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भारतीय मानक मसौदा

नमूनाकरण और परीक्षण पद्धतियाँ — वेजिटेबल गोंद

(आई. एस. 7437 का पहला *पुनरीक्षण*)

Draft Indian Standard

METHODS OF SAMPLING AND TEST — VEGETABLE ADHESIVES

(*First Revision of* IS 7437)

(ICS 83.180)

Methods of Sampling and Test for Plastics Sectional Committee, PCD 27	Last date for Comments: **12 September 2025**

FOREWORD

(*Formal clauses to be added later*)

This standard was originally published in 1974. This revision (first) has been undertaken to:

— update the cross referred standards; and
— make the editorial corrections.

The methods are intended to apply to adhesives manufactured from materials of vegetable origin, such as starch or materials containing a high proportion of starch, gum arabic, alginates and water soluble ethers of cellulose. Typical examples of such adhesives are crystal gums, dextrin and water-soluble pastes and powders. This standard is not intended to apply to rubber latex or to adhesives used for miscellaneous purposes, such as binders for pigments and coatings for papers.

The relative importance of the tests described depends on the purpose for which the adhesive is intended. In practice, it is customary for the user to determine by experience the most suitable type of adhesive for this purpose. The problem then reduces itself to checking future deliveries in respect of those properties which are relevant. Some of the physical tests, for example, the foam test, are of an empirical nature, but if these methods are carefully followed in every detail, it is possible to obtain consistent results which will give useful information both to the manufacturer and the user.

Original standard was published by taking reference from BS 844: 1965 ‘Methods of sampling and testing vegetable adhesives’, published by the British Standards Institution.

In reporting the result of a test 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: 2022 ‘Rules for rounding off numerical values (*second revision*)’.

1 SCOPE

This standard prescribes the methods of sampling and test for adhesives manufactured from materials of vegetable origin. It applies to adhesives in the form of liquid, pastes, powders or granular solids.

2 REFERENCES

The standards given below 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 edition of these standards.

IS No.	*Title*
IS 266 : 2024	Sulphuric acid — Specification (*fourth revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 460 (Part 1) : 2020	Test Sieves — Specification : Part 1 Wire cloth test sieves (*fourth revision*)
IS 460 (Part 2) : 2020	Test Sieves — Specification : Part 2 Perforated plate test sieves (*fourth revision*)
IS 878 : 2008	Laboratory glassware — Graduated measuring cylinders (*second revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 2088 : 2023	Methods for determination of arsenic (*third revision*)
IS 3434 : 1984	Glossary of terms for adhesives and pressure sensitive adhesive tapes (*first revision*)
IS 4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 3434 shall apply.

4 QUALITY OF REAGENTS

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

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

5 SAMPLING

5.1 General Requirements of Sampling

5.1.1 In drawing, preparing, strong and handling test samples, the following precautions and directions shall be observed.

5.1.1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the container for samples from adventitious contamination.

5.1.1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

5.1.1.3 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

5.1.1.4 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

5.2 Scale of Sampling

5.2.1 *Lot*

All the containers in a single consignment of the material of the same grade drawn from a single batch of processing shall constitute a lot. If a consignment is declared or known to consist of different batches of processing, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

5.2.1.1 Tests for determining the conformity of the lot to the requirements of the specifications shall be done on each lot separately.

5.2.2 The number of containers (n) to be sampled shall be in accordance with col (2) and (3) of Table 1.

5.2.3 The containers shall be selected at random and in order to ensure the randomness of selection, a random number table (*see* IS 4905) shall be used. In case such a table is not available, the following procedure may be adopted:

> Starting from any container, count them 1, 2, 3. . . etc., *up* to r and so on in a systematic manner, where r is the integral part of N/n. Every r^{th} container thus counted shall be withdrawn from the lot.

Table 1 Number of Containers to be Selected for Sampling
(*Clauses* 5.2.2 and 5.3.1)

Sl No. (1)	Lot Size N (2)	Number of Containers to be Selected n (3)
i)	3 to 50	3
ii)	51 to 200	4
iii)	201 to 400	5
iv)	401 to 650	6
v)	651 to 1 000	7

5.3 Test Samples and Referee Sample

5.3.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 1). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics and shall be not less than 0.2 kg.

