SPECIFICATION

IS : 3708 (Part I) - 1966

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER LATEX

PART I DRY RUBBER CONTENT, TOTAL SOLIDS, COAGULUM CONTENT, VISCOSITY, SLUDGE CONTENT, DENSITY, TOTAL ALKALINITY, KOH-NUMBER, MECHANICAL STABILITY, VOLATILE FATTY ACID NUMBER, *p*H, TOTAL NITROGEN, TOTAL COPPER, TOTAL IRON, TOTAL MANGANESE & TOTAL ASH

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 February 1966, after the draft finalized by the Rubber Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard covers different test methods including the preparation of laboratory test samples of natural rubber latex of industrial importance, *Hevea brasiliensis* latex.

0.3 Tests for total solids, dry rubber, and coagulum content of latex, though simple in nature, are important as they measure quantities on which the purchase of latex may be made. Volatile fatty acid (VFA) and KOH-numbers are to a large extent indicative of the state of preservation of latex. VFA measurement, to a large extent, has replaced the electrical conductivity measurement of latex as a quick measure of preservation. Mechanical stability is one of the vital tests for latex contents. It is an emperical test and measures an effect rather than a cause and is useful in providing an index of latex quality with respect to its average processing behaviour. The estimation of copper and manganese is of importance in avoiding danger of active contamination leading to degradation in the final rubber. Discolouration is usually indicative of iron contamination which has reacted with the high sulphur content of the latex protein. Nitrogen measurement gives an indication as to the amount of latex protein and other nitrogenous compounds. Other determinations like ammonia content, *p*H and alkalinity are with subtle significance nevertheless in vogue for their indirect influence on other important determinations in testing of latex. Viscosity measurement has a greater significance to synthetic rubber latex than natural rubber latex, for it reflects average particle size of the latex.

0.4 In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries. This has been met by basing this standard on the following documents:

ISO/R 35-1957 Determination of the mechanical stability of latex. International Organization for Standardization.

ISO/R 123-1959 Sampling of latex. International Organization for Standardization.

ISO/R 124-1959 Determination of total solids of latex. International Organization for Standardization.

ISO/R 125-1959 Determination of alkalinity of latex. International Organization for Standardization.

ISO/R 126-1959 Determination of dry rubber content of latex. International Organization for Standardization.

ISO/R 127-1959 Determination of KOH-number of latex. International Organization for Standardization.

ISO/DR 616 Determination of nitrogen in latex. International Organization for Standardization.

ISO/DR 618 Method for the determination of density of latex. International Organization for Standardization.

ISO/DR 620 Determination of coagulum content of latex. International Organization for Standardization.

ISO/DR 621 Determination of *p*H of latex. International Organization for Standardization.

ISO/DR 626 Determination of volatile fatty acid number of latex. International Organization for Standardization.

ASTM D 1076-1964 Specification and tests for concentrated ammonia preserved, creamed and centrifuged natural rubber latex. American Society for Testing and Materials.

0.5 At the time of finalizing this draft the Committee felt the need for the inclusion of certain additional tests, such as determination of boric acid and magnesium in latex. These additional tests will be published at a later stage as Part II of this standard.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of test for natural rubber latex of industrial importance, namely, *Hevea brasiliensis* latex. It covers definition of terms and test procedures for determining dry rubber content (NRL : 1), total solids (NRL : 2), coagulum content (NRL : 3), viscosity (NRL : 4), sludge content (NRL : 5), density (NRL : 6), total alkalinity (NRL : 7), KOH-number (NRL : 8), mechanical stability (NRL : 9), volatile fatty acid number (NRL : 10), *p*H (NRL : 11), total nitrogen (NRL : 12), total copper (NRL : 13), total iron (NRL : 14), total manganese (NRL : 15), and total ash (NRL : 16).

2. TERMINOLOGY

2.0 For the purpose of this test, the following definitions shall apply.

2.1 KOH-Number — Number of grams of KOH required to neutralize the acids present in 100 g of the total solids in latex.

2.2 Latex — Colloidal aqueous dispersion of elastomer.

3. METHOD FOR TAKING OUT TEST SAMPLES

3.0 Rubber latex tends to cream on standing. Once stratification has occured, the latex should be thoroughly agitated to obtain a homogenous blend suitable for the withdrawl of sample for test. The procedure differs with the type of container and facilities available.

3.1 Apparatus — motor-driven stirrer.

3.1.1 A suitable type of stirrer for use in drum supplies consists of a collapsible two-bladed stainless steel propeller of 11 cm minimum diameter when fully opened, mounted on a stainless steel shaft sufficiently long for the propeller to be distant about one quarter the height of the latex from the bottom of the drum. The stirrer is operated at a minimum speed of 100 rpm if desired, two propellers may be used on the same shaft, in which case the top one should meet the foregoing requirements as regards position. The shaft speed should give a brisk turnover without creating a vortex. The part of the equipment immersed in the latex should not contain copper.

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*Rules for rounding off numerical values (*revised*).

3.2 Procedure

3.2.1 *Drum Supplies* — The material shall be blended by either 'Method A' or 'Method B'.

a) *Method A* — If the drum is fitted with a bung and contains at least two percent airspace, lay on its side and roll to and fro briskly for not less than 10 minutes. Then, turn the drum so as to stand on the end which was originally uppermost and allow to stand for about 15 minutes and repeat the rolling operation for further ten minutes.

If the drum contains less than two percent air space, transfer the content to a larger vessel and thoroughly stir, preferably by means of a perforated steel disk plunger (normally about ten minutes will suffice).

If the drum is of the open head type, remove the end head and stir the contents thoroughly, preferably by means of a perforated steel disk plunger (normally about ten minutes will suffice).

b) *Method B* — Agitate the contents by means of a suitable motor-driven stirrer so long as is necessary to disperse the cream. Avoid excessive stirring and unnecessary exposure of the latex to air.

3.2.1.1 Take out the sample without delay. Insert a clean, dry, glass tube of 10 to 15 mm internal diameter whose both ends are open, until it reaches the bottom of the container. Close the upper end of the tube and transfer the contents to a clean dry sample bottle. Repeat the operation until sufficient latex is obtained for testing.

Note — Alternatively, a specially constructed metal sampling tube may be used, the bottom of which may be closed by remote control. No copper or brass shall be used in any part of its construction. A suitable type of sampling device is shown in Fig. 1.

3.2.2 *Tank Supplies* — Take three samples of similar volume, one at the centre of the tank, another half way between the centre and the bottom, and the third half way between the centre and the top. Take the top sample first, then the centre 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.

Note — If there is reason to suspect that stratification has occured, separate samples, 75 mm from the top surface and 75 mm from the bottom of the tank, are taken. If results from the top and bottom samples do not agree within one percent total solids, the contents are thoroughly agitated until samples obtained do agree within this tolerance. After this has been accomplished, the sampling is carried out as described in **3.2.2**.

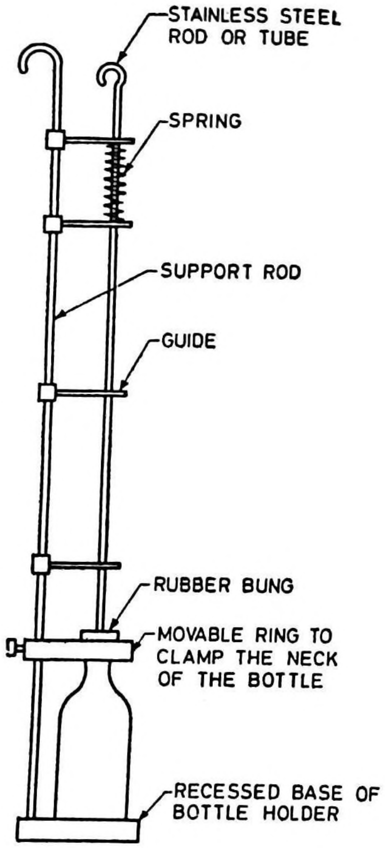

FIG. 1 SAMPLING DEVICE WITH REMOTE CONTROL

3.2.3 *Bulk Supplies* — Where samples are to be drawn from several containers or where samples are to be taken from different depths of tanks, bulk the samples and thoroughly blend by stirring or shaking immediately before taking the final sample.

4. QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

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*Specification for water, distilled quality (*revised*). (Since revised).

DRY RUBBER CONTENT

(NRL : 1)

1. SCOPE

1.1 This method is intended for the determination of dry rubber content of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 The total solid content of the latex is approximately adjusted to 20 percent and then it is coagulated with a suitable acid. The coagulum is dried at constant temperature and weighed.

3. REAGENTS

3.1 Acetic Acid — two percent solution, (*w/v*) (*see* IS : 695-1955[*]).

3.2 Rectified Spirit — (*See* IS : 323-1959[†].)

4. PROCEDURE

4.1 Duplicate determination shall be done on each sample.

4.2 Weigh about 10 g of the well-mixed sample accurately to 1 mg in a suitable container such as a beaker. Add water until the total solids content of the latex is about 20 percent. Add up to 8 ml of acetic acid per gram of sample, gently stirring over a period of 5 min. Keep the solution undisturbed for about 15 min at a temperature a little below boiling. If the serum remains milky, add about 0·5 ml of rectified spirit per gram of the sample and gently stir. When the serum is clear, collect any small particles of coagulum by rubbing with the main bulk. Wash the coagulum with running water or with at least six changes of water until neutral to litmus, and reduce its thickness to less than 2 mm either by hand roller or mechanical rollers. Dry the coagulum at a temperature of approximately 70 ± 2°C. Cool in a desiccator and weigh. Repeat drying operation until the loss in weight is less than 1 mg.

NOTE — Freshly preserved latex may be coagulated with 0·5 percent acetic acid by adding up to 15 ml of acid per gram of sample.

