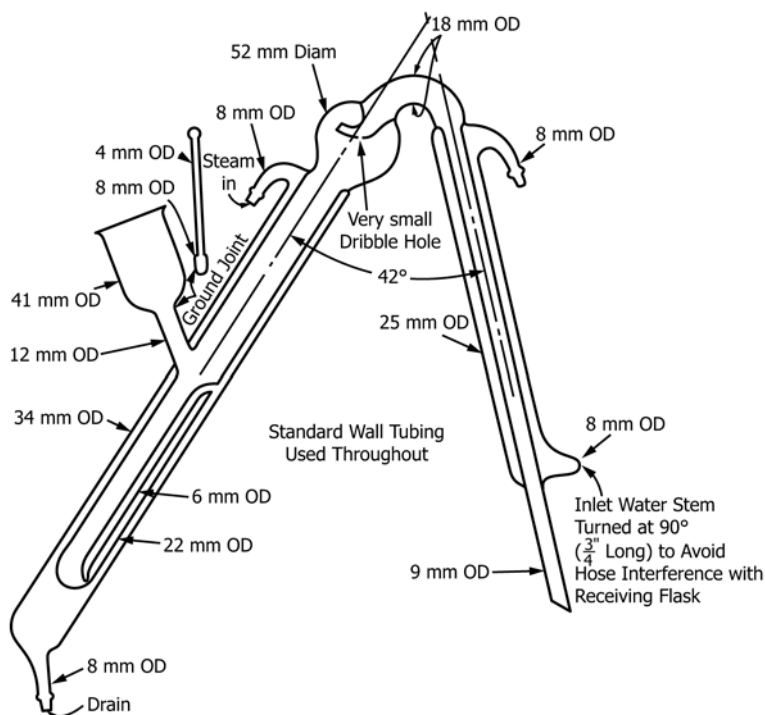
where:

- $D_t$  = density of the latex in mg/cm<sup>3</sup> at temperature  $t$ ,
- $t$  = temperature of the latex and water mixture in the volumetric flask,
- $B$  = mass of the flask plus the latex,
- $A$  = mass of the empty flask,
- $V$  = volume of the flask to the calibrated mark on the stem,
- $C$  = mass of the flask, latex, and water to the calibrated mark on the stem, and
- $d_t$  = density of the distilled water in grams per cubic centimetre at temperature  $t$ .

NOTE 10—Sample Calculation:

$B$	101.426 g
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NOTE 1—Standard-wall tubing used throughout.

FIG. 1 Markham-Type Still for Volatile Fatty Acid Test

A	<u>52.997</u>
Mass of latex	48.429 g
C	153.187 g
B	<u>101.426</u>
Mass of water	51.761 g
Temperature of mixture	23.3°C
Density of water at 23.3°C	0.99749 Mg/m <sup>3</sup>
Volume of water at 23.3°C:	
	51.761/0.99749 = 51.891 cm <sup>3</sup>
Volume of flask, V	103.307 cm <sup>3</sup>
Volume of water (23.3°C)	<u>51.891</u>
Volume of latex (23.3°C)	51.416 cm <sup>3</sup>
	$D_{23.3} = 48.429/51.416 = 0.9419 \text{ Mg/m}^3$
is the density of the latex at 23.3°C.	
Volume expansivity of latex 0.00055 Mg/m <sup>3</sup>	
	25.0 - 23.3 = 1.7°C change
	$1.7 \times 0.00055 = 0.0009 \text{ Mg/m}^3$
	$D_{25} = 0.9419 - 0.0009 = 0.9410 \text{ Mg/m}^3$

is the density of the latex at 25.0°C. Consult Section 21 for conversions of this density to other units.

## VOLATILE FATTY ACIDS

### 32. Definition

32.1 *volatile fatty acid number*—The number of grams of potassium hydroxide (KOH) required to neutralize the volatile fatty acid in a latex sample containing 100 g of total solids.

