Designation: D1076 – 21

Standard Specification for Rubber—Concentrated, Ammonia Stabilized, Creamed, and Centrifuged Natural Latex[1]

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 (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 categories:

Category 1—Centrifuged *Hevea* natural latex stabilized with ammonia only.

Category 2—Creamed *Hevea* natural latex stabilized with ammonia only.

Category 3—Centrifuged *Hevea* natural latex stabilized with low ammonia.

Category 4—Centrifuged, or centrifuged and creamed, guayule latex, or other natural rubber latex, containing less than 200 µg total protein per gram dry weight of latex, with ammonia or other hydroxide.

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

1.2 This specification is not necessarily applicable to latices prepared, stabilized, 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.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 Immunological Measurement of Antigenic Protein in Hevea Natural Rubber (HNR) 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.

[1] This specification is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.22 on Natural Rubber.

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

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),[A] 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)					
Total protein by D5712				200 max	
Hevea antigenic protein by D6499				None detectable	
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[B]	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[C]			
Odor after neutralization with boric acid		no putrefactive odor			

[A] Dry rubber content by definition and use is the acid coagulable portion of latex after washing and drying.
[B] 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.
[C] Blue or gray color usually denotes iron contamination caused by improper storage in containers.

4. Significance and Use

4.1 This specification denotes limits on the 5 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.

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/Tank Trucks*—Samples shall be taken from the top and bottom of the car/truck. 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/Tank Trucks*—Separate samples of at least 475 cm^3 each shall be taken from the top, center, and bottom of the tank car/tank truck. 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^3 of this composite sample shall be used for test. One composite sample only is required from each tank car/tank truck.

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 cm^2 (5 $in.^2$). This may be facilitated by carefully adding approximately 1 cm^3 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 ± 2°C or 2 h at 100 ± 2°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^3).

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^3 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:

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

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^3 capacity, having a ground glass cap, and weigh to the nearest 1 mg. Pour the specimen into a beaker containing approximately 300 cm^3 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^3 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_3 based on grams of NH_3 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,