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[*] Specification for acetic acid, glacial, pure, pharmaceutical and technical. (**Since revised**).

[†] Specification for rectified spirit (*revised*).

5. CALCULATION

5.1 Calculate the dry rubber content (*DRC*) of the sample as follows:

$$\text{Dry rubber content, percent by weight} = \frac{W_1}{W_2} \times 100$$

where

W_1 = weight in g of dry coagulum, and

W_2 = weight in g of the sample taken for the test.

Note — A difference of 0·2 percent between the results of duplicate determinations on the sample is not considered significant.

TOTAL SOLIDS
(NRL : 2)

1. SCOPE

1.1 This method is intended for the determination of total solids content of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 A known portion is dried in air at 70 to 100°C and weighed.

3. APPARATUS

3.1 Air Oven — a vented air oven, to maintain uniform temperature, *in the range of 70 to 125°C.*

3.2 Container for Weighing — A light, substantially horizontal, flat bottomed open vessel of about 60 mm diameter and provided with a suitable cover for use when weighing. This shall be made of non-volatile inert material.

4. PROCEDURE

4.1 Carry out determinations in duplicate on each sample.

4.2 Weigh approximately 2·0 g of the well mixed sample (to obtain about 1·0 g total solids) accurately to 1 mg into the vessel. Swirl the vessel gently to ensure that the latex covers the bottom of the dish. Dry the test portion in the vented air oven at a temperature of 70 to 100°C. Cool in a desiccator and weigh as quickly as possible. Repeat the drying operation until the loss in weight is less than 1 mg during a period of 30 minutes for temperature between 70°C and 85°C, or during a period of 15 minutes for temperatures between 85°C and 100°C.

NOTE — Few drops of *iso*-propyl alcohol may be used to facilitate drying.

5. CALCULATION

5.1 Calculate total solids (*TS*) of the sample as follows:

$$TS\text{, percent by weight} = \frac{W_1}{W_2} \times 100$$

where

W_1 = weight in g of dried film, and
W_2 = weight in g of the sample taken for the test.

NOTE — A difference of 0·2 between the results of duplicate determinations on the same sample is not considered significant.

COAGULUM CONTENT
(NRL : 3)

1. SCOPE

1.1 This method is intended for the determination of coagulum content of natural rubber latex of *Hevea brasiliensis,* uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. TERMINOLOGY

2.0 For the purpose of this test, the following definition shall apply.

2.1 Coagulum — The material retained on a test sieve with apertures 0·18 ± 0·02 mm under the conditions of the test. It comprises pieces of rubber coagulum, latex skin and coarse foreign matter.

3. OUTLINE OF THE METHOD

3.1 A known quantity of latex is diluted with a known volume of alkaline soap solution filtered through a mesh screen, and the coagulum retained on the sieve is washed dried and weighed.

4. APPARATUS

4.1 Stainless Steel Sieve — 180 micron IS Sieve (*see* IS : 460-1962*).

NOTE — The aperture of BS Sieve 85 and ASTM Sieve 80 (also known as 177 μ US Standard Sieve) are within the limits laid down for the specified IS test sieve and may, therefore be used as 125-micron IS Sieve.

5. REAGENTS

5.1 Nitric Acid — sp gr 1·42 (*see* IS : 264-1950†).

5.2 Soap Solution — 50 g of potassium oleate in one litre of water.

6. PROCEDURE

6.1 Clean the gauze by immersion for two minutes in boiling nitric acid. Wash the gauze with water and dry in an oven at 98 to 100°C for 30 minutes and weigh. Repeat the drying procedure for periods of 15 minutes until the change in weight is less than 1 mg. Weigh 200 g of the well stirred sample into a 600-ml beaker with spout. Add 200 ml of soap solution and thoroughly mix. Filter the mixture through the clean, dry gauze which has been previously weighed, and firmly clamped

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*Specification for test sieves (*revised*).

†Specification for nitric acid. (Since revised).

between the rings. Wash the residue on the gauze with soap solution until it is free from latex and then with water until the washings are neutral to litmus. Carefully remove the gauze containing the wet solid matter and swab the bottom side with filter paper. Dry at 98 to 100°C until the change in weight is less than 1 mg during 15 minutes drying.

7. CALCULATION

7.1 Subtract the original weight of gauze to obtain the weight of coagulum. Report the result as a percentage of coagulum content on the total solids content of the latex or as a percentage of the weight of latex, calculated as follows:

$$\text{Coagulum content, (expressed as percent of total solids)} = \frac{W_1 \times 10\,000}{W_2 \times TS}$$

or

$$\text{Coagulum content, (expressed as percent of weight of latex)} = \frac{W_1 \times 100}{W_2}$$

where

W_1 = weight in g of coagulum and foreign matter,
W_2 = weight in g of latex sample, and
TS = percent of total solids.

7.2 The report shall state whether the result is quoted as a percentage of total solids (*TS*) content or of latex weight.

7.2.1 In either case, the total solids (*TS*) content of the latex shall be given.

NOTE — Some latices will not tolerate potassium oleate solution without coagulating, for example, cationic latices. Such latices are diluted and washed with water. If properties of the latex are not known, check a sample for dilutability with soap solution and with water.

VISCOSITY

(NRL : 4)

1. SCOPE

1.1 This method is intended for the determination of viscosity of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 This method consists of measuring the rate of efflux of the liquid to be tested through a capillary tube of known length and known radius under two known pressure heads. The limiting coefficient of viscosity is calculated from these data and is expressed in centipoises at 27°C.

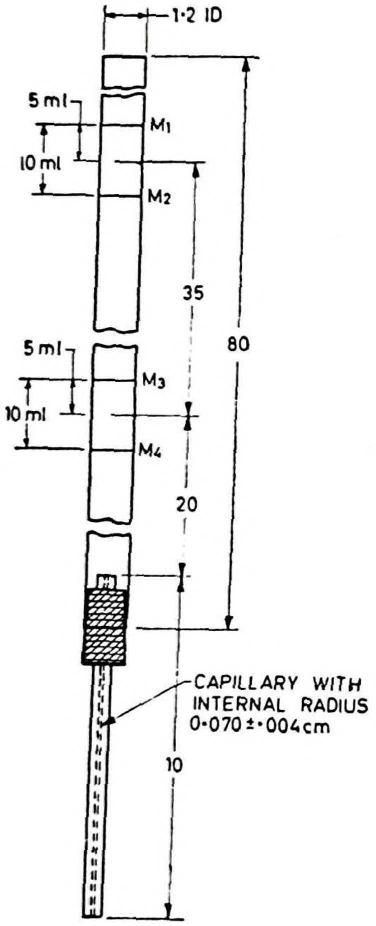
3. APPARATUS

3.1 The apparatus, shown in Fig. 1, consists of two parts, a glass tube 80 cm in length and 1·2 cm in inside diameter, and a capillary glass tube 10 cm in length with an internal radius of 0·070 ± 0·004 cm. The marks M_1, M_2, M_3 and M_4, on the large glass tube, as shown in Fig. 1, shall be so placed that the volume included between M_1 and M_2 is 10 ± 0·05 ml, and an equal volume between M_3 and M_4. The mid-points of the intervals $M_1 M_2$ and $M_3 M_4$ shall be separated by a distance of 35 cm. The capillary tube shall be inserted in the lower end of the 80-cm cylinder by means of a one-hole stopper. It shall be adjusted so that the upper end of the capillary is at a distance of 20 cm below the mid-point of section $M_3 M_4$ and 55 cm below the mid-point of section $M_1 M_2$.

4. CALIBRATION OF CAPILLARY

4.1 Clean the capillary with chromic acid solution, wash with water, and finally dry. Clamp the capillary in a nearly horizontal position and slowly pipette clean mercury into the upper end until it just flows out at the lower end. Scrape off the protruding meniscus by means of a spatula, and then hold the spatula tightly against the capillary, and withdraw the pipette so that the capillary is completely filled with mercury. Likewise scrape off the meniscus protruding from the upper end. Empty the mercury contained in the capillary into a weighing bottle and weigh. Duplicate determinations should agree within 0·1 percent. Determine the length of the capillary by means of callipers or travelling microscope to the nearest 0·1 cm. Calculate the radius of the capillary as follows:

$$R = \sqrt{\frac{W}{\pi L D}}$$



All dimensions in centimetres.

FIG. 1 VISCOSITY TEST APPARATUS

where

R = radius of capillary in centimetres,

W = weight of mercury contained when the capillary is full,

L = length of capillary in centimetres, and

D = density of mercury at temperature of measurement.

NOTE — It is of great importance that the capillary be of uniform bore throughout its length. This may be checked as follows, with the aid of a microscope having a travelling stage:

> Place enough mercury in the clean capillary to fill it about half full. Measure the length of the mercury column in a random position in the tube by means of the microscope. Then tilt the tube so as to shift the position of the column of mercury to another section of the tube, taking care not to lose any mercury, and again measure its length. Repeat this several times. The length of the thread of mercury should not vary from place to place by more than 0·1 percent.

5. PREPARATION OF SAMPLE

5.1 All viscosity measurements shall be made at 60 ± 0·19 percent total solids. After the adjustment of the total solids, strain the sample through a stainless steel screen having 32 meshes to the cm. Allow it to stand in a closed glass vessel for a period of not less than 12 h in order to permit air elimination. In case, partial creaming takes place during the standing period, gently swirl the container to stir in the cream. Care shall be taken that no bubbles are formed.

