

**BUREAU OF INDIAN STANDARDS**

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*Draft Indian Standard*

**CASHEWNUT SHELL LIQUID (CNSL) — SPECIFICATION**

*(Third Revision of IS 840)*

(ICS 86.060)

Raw materials for Paints, Varnishes and  
Related Product Sectional Committee, CHD 21

**Last Date for Comments 24 November 2024**

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**FOREWORD**

*(Formal Clause shall be added later)*

This standard was initially published in 1956. Subsequently, the first revision was undertaken in 1964. In that revision the catalytic method was added as an alternate to Wij's method for determination of iodine value. Due to significant differences in the values produced by these method different values, two different iodine values were prescribed according to the methods followed. Additionally, the limit for iodine value by following Wij's method was raised from 220 to 250, loss in weight on heating and viscosity after acid washing were added, and the requirement for acid value was deleted.

In the second revision in 1986, the catalytic method was replaced by the Rosenmund-Kuhnenn (R.K.) method, with the iodine value limits modified accordingly.

This third revision has been undertaken to align the product standards with current market demands, enhancing its acceptance and relevance. Notable changes include:

- a) The test method for moisture content determination has been updated to the Karl Fischer method due to its better accuracy and adaptability within the industry. Additionally, the alternative R K method for determining the Iodine value has been removed as it is no longer in use.

- b) To address health and safety concerns, restriction for lead has been tightened and restriction for toxic heavy metals has been introduced;
- c) A suitable precautionary note has been added in the marking clause in order to prevent unforeseen events, and;
- d) Additionally, various editorial corrections, and references have been updated to ensure accuracy and relevance in the revised standards.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 (*second revision*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Draft Indian Standard*

CASHEWNUT SHELL LIQUID (CNSL) — SPECIFICATION

*(Third Revision)*

## 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for cashewnut shell liquid. The material is used as a phenolic component in many commercial resins, resinous compositions, moulding compositions, protective coatings and insulating varnishes.

## 2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

## 3 REQUIREMENTS

### 3.1 Description

**3.1.1 Source** — The material shall be produced from the shells of cashewnuts (*Anacardium occidentale*).

**3.1.2** The material shall be free from separated water and extraneous matter.

**3.1.3 Colour** — The material shall be not deeper than dark brown when viewed by transmitted light.

### 3.2 Lead Restriction

The material shall be tested for restriction from lead in accordance with ICP-OES or AAS Method of IS 101 (Part 8/Sec 5). When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm (*see Note*).

### 3.3 Toxic Heavy Metal Restriction

Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony, and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material when tested by the relevant Absorption/Emission Spectroscopic methods.

**3.4** The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Annex B and ISs. Reference to relevant clauses of Annex B is given in col 4 of the table.

**TABLE 1 REQUIREMENTS FOR CASHEWNUT SHELL LIQUID (CNSL)**

| SL No. | CHARACTERISTIC  | REQUIREMENT    | METHOD OF TEST ( REF TO CL NO. IN ANNEX B OR IS ) |
|--------|---|----------------|---|
| (1)    | (2)   | (3)            | (4)   |
| i)     | Specific gravity, 30/30°C   | 0.950 to 0.970 | B-2   |
| ii)    | Viscosity at 30°C, in centipoises, <i>Max</i>                       | 550            | B-3   |
| iii)   | Moisture, percent by weight, <i>Max</i>                             | 1.0            | IS 2362   |
| iv)    | Matter insoluble in toluene, percent by weight, <i>Max</i>          | 1.0            | B-4   |
| v)     | Loss in weight on heating, percent by weight, <i>Max</i>            | 2.0            | B-5   |
| vi)    | Ash, percent by weight, <i>Max</i>                                  | 1.0            | B-6   |
| vii)   | Iodine value ( <i>see 0.2</i> ), <i>Min</i>                         | 250            | B-7   |
| viii)  | Polymerization  |                |   |
|        | a) Time in minutes, <i>Max</i>                                      | 4              | B-8   |
|        | b) Viscosity at 30°C, in centipoises, <i>Min</i>                    | 30             | B-9   |
|        | c) Viscosity after acid washing at 30°C, in centipoises, <i>Min</i> | 200            | B-10  |

#### **4 PACKING AND MARKING**

**4.1** The material shall be packed in containers or transported in bulk as agreed to between the purchaser and the supplier.

**4.2** Each container shall be marked with the name of the manufacturer; weight of the material in the container; recognized trade-mark, if any; and the month and year of manufacture.

**4.2.1** The containers may also be marked with the Standard Mark.

##### **4.2.2 BIS Certification Marking**

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016*

and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

## **5 SAMPLING**

### **5.1 Preparation of Test Samples**

The method of drawing representative test samples of the material and the criteria for conformity shall be as prescribed in Annex C.