### 33. Apparatus

33.1 *Markham Semi-Micro Still or Modified Markham Semi-Micro Still*, as shown in Fig. 1.<sup>3</sup>

<sup>3</sup> The sole source of supply of the apparatus known to the committee at this time is Ace Glass Inc., 1430 Northwest Blvd., Vineland, NJ 08360. If you are aware 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,<sup>1</sup> which you may attend.

33.2 *Micro Buret*, 10-cm<sup>3</sup>.

33.3 *Steam Generator*, consisting of a 2 to 3-dm<sup>3</sup> flask, a hot plate with a temperature control, and suitable glass and rubber-tube connections. Carborundum crystals or similar material shall be used to prevent bumping.

### 34. Reagents

34.1 *Ammonium Sulfate Solution (350 kg/m<sup>3</sup>)*—Dissolve 350 g of reagent grade ammonium sulfate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) in distilled water and dilute to 1 dm<sup>3</sup>.

34.2 *Barium Hydroxide Solution (0.01 mol)*—Use reagent grade barium hydroxide (Ba(OH)<sub>2</sub>·8H<sub>2</sub>O) that has not been unduly exposed to air. Weigh out 1.58 g of the Ba(OH)<sub>2</sub>·8H<sub>2</sub>O and dissolve in 1 dm<sup>3</sup> of freshly boiled and cooled distilled water. Decant the solution after settling, or filter it, and then standardize. It should preferably be stored in a polyethylene bottle, and in any case it should be protected from absorption of carbon dioxide (CO<sub>2</sub>) from the air. The solution should be standardized frequently for changes taking place during storage.

34.3 *Bromothymol Blue Indicator (10 g/dm<sup>3</sup>)*—Dissolve 1 g of bromothymol blue in 75 cm<sup>3</sup> of ethyl alcohol (50 %). Adjust the pH to 6.5 to 7.0 with dilute sodium hydroxide (NaOH) solution. Dilute the solution to 100 cm<sup>3</sup>.

34.4 *Silicone-Type Antifoam Material*.

34.5 *Sulfuric Acid (2 + 5)*—Slowly, and with care, add 2 volumes of ACS grade H<sub>2</sub>SO<sub>4</sub> (density 1.84) to 5 volumes of distilled water and allow to come to room temperature before using.

### 35. Procedure

35.1 To  $50 \pm 0.2$  g of concentrated latex in a 250-cm<sup>3</sup> beaker, add 50 cm<sup>3</sup> of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution and stir with a glass rod. Immerse the beaker in a water bath at approximately 70°C for 3 to 5 min to coagulate the latex. Filter the serum through a low-ash, medium-texture dry filter paper into a 50-cm<sup>3</sup> Erlenmeyer flask. Squeeze the coagulum in the beaker with a glass rod to remove the remainder of the serum. Do not wash the filter.

35.2 Pipet 25 cm<sup>3</sup> of the filtered serum into a second 50-cm<sup>3</sup> flask. Pipet 5 cm<sup>3</sup> of H<sub>2</sub>SO<sub>4</sub> (2 + 5) into the flask. Stopper and swirl to mix.

35.3 Purge the still by passing steam (Note 10) through it for a period of 15 min or longer before starting a series of tests. Empty the inner chamber by siphon action as follows: Vent the steam generator, shut off the steam supply to the still and open the bottom drain. The discharge of water from the bottom drain will create negative pressure to empty the inner chamber. Flush with distilled water.

35.4 To start distillation, open the steam supply to the still and close the vent on the steam generator. Leave the bottom drain open temporarily. Pipet 10 cm<sup>3</sup> of acidified serum, along with a drop of silicone antifoam agent, into the inner chamber and replace the glass stopper. Place a 100-cm<sup>3</sup> graduated cylinder under the condenser to collect the distillate and close the bottom drain slowly, thereby directing steam through the sample in the inner chamber. Adjust the steam flow to produce distillate at a rate of 3 to 6 cm<sup>3</sup>/min, and collect 100 cm<sup>3</sup> of the distillate. Aerate the distillate with air free of CO<sub>2</sub>. Add a drop of bromothymol blue indicator and titrate rapidly with 0.01 mol Ba(OH)<sub>2</sub> solution to a blue color that persists for about 10 to 20 s before turning green.