6. PROCEDURE

6.1 Assemble the glass tubes as shown in Fig. 1. Hold one finger over the lower end of the capillary and fill the tube with latex to a point about 5 cm above M_1. Measure the temperature of the latex to the nearest 0·5°C just prior to making the viscosity determination. Place a beaker under the lower end of the capillary and allow the latex to run out through the capillary. By means of a stop watch, determine to the nearest tenth of a second the time required for the meniscus to pass through the interval in the 80-cm tube bounded by marks M_1 and M_2. Similarly determine the time for the meniscus to pass from M_3 to M_4. If difficulty is experienced in seeing the meniscus, a small flashlight placed behind the tube will be found helpful. During the passage of the meniscus from M_3 to M_4, efflux from the capillary may take place dropwise. This is undesirable and may be prevented by bringing the lower end of the capillary nearly into contact with the latex in the receiving beaker.

7. CALCULATION

7.1 Calculate the limiting coefficient of viscosity in centipoises at 25°C as follows:

$$n' = n'_T [\, 1 - 0{\cdot}02 (25 - T) \,]$$

where

n' = limiting coefficient of viscosity in centipoises at 25°C;

n'_T = limiting coefficient of viscosity in centipoises at temperature, T; and

T = temperature of the latex in °C, at the time of measurement.

n'_T in turn is calculated as follows:

$$n'_T = K_1 \frac{t_1 t_2}{t_2 - t_1}$$

where

$$K_1 = \frac{\pi R^4 g\, d (h_1 - h_2) \times 100}{8\, LV}$$

t_1 and t_2 = time in seconds for meniscus to pass through intervals M_1M_2 and M_3M_4, respectively;

R = radius of capillary in centimetres;

g = acceleration due to gravity in centimetres per second;

d = density of latex in grams per millilitre;

h_1 and h_2 = heights in centimetres of mid-points of intervals M_1M_2 and M_3M_4, above bottom capillary, respectively;

L = length of capillary in centimetres; and

V = volume in millilitres of each of the two intervals M_1M_2 and M_3M_4.

SLUDGE CONTENT
(NRL : 5)

1. SCOPE

1.1 This method is intended for the determination of sludge content of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 A known quantity of latex is centrifuged, and the residue is dried and weighed.

3. APPARATUS

3.1 Centrifuge — operated at 2 300 rev/min.

4. REAGENTS

4.1 Ammonia-Alcohol Mixture — Prepare the mixture with the following:

Ammonia hydroxide, sp gr 0·93	28 ml
Denatured alcohol (*see* IS : 324-1959* or rectified spirit (*see* IS : 323-1959†)	946 ml
Water	2 810 ml

5. PROCEDURE

5.1 Weigh 45 to 50 g of the sample into each of two 50-ml centrifuge tubes and centrifuge them for 20 min at approximately 2 300 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 long handled porcelain spoon before pipetting. Using a pipette with an end opening of about 2 mm draw off the supernatant latex to approximately 1 cm above the top of the sludge. Fill the tubes to the top with the ammonia-alcohol mixture, and balance them in pairs. Recentrifuge at approximately 2 300 rpm for 25 min. Again pipette off the supernatant liquid to approximately 1 cm 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 150-ml heat-resistant

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*Specification for ordinary denatured spirit (*revised*).

†Specification for rectified spirit (*revised*).

beakers using a rubber policeman and wash-bottle containing some of the ammonia-alcohol mixture. Evaporate on a hot plate to a low level, complete the drying at 70 ± 2°C, and weigh.

5.1.1 The weight of the dried residues run in duplicate shall agree within 1 mg.

6. CALCULATION

6.1 Calculate the sludge content as follows:

$$\text{Sludge content, percent by weight} = \frac{W_1}{W_2} \times 100$$

where

W_1 = weight in g of the dried residue, and

W_2 = weight in g of the sample.

DENSITY
(NRL : 6)

1. SCOPE

1.1 This method is intended for the determination of density of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. TERMINOLOGY

2.0 For the purpose of this test, the following definition shall apply.

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

3. OUTLINE OF THE METHOD

3.1 The density of latex is determined by density bottle method and the result is expressed as g/ml at a definite temperature. A correction is applied, if the temperature of bulk of the latex differs from the temperature of sample taken for density determination.

4. APPARATUS

4.1 **Balance** — to weigh accurately to the nearest 0·001 g.

4.2 **Constant Temperature Bath** — Accurate to within 0·2°C and adjustable to a temperature above or below room temperature.

4.3 **Density Bottle** — 50 ml capacity having a ground glass stopper perforated by a capillary and a ground glass cap and, if desired, an evacuated jacket.

4.4 **Two Conical Flasks** — of at least 200 ml capacity, each fitted with a rubber stopper, a short glass inlet tube with a rubber blowing ball at the external end and a glass outlet tube reaching nearly to the bottom of the flask.

5. PROCEDURE

5.1 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. Partly fill the second conical flask likewise with freshly boiled 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. Bring the density bottle, and, the latex and water in the two conical flasks to the temperature of the constant temperature bath. This will require a minimum of about three hours in the case of a jacketed bottle. First blow a few millilitres of latex from the conical flask containing the latex and discard. Blow sufficient latex from the conical flask into the density bottle to fill it completely. Place the stopper in place and wipe the top surface immediately 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 to the nearest 0·001 g. Empty the density bottle and wash free from latex with water. Immerse the bottle again in the bath as before. Fill the bottle with water by blowing from the second conical flask and allow to stand for five minutes. Empty the bottle and completely refill, while still immersed in the bath by the same procedure.

5.1.1 Put the stopper in place and wipe the top surface immediately 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 shall agree within 0·001 g/ml.

6. CALCULATION

6.1 Calculate the density from the formula:

$$D = \frac{M_L \times D_W}{M_W}$$

where

D = the density of latex at the temperature of the constant temperature bath in g/ml,

M_L = the weight in g of latex in the density bottle,

D_W = the density of water at the bath temperature in g/ml, and

M_W = the weight in g of water in the density bottle.

6.2 If the temperature of the density determination of the sample tubes differs from that of the bulk of the latex, apply a correction by calculation using Table 1:

$$D = A + B - C$$

TABLE 1 DENSITY CONVERSION VALUES FOR NATURAL RUBBER LATEX

(*Clause* 6.2)

TEMPERATURE °C	DENSITY OF WATER	CORRECTION FOR DRY RUBBER CONTENT									
		20 per-cent	25 per-cent	30 per-cent	35 per-cent	40 per-cent	45 per-cent	50 per-cent	55 per-cent	60 per-cent	65 per-cent
	g/ml	g/ml	g/ml	g/ml	g/ml	g/ml	g/ml	g/ml	g/ml	g/ml	g/ml
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
0	0·999 87	0·000 0	0·000 0	0·000 0	0·000 0	0·000 0	0·000 0	0·000 0	0·000 0	0·000 0	0·000 0
2	0·999 97	0·000 2	0·000 3	0·000 4	0·000 4	0·000 5	0·000 6	0·000 6	0·000 7	0·000 8	0·000 8
4	1·000 00	0·000 5	0·000 6	0·000 8	0·000 9	0·001 0	0·001 2	0·001 3	0·001 4	0·001 6	0·001 7
6	0·999 97	0·000 8	0·001 0	0·001 2	0·001 4	0·001 6	0·001 8	0·002 0	0·002 2	0·002 4	0·002 6
8	0·999 88	0·001 1	0·001 4	0·001 7	0·001 9	0·002 2	0·002 5	0·002 7	0·003 0	0·003 2	0·003 5
10	0·999 73	0·001 5	0·001 9	0·002 2	0·002 5	0·002 8	0·003 2	0·003 5	0·003 8	0·004 1	0·004 4
12	0·999 52	0·002 0	0·002 4	0·002 8	0·003 1	0·003 5	0·003 9	0·004 2	0·004 6	0·004 9	0·005 3
14	0·999 27	0·002 5	0·002 9	0·003 4	0·003 8	0·004 2	0·004 6	0·005 0	0·005 4	0·005 8	0·006 2
16	0·998 97	0·003 0	0·003 5	0·004 0	0·004 5	0·004 9	0·005 4	0·005 9	0·006 3	0·006 8	0·007 2
18	0·998 62	0·003 5	0·004 1	0·004 6	0·005 2	0·005 7	0·006 2	0·006 7	0·007 2	0·007 7	0·008 2
20	0·998 23	0·004 1	0·004 7	0·005 3	0·005 9	0·006 5	0·007 0	0·007 6	0·008 1	0·008 6	0·009 2
22	0·997 80	0·004 8	0·005 4	0·006 0	0·006 6	0·007 3	0·007 9	0·008 5	0·009 0	0·009 6	0·010 2
24	0·997 32	0·005 4	0·006 1	0·006 8	0·007 4	0·008 1	0·008 7	0·009 4	0·010 0	0·010 6	0·011 2
26	0·996 81	0·006 1	0·006 8	0·007 5	0·008 2	0·008 9	0·009 6	0·010 3	0·010 9	0·011 6	0·012 2
28	0·996 26	0·006 8	0·007 6	0·008 3	0·009 0	0·009 8	0·010 5	0·011 2	0·011 9	0·012 6	0·013 3
30	0·995 67	0·007 6	0·008 4	0·009 1	0·009 9	0·010 7	0·011 4	0·012 2	0·012 9	0·013 6	0·014 3
32	0·995 05	0·008 3	0·009 2	0·010 0	0·010 8	0·011 6	0·012 4	0·013 1	0·013 9	0·014 6	0·015 4
34	0·994 40	0·009 1	0·010 0	0·010 8	0·011 7	0·012 5	0·013 3	0·014 1	0·014 9	0·015 7	0·016 4
36	0·993 71	0·009 9	0·010 8	0·011 7	0·012 6	0·013 4	0·014 3	0·015 1	0·015 9	0·016 7	0·017 5
38	0·992 99	0·010 8	0·011 7	0·012 6	0·013 5	0·014 4	0·015 3	0·016 1	0·017 0	0·017 8	0·018 6
40	0·992 24	0·011 7	0·012 6	0·013 5	0·014 5	0·015 4	0·016 3	0·017 2	0·018 1	0·018 9	0·019 7

where

D = density of latex,

A = density measured at temperature of test,

B = correction value from table for temperature of test and *DRC* of latex, and

C = correction value from table for temperature of bulk of latex and *DRC* of latex.

