



Designation: D 1076 – 02

Standard Specification for Rubber—Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex¹

This standard is issued under the fixed designation D 1076; 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 This specification covers requirements for first grade concentrated natural rubber latex (see Table 1) of the following types:

Type 1—Centrifuged natural latex preserved with ammonia only or by formaldehyde followed by ammonia.

Type 2—Creamed natural latex preserved with ammonia only or by formaldehyde followed by ammonia.

Type 3—Centrifuged natural latex preserved with low ammonia with other necessary preservatives.

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 types of latices. It does not apply to natural latex sources other than *Hevea brasiliensis* nor 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:

¹ 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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TABLE 1 Requirements for Specified Latex Types

	Type 1	Type 2	Type 3
Total solids, min, %	61.3	66.0	61.3
Dry rubber content (DRC), ^A min, %	59.8	64.0	59.8
Total solids content minus dry rubber content, max, %	2.0	2.0	2.0
Total alkalinity calculated as ammonia, as % latex	0.60 min	0.55 min	0.29 max
Sludge content, max, %	0.10	0.10	0.10
Coagulum content, max, %	0.050	0.050	0.050
KOH number, max ^B	0.80	0.80	0.80
Mechanical stability, s, min	650	650	650
Copper content, max, % of total solids	0.0008	0.0008	0.0008
Manganese content, max, % of total solids	0.0008	0.0008	0.0008
Color on visual inspection	no pronounced blue or gray ^C		
Odor after neutralization with boric acid	no putrefactive odor		

^ADry rubber content by definition and use is the acid coagulable portion of latex after washing and drying.

^BIt 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.

^CBlue or gray color usually denotes iron contamination caused by improper storage in containers.

D 1278 Test Methods for Rubber from Natural Sources—Chemical Analysis²

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

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode³

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 3 types of latex as defined in the scope and defines the test methods to use for

² Annual Book of ASTM Standards, Vol 09.01.

³ Annual Book of ASTM Standards, Vol 15.05.



the specified properties. These test methods may be used for production control or for referee purposes.

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:

7.1.1 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:

7.2.1 Separate samples of at least 475 cm³ 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 cm³ 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 ± 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 cm² (5 in.²). This may be facilitated by carefully adding approximately 1 cm³ 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/m³).

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 cm³ should be sufficient).

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 $70 \pm 2^\circ\text{C}$ in a vented air oven atmosphere. If oxidation occurs, the test may be run with the option of using a drying temperature of $55 \pm 2^\circ\text{C}$, 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 $55 \pm 2^\circ\text{C}$. 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:

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

10. Total Alkalinity

10.1 *Apparatus*—Glass electrode pH meter.

10.2 *Reagent*—0.1 mol standard HCl.





10.3 Preparation of Specimen—Place approximately 5 g of latex into a glass weighing bottle of approximately 10-cm³ capacity, having a ground glass cap, and weigh to the nearest 1 mg. Pour the specimen into a beaker containing approximately 300 cm³ 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.

10.4 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 E 70, 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-cm³ increments, and a pH reading taken 10 s after each addition. As the pH of 6.0 is approached, smaller increments should be added.

10.5 Alternative Procedure—Prepare the sample as described in 10.3. 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.

10.6 Calculation—Calculate (Note 1) the total alkalinity, reported as NH₃ based on grams of NH₃ 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, cm³, and
 W = original mass of the latex, g.

NOTE 1—This calculation applies to both procedures.

NOTE 2—If total alkalinity is wanted based on the water phase of the latex, calculate as follows:

$$\text{Total alkalinity, as \% of water} = (1.7 \times M \times n)/W(1 - TS/100) \quad (4)$$

where:

TS = percent total solids,
 M = mole of the standard HCl,
 n = volume of standard HCl required, cm³, and
 W = original mass of the latex, g.

11. Viscosity

11.1 Apparatus:

11.1.1 Viscometer⁴—The apparatus shall consist of a synchronous induction-type motor capable of driving at constant

rotational speeds of 0.63 and 6.3 rad/s (6 and 60 rpm) a shaft to which spindles of different shapes and dimensions may be attached, a gear train to control speed of rotation of the spindles and a beryllium copper spring. The spindle, when rotating, is driven through the beryllium copper spring which winds up when a drag is exerted on it. The amount of drag is indicated by a red pointer on the viscometer dial. This reading is proportional to the viscosity for any given speed and spindle.⁴

NOTE 3—In Newtonian liquids, the viscometer will give the same viscosity value regardless of speed and spindle, if within the range of these, when the proper factor for each spindle and speed combination is used. However, in the case of latex, which is not a Newtonian fluid, the viscosity value computed from the factor will be different for each speed. By using different speeds one can assess the non-Newtonian characteristics of the latex.