## ANNEX A

( Clause 2 )

### LIST OF REFERRED STANDARDS

| <i>IS No.</i>   | <i>Title</i>   |
|-----------------|--|
| IS 265 : 2021   | Hydrochloric acid specification ( <i>fifth revision</i> )                              |
| IS 321 : 1964   | Specification for Absolute Alcohol ( <i>first revision</i> )                           |
| IS 336 : 2021   | Ether — Specification ( <i>third revision</i> )  |
| IS 534 : 2021   | Benzene — Specification ( <i>fifth revision</i> )                                      |
| IS 537 : 2011   | Toluene — Specification ( <i>second revision</i> )                                     |
| IS 695 : 2020   | Acetic Acid — Specification ( <i>fourth revision</i> )                                 |
| IS 718 : 2023   | Carbon Tetrachloride — Specification ( <i>third revision</i> )                         |
| IS 1070 : 2023  | Reagent Grade Water — Specification ( <i>fourth revision</i> )                         |
| IS 1745 : 2018  | Petroleum hydrocarbon solvents — Specification ( <i>third revision</i> )               |
| IS 2362 : 1993  | Determination of water by karl fischer method - Test method ( <i>second revision</i> ) |
| IS 2618 : 2016  | Laboratory glassware - Test tubes ( <i>third revision</i> )                            |
| ISO 4142 : 2002 |  |
| IS 2619 : 2018  | Glass beakers — Specification ( <i>third revision</i> )                                |

## ANNEX B

( Clause 3.4 and Table 1 )

### ANALYSIS OF CASHEWNUT SHELL LIQUID ( CNSL )

#### B-1 QUALITY OF REAGENTS

**B-1.1** Unless specified otherwise, pure chemicals and distilled water (*see IS 1070* ) shall be used in all tests.

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

#### B-2 DETERMINATION OF SPECIFIC GRAVITY

##### B-2.1 Principle

The specific gravity of the material determined with a specific gravity bottle.

##### B-2.2 Preparation of the Sample

Filter the material through filter paper, glass wool or any other suitable filtering medium to remove traces of suspended impurities. Suction may be used if necessary.

### **B-2.3 Apparatus**

#### **B-2.3.1 *Thermometer***

Any convenient thermometer of a suitable range with 0.1 or 0.2°C subdivisions.

#### **B-2.3.2 *Water-Bath***

#### **B-2.3.3 *Specific Gravity Bottle***

A specific gravity bottle of about 50-ml capacity with well-fitted ground glass joints and of the shape as shown in Fig. 1 is recommended. To calibrate, clean and dry the bottle thoroughly, weigh and then fill with boiled and cooled water at 30°C. Fill to overflowing by holding the bottle on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper and immerse in a water-bath at  $(30 \pm 0.2)$  °C. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool to room temperature and weigh. Calculate the weight of water. This is a constant for the bottle but should be checked periodically.

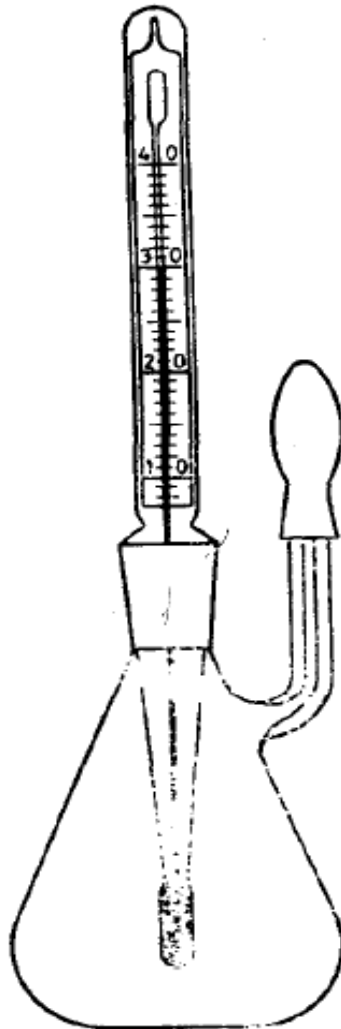


FIG. 1 50 ML SPECIFIC GRAVITY BOTTLE

#### B-2.4 Procedure

Fill the specific gravity bottle with the material to over-flowing, holding the bottle on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper, immerse in the water-Bath maintained at  $(30.0 \pm 0.2)^{\circ}\text{C}$  and hold for 30 minutes. Carefully wipe off any oil which has come through the capillary opening. Remove the bottle from the bath and wipe it completely dry. Cool to room temperature and weigh.

#### B-2.5 Calculation

$$\text{Specific gravity at } 30/30^{\circ}\text{C} = \frac{A - B}{C - B}$$

where



$A$  = weight in g of the specific gravity bottle with material at 30°C,

$B$  = weight in g of the empty specific gravity bottle, and

$C$  = weight in g of the specific gravity bottle with water at 30°C.

## **B-3 DETERMINATION OF VISCOSITY**

### **B-3.1 Principle**

The viscosity of the material is determined with a U-tube viscometer.