TOTAL ALKALINITY

(NRL : 7)

1. SCOPE

1.1 This method is intended for the determination of total alkalinity of natural rubber latex of *Hevea brasiliensis,* uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 A known quantity of latex, in presence of a non-ionic or anionic emulsion stabilizer (*p*H 5·2 to 6·8), is titrated against standard acid using a suitable indicator or determined electrometrically.

3. APPARATUS

3.1 *p*H Meter — *p*H meter fitted with glass electrode.

3.2 Weighing Bottle

4. REAGENTS

4.1 Emulsion Stabilizer — 5 percent aqueous solution of non-ionic or suitable anionic emulsion stabilizer, having a *p*H value of 5·2 to 6·8, which is the value taken for the titration end-point.

4.2 Sulphuric Acid or Hydrochloric Acid — approximately 0·1 N (*see* IS : 266-1961* and IS : 265-1962†).

4.3 Indicator Solution — methyl red or bromothymol blue (*see* IS : 2263-1962‡).

5. PROCEDURE

5.1 Take between 5 and 10 g of the well mixed sample in a weighing bottle fitted with a stopper. Weigh to the nearest 0·01 g. To a 600 ml squat beaker add 200 ml of water. Mix with the water 10 ml of non-ionic or suitable anionic emulsion stabilizer. Stir the water with a glass rod to distribute the stabilizer. Pour the latex into the beaker from the weighing bottle, restopper the bottle and reweigh to obtain the weight of latex added by difference. Stir to distribute the latex in the stabilizer solution. The contents are titrated with sulphuric acid or hydrochloric acid. If methyl red is used as indicator, the colour change to pink will

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*Specification for sulphuric acid (*revised*).

†Specification for hydrochloric acid (*revised*).

‡Methods of preparation of indicator solutions for volumetric analysis.

occur at a *p*H value of approximately 5·2. If bromothymol blue is used, the colour change to yellow will occur at a *p*H value of approximately 6·8.

5.2 If the end-point is determined electrometrically, a *p*H value between 5·2 and 6·3 may be taken.

NOTE — The object of the stabiliser is to prevent coagulation of the latex on addition of acid.

6. CALCULATION

6.1 Alkalinity, in milli-equivalents per 100 g of water in the latex $= \frac{100}{(100-TS)} \times \frac{100\,N\,V}{W}$

where

TS = total solids, percent, in test portion;
N = normality of acid;
V = millilitres of acid used; and
W = weight of test portion in g.

6.2 Where the alkali is only ammonia, the alkalinity may be expressed as grams of ammonia (NH_3) per 100 g of water in the latex as follows:

$$\text{Alkalinity, percent, } NH_3 = \frac{170\,N\,V}{W\,(100-TS)}$$

6.3 In all cases, the criterion of the end point shall be stated in the report.

KOH-NUMBER

(NRL : 8)

1. SCOPE

1.1 This method is intended for the determination of KOH-number of natural rubber latex *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. TERMINOLOGY

2.0 For the purpose of this test, the following definition shall apply.

2.1 KOH-Number — Number of grams of potassium hydroxide required to neutralize the acids present in 100 g of the total solids in latex.

3. OUTLINE OF THE METHOD

3.1 A portion of latex of known solids content and alkalinity is weighed, the ammonia content is adjusted to 0·5 percent calculated on water phase with formaldehyde and titrated with KOH of known strength potentiometrically.

4. APPARATUS

4.1 The titration assembly shall consist of the following essential parts:

a) *pH meter* — dependent on electrometric measurements and capable of being read to 0·02 unit.

b) *Glass Electrode* — with a linear response up to *p*H 10.

c) *Suitable Half Cell* — A calomel electrode is recommended.

d) *Mechanical Stirrer* — equipped with glass paddle.

5. REAGENTS

5.1 Formaldehyde Solution (5 Percent, Acid Free) — This solution is prepared by diluting concentrated formaldehyde of pharmacopoeia guaranteed purity to 5 percent strength by adding six times its volume of water and neutralizing with 0·1 N potassium hydroxide solution, using phenolphthalein as indicator.

NOTE — As formaldehyde is not very stable, it may be necessary, if not freshly prepared, to determine its strength by adding a known volume of excess ammonia of known alkalinity and determining the residual alkalinity after 15 min. If the strength of the solution is less than 4·5 percent, the calculated amount of concentrated formaldehyde should be added to bring it to 5 percent. (180 g of formaldehyde are equivalent to 68 g ammonia.)

5.2 Potassium Hydroxide Solution — 0·5 N; carbonate free.

5.2.1 To ensure that the solution is reasonably free from carbonate, the procedure given in **5.2.1.1** to **5.2.1.3** is recommended.

5.2.1.1 First prepare a fairly concentrated solution of approximately known strength. For this purpose, hold each stick of pure potassium hydroxide by means of clean stainless steel tongs and rinse rapidly in a stream of freshly boiled and cooled water. Add bulk of boiled water immediately until approximately the required quantity has been added. If there is reason to suspect that more than traces of carbonate are present, treat a small quantity of the solution, say 10 ml, with 10 percent barium chloride solution. Ignore, if a very slight precipitate appears. If carbonate is definitely present, add 10 percent barium chloride solution until no further precipitate occurs and then add calculated amount of barium chloride solution to the main bulk solution of potassium hydroxide to precipitate all the carbonate. Then retest the bulk solution for carbonate and also for excess of barium. If the latter is present, remove it by adding 10 percent potassium sulphate solution dropwise. Before dilution of the bulk solution, allow the precipitate to settle and decant off the clear supernatant liquid. Spare the potassium hydroxide solution in wax-lined or polyethylene bottles.

5.2.1.2 For dilution, freshly boiled and cooled water shall be used.

5.2.1.3 Solutions which have to be stored shall be kept in air-tight bottles.

6. PROCEDURE

6.1 The *p*H meter is standardized with a buffer of known *p*H about 9. A suitable buffer is 0·06 N solution of sodium borate (*p*H = 9·18 at 27 ± 2°C).

6.2 Weigh a portion of latex of known solids content and alkalinity, containing approximately 50 g of solids accurately into a 400 ml beaker. Adjust the ammonia content to 0·5 percent calculated on the water phase by adding five percent formaldehyde solution (1 ml = 0·0189 g NH_3), while stirring. Add enough boiled and cooled water to dilute the latex to about 30 percent solids. Insert the electrodes and add slowly 5 ml of 0·5 N potassium hydroxide, while stirring the solution slowly with a glass paddle. Record the reading of the *p*H meter. Continue additions of 1 ml increments of 0·5 N potassium hydroxide solution stirring at the same time. Record the *p*H after each addition. To keep electrical conditions constant, do not turn off the stirrer until the titration is finished. The end-point of the titration is the point of inflection of the *p*H-ml potassium hydroxide titration curve. At this point, the slope of the curve, that is, the first differential, reaches a maximum and the second differential changes from a positive to a negative value. The end-point may be calculated with reasonable accuracy from the second differential, assuming that the change from a positive to a negative value bears a linear relation to the addition of 0·5 N potassium hydroxide during the 1-ml interval involved.

NOTE — Careful earthing of the *p*H meter and of the motor (should one be used to drive the glass stirrer) is necessary to prevent the wandering of the released galvanometer needle about its zero point, due to external electrical and electrostatic interferences.

7. CALCULATION

7.1 Calculate the KOH-number as follows:

$$\text{KOH-number} = \frac{V \times N \times 561}{TS \times W}$$

where

V = ml of standard potassium hydroxide solution,
N = normality of standard potassium hydroxide solution,
TS = percentage of total solids, and
W = weight in grams of the sample.

MECHANICAL STABILITY

(NRL : 9)

1. SCOPE

1.1 This method is intended for the determination of mechanical stabilit of natural rubber latex of *Hevea brasiliensis*, uncompounded and un vulcanized, and subjected to some type of concentration process othe than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 Latex is stirred at a high speed and the time required to produc obvious signs of clotting is recorded as a measure of mechanical stability

3. APPARATUS

3.0 The apparatus shall consist of the following.

3.1 Container — a latex container consisting of a flat-bottome cylindrical vessel 127 mm high and having an inside diameter of 56 mn to 60 mm. The inner surface shall be smooth, and a transparen container is preferred.

3.2 Stirring Device — The stirring device consists of a vertical stainles steel shaft 15·2 cm long and tapering to 6·35 mm at its lower end, wher a horizontal smooth stainless steel disk 21 ± 0·025 mm in diameter an 1·55 ± 0·05 mm thick is attached by means of a threaded stud at th exact centre of the disk.

3.3 Holding Device — An arrangement, for holding the latex containe so that the axis of the rotating shaft is concentric with that of the late container and the bottom of the stirring disk, is 12·5 ± 2·5 mm from th bottom inside of the latex container.

3.4 The apparatus is designed to maintain a stirring speed of 14 000 rev min ± 200 rev/min throughout the test. The speed should be checke while a test is in progress. At this speed the shaft shall not run mor than 0·25 mm out of true.

4. REAGENT

4.1 Dilute Ammonia — 1·6 percent ammonia.

4.2 Stainless Steel Sieves — 150-micron IS Sieve or 180-micron IS Siev (*see* IS : 460-1962*).

NOTE — The apertures of BS Sieves 100 or 85, and ASTM Sieves 100 or 8 (also known as 149 μ or 177 μ-US Standard Sieves) are within the limits lai down for the specified IS test sieves and may, therefore, be used as 150-micron o 180-micron IS Sives.

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*Specification for test sieves (*revised*).