NOTE 11—Use distilled water in the steam generator.

35.5 *Blank*—To check the reagents and the technique, a blank may be run as above by substituting 20 cm<sup>3</sup> of distilled water for the 50 g of concentrated latex.

### 36. Calculations

36.1 Calculate the volatile fatty acid number as follows:

$$\text{Volatile fatty acid number} = (AM \times 561)/(W \times TS) \quad (15)$$

where:

- A* = cubic centimetres of Ba(OH)<sub>2</sub> solution required for titration of the sample,
- M* = mole of the Ba(OH)<sub>2</sub> solution,
- W* = mass of latex corresponding to 10 cm<sup>3</sup> of acidified serum, and
- TS* = percentage of total solids in the latex.

36.2 Calculate the factor *W* as follows:

$$W (50 \times 25)/[(50 + S) \times 3] \quad (16)$$

where:

- 50 = gram of latex weighed out,
- 25 = cubic centimetres of serum used,
- 50 + *S* = cubic centimetres of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution plus the cubic centimetres of serum in 50 g of latex, and

3 = ratio 30/10, where 30 is equal to 25 cm<sup>3</sup> of filtered serum plus 5 cm<sup>3</sup> of H<sub>2</sub>SO<sub>4</sub>, and 10 is equal to the 10-cm<sup>3</sup> aliquot.

The value of *W* is dependent on the total solids and the dry rubber content of the latex, but it need be recalculated only for significant differences in these values. The following are several typical values of *W*:

	<i>TS</i>	<i>DRC</i>	<i>W</i>
Centrifuged latex	62.5	61.0	6.03
Creamed latex	68.0	66.5	6.28
Normal latex	40.0	36.0	5.12

36.3 Calculate the volume of serum, *S*, as follows:

$$S = (100 - DRC)/(1.02 \times 2) \quad (17)$$

where:

- DRC* = percentage of dry rubber content of the latex, and
- 1.02 = specific gravity of the serum.

### 37. Boric Acid

37.1 *Scope*—This method covers the testing of natural rubber latices that contain a preservative agent of boric acid and that have been subjected to some kind of concentration process.

37.2 *Summary of Method*—A quantity of latex containing approximately 0.02 g boric acid is adjusted to pH 7.5 at which boric acid exists substantially in the undissociated form. Mannitol is then added in excess to form the strongly acidic boric acid-mannitol complex. Hydrogen ions equivalent to the boric acid present in the latex are thus liberated and the pH falls. Boric acid is determined from the amount of alkali required to restore the pH of the latex to its original value, 7.5.

37.3 *Reagents*—All reagents should be of recognized analytical reagent quality. Distilled water or water of equivalent purity should be used whenever water is specified.

37.3.1 *Sodium Hydroxide (0.05 mol)*—Prepare a 0.05 mol solution of sodium hydroxide (NaOH) and standardize as follows:

37.3.1.1 *Standardization*—Pipet 5 cm<sup>3</sup> of the boric acid solution into a 250-cm<sup>3</sup> beaker; add 2 cm<sup>3</sup> of stabilizer solution and 50 cm<sup>3</sup> of water. If the pH of the solution measured electrometrically exceeds pH 5.5, add dilute hydrochloric acid dropwise with constant stirring to reduce the pH to a value between 5.5 and 2.5. Allow solution to stand for 15 min. Add the NaOH solution from a buret, with constant stirring, until the pH is 7.50. Add 4 g of mannitol with continued stirring, causing the pH to fall. Again add NaOH solution and record the volume of solution required to restore the pH to 7.50.

37.3.1.2 Calculate the mole, *M*, of the NaOH solution as follows:

$$M = 0.081 m/T \quad (18)$$

where:

- m* = mass of boric acid in 1000 cm<sup>3</sup> of boric acid solution, g, and
- T* = volume of NaOH solution required to restore the pH to 7.50, cm<sup>3</sup>.