### **B-3.2 Apparatus**

#### **B-3.2.1 *U-Tube Viscometer***

The U-tube viscometer shown in Fig. 2 shall be of homogeneous transparent glass and shall be free from mechanical imperfections. All glass tubing employed in the construction of the viscometer shall be of the same composition and the finished instrument shall be thoroughly annealed.

#### **B-3.2.2 *Thermometer***

Any convenient thermometer of a suitable range with subdivisions of 0.2 °C.

#### **B-3.2.3 *Bath***

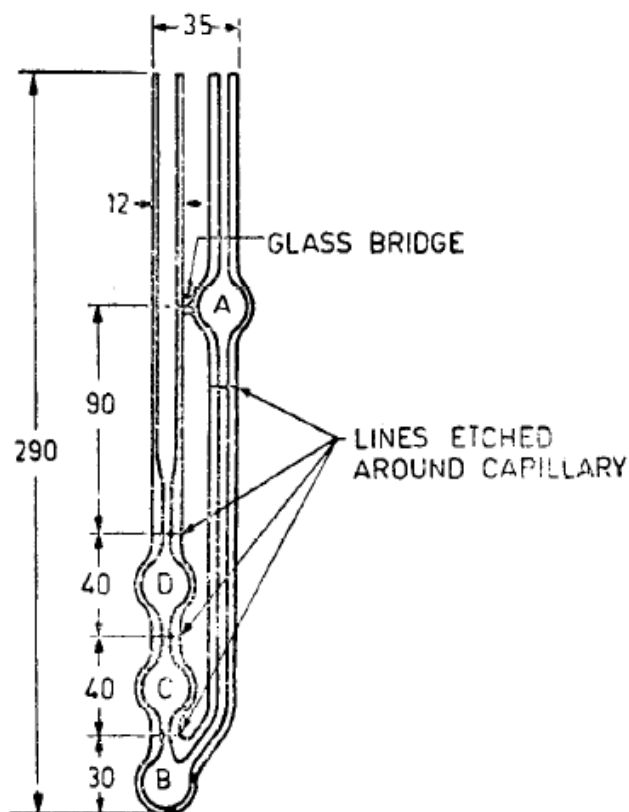
A water bath, suitable for immersion of the viscometer to within 5 cm of the top, with provision for visibility of the instrument and the thermometer, shall be provided. The viscometer may be fixed as an integral part of the bath. The thermometer shall be mounted with the bulb near the centre of the viscometer. The bath shall be regulated so that the variation of the thermometer reading does not exceed 0.2°C. Use of thermoregulatory is recommended.

#### **B-3.2.4 *Time-Recording Device***

a suitable stop watch.

#### **B-3.2.5 *Frame***

Suitable frames shall be provided so that the viscometers may be suspended securely in the bath in a vertical position.



All dimensions in millimetres.

FIG. 2 U-TUBE VISCOMETER

### B-3.3 Procedure

**B-3.3.1** Clean the viscometer by rinsing with suitable solvents, such as benzene ( *see* IS 534 : 1974\* ) followed by ethyl ether ( *see* IS 336 : 1973† ) or petroleum hydrocarbon solvents ( *see* IS 1745 : 1978‡ ). Remove each solvent by passing a current of dry air through it and take care that no moisture remains inside the instrument.

**B-3.3.2** Fill the viscometer by holding it in an inverted position with the capillary side submerged in the material under test. Apply suction to the arm of the viscometer either by sucking through a piece of rubber tubing or a water aspirator. Fill the main line reservoir A and bring the liquid into the capillary to the etched line just below A. Wipe the excess liquid off the end of the capillary arm and incline the instrument slightly to cause the oil to flow by gravity from the upper capillary into A. Discharge this small amount of liquid into the main capillary leading to bulb B. Slip a short piece of rubber tubing with a pinch clamp over the open end of the upper capillary to close one end. Turn the viscometer to a vertical position and place in the constant temperature bath maintained at  $(30 \pm 0.2)^{\circ}\text{C}$ . During this time, prevent the entrance of dust and moisture. Remove the pinch clamp when the equilibrium is reached and allow the liquid to flow by gravity. Start the stop watch just when the liquid passes the etched line between B and C. Note the time required for

the meniscus to reach the etched line above  $C$  [ efflux time (i) ] and then to the etched line above  $D$  [efflux time (ii) ].

**B-3.3.3** The bulb  $B$ , which the liquid enters at the base of capillary is for the purpose of allowing some liquid to discharge from the upper reservoir  $A$  and thus the diameter of the meniscus in this bulb is large throughout the test. This reduces both the effect of loading errors and surface tension corrections.

### **B-3.4 Calculation**

**B-3.4.1** Calculate the kinematic viscosity in centistokes from each efflux time by the formula given below. The viscosities so calculated separately for each bulb should agree:

$$V = C t$$

where

$V$  = kinematic viscosity in centistokes,

$C$  = the determined calibration constant for the instrument ( *see* **B-3.5** ), and

$t$  = time of flow in seconds.