5. PROCEDURE

5.1 Dilute the latex with dilute ammonia to 55·0 ± 0·2 percent total solids. Strain through stainless steel sieve. Within 6 hours, weigh 80 ± 1 g of the diluted, strained latex into the container and adjust the temperature to 35 ± 1°C. Do not subject the latex to unnecessary prolonged warming which may tend to form a surface skin by evaporation. For a convenient method of obtaining the required temperature, pass hot water through a glass tube bent to a suitable shape and very gently stir the latex with it for about 5 minutes, while still in the container. Place the container in position and stir the contents at 14 000 ± 200 rev/min until the end-point is reached. Note the end-point, when small pieces of coagulated rubber are first readily seen in the thin film of liquid formed, when a glass rod is dipped into the latex and lightly drawn over the palm of the hand, or when a curdy or rippled appearance is first visible over most of the surface of the latex in the container.

NOTE — The nearing of the end-point is marked by a notable decrease in vortex. Curdiness is best seen by reflected light on the surface.

5.1.1 In case of dispute, the end-point is reached when, on straining the latex through stainless steel 150-micron IS Sieve or 180-micron IS Sieve the amount of dry coagulum obtained first exceeds 0·5 g.

6. EXPRESSION OF RESULTS

6.1 In reporting the result of the test, state the condition of the end point used. Express the mechanical stability of the latex as the number of seconds between the commencement of stirring and the end point.

6.2 The results of duplicate tests shall not differ by more than 5 percent.

VOLATILE FATTY ACID NUMBER
(NRL : 10)

0. GENERAL

0.1 The two methods of distillation described in this test are identical in principle but differ in scale. Method A, in which the distillation is performed in a microchemical distillation apparatus of specified design, is preferred because it may be completed in a shorter time than Method B and is more convenient for serial analysis. The microchemical distillation apparatus of Method A shall be not less efficient than the apparatus described (Markham still) and it is important to note that some microchemical stills which have been designed primarily for the distillation of ammonia have not the high efficiency necessary for the distillation of volatile fatty acids. Method B, which requires only conventional chemical laboratory equipment, will be the method of choice when a microchemical apparatus suitable for carrying out Method A is not available.

1. SCOPE

1.1 This method is intended for the determination of volatile fatty acid number of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than the heat concentration and which may contain preservatives.

2. TERMINOLOGY

2.0 For the purpose of this test, the following definition shall apply.

2.1 Volatile Fatty Acid Number (*VFA*) — The number of grams of potassium hydroxide equivalent to the volatile fatty acids in latex containing 100 g of total solids.

NOTE — If substances have been added to the latex which will produce volatile acids on acidification with sulphuric acid, the volatile fatty acid number will be high and will not represent the volatile fatty acid content without correction.

3. OUTLINE OF THE METHOD

3.1 In this method of test, the volatile acids (mainly acetic acid) present in rubber latex are determined by steam-distilling rubber-free latex serum and estimating by acidimetric titration the total amount of volatile acids distilled either by a semi-micro method given in **5** or a micro method given in **6**.

4. REAGENTS

4.1 Ammonium Sulphate Solution — 30 percent (w/w) aqueous solution.

4.2 Sulphuric Acid Dilute — approximately 50 percent (*w/w*) aqueous solution.

4.3 Standard Barium Hydroxide Solution — 0·01 to 0·02 N. The solution is standardized by titration with potassium hydrogen phthalate. and stored. It is transferred to a burette in the absence of carbon dioxide.

4.4 Indicator — The indicators bromothymol blue or phenolphthalein may be used. Bromothymol blue shall be used as 0·5 percent solution of the indicator in water and phenolphthalein shall be used as 0·5 percent solution in a mixture of equal volumes of ethanol and water.

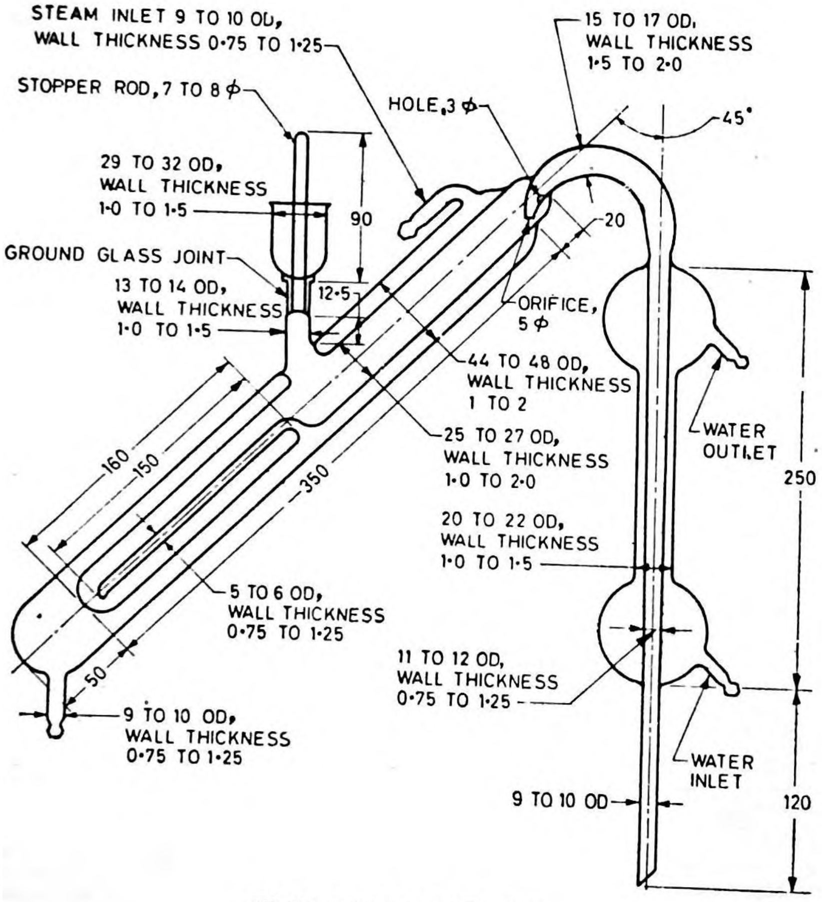
4.5 Antifoaming Agent -- silicone type.

5. SEMI-MICRO METHOD (METHOD A)

5.1 Apparatus — a steam-jacketed distillation apparatus (Markham still) conforming essentially to Fig. 1. As an alternative to the one-piece apparatus illustrated, a ground glass joint may be inserted between the distillation vessel and the condenser.

5.2 Procedure — Weigh about 50 g to the nearest 0·1 g of latex of known dry rubber and total solids content into a beaker. Add 50 ml of ammonium sulphate solution and swirl the mixture warming over a water-bath until the latex thickens and coagulates. Allow the beaker containing the coagulum to stand on the water-bath for a further 10 to 15 minutes. Press the serum out of the coagulum by kneading with a glass rod or pestle and filter through a dry filter. Transfer 25 ml of the filtered serum to a dry 50 ml conical flask and acidify with 5 ml of dilute sulphuric acid. Introduce 10 ml of the acidified serum by pipette with steam passing through the outer jacket of the apparatus (steam outlet open) into the inner tube. Add one drop of a suitable silicone foaming agent. Place a 150 ml conical flask under the tip of the condenser to receive the distillate. Close the steam outlet partly to divert steam into the inner tube. Pass steam gently at first, then close the steam outlet fully and continue distillation at a rate of approximately 30 ml per hour until 100 ml of distillate collects. Aerate the distillate by passing through it a stream of carbon dioxide-free air at a rate of 200 to 300 ml per minute for approximately 3 minutes and titrate with barium hydroxide. Run a blank test by substituting 20 ml of water for the 50 g of concentrated latex.

NOTE — The serum required for the distillation is prepared by treatment of latex with a non-volatile coagulant. The selected coagulant is ammonium sulphate because it induces a slow regular type of coagulation when added to latex, unlike acid coagulants which form local clots instantaneously. With ammonium sulphate, even distribution of coagulant throughout the latex may be ensured before the onset of thickening or gelling. (Moreover, since ammonium sulphate effectively removes proteins by precipitation, its use precludes the possibility of error due to the presence in the distillate of volatile products derived from the decomposition of proteins during distillation. By contrast, acids, such as sulphuric or phoshphoric, do not precipitate serum proteins completely and, if used as coagulants, shall be supplemented by a specific protein precipitant, such as phosphotungstic acid.)



All dimensions in millimetres.

FIG. 1 MARKHAM STILL FOR SEMI-MICRO DISTILLATION

5.3 Calculation — The volatile fatty acid (*VFA*) number is calculated as follows:

$$VFA = \frac{561 \times N \times V [50 + (100 - DRC) W/100\, D]}{25 \times W \times TS \times \frac{1}{3}}$$

where

N = normality of the barium hydroxide solution;

V = final volume of barium hydroxide solution required to neutralize the distillate in millilitres, after correction for blank;

DRC = percentage dry rubber in the latex;

W = weight of test portion in grams;

D = density of serum in g/ml

= 1·02 for any latex except heat concentrated latex; and

TS = percentage total solids in the latex.

5.3.1 Calculate *VFA* as a percentage of the aqueous phase as follows:

$$VFA \text{ (aqueous phase)} = \frac{VFA \times TS}{100 - TS}$$

where *VFA* is the value given in **5.3**.

5.3.2 A difference in *VFA* number between duplicate determinations on the same sample is not considered significant, if it is less than 0·01 unit where the actual *VFA* number is 0·10 or less, or if it is less than 10 percent of the actual *VFA* number where the *VFA* number is greater than 0·10 unit.

6. MACRO METHOD (METHOD B)

6.1 Apparatus — a steam-distillation apparatus with a long-necked 500-ml centre flask, a long condenser fitted with ground glass joints. The apparatus does not include a steam trap.