37.3.2 *Hydrochloric Acid (2 mass %)*.

37.3.3 *Stabilizer Solution (5 mass %)*. Prepare a 5 mass % solution of a suitable nonionic stabilizer of the ethylene oxide condensate type.

37.3.4 *Mannitol*.

37.3.5 *Boric Acid Solution (5 g/dm<sup>3</sup>)*—Dissolve 5 g of boric acid (H<sub>3</sub>BO<sub>3</sub>) in water and dilute accurately to 1000 cm<sup>3</sup>.

37.4 *Procedure*—Weigh approximately 10 g of latex to the nearest 0.1 g in a 250-cm<sup>3</sup> beaker; add 2 cm<sup>3</sup> of stabilizer solution and 50 mL of water. Add dilute hydrochloric acid dropwise, with constant stirring, until the pH of the latex measured electrometrically is below 5.5 and above 2.5. Allow the solution to stand for 15 min. Adjust the pH to 7.50 by adding NaOH solution with constant stirring; add 4 g of mannitol with continued stirring, causing the pH to fall. Again, add NaOH solution and record the volume NaOH solution required to restore the pH to 7.50.

37.5 *Calculation*—Calculate the percentage (mass basis) of boric acid in the latex as follows:

$$\text{Boric acid (H}_3\text{BO}_3) = 6.18 \times M \times V/M \quad (19)$$

where:

$M$  = mole of the NaOH solution,

$V$  = volume of NaOH solution required to restore the pH of the latex to 7.50, cm<sup>3</sup>, and

$M$  = mass of the latex specimen, g.

37.5.1 A difference of 0.01 % boric acid between the results of duplicate determinations is not considered significant.

## 38. Dry Films

38.1 *Scope*—This method covers the preparation of air-free, dry, homogeneous films from concentrated *Hevea* natural rubber latex, which contains preservative agents. The procedure also is suitable for latices from natural sources other than *Hevea brasiliensis*, such as guayule.

38.2 *Apparatus*:

38.2.1 *Mold*—A suitable mold in which the film can be cast. It should be constructed by cementing rigid plastic strips 6 mm wide and 1.5 mm thick on a flat glass plate to form a cavity surface that is preferably from 125 to 150 mm square. Dry films of about 1 mm thick will result when the mold is filled with latex of 62 % total solids content or about 0.7 mm thick when the mold is filled with latex of 48 % total solids content. The plastic strips may be cemented to the glass plate with epoxide resin adhesive or polyvinyl acetate dissolved in methyl ethyl ketone.

38.2.2 *Straightedge*—A wood or stainless steel straightedge with which to scrape the surface of latex in the mold free of air bubbles.

38.2.3 *Cabinet*—A clean, dry and dust-free cabinet or covered space with a level surface on which to place the mold.

38.2.4 *Film Covers*—Thin transparent cellulosic film sheets to cover and protect the dry rubber films.

NOTE 12—Dry films of about 1 mm thick will result when the mold is filled with latex of 62 % total solids content.

38.3 *Procedure*:

38.3.1 Prepare the film without dilution if the total solids content is 62 % or less. If the total solids content is above

62 %, bring it to this value by dilution with distilled water. Mix the latex well in the sampling bottle and allow it to stand for 5 min. Carefully strain the latex through a 180- $\mu$ m stainless steel sieve with a nominal aperture of  $0.180 \pm 0.009$  mm ( $0.0070 \pm 0.0004$  in.) into a 50-cm<sup>3</sup> glass beaker. Cover the beaker to minimize surface drying and allow it to stand for 5 min before pouring the latex into the mold. Place the mold into the position in which the film will be left to dry. Immediately before pouring the latex into the mold, remove the cover from the beaker and scrape the surface of the latex free of foam with a piece of filter paper. Keeping the beaker close to the plate, carefully pour the latex into the mold in a continuous stream, disturbing the latex evenly in the mold cavity. Pour a slight excess over that required to fill the mold completely. Allow the latex to stand in the mold for 1 min, then scrape the excess latex off with a clean wood or stainless steel straightedge. Move the straightedge evenly, once only, across the mold at a speed of up to 25 mm/s.