**B-3.4.2** Calculate the dynamic viscosity in centipoises from the following:

Dynamic viscosity in cp = Kinematic viscosity in cs  $\div$  specific gravity ( *see* **B-2.5** )

### **B-3.5 Determination of Calibration Constant**

**B-3.5.1** Unless the calibration constant is already known, calibrate the viscometer using, as primary standard, oil samples the viscosities of which have been determined by the National Physical Laboratory, New Delhi, or any other Institution recognized by the Government of India.

**B-3.5.2** Determine, in the viscometer being calibrated, the flow time of the appropriate standard, which shall not be less than 200 seconds.

**B-3.5.3** Then calculate the calibration constant  $C$ , by substituting in the equation under **B-3.4.1** the flow time and the kinematic viscosity of the oil.

**B-3.5.4** The calibration constant shall be determined at a temperature of  $(30 \pm 0.2)^\circ\text{C}$ .

## **B-4 DETERMINATION OF MATTER INSOLUBLE IN TOLUENE**

### **B-4.1 Reagent**

**B-4.1.1** *Toluene* — conforming to IS 537.

**B-4.2 Procedure** - Weigh accurately about 50 g of the well-mixed material into a conical flask and dissolve by warming on a water-bath, in four times its volume of toluene, the flask being loosely covered. Then filter the solution through a weighed sintered glass crucible ( G No. 1 ), previously dried at  $(100 \pm 2)^\circ\text{C}$  ( *see* Note ). Transfer any insoluble residue to the crucible by

means of additional toluene and wash the residue with toluene until a few drops of the filtrate yield no residue on evaporation. Dry the crucible at  $(100 \pm 2)^{\circ}\text{C}$  to constant weight.

NOTE — In place of sintered glass crucible, counterpoised double filter papers ( for example, Whatman No. 5 ) about 150 mm in diameter may be used. Reduce the two folded filter papers to equal weight by removing the apex of the heavier filter paper. Heat the two to a temperature of  $(100 \pm 2)^{\circ}\text{C}$ . For filtering, use the uncut paper inside the originally heavier paper so that the separated material is retained by the inner paper, yet both of them are equally subjected to any action exerted by the oil and toluene.

### B-4.3 Calculation

$$\text{Matter insoluble in toluene, percent by weight} = \frac{100 w}{W}$$

where

$w$  = weight in g of residue, and

$W$  = weight in g of the material taken for the test.

## B-5 DETERMINATION OF LOSS IN WEIGHT ON HEATING

### B-5.1 Procedure

Weigh accurately 100 g of the material in a 400-ml beaker. Heat the beaker together with the material for 30 minutes on an electric hot-plate maintained at  $(205 \pm 5)^{\circ}\text{C}$  and stir the material after every two minutes. At the end of this period, remove the beaker, allow it to cool to room temperature and weigh again.

### B-5.2 Calculation

$$\text{Loss in weight on heating, percent by weight} = \frac{100 w}{W}$$

where

$w$  = loss in weight in g of the material on heating, and

$W$  = weight in g of the material taken for the test.

## B-6 DETERMINATION OF ASH

### B-6.1 Apparatus

**B-6.1.1 Dish** — made of platinum, porcelain, or silica, or any wide-form crucible of suitable capacity.

### B-6.2 Reagents

**B-6.2.1 Ethyl Alcohol** — conforming to IS 321

**B-6.2.2 Benzene** — conforming to IS 1840

### B-6.3 Procedure

Heat the dish to redness. Allow it to cool in a desiccator and weigh it to the nearest 0.1 mg. Weigh accurately, to the nearest 0.1 mg, about 2 g of the material from a weighing bottle into the dish. Heat the dish gently by means of a burner until the material can be ignited at the surface ( *see Note* ). Allow the combustible matter to burn off slowly and heat the residue with a strong flame or in a muffle furnace until the ash is free from carbonaceous matter. Cool the dish and its contents in a desiccator and weigh. Repeat heating and cooling till constant weight is obtained.

NOTE — In the case of materials containing sufficient moisture to cause foaming and loss, add one to two millilitres of ethyl alcohol before heating. If excessive foaming still occurs notwithstanding this treatment, add 10 ml of a mixture of equal volumes of benzene and ethyl alcohol and stir thoroughly with the material. Insert several strips of ashless filter paper into the mixture and then ignite. When most of the moisture is removed, the paper itself begins to burn procedure given in **B-6.3** shall be followed. After this stage, the procedure given in **B-6.3** shall be followed.

#### B-6.4 Calculation

$$\text{Ash, percent by weight} = \frac{100 w}{W}$$

where

$w$  = weight in g of the ash, and

$W$  = weight in g of the material 'taken for the test.

### B-7 DETERMINATION OF IODINE VALUE

#### B-7.1 Principle

The material is treated in carbon tetrachloride medium, with a known excess of iodine monochloride solution in glacial acetic acid (Wij's solution). The excess of iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulphate solution.