5.3.1.1 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 0.5 kg. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

5.3.2 The referee sample shall consist of the composite sample marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place for a period as agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

5.4 Criteria for Conformity — Criteria for conformity shall be given in the individual specifications.

6 DETERMINATION OF VOLATILE MATTER

6.1 Terminology

6.1.1 *Volatile Matter* — The content of volatile matter is defined as the percentage loss in mass of the sample as received, when the adhesive is dried at (105 ± 2) °C to constant mass.

6.2 Apparatus

6.2.1 *Stainless Steel Dish* — flat bottomed, of 70 mm diameter and 15 mm high, weighing about 30 g, fitted with a stainless steel cover

6.2.2 *Sieve* — 425 micron IS Sieve.

6.2.3 *Oven* — maintained at (105 ± 2) °C.

6.2.4 *Water-Bath* — maintained at boiling point.

6.3 Procedure

6.3.1 *For Solid Adhesives* — Weigh approximately 1 g of the adhesive to the nearest 0.001 g into a clean, dry dish. If necessary, grind the sample so that it passes through a 425 micron IS sieve.

NOTE — In preparing the sample for the test it should be remembered that the adhesive, especially in the finely divided state, may alter rapidly in moisture content according to its nature and the humidity of the air to which it is exposed. It is, therefore, important that the operations described above should be carried out promptly and in surroundings which are not excessively humid or dry. There should be no delay in transferring the sample to an air-tight container pending weighing.

6.3.1.1 Remove the cover and heat the sample at (105 ± 2) °C in the oven for 2 h. Cover the dish, cool in a desiccator and weigh. Replace the dish in the oven, remove the cover and continue heating for 1 h . Again, cover the dish, cool in a desiccator and weigh. Repeat the cycle of heating for 1 h, cooling and weighing, until two successive weighing's do not differ by more than 0.001 g. The adhesive is then considered to be free from all volatile matter.

6.3.2 *For Liquids and Pastes* — Weigh, to the nearest 0.001 g, a quantity of the adhesive containing about 1 g of volatile-free material. A preliminary test may be performed to determine the approximate content of volatile matter. Transfer the sample quickly to a suitable tared dish fitted with a lid and spread it roughly over the bottom of the dish with a rod or spatula (which is then discarded). Replace the lid and re-weigh the dish with contents without delay. Add 10 ml of hot water. Heat on a boiling water bath until the adhesive is homogeneous and forms a uniform thin film on the bottom of the dish. Take care to avoid loss of material during the process. Continue to heat on the boiling water bath until the adhesive is apparently dry.

6.3.2.1 Place the dish and contents in the air oven at (105 ± 2) °C and heat for 3 h. Cover the dish, cool it in a desiccator and weigh. Replace the dish in the oven, remove the cover and continue the heating for 1 h . Again cover the dish, cool it in a desiccator, and weigh. Repeat the cycle of heating for 1 h, cooling and weighing, until two successive weighing's do not differ by more than 0.001 g. The adhesive is then considered to be dry.

6.4 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass, in g, of the sample taken for the test; and
M_2 = mass, in g, of direct sample.

7 DETERMINATEON OF ASH

7.1 Procedure

Accurately weigh about 3 g to 5 g of the test sample in a tared silica dish. Gently heat the dish on a hot plate until the sample is thoroughly carbonized and then increase the heat until carbonization is complete. Transfer the dish and its contents to a muffle furnace and ash at about 550 °C to a constant mass.

7.2 Calculation

$$\text{Ash, percent by mass (on dry basis)} = \frac{10\,000}{(100 - M)} \times \frac{M_1}{M_2}$$

where

M_1 = mass, in g, of the ash;
M = percent volatile matter content (as determined in **6**); and

M_2 = mass, in g, of the sample taken for the test.

8 DETERMINATION OF ACID-INSOLUBLE ASH

8.1 Reagents

8.1.1 *Rectified Spirit* — conforming to IS 323.

8.1.2 *Dilute Hydrochloric Acid* — approximately 4 N.