38.3.2 Dry the cast film in a normal dust-free atmosphere. After drying at room temperature, dry the film in an oven at a temperature not exceeding 35°C. When sufficiently dry to remove the film from the mold without distortion, strip the film from the mold taking care to handle the surface of the film as little as possible. Turn the film over and place it flat on a piece of thin transparent cellulosic sheet. Allow the film to dry for at least another 24 h at a temperature not exceeding 35°C. When completely dry, cover the remaining side of the film with another piece of cellulosic sheet. Film dryness may be judged by clarity, which increases as the film becomes dry. If there is any doubt about dryness with visual examination, dry the film to constant mass at a temperature not exceeding 35°C in a dry atmosphere.

38.3.3 Store the dry film until required for testing in a cool dark place in an air-tight container or desiccator to prevent absorption of moisture.

## 39. Precision and Bias

39.1 These precision statements have been prepared in accordance with Practice D4483. Please refer to this practice for terminology and other testing and statistical concept explanations.

39.2 The precision of each test method was estimated from an interlaboratory study of three different natural rubber latices from *Hevea brasiliensis*. The number of laboratories that tested each material on two days is given in the precision statement summary along with range of each property tested.

39.3 A Category 1 precision was evaluated and the test precision for these methods is expressed by the data given in Table 2.

39.4 The precision of this test method may be expressed in the format of the following statements that use an appropriate value of  $r$ ,  $R$ , ( $r$ ), or ( $R$ ), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of  $r$  or  $R$  associated with a mean level in the precision tables closest to the mean level (under consideration at any given time, for any given material) in routine testing operations.

**TABLE 2 Test Precision—Category 1**

NOTE 1—

- $S_r$  = within laboratory standard deviation.  
 $r$  = repeatability (in measurement units).  
 $(r)$  = repeatability (in percent).  
 $S_R$  = between laboratory standard deviation.  
 $R$  = reproducibility (in measurement units).  
 $(R)$  = reproducibility (in percent).

 FOR  $(r)$  AND  $(R)$  THE PRECISION IS RELATIVE PERCENT (THAT IS, PERCENT OF A PERCENT IN THOSE CASES WHERE PERCENT IS THE MEASURED PROPERTY).

Test Method	Number of Laboratories in Property		Within Laboratories			Between Laboratories		
	Program	Range	$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$
Total solids, %:								
Dried 16 h at 70 ± 2°C	13	59.1–68.3	0.11	0.31	0.49	0.16	0.45	0.71
Dried 2 h at 100 ± 2°C	13	60.0–68.3	0.09	0.26	0.41	0.18	0.51	0.80
Dry rubber content, %	11	58.0–66.8	0.35	0.99	1.59	1.23	3.48	5.58
Total alkalinity, %								
Recommended procedure	12	0.137–0.780	0.011	0.030	6.63	0.032	0.090	19.72
Alternate procedure	12	0.172–0.800	0.010	0.030	6.09	0.024	0.068	13.99
Sludge content, %	8	0.0008–0.0785	0.0060	0.0169	43.33	0.0148	0.0419	105.54
Coagulum content, %	10	0.002–0.076	0.005	0.014	35.90	0.014	0.040	102.56
KOH number	12	0.49–0.79	0.01	0.03	4.69	0.03	0.08	12.50
Mechanical stability, s	8	1020–2650	37	105	5.7	177	501	27.3

39.5 *Repeatability*—The repeatability  $r$ , of this test method has been established as the *appropriate value* tabulated in the precision tables. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated  $r$  (for any given level) must be considered as derived from different or non-identical sample populations.