#### B-7.2 Reagents

- a) *Potassium Dichromate* — conforming to IS 250.
- b) *Concentrated Hydrochloric Acid* — conforming to IS 265.
- c) *Potassium Iodide Solution* — Prepare a fresh solution by dissolving 10 g of potassium iodide free from potassium iodate, in 5.0 ml of water.
- d) *Starch Solution* — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.
- e) *Standard Sodium Thiosulphate Solution* — approximately 0.1 N. Dissolve approximately 24.8 g of sodium thiosulphate crystals (  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  ) in water which has been well boiled to free it from carbon dioxide and make up to 1000 ml. Store the solution in a cool place in a dark coloured stock bottle with a guard tube filled with soda lime. After storing the solution for about two weeks, filter if necessary and standardize it as prescribed below:

Weigh accurately about 5.0 g of finely ground potassium dichromate which has been previously dried to a constant weight at  $(105 \pm 2)^{\circ}\text{C}$  into a clean one-litre volumetric flask. Dissolve in water, make up to the mark; shake thoroughly and keep the solution in a cool dark place. For standardization of sodium thiosulphate, pipette 25 ml of this solution into a clean glass stoppered 250-ml conical flask or bottle. Add 5 ml of concentrated hydrochloric acid and 15 ml of a 10 percent potassium iodide solution. Allow to stand in the dark for 5 minutes and titrate the mixture with the solution of sodium thiosulphate, using starch solution as an internal indicator towards the end. The end point is taken when the blue colour changes to green. Calculate the normality ( $N$ ) of the sodium thiosulphate solution as follows:

$$N = \frac{25 W}{49.03 V}$$

where

$W$  = Weight in g of the potassium dichromate, and

$V$  = volume in ml of sodium thiosulphate solution required for the titration.

- f) *Iodine Crystals* — re-sublimed.
- g) *Acetic Acid* — glacial, 99 percent ( *see* IS 695 ) and free from reducing impurities. Test for reducing impurities is as follows:

Dilute 2 ml of the acetic acid with 10 ml of water and add 0.1 N potassium permanganate solution and maintain at  $(27 \pm 2)^{\circ}\text{C}$ . The test shall be taken as having been satisfied if the pink colour is not discharged at the end of two hours.

- h) *Chlorine Gas* — dry.
- j) *Iodine Trichloride* ( $\text{ICl}_3$ )
- k) *Iodine Monochloride* ( $\text{ICl}$ ) — 98 percent, chemically pure.
- m) *Wij's Iodine Monochloride Solution* — Prepare this solution by one of the following two methods, and store in a glass stoppered bottle in a cool place, protected from light:
  - i) Dissolve 13 g of iodine in one litre of acetic acid, using gentle heat, if necessary, and determine the strength by Titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine-acetic acid solution. A slight excess of iodine does no harm, but avoid an excess of chlorine.  
*Example:*  
If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate 20 ml of the finished Wij's solution shall require between 43 to 44 ml ( and not more than 44 ml ) of the same sodium thiosulphate solution.
  - ii) Dissolve eight grams of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately nine grams of iodine in 450 ml of acetic acid, using heat, if necessary. Add gradually the iodine solution to the iodine trichloride until the colour has changed to reddish-brown. Add 50 ml more of iodine solution and dilute

the mixture with acetic acid till 10 ml of the mixture is equivalent to 20 ml of standard thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to 100°C for 20 minutes, and cook. Prevent access of water vapour in preparing the solution.

- n) *Carbon Tetrachloride or Chloroform* — inert to Wij's solution.

### B-7.3 Procedure

Take 50 g of the cashewnut shell liquid in a 250-ml beaker and heat slowly to  $(205 \pm 5)^\circ\text{C}$  on an electric hot plate stirring thoroughly to prevent violent foaming. As soon as this temperature is reached cover the beaker and cool it undisturbed to room temperature. Filter the material through a filter paper to remove any impurities. Make sure that the glass apparatus used is absolutely clean and dry. Weigh accurately by difference about 0.10 to 0.12 g of the filtered material in a clean dry 500-ml iodine flask or well ground glass-stoppered bottle to which 25 ml of carbon tetrachloride has been added and agitate to dissolve the contents. The weight of the sample shall be such that there is an excess of Wij's solution 50 to 60 percent of the amount added. Add 25 ml of the Wij's solution and replace the glass stopper after wetting with potassium iodide solution; swirl for intimate mixing, and allow to stand in the dark for one hour. Carry out a blank test simultaneously under similar experimental conditions. After standing, add 15 ml of potassium iodide solution and 100 ml of water, rinsing in the stopper also, and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the contents of the bottle continuously to avoid any local excess until the colour of the solution is straw yellow. Add one millilitre of the starch solution and continue the titration until the blue colour formed disappears after thorough shaking with the stopper on.