8.2 Procedure

8.2.1 Accurately weigh about 3 g of the ground sample in a tared crucible and incinerate at a low temperature until free from carbon. If a carbon-free ash is not obtained, cool the crucible. Add 15 ml of rectified spirit, break up the ash with a glass rod, burn off the alcohol and again heat the whole mass to low redness.

8.2.2 Boil the ash obtained with 25 ml of dilute hydrochloric acid for 5 min. Collect the insoluble matter in a tared Gooch crucible or an ash less filter paper. Wash with hot water, ignite and weigh.

8.3 Calculation

$$\text{Acid-insoluble ash, percent by mass (on dry basis)} = \frac{10\,000\,M}{M_1(100 - M_2)}$$

where

M = mass, in g, of the acid-insoluble residue,
M_1 = mass, in g, of the material taken for the test; and
M_2 = percent volatile matter in the material (*see* **6**).

9 DETERMINATION OF COLD WATER SOLUBLES

9.1 Procedure

Accurately weigh about 5 g of the test sample and make it into a uniform slurry with about 80 ml of water at 30 °C. Dilute to about 200 ml in a 250 ml volumetric flask. Shake it for an hour and make up the volume to 250 ml. Filter the contents discarding the first 20 ml of the filtrate and collect exactly 50 ml of the filtrate in a tared dish. Evaporate to dryness on a steam-bath and dry the residue to constant mass in a drying oven at 100 $^{\circ}$C to 105 $^{\circ}$C.

9.2 Calculation

$$\text{Cold water solubles, percent by mass (on dry basis)} = \frac{M \times 5 \times 10\,000}{M_1(100 - M_2)}$$

where,

M = mass, in g, of the residue,

M_1 = mass, in g, of the sample taken for the test; and
M_2 = percent volatile matter content of the sample (*see* **6**).

10 DETERMINATION OF *p*H

10.1 Procedure

Weigh to the nearest 0.05 g a quantity of the adhesive containing about 1 g of volatile-free matter. Disperse this in freshly boiled water, cool and dilute to a final volume of 100 ml. Determine *p*H of this dispersion at 27 ^{o}C to the nearest 0.1 *p*H unit, preferably using a glass electrode.

NOTE — In practice, it is sometimes desirable to obtain a reading of the *p*H value of the adhesive as it is actually used rather than on dilution.

11 DETERMINATION OF ACIDITY

11.1 Reagents

11.1.1 *Standard Sodium Hydroxide Solution* — 0.1 N

11.1.2 *Phenolphthalein Indicator Solution* — 0.1 percent solution in rectified spirit.

11.1.3 *Neutral Distilled Water*

To 100 ml of freshly boiled and cooled distilled water, add a few drops of phenolphthalein indicator solution and then add, drop by drop, standard sodium hydroxide solution until a permanent faint pink colour is produced.

11.2 Procedure

Accurately weigh about 10 g of the test sample into a 250 ml beaker. Add about 100 ml of neutral distilled water and 2 drops of phenolphthalein indicator solution. Stir the contents well with the glass rod taking care that no drops splash out. Titrate with standard sodium hydroxide solution with the solution just changes to permanent pink.

11.3 Calculation

$$\text{Acidity (as hydrochloric acid), percent by mass (on dry basis)} = \frac{365\,V\,N}{M(100-M_1)}$$

where

V = volume, in ml, of standard sodium hydroxide solution used in the titration;
N = normality of standard sodium hydroxide solution;
M = mass, in g, of the sample taken for the test; and
M_1 = percent volatile matter content of the sample (as determined in **6**).

12 DETERMINATION OF ALKALINITY

12.1 Reagents

12.1.1 *Hydrochloric Acid* — 0.1 N, standardized freshly.

12.1.2 *Methyl Orange Indicator* — 0.05 percent solution.

12.1.3 *Neutral Distilled Water* — same as in **11.1.3.**

12.2 Procedure

Accurately weigh about 10 g of the test sample into a 250 ml beaker. Add about 100 ml of neutral distilled water and 2 drops of methyl orange indicator solution. Stir the contents well with a glass rod, taking care that no drops splash out. Titrate with standard hydrochloric acid until end point is reached.