39.6 *Reproducibility*—The reproducibility  $R$ , of this test method has been established as the *appropriate value* tabulated in the precision tables. 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 level) must be considered to have come from different or non-identical sample populations.

39.7 Repeatability and reproducibility expressed as a percentage 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 percentage of the arithmetic mean of the two test results.

39.8 *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 this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

## APPENDIX

### (Nonmandatory Information)

#### X1. Table X1.1

**TABLE X1.1 Density Conversion Values for Natural Rubber Latex from *Hevea brasiliensis***

 NOTE 1—The table is based on a maximum density of water of 999.972 Kg/m<sup>3</sup> and taken from *Handbook of Chemistry and Physics*, 65th Edition 1984, CRC Press, Inc., Boca Raton, FL, USA.

Temperature, °C	Density of water, Mg/m <sup>3</sup>	Correction for Dry Rubber Content of, %									
		20	25	30	35	40	45	50	55	60	65
0	0.99984	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
2	0.99994	0.0002	0.0003	0.0004	0.0004	0.0005	0.0006	0.0006	0.0007	0.0008	0.0008
4	0.99998	0.0005	0.0006	0.0008	0.0009	0.0010	0.0012	0.0013	0.0014	0.0016	0.0017
6	0.99994	0.0008	0.0010	0.0012	0.0014	0.0016	0.0018	0.0020	0.0022	0.0024	0.0026
8	0.99985	0.0011	0.0014	0.0017	0.0019	0.0022	0.0025	0.0027	0.0030	0.0032	0.0035
10	0.99970	0.0015	0.0019	0.0022	0.0025	0.0028	0.0032	0.0035	0.0038	0.0041	0.0044
12	0.99950	0.0020	0.0024	0.0028	0.0031	0.0035	0.0039	0.0042	0.0046	0.0049	0.0053
14	0.99924	0.0025	0.0029	0.0034	0.0038	0.0042	0.0046	0.0050	0.0054	0.0058	0.0062
16	0.99894	0.0030	0.0035	0.0040	0.0045	0.0049	0.0054	0.0059	0.0063	0.0068	0.0072
18	0.99860	0.0035	0.0041	0.0046	0.0052	0.0057	0.0062	0.0067	0.0072	0.0077	0.0082



**TABLE X1.1** *Continued*

Temperature, ° C	Density of water, Mg/m <sup>3</sup>	Correction for Dry Rubber Content of, %									
		20	25	30	35	40	45	50	55	60	65
20	0.99820	0.0041	0.0047	0.0053	0.0059	0.0065	0.0070	0.0076	0.0081	0.0086	0.0092
22	0.99777	0.0048	0.0054	0.0060	0.0066	0.0073	0.0079	0.0085	0.0090	0.0096	0.0102
24	0.99730	0.0054	0.0061	0.0068	0.0074	0.0081	0.0087	0.0094	0.0100	0.0106	0.0112
26	0.99678	0.0061	0.0068	0.0075	0.0082	0.0089	0.0096	0.0103	0.0109	0.0116	0.0122
28	0.99623	0.0068	0.0076	0.0083	0.0090	0.0098	0.0105	0.0112	0.0119	0.0126	0.0133
30	0.99565	0.0076	0.0084	0.0091	0.0099	0.0107	0.0114	0.0122	0.0129	0.0136	0.0143
32	0.99503	0.0083	0.0092	0.0100	0.0108	0.0116	0.0124	0.0131	0.0139	0.0146	0.0154
34	0.99437	0.0091	0.0100	0.0108	0.0117	0.0125	0.0133	0.0141	0.0149	0.0157	0.0164
36	0.99368	0.0099	0.0108	0.0117	0.0126	0.0134	0.0143	0.0151	0.0159	0.0167	0.0175
38	0.99297	0.0108	0.0117	0.0126	0.0135	0.0144	0.0153	0.0161	0.0170	0.0178	0.0186
40	0.99222	0.0117	0.0126	0.0135	0.0145	0.0154	0.0163	0.0172	0.0181	0.0189	0.0197

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