### B-7.4 Calculation

$$\text{Iodine value} = \frac{12.69 (B - S) N}{W}$$

where

$B$  = volume in ml of standard sodium thiosulphate solution required for the blank,

$S$  = volume in ml of standard sodium thiosulphate solution required for the sample,

$N$  = normality of the standard sodium thiosulphate solution, and

$W$  = weight in g of the material taken for the test.

## B-8 DETERMINATION OF POLYMERIZATION TIME

### B-8.1 Principle

It is the time taken by de-hydrated cashewnut shell liquid to gel after the addition of concentrated sulphuric acid in diethyl sulphate.

### B-8.2 Reagents

#### B-8.2.1 Concentrated Sulphuric Acid

### **B-8.2.2** *Diethyl Sulphate*

NOTE — Diethyl sulphate is poisonous and should be handled with care.

## **B-8.3 Procedure**

### **B-8.3.1** *Dehydration of Material*

Pour 50 g of the material into a 250-ml beaker ( *see* IS 2619 ) and heat slowly to  $(205 \pm 5)^{\circ}\text{C}$  with continuous stirring till foaming ceases.

**B-8.3.2** *Polymerization Test* — Take  $(5.0 \pm 0.1)$  g of the dehydrated oil into a  $150 \times 15$  mm test-tube ( *see* IS 2618 ). Add through a dropping pipette 0.5 ml of 25 percent solution ( *v/v* ) of concentrated sulphuric acid in diethyl sulphate and stir the mixture thoroughly with a glass rod of three millimetres in diameter and rounded at the end. Make sure that a thorough mixture is obtained after the addition of the sulphuric acid-diethyl sulphate solution, to avoid local resin formation, which would vitiate the result. Insert the tube into an oil bath which has been heated to  $(176 \pm 1)^{\circ}\text{C}$  and note the time of insertion with a stop watch. Stir the material slowly. It will be noted that the sample becomes more and more viscous. Note the time when the material suddenly ‘gels’ or sets to a stiff rubbery pasty mass or a dry rubbery mass. Report the time taken as the polymerization time.

## **B-9 DETERMINATION OF POLYMERIZATION BY VISCOSITY METHOD**

### **B-9.1 Principle**

It is determined by finding out the viscosity of the material when mixed with diethyl sulphate and kept in an oven at  $(185 \pm 1)^{\circ}\text{C}$  for one hour and finally dissolved in xylene.

### **B-9.2 Reagents**

#### **B-9.2.1** *Diethyl Sulphate*

NOTE — Diethyl sulphate is poisonous and should be handled with care.

#### **B-9.2.2** *Xylene*

**B-9.3 Procedure** — Weigh 200 g of the material into a 500-ml beaker ( *see* IS 2619 ) and add eight grams of diethyl sulphate. Reweigh the beaker and contents. Mix the contents thoroughly and place the beaker in an oven at  $(185 \pm 1)^{\circ}\text{C}$  for one hour. Remove the beaker from the oven and add 200 g of xylene. Mix well and cool to room temperature. Make up for any loss in xylene so as to have exactly 200 g of xylene in the mixture. Determine the viscosity of the diluted solution at  $(30 \pm 0.2)^{\circ}\text{C}$  using a U-tube viscometer as prescribed in **B-3** or any other suitable viscometer.

## **B-10 DETERMINATION OF POLYMERIZATION BY VISCOSITY METHOD AFTER ACID WASHING**

### **B-10.1 Principle**

It is determined by finding out the viscosity of the material after washing with dilute sulphuric acid which is then mixed with diethyl sulphate and kept in an oven at  $(185 \pm 1)^{\circ}\text{C}$  for one hour and finally dissolved in xylene.



## **B-10.2 Reagents**

**B-10.2.1 Dilute Sulphuric Acid** — 5 percent solution.

**B-10.2.2 Diethyl Sulphate**

NOTE — Diethyl sulphate is a poisonous material and should be handled with care.

**B-10.2.3 Xylene**

## **B-10.3 Procedure**

Take approximately 500 g of the material in 1000-ml beaker or flask. Add 200 ml of dilute sulphuric acid and mix thoroughly. Heat the solution on a water-bath maintained at (90 to 95) °C for five minutes and then separate the two layers by any suitable method. The separation may best be achieved by centrifuging the hot mixture. Transfer this mixture to the separating funnel and allow it to stand for ten minutes. Discard the lower aqueous layer and collect the upper layer of the cashewnut shell liquid from the separating funnel. Repeat treatment of the separated CNSL layer with sulphuric acid and separate the two layers. Add 200 ml of water to the separated CNSL, mix it thoroughly and heat the mixture of (90 to 95) °C for five minutes. Separate the layers by centrifuging or any other suitable method. Test the pH of the aqueous layer is below five, repeat the treatment of the above separated CNSL layer with further quantity of 200 ml of water till the pH of the aqueous layer obtained is above five. Weigh 200 g of the separated CNSL into a 500 ml beaker ( *see* IS 2619 : 1971) and add eight grams of diethyl sulphate. Reweigh the beaker and the contents. Mix the contents thoroughly and place the beaker in an oven maintained at (185 ± 1) °C for one hour. Remove the beaker from the oven and add 200 ml of xylene. Mix well and cool to room temperature. Make up for any loss in xylene so as to have 200 g of xylene in the mixture. Determine the viscosity of the solution at (30 ± 0.2) °C by using the U-tube viscometer as prescribed in **B-3** or any other suitable viscometer.