12.3 Calculation

Alkalinity (as sodium hydroxide), percent by mass (on dry basis) = $\frac{400\,V\,N}{M\,(100 - M_1)}$

where

V = volume, in ml, of standard hydrochloric acid used in the titration;
N = normality of standard hydrochloric acid;
M = mass, in g, of the sample taken for the test; and
M_1= percent volatile matter content of the sample (*see* **6**).

13 TEST FOR CHLORIDES

13.1 Apparatus

13.1.1 *Nessler Tubes* — 50 ml capacity.

13.2 Reagents

13.2.1 *Dilute Nitric Acid* — approximately 4 N.

13.2.2 *Silver Nitrate Solution* — 4 percent.

13.2.3 *Standard Hydrochloric Acid* — exactly 0.02 N.

13.3 Procedure

13.3.1 Weigh 2 g of the material and transfer it to a beaker containing 50 ml of a mixture of equal volumes of dilute nitric acid and water. Cover with a watch glass and boil gently until the mixture loses its viscosity (about 30 min). Filter through a filter paper, wash the residue with water till sulphates and chlorides are washed out, and make up the volume of the combined filtrate and washings to 100 ml. Use this solution for tests of chlorides and sulphates (**14**).

13.3.2 Transfer 25 ml of the solution (**13.3.1**) to a Nessler tube, add 1 ml each of dilute nitric acid and silver nitrate solution, dilute to 50 ml and mix well. Allow to stand for 5 min, protected from direct sunlight. Carry out simultaneously a control test in another Nessler tube using 0.2 ml of standard hydrochloric acid and compare the turbidity in the two Nessler tubes.

13.3.3 The material shall be taken to have passed the test if the turbidity with the material is not greater than that produced in the control test.

14 TEST FOR SULPHATES

14.1 Apparatus

14.1.1 *Nessler Tubes* — 50 ml capacity.

14.2 Reagents

14.2.1 *Dilute Hydrochloric Acid* — approximately 4 N.

13.2.2 *Barium Chloride Solution* — 10 percent.

14.3 Procedure

Transfer 25 ml of the solution prepared in **13.3.1** to a Nessler tube. Add 1 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Dilute to 50 ml and mix well. Allow to stand for 10 min.

14.3.1 The material shall be taken to have passed the test if no precipitate or turbidity is produced.

15 DETERMINATION OF REDUCIBLE SULPHUR

15.1 Apparatus

15.1.1 *Flat-Bottomed Flask* — 250 ml, having a neck 8 cm long and 25 mm in internal diameter, with a ground flange.

15.1.2 *Glass Tube* — 5 cm long and 25 mm in internal diameter, having a flange ground to a close fit with the flange on the flask.

15.1.3 *Spray Trap* — The spray trap consists of a glass rod with a disc pressed in it so as to fit the neck of the flask closely near to the bottom (*see* Fig. 1).

15.2 Reagents

15.2.1 *Zinc, Granulated* — sulphur-free.

15.2.2 *Orthophosphoric Acid* — sulphur-free.

15.2.3 *Lead Acetate Paper* — Filter paper impregnated with a 10 percent solution of lead acetate and subsequently dried.

15.2.4 *Standard Stains*

Dilute 3 ml of 0.01 N sodium thiosulphate solution to 1 000 ml with water. One millilitre of this solution is equivalent to 1 part per million of reducible sulphur per 2 g of volatile-free adhesive. Prepare standard stains by using aliquot volumes of this solution by the procedure given in **15.3**.

15.3 Procedure

Weigh, to the nearest 0.02 g, a quantity of the adhesive containing approximately 2 g of the volatile-free material, disperse it in 2.5 ml of water and transfer the solution to the flask. Introduce into the flask 10 ml of orthophosphoric acid and 1 g of zinc. Clamp securely a piece of lead acetate paper between the ground surfaces of the neck of the flask and the glass tube, for example, by connecting the clamping hooks on the flask to those on the tube in such a way that all the gases evolved pass through the filter paper. Place a loose wad of cotton wool in the upper end of the glass tube. Heat the flask on a steam-bath for 1 h and, at the end of this period, remove the lead acetate paper and dry it. Compare the sulphide stain produced with the standard stains. Carry out a blank determination using the same conditions of test.