11.1.2 Calibration—Calibration can readily be made by using fresh oils from the National Bureau of Standards. The temperature of the oil shall be read with a certified thermometer to within $\pm 0.02^\circ\text{C}$. Silicone oils may be used as simple reference fluids. Although not provided to exact values, their stability and relative insensitivity to temperature make them ideal for this purpose.

11.2 Preparation of Specimen—Strain the specimen through a standard 180- μm 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. The specimen shall be conditioned to the desired test temperature of $25 \pm 2^\circ\text{C}$ in a suitable bath for a period of 2 h in order to eliminate air from the latex.

11.3 Procedure—Pour a specimen of latex prepared as above slowly down the side of a 600-cm³ beaker, previously cooled to 25°C , preventing incorporation of air. Remove guard. This is done to reduce later cleaning problems. Attach the longest spindle, denoted as No. 1. This is best done by lifting the shaft slightly while holding it firmly in one hand and screwing the spindle on with the other hand. Remember that this is a left-hand thread. In order to preserve its alignment, do not apply side thrust on the shaft. Tilt the instrument slightly, while immersing the spindle, to avoid trapping air bubbles on the disk surfaces. 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. Do not hit the spindle against the side of the beaker while it is attached to the viscometer, as this can damage the shaft alignment. Level the viscometer so the spindle is plumb. Set the speed control at the desired speed. Depress the clutch and turn on the viscometer motor. (This procedure will reduce wear and tear on the equipment.) Release the clutch and allow the dial to rotate until the pointer stabilizes at a fixed position on the dial. This will take only a few seconds. Depress the clutch and shut off the motor. If the pointer is not in view, keep the clutch depressed and start and stop the motor until it can be seen. With a little practice, this can be done with the first stopping of the motor. Take a reading on the 100 scale at 0.63 and 6.3 rad/s (6 and 60 rpm) using spindle No. 1. If the viscosity is greater than the limit of spindle No. 1, then substitute spindle No. 2, noting in the report that spindle No. 2 was used.

⁴ The sole source of supply of the Brookfield Viscometers, Models LVF and LVT 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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.



11.4 *Calculation*—Multiply the reading on the 100 scale as follows:

No. 1 spindle, 0.63 rad/s (6 rpm)	10
No. 1 spindle, 6.3 rad/s (60 rpm)	1
No. 2 spindle, 0.63 rad/s (6 rpm)	50
No. 2 spindle, 6.3 rad/s (60 rpm)	5

In general, the difference in spindles will not affect the results. Record the viscosity in millipascals-second (mPa·s) which is equivalent to centipoises. In reporting the viscosity record the spindle number and speed.

12. Sludge Content

12.1 *Apparatus*—Centrifuge operating at 12 000 m/s² with two 50-cm³ pear-shaped centrifuge tubes held in a centrifuge accessory.

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

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

12.3 *Procedure*—Weigh 45 to 50 g of the latex into each of two 50-cm³ 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-cm³ 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.

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

$$\text{Sludge content, \%} = (\text{mass of dried residue/mass of sample}) \times 100 \quad (5)$$

13. Coagulum Content

13.1 *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).

13.2 *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.

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

$$\text{Coagulum content, \%} = (m_1/m_0) \times 100 \quad (6)$$

where:

m_0 = mass of test portion, g, and

m_1 = mass of coagulum, g.

14. KOH Number

14.1 *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.

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

14.2.1 *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.

14.2.2 *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-cm³ portion into a short 25-cm³ graduate, add 10 % BaCl₂ solution until no further precipitation takes place upon allowing the BaCO₃ to settle, and then add one more drop of BaCl₂ solution to the clear supernatant liquid. Several 10-cm³ portions of the 40 % KOH solution may be titrated with the 10 % BaCl₂ solution as needed to determine the exact equivalent amount of BaCl₂. From these results the proper amount of BaCl₂ 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-cm³ sample of the BaCl₂-treated KOH solution with 10 cm³ of the distilled water and divide it into two portions. Add one drop of 10 % BaCl₂ solution to one portion and one drop of 10 % K₂SO₄ solution to the other. A precipitate in the first case shows incomplete precipitation of carbonate. A precipitate with the K₂SO₄ 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 % K₂SO₄ solution dropwise, until one drop gives no further precipitation. Any excess of carbonate should likewise be removed by dropwise addition of BaCl₂ 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.