## **ANNEX C**

( *Clause 5.1* )

### **SAMPLING OF CASHEWNUIT SHELL LIQUID ( CNSL )**

#### **C-1 GENERAL REQUIREMENTS OF SAMPLING**

**C-1.1** In drawing samples the following precautions and directions shall be observed.

**C-1.2** As the material is vesicant, care shall be taken in opening the drums for sampling.

**C-1.3** Samples shall not be taken in an exposed place.

**C-1.4** The sampling instruments shall be clean and dry when used.

**C-1.5** Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

**C-1.6** The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

**C-1.7** The sample containers shall be of such a size that they are almost completely filled by the sample.

**C-1.8** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the month and year of manufacture of the material.

**C-1.9** All sampling instruments, if made of copper, brass or bronze, shall be nickel plated.

## **C-2 SAMPLING INSTRUMENTS**

**C-2.1 Sampling Bottle or Can (see Fig. 4)** — This instrument is suitable for taking samples from various depths in tanks. It consists of a weighed bottle or metal container with removable stopper or cap, to which is attached a suitable chain, pole or cord. This device is lowered to the various desired depths at which the stopper is removed and the container is allowed to fill.

**C-2.2 Open type Sampling Tube (see Fig. 5)** — It is made of metal or thick glass and may be of 20 to 40 mm diameter and 400 to 800 mm in length (see Note). The upper and lower ends are conical and narrow down to 5 to 10 mm diameter. Handling is facilitated by two rings at the upper end. For taking a sample, the instrument is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material, and finally closed and withdrawn.

NOTE — For small containers, the size of the sampling tube may be altered suitably.

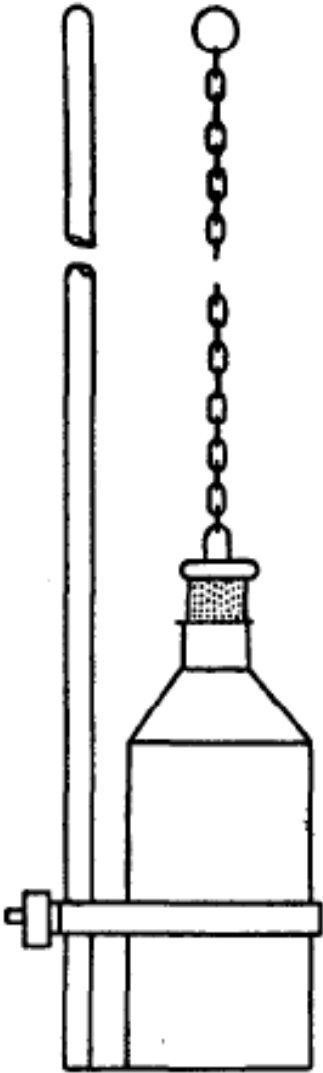
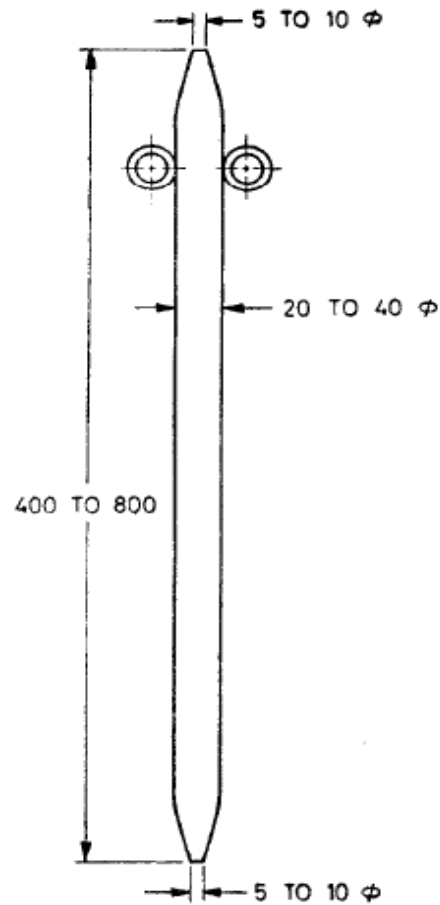


FIG. 4 SAMPLING BOTTLE OR CAN



All dimensions in millimeters.