NOTE — When amounts of sulphur greater than 10 parts per million are present, use smaller amounts of the adhesive. Take stringent precautions to keep the adhesive free from dust and laboratory fumes.

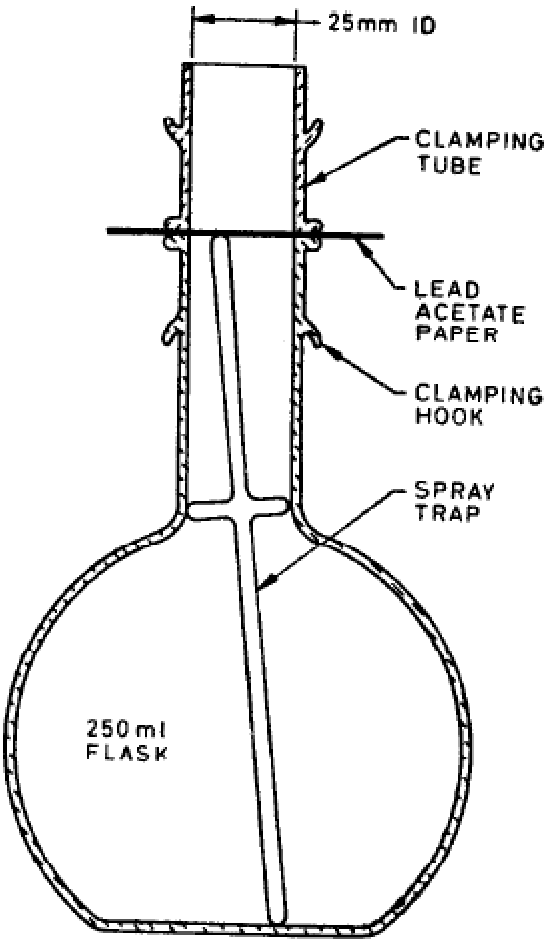

FIG. 1 APPARATUS FOR THE DETERMINATION OF REDUCIBLE SULPHUR

15.4 Report

Report the sulphur content corresponding to the standard stain which most nearly matches the test stain as the reducible sulphur content, in parts per million, of the volatile-free adhesive.

16 DETERMINATION OF BORATE

16.1 Methods

Two methods have been prescribed:

- Method A is recommended for adhesives which are known to be free from silicates or phosphates; and
- Method B for adhesives which contain silicates or phosphates.

16.2 Method A

16.2.1 *Apparatus*

16.2.1.1 *Flask, wide-necked* — 250 ml capacity.

16.2.2 *Reagents*

16.2.2.1 *Mannitol*

16.2.2.2 *Standard sulphuric acid* — 0.2 N

16.2.2.3 *Standard sodium hydroxide solution* — 0.2 N

16.2.2.4 *Phenolphthalein indicator solution* — 0.1 percent

16.2.2.5 *Methyl orange indicator solution* — 0.05 percent

16.2.3 *Procedure*

16.2.3.1 Weigh into the flask, to the nearest 0.001 g, a quantity of the adhesive containing approximately 2 g of the volatile-free material. Disperse this in approximately 30 ml of water by warming. Cool and titrate with standard sulphuric acid using methyl orange as indicator. Boil for a short time only to expel carbon dioxide, then cool and add standard sodium hydroxide solution till the mixture just assumes a yellow tinge. Add mannitol in such proportion that the solution after titration will contain at least 30 percent, then titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

16.2.3.2 If the adhesive is very dark in colour, the end point in the above titration may be obscured; it is then advisable to reduce the 2 g sample to ash before starting the determination.

16.2.4 *Calculation*

$$\text{Borate (as } B_2O_3\text{), percent by mass (on dry basis)} = \frac{348.2\, V\, N}{M(100 - M_1)}$$

where

V = volume, in ml, of standard sodium hydroxide solution;
N = normality of standard sodium hydroxide solution;
M = mass, in g, of the sample taken for the test; and

M_1 = percent volatile matter content of the sample (*see* **6**).

NOTE — The presence of boric acid or metaborate is also shown by the above test. If the number of millilitres of sodium hydroxide solution consumed in the phenolphthalein titration is more