6.2 Procedure — Weigh about 100 g to the nearest 0·2 g of latex of known dry rubber and total solids content into a beaker. Add 100 ml of ammonium sulphate solution and swirl the mixture warming over a water-bath until the latex thickens and coagulates. Allow to stand the beaker containing the coagulum on the water-bath for a further 10 to 15 minutes. Press the serum out by kneading the coagulum with a glass rod or pestle and filter through a dry filter. Detach the centre flask from the steam-distillation apparatus, which has already been steamed out, and pipette out 50 ml of the filtrate into it followed by 10 ml of dilute sulphuric acid. Mix the contents by swirling and blow a current of air over the liquid in the flask. Re-connect the centre flask to the apparatus. Steam distil the solution with a small steam supply and a strong flame under the centre flask until the volume of liquid is reduced to 35 to 40 ml; this normally takes approximately 10 minutes. Then steam distil at a rate of approximately 30 ml per hour and at approximately constant volume by reducing the flame under the centre flask and increasing the steam supply. Collect 250 ml of distillate. Aerate the distillate by passing through it a stream of carbon dioxide-free air at a rate of 200 to 300 ml per minute for approximately 3 minutes and titrate with 0·02 N barium hydroxide.

NOTE — The serum required for the distillation is prepared by treatment of latex with a non-volatile coagulant. The selected coagulant is ammonium sulphate because it induces a slow regular type of coagulation when added to latex, unlike acid coagulants which form local clots instantaneously. With ammonium sulphate, even distribution of coagulant throughout the latex may be ensured before the onset of thickening or gelling. Moreover, since ammonium sulphate effectively removes proteins by precipitation, its use precludes the possibility of error due to the presence in the distillate of volatile products derived from the decomposition of proteins during distillation. By contrast, acids, such as sulphuric or phosphoric, do not precipitate serum proteins completely and, if used as coagulants, shall be supplemented by a specific protein precipitant such as phosphotungstic acid.

6.3 Calculation — Calculate the volatile fatty acid (*VFA*) number as follows:

$$VFA = \frac{561 \times N \times V \left[\sqrt{100} + (100 - DRC)\, W/100\, D \right]}{50 \times W \times TS}$$

where

N = normality of the barium hydroxide solution,
V = volume of barium hydroxide solution required to neutralize the distillate in millilitres,
DRC = percentage dry rubber content in the latex,
W = weight of test portion in grams,
D = density of serum in g/ml
= 1·02 for any latex except heat concentrated latex, and
TS = percentage total solids in the latex.

6.3.1 Calculate *VFA* as a percentage of the aqueous phase as follows:

$$VFA \text{ (aqueous phase)} = \frac{VFA \times TS}{100 - TS}$$

where *VFA* is the value given in **6.3**.

6.3.2 A difference in *VFA* number between duplicate determinations on the same sample is not considered significant, if it is less than 0·01 unit where the actual *VFA* number is 0·10 unit or less, or if it is less than 10 percent of the actual *VFA* number where the *VFA* number is greater than 0·10 unit.

*p*H MEASUREMENT
(NRL : 11)

1. SCOPE

1.1 This method is intended for the determination of *p*H of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 *p*H of latex is determined electrometrically by a *p*H meter fitted with glass electrode.

3. APPARATUS

3.1 Any *p*H meter dependent on electrometric measurements, and a glass electrode-calomel assembly for determining *p*H may be used. A flowing calomel electrode has been found particularly suitable for this use. The glass electrode shall be of the type applicable for a *p*H range 8 to 14.

4. PROCEDURE

4.1 Calibrate the *p*H meter. Take any convenient size of sample and adjust the temperature to a range of 27 ± 2°C by mildly agitating the sample-container in a water bath at a suitable temperature. Determine the *p*H.

5. REPORT

5.1 Report both the *p*H and temperature of the latex sample.

TOTAL NITROGEN

(NRL : 12)

1. SCOPE

1.1 This method is intended for the determination of total nitrogen content of natural rubber latex of *Hevea brasiliensis,* uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 Nitrogen is determined by a semi-micro-Kjeldahl procedure. The rubber is oxidized by heating with a mixture of potassium sulphate and concentrated sulphuric acid together with a catalyst, thereby converting nitrogen compounds into ammonia which is removed by distillation after making the mixture alkaline. The distilled ammonia is absorbed in sulphuric acid, boiled to remove carbon dioxide and titrated with sodium hydroxide.

2.2 Alternatively, the distilled ammonia is absorbed in boric acid solution and titrated with standard acid.

3. APPARATUS

3.1 Semi-micro-Kjeldahl Digestion and Distillation Apparatus — with a condenser tube made of borosilicate glass, pure tin or silver.

3.2 5-ml Burette — calibrated at every 0·02 ml. A burette with storage bottle and automatic zero setting is suitable.

4. REAGENTS

4.1 Boric Acid Solution — Dissolve 40 g of boric acid in water warming, if necessary, and make up the volume to two litres. Add 20 ml of indicator solution and mix well.

4.2 Catalyst Mixture — Prepare a finely-divided and intimate mixture of the following:

> Potassium sulphate anhydrous 30 parts; Copper sulphate pentahydrate 4 parts; Selenium 1 part or Sodium selenate 2 parts.

4.3 Mixed Indicator Solution — 0·1 g of methyl red and 0·05 g of methylene blue in 100 ml of ethanol.

NOTE — This indicator may deteriorate on storage and in such cases shall be freshly prepared.

4.4 Standard Sodium Hydroxide Solution — 0·02 N, carbonate-free.

4.5 Sodium Hydroxide Concentrated Solution — 56 g in 100 ml of water.

4.6 Concentrated Sulphuric Acid — conforming to IS : 266-1961*.

4.7 Standard Sulphuric Acid — 0·02 N.

5. PROCEDURE

5.1 Blend the rubber obtained in the determination of total solids by passing six times between the tightly closed rolls of a laboratory mill, rolling into a cylinder after each pass and presenting the cylinder end-on to the rolls. Weigh accurately about 0·1 g of the blended rubber into a clean dry micro-Kjeldahl flask. Add approximately 0·65 g of the catalyst mixture and 3·0 ml of concentrated sulphuric acid. Boil gently. Boil for about half an hour after the digest has become a clear green colour with no yellow tint.

5.1.1 *Standard Distillation Procedure (Reference Method)* — From a semi-micro burette, run 5 ml or appropriately less of standard sulphuric acid into the steamed-out receiver of the distillation apparatus, add two drops of indicator solution and place the receiver under the condenser so that the end of the condenser dips below the surface of the acid. Add approximately 10 ml of concentrated sodium hydroxide solution to the distillation flask and wash it with not more than 5 ml of water. Pass steam from the generating flask through the distillation flask for 4 to 7 minutes at such a rate that the final volume of solution in the receiver is about 30 ml. If necessary, during distillation add more standard sulphuric acid to maintain an excess in the receiver. At the conclusion of the distillation, lower the receiver until the tip of the condenser is well above the level of the acid. Continue distilling for one minute and then wash the end of the condenser with water into the distillate. Boil the contents of the receiver for half a minute and cool with exclusion of carbon dioxide. Titrate the residual acid with standard sodium hydroxide using the 5 ml burette.

5.1.1.1 Carry out a blank determination using the same quantities of reagents and conditions of test and correct accordingly.

5.1.2 *Alternative Procedure Using Boric Acid* — Add 10 ml of boric acid solution to the steamed-out receiver of the distillation apparatus, add two drops of indicator solution and place the receiver under the condenser so that the end of the condenser dips below the surface of the boric acid solution. Add approximately 10 ml of concentrated sodium hydroxide solution to the distillation flask and wash in with not more than 5 ml of water.

5.1.2.1 Pass steam from the generating flask through the distillation

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*Specification for sulphuric acid (*revised*).

flask for four minutes and then lower the receiver until the tip of the condenser is well above the level of the acid. Continue distilling for one minute and then wash the end with water into the distillate. The final volume of solution in the receiver shall be between 30 and 35 ml. Immediately titrate the contents of the receiver with standard sulphuric acid.

5.1.2.2 Carry out a blank determination using the same quantities of reagents and conditions of test and correct accordingly.

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6. CALCULATION

6.1 Calculate the nitrogen content of latex from the formula:

$$\text{Nitrogen, percent by weight} = \frac{0{\cdot}028\,V}{W}$$

where

V = corrected volume in millilitres of standard sodium hydroxide (procedure described in **5.1.1** or corrected volume in millilitres of standard sulphuric acid (procedure described in **5.1.2**), and

W = weight of test portion in g.

TOTAL COPPER
(NRL : 13)

0. GENERAL

0.1 The method measures the total amount of copper in the rubber. While it is recognized that some compounds of copper may not be harmful to rubber, there are at present no generally accepted means of distinguishing analytically between harmful and safe forms when present in rubber. There is also the possibility, depending on conditions of service and other contingencies that acknowledged safe forms of copper may undergo change to active forms which may accelerate the degradation of the rubber.

1. SCOPE

1.1 This method is intended for the determination of total copper content of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 Five grams of the dried latex solids or of the raw rubber is ashed in a crucible. The ash with a hydrochloric-nitric acid mixture is extracted and made alkaline with ammonium hydroxide. After removing the iron complex formed in presence of ammonium citrate, the aqueous solution is then shaken with a chloroform solution of diethyldithiocarbamate to form a yellow copper complex. The optical density of this solution is measured photometrically, which is proportional to the concentration of copper.

3. APPARATUS

3.1 Photoelectric Absorptiometer — filter-photometer or spectophotometer.

3.2 Silica Crucibles — 50 or 80 ml capacity.

4. REAGENTS

4.1 Light Magnesium Oxide

4.2 Hydrochloric Acid-Nitric Acid Mixture — The following are mixed together:

2 volumes of hydrochloric acid (*see* IS : 265-1962*),
1 volume of nitric acid (*see* IS : 264-1950†), and
3 volumes of water.

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*Specification for hydrochloric acid (*revised*).
†Specification for nitric acid. (Since revised).

4.3 Citric Acid Solution — Fifty grams of solid citric acid are dissolved in 100 ml of water.

4.4 Ammonia Solution — 20 percent (*w*/*w*).

4.5 Litmus Paper

4.6 Zinc Diethyldithiocarbamate Reagent — one gram of solid zinc diethyldithiocarbamate is dissolved in 1 litre of chloroform. If zinc diethyldithiocarbamate is not available, the reagent may be prepared as follows:

One gram of sodium diethyldithiocarbamate is dissolved in water to which 2 g of zinc sulphate are then added. The resulting zinc diethyldithiocarbamate is extracted by shaking with 100 ml of chloroform and the chloroform solution is separated and diluted to 1 litre. Stored in an amber coloured bottle this reagent is stable for at least six months.

4.7 Sodium Sulphate, Anhydrous

4.8 Standard Copper Solution — 0·393 g of copper sulphate pentahydrate ($CuSO_4,5H_2O$) is weighed into a small beaker and dissolved in water. To this 3 ml of concentrated sulphuric acid are added. The solution is transferred to a 1 000-ml volumetric flask and diluted with water to the graduation mark to form the stock solution. 10 ml of this stock solution is pipetted into a 100-ml volumetric flask and diluted with water to the graduation mark. This solution contains the equivalent of 0·01 mg Cu per ml and shall be freshly prepared from the stock solution when required.

5. PROCEDURE

5.1 Preparation of Sample — For the determination of copper in latex, prepare a specimen, the total solids of which weigh at least 5 g as described in total solids determination (*see* NRL : 2). Blend the rubber so obtained by passing it six times between the tightly closed rolls of a laboratory mill, rolling into a cylinder after each pass and presenting the cylinder end-on to the rolls.

5.2 Ashing — Weigh a 5-g test portion of the dried latex film to the nearest 5 mg and place in a silica crucible containing 0·1 g of light magnesium oxide distributed over the base and partly on the top of the crucible. Support the crucible in a hole, cut in an asbestos board, so that about two-thirds of the crucible project below the asbestos. Start a blank determination in a similar crucible using the same amount of magnesium oxide. Henceforth treat identically the test and blank determinations. Heat the crucible and contents with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to a muffle furnace at a temperature of 550 ± 25°C and heat until all carbon is oxidized. Remove the crucible and allow it to cool.

5.2.1 Alternatively, weigh 5 g of test portion, to the nearest 5 mg, wrap in a piece of ashless filter paper about 15 cm in diameter and place in transparent silica crucible having clean unetched walls. Place the crucible with its contents in a furnace at 550 ± 25°C and close the door. The furnace door should not be opened during the first hour. Start a blank determination also using a similar filter paper and crucible, and henceforth treat identically test and blank determinations. When all the carbon is oxidized, remove the crucible and allow it to cool.

5.3 Moisten the contents of the crucible with 0·5 to 1 ml water, then add 10 ml of dilute hydrochloric acid-nitric acid mixture and cover the crucible, with a glass, and heat on a steam bath for 30-60 minutes. Wash the contents of the crucible into a small beaker or flask, add 5 ml of citric acid solution and then add ammonia solution dropwise until the solution is just alkaline to litmus paper. Cool the solution by immersion in running water, or otherwise transfer to a separating funnel and add a further 2 ml of ammonia solution, and then dilute to about 40 ml with water. Pipette out 25 ml of zinc diethyldithiocarbamate reagent into the solution and shake for 2 minutes. After separation, draw the chloroform layer immediately into a stoppered flask containing about 0·1 g anhydrous sodium sulphate. If turbidity persists after standing for about 30 minutes, make further small additions of anhydrous sodium sulphate until the solution becomes clear.

5.4 Photometric Measurement of Colour — Decant the chloroform solution through a plug of glass wool, or a small filter paper, into the cell of a photoelectric absorptiometer and measure the optical density at approximately 435 mμ. Correct the reading by subtracting the value for the optical density of the blank solution and, from the corrected reading and the calibration curve of the instrument, obtain the concentration of copper in the test solution.

5.4.1 Prepare a calibration curve as follows.

5.4.1.1 Make up a series of standard solutions each containing, if the magnesium oxide method of ashing is used, 0·1 g magnesium oxide dissolved in 10 ml dilute hydrochloric acid-nitric acid mixture. To these solutions, add portions of the standard copper solution ranging from 1 to 10 ml, followed by 5 ml of citric acid solution and 2 ml excess of ammonia solution. If the rubber was wrapped in filter paper and placed directly in the muffle, then omit the magnesium oxide. Simultaneously prepare a blank solution with the standard solutions using the same quantities of reagents but without added copper. Dilute the solutions to 40 ml with water, extract with 25 ml zinc diethyldithiocarbamate reagent, and measure the optical density of each dried chloroform extract. Correct reading for each standard solution by subtracting the blank value and obtain the calibrated curve by plotting the relationship between copper concentration and optical density.

NOTE — The calibration curve should be checked as necessary depending on local conditions and on the type of instrument used.

TOTAL IRON
(NRL : 14)

1. SCOPE

1.1 This method is intended for the determination of total iron content of natural rubber latex ~~of~~ *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

[illegible]

2. OUTLINE OF THE METHOD

2.1 After removal of the organic matter by dry ashing, the iron is reacted with *o*-phenanthroline and is determined absorptiometrically.

3. APPARATUS

3.1 Photoelectric Absorptiometer — filter photometer or spectrophotometer.

3.2 Silica or Porcelain Crucible — nominal capacity 50 to 80 ml.

3.3 Muffle Furnace — with pyrometer and thermostatic control.

4. REAGENTS

4.1 Hydrochloric Acid, Concentrated — (*see* IS : 265-1962*.)

4.2 Buffer Solution — Dissolve 123 g of anhydrous sodium acetate in water, add 90 ml of glacial acetic acid (sp gr 1·05), and dilute the mixture with water to 500 ml, filter, if the solution is cloudy.

NOTE — If this buffer solution gives highly coloured reference solutions, alternative buffer solutions may be prepared by dissolving 60 g of sodium hydroxide or 80 g of anhydrous sodium carbonate in 200 ml of water, adding 180 ml of glacial acetic acid (sp gr = 105), and diluting to 500 ml.

4.3 Hydroxylamine Hydrochloride Solution — Dissolve 10 g of hydroxylamine hydrochloride in 100 ml of water.

4.4 *o*-Phenanthroline Solution — Dissolve 0·5 g of *o*-phenanthroline monohydrate in hot water and dilute to 500 ml.

4.5 Standard Iron Solution (1 ml = 0·1 mg Fe) — Dissolve 0·702 1 g of ferrous ammonium sulphate [$Fe(NH_4)_2 SO_4 \cdot 6H_2O$] in water in a 1 000-ml volumetric flask. Add three millilitres of concentrated hydrochloric acid (sp gr 1·10) and dilute to the mark with water. This solution should remain stable for at least a month.

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*Specification for hydrochloric acid (*revised*).

4.6 Standard Iron Working Solution (1 ml = 0·01 Fe) — Take 10 ml of the standard iron solution, transfer it to a 100 ml volumetric flask and dilute with water to the mark. Carry out dilution just before use.

5. PROCEDURE

5.1 For the determination of iron in latex, prepare a specimen of the total solids, according to the method described under determination of total solids (*see* NRL : 2). Blend the rubber so obtained by passing six times between the tightly closed rolls of a laboratory mill, rolling into a cylinder after each pass and presenting the cylinder end-on to the rolls.

5.2 Weigh accurately about 10 g of the sample prepared as above. Place in a silica or porcelain unetched crucible and support the crucible in a hole, cut in an asbestos board, so that about two-thirds of the crucible project below the asbestos. Heat gently with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to a muffle furnace at a temperature of 550 ± 25°C. When all the carbon has been oxidized, remove the crucible and allow it to cool.

5.3 Alternatively wrap the weighed sample in a piece of ashless filter paper about 15 cm in diameter and place in the crucible. Place the crucible and contents in a furnace at 550 ± 25°C and close the door. The furnace door should not be opened for at least 1 hour while inflammable vapours are evolved. Start a blank determination using a similar filter paper and crucible, and treat identically henceforth as in the case of determinations with the sample. When all the carbon is oxidized, remove the crucible and allow it to cool.

5.4 Add 5 ml of hydrochloric acid and 5 ml of water to the crucible and digest the mixture on a steam plate for 30 to 60 minutes. If the solution has a deep yellow colour, indicating the presence of much iron, add 5 ml more of hydrochloric acid and continue the digestion for 30 minutes more. Filter the solution through a sintered glass pad, collect the filtrate in a 50 ml volumetric flask and dilute to the mark.

5.4.1 Transfer an aliquot containing not more than 2 ml of hydrochloric acid to a 50 ml volumetric flask. Add 10 ml of the buffer solution, then 1 ml of hydroxylamine solution, and 10 ml of *o*-phenanthroline solution.

5.4.2 Prepare a blank with the same reagents and following the same procedure.

5.5 After 10 minutes, carry out the photometric measurement at about 510 mμ. Correct the reading by subtracting the value for the optical density of the blank solution, and obtain from the corrected reading and the calibration curve for the instrument, the concentration of iron in the test solution. Express the value in parts per million of iron for the entire specimen.

5.5.1 Prepare a calibration curve as follows.

5.5.1.1 Introduce into a series of 50-ml volumetric flasks; 0; 0·5; 5; 10; 15; 20 ml of standard iron working solution. To each add 1 ml of hydrochloric acid, 10 ml of buffer solution, 1 ml of hydroxylamine solution and 10 ml of *o*-phenanthroline solution. Dilute with water to the mark. After 10 minutes, measure the optical density of each standard solution at about 510 mμ and correct this value by subtracting the value of compensation solution. The calibration curve is obtained by plotting the relationship between iron concentration and optical density. The calibration curve should be checked, as necessary, depending on local conditions and on the type of instrument used.

TOTAL MANGANESE

(NRL : 15)

0. GENERAL

0.1 The method described here, measures the total amount of manganese in the rubber. While it is recognized that some compounds of manganese may not be harmful to rubber, there are at present no generally accepted means of distinguishing analytically between harmful and safe forms when present in rubber. There is also the possibility, depending on conditions of service and other contingencies that acknowledged safe forms of manganese may undergo change to active forms which may accelerate the degradation of the rubber.

1. SCOPE

1.1 This method is intended for the determination of total manganese content of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 Ten grams of the dried latex solids are ashed in a crucible and the ash treated with potassium hydrogen sulphate and sulphuric acid to convert the manganese to a soluble form. After dissolving the ash in dilute sulphuric acid, any iron present is complexed with orthophosphoric acid and the manganese oxidized to permanganate by boiling with potassium periodate. The optical density of the solution is measured photometrically which is proportional to the concentration of manganese.

3. APPARATUS

3.1 Photoelectric Absorptiometer — filter photometer or spectrophotometer.

3.2 Silica Crucibles — nominal capacity 50 or 80 ml.

4. REAGENTS

4.1 Sulphuric Acid Concentrated — (*see* IS : 266-1961*.)

4.2 Potassium Hydrogen Sulphate

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*Specification for sulphuric acid (*revised*).

4.3 Dilute Sulphuric Acid 5% — One volume of concentrated acid is mixed with 19 volumes of water.

4.4 Orthophosphoric Acid — 85 to 90 percent purity.

4.5 Stabilized Water — About 0·1 g potassium permanganate is dissolved in one litre of water to which a few drops of sulphuric acid have been added. The water is distilled through an effective spray trap discarding the first and last 50 ml of distillate. The rest of the distillate is collected and stored in a glass-stoppered bottle.

4.6 Potassium Permanganate — approximately 0·001 N.

4.7 Standard Manganese Solution

4.7.1 Into a small beaker, 0·720 g potassium permanganate is weighed and dissolved in water containing two millilitres sulphuric acid. Sulphur dioxide saturated water is added until the solution is colourless. The solution is boiled for 15 minutes, cooled, transferred to a 500-ml volumetric flask and diluted to the mark. Ten millilitres of this solution is pipetted into a second 500-ml flask and again diluted to the mark. This dilute solution contains the equivalent of 0·01 mg manganese (as Mn) per ml and shall be freshly prepared from the stock solution when required.

4.7.2 Into a small beaker, 0·770 g manganese sulphate ($MnSO_4$, $4H_2O$) is weighed and dissolved in water containing two millilitres sulphuric acid. The solution is transferred to a 500-ml volumetric flask and diluted to the mark. This solution shall be stable for at least a month. Ten millilitres of this solution is pipetted into a second 500-ml flask and again diluted to the mark. This dilute solution contains the equivalent of 0·01 mg manganese (as Mn) per ml and shall be freshly prepared from the stock solution when required.

5. PROCEDURE

5.1 For the determination of manganese in latex, prepare a specimen of the total solids weighing at least 10 g as described in determination of total solids (*see* NRL : 2). Blend the rubber so obtained by passing it six times between the tightly closed rolls of a laboratory mill, rolling into a cylinder after each pass and presenting the cylinder end-on to the rolls.

5.2 Weigh a 10 g test portion of the dried latex film to the nearest 10 mg, cut into small pieces and place in a crucible, which is supported in a hole, cut in an asbestos board, so that about two-thirds of the crucible projects below the asbestos. Start a blank determination using a similar crucible at the same time and identically treat throughout as in the case of determinations with the sample. Heat the crucible and contents with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to a muffle furnace at a temperature of 550 ± 25°C, heat until all carbon has been oxidized. Remove the crucible and allow it to cool. Add concentrated sulphuric acid delivered from a pipette with a fine jet, dropwise round the sides of the crucible in amount just sufficient to moisten the ash, fume off the excess acid by gentle heating, and place the crucible again in the muffle furnace at 550 ± 25°C to remove the last traces of carbon. Give the ash a second treatment with sulphuric acid adding the acid as before from a fine pipette, but heat only till fuming ceases in order to retain the ash, as far as possible, in the form of sulphate. Then cool the crucible, add 2 to 3 g potassium hydrogen sulphate, apply strong heat from a burner supporting the crucible on the asbestos board until a clear melt is obtained. Then cool the crucible and its contents.

5.3 As an alternative to the above method of ashing, weigh a 10 g test portion to the nearest 10 mg, in a piece of ashless filter paper about 15 cm in diameter and place in a crucible of about 80 ml nominal capacity. Place about 5 g potassium hydrogen sulphate on the top of the wrapped test portion, place the crucible in a furnace at 550 ± 25°C and close the door. The furnace door shall not be opened during the first hour. Start a blank determination also using a similar filter paper and crucible and identically treat throughout the blank as in the case of test determination. When all the carbon has been oxidized, allow the crucible and contents to cool.

5.4 Add to the ash produced by either of the above procedures, 20 ml dilute sulphuric acid, and heat the crucible on a steam bath until the solid material is dissolved or loosened from the walls of the crucible. Wash the contents in a small beaker using a glass rod to dislodge undissolved solid and boil the solution gently until no more will dissolve. Filter the solution through a sintered glass filter pad into a small conical flask, and wash the filter and insoluble material with two or three portions of water. Add 3 ml of orthophosphoric acid to the solution in the flask making further additions of 1 ml as may be necessary to remove any yellow colour due to iron. To the solution, add 0·3 g potassium periodate and raise the solution to boiling and maintain at the boiling point for 10 minutes. Maintain the temperature above 90°C for a further 10 minutes to ensure full development of the permanganate colour. After cooling, transfer the solution to a 50-ml volumetric flask and dilute to the mark with stabilized water at 27°C. After mixing the colour should be stable for several hours; any tendency to fading indicates the incomplete removal of organic matter or chloride.

5.5 Rinse the cells of the photoelectric absorptiometer first with approximately 0·001 N potassium permanganate and then with stabilized water. Rinse one cell with the test solution and the other with the blank and fill each cell with the appropriate solution. Measure the optical density of the test solution against the blank as reference solution at a wave length of approximately 525 mμ. Obtain the concentration of manganese from the observed optical density and the calibration graph of the instrument.

5.5.1 Prepare a calibration graph as follows.

5.5.1.1 Make up a series of standard solutions, each containing 20 ml dilute sulphuric acid, three millilitres orthophosphoric acid and 25 ml stabilized water. Add increasing volumes of 0, 2·0, 4·0, 6·0, 8·0 and 10·0 ml of standard manganese solution, followed in each case by 0·3 g potassium periodate. Boil the solutions and treat exactly as described for the test solution finally cooling and diluting to 50 ml in volumetric flasks. After rinsing the cells of the instrument first with approximately 0·001 N potassium permanganate, next with stabilized water add then with the appropriate test solution, measure the optical density of each solution at about 525 mμ. Correct all readings for the blank value and obtain the calibration curve by plotting the relationship between concentration of manganese and optical density.

NOTE 1 — The calibration curve shall be checked as necessary depending on local conditions and on the type of instrument used.

NOTE 2 — All precautions and safeguard required for carrying out of trace metal analysis shall be observed.

TOTAL ASH

(NRL : 16)

1. SCOPE

1.1 This method is intended for the determination of total ash content of natural rubber latex of *Hevea brasiliensis*, uncompounded and unvulcanized, and subjected to some type of concentration process other than heat concentration and which may contain preservatives.

2. OUTLINE OF THE METHOD

2.1 A known quantity rubber is obtained as described under total solids, blended and the dry film is ashed in a muffle furnace at 550 ± 25°C, cooled and weighed.

3. APPARATUS

3.1 Smooth Unetched Crucible — 50 ml capacity, silica or porcelain.

NOTE — Silica crucible shall be used if copper or manganese is subsequently to be determined in the ash.

3.2 Muffle Furnace — with pyrometer and thermostatic control.

3.3 Asbestos Board — This board is about 10 cm square and 0·5 cm thick, with a central hole to support the crucible so that about two-thirds of its height protrude below the board.

3.4 Ashless Filter Paper — about 15 cm in diameter.

4. PROCEDURE

4.1 For the determination of ash content of latex, prepare a specimen of 5 g as described under total solids (*see* NRL : 2). Blend the rubber so obtained by passing it six times between the tightly closed rolls of a laboratory mill rolling into a cylinder after each pass and presenting the cylinder end-on to the rolls. Then ash and weigh as described under Method A or Method B.

4.1.1 *Method A* — Weigh a test portion of 5 g of the dry latex film to the nearest 1 mg and place in a previously ignited weighed crucible. Rest the crucible in the hole in the asbestos board and heat gently over a small flame, taking care not to ignite the rubber and to avoid spurting when it is liquid. When the rubber is completely decomposed to a charred mass, transfer the crucible to the muffle furnace and heat the ash at a temperature of 550 ± 25°C until free from carbon. When ashing is complete cool the crucible in a desiccator and weigh to the nearest 1 mg.

4.1.2 Weigh a test portion of 5 g of the dry latex film to the nearest 1 mg, wrapped in ashless filter paper and place in a previously ignited weighed crucible. Then place the crucible in the muffle furnace at a temperature of 550 ± 25°C, with the door and vents closed. After about one hour in the furnace, open the door and vents and allow the test portion to burn until free from carbon. When ashing is complete, cool the crucible in a desiccator and weigh to the nearest 1 mg.

5. RESULTS

5.1 Express the results as a percentage of the weight of test portion of latex.