FIG. 5 OPEN TYPES SAMPLING TUBE

### C-3 SCALE OF SAMPLING

#### C-3.1 Lot

In a single consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of containers of different sizes or of different batches of manufacture, then the containers belonging to the same size and batch of manufacture & all be grouped together and each such group shall constitute a separate lot. In case the consignment is in tanks, the tanks belonging to the same batch of manufacture shall constitute a lot.

**C-3.1.1** For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately.

#### C-3.2 Sampling from Containers

The number ( $n$ ) of containers to be selected for sampling shall depend on the size ( $N$ ) of the lot and shall be in accordance with Table 3. In the case of very small lots where the selection of three

containers may be uneconomical, the method of judging the conformity of the lot to the requirements of the specification shall be as agreed to between the purchaser and the supplier.

**TABLE 3 SCALE OF SAMPLING FOR CONTAINERS**

| SIZES OF THE LOT | NUMBER OF CONTAINERS TO BE<br>SELECTED |
|------------------|--|
| $N$<br>(1)       | $n$<br>(2)                             |
| Up to 20         | 3                                      |
| 21 to 40         | 4                                      |
| 41 to 80         | 5                                      |
| 81 to 120        | 6                                      |
| 121 to 200       | 8                                      |
| 201 and above    | 10                                     |

**C-3.2.1** The containers shall be selected at random and to ensure the randomness of selection, random number tables may be used. In case, such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1,2,3,..., etc, up to  $Y$  and so on, where  $r$  is the integral part of  $N/n$  ( $N$  being the lot size and  $n$  the number of containers to be selected ). Every  $r$ th container thus counted shall be withdrawn to give sample for test.

**C-3.3 Sampling from Tanks**

Each tank in the lot shall be sampled separately for determining the conformity of the lot to the requirements of the specification.

**C-4 PREPARATION OF TEST SAMPLES**

**C-4.1 Test Samples from Containers** — To ensure that the sample; taken from each container are fairly representative, the contents shall be mixed thoroughly by shaking or stirring or rolling. Draw small samples of the material from various depths with the help of the sampling tube ( *see* Fig. 5 ). The approximate quantity of the material to be drawn from a container shall nearly be equal to thrice the quantity required for test purpose.

**C-4.1.1** Out of the material drawn from individual containers, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

**C-4.1.2** The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  containers selected shall be for the purchaser, another for the supplier and the third for the referee.

**C-4.1.3** All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars given in **C-1.8**.

**C-4.1.4** The referee samples, consisting of a composite sample and a set of II individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

#### **C-4.2 Test Samples from Tanks**

**C-4.2.1** For drawing a sample from a tank, lower the closed sampling bottle or can ( *see C-2.1* ) slowly to the required depth, open and fill it at that depth. Three samples shall be obtained at levels of one-tenth of the depth of the liquid from the top surface ( top sample ), one-half of the depth ( middle sample ) and nine-tenths of the depth of the liquid from the top surface ( lower sample ). All the three samples thus obtained from a tank shall be mixed together in a clean dry container and shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. Each tank in the lot shall be sampled in the above manner and separate samples obtained from each tank. The approximate quantity of the material to be drawn from a tank shall nearly be equal to thrice the quantity required for carrying out tests for the requirement prescribed in this standard.

**C-4.2.2** All the samples thus obtained from tanks in the lot shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **C-1.8**.

**C-4.2.3** The referee test samples, consisting of the samples from the tanks in the lot, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

#### **C-5 NUMBER OF TESTS**

##### **C-5.1 For Samples from Containers**

**C-5.1.1** Test for the determination of iodine value shall be conducted on each of the individual samples separately.

**C-5.1.2** Tests for the determination of all the remaining characteristics in the specification shall be conducted on the composite sample.

**C-5.2 For Samples from Tanks** — Tests for the determination of all the characteristics in the specification shall be conducted on the samples from different tanks separately.

##### **C-5.3 Criteria for Conformity**

###### **C-5.3.1 For Containers**

**C-5.3.1.1 For individual samples** — For the iodine value which shall be determined on the individual samples, the mean (  $\bar{X}$  ) and range (  $R$  ) of the test results shall be calculated as follows:

$$\text{Mean ( } \bar{X} \text{ )} = \frac{\text{the sum of the test results}}{\text{number of the test results}}$$

Range (  $R$  ) = the difference between the maximum and the minimum values of the test results

NOTE — For declaring the conformity of the lot to the requirement of iodine value, the value of the expressions (  $\bar{X} - 0.6 R$  ) as calculated from the relevant test, results shall be not less than 250 or 375, as the case may be.

**C-5.3.1.2** *For composite samples*

For declaring the conformity of the lot to the requirements of all the other characteristics determined on the composite samples, the test results for each of the characteristic shall satisfy the relevant requirements given in the specification.

**C-5.3.2** *For Tanks*

**C-5.3.2.1** The lot shall be declared as conforming to the specification requirements of various characteristics, if each of the test results satisfies individually the relevant requirements specified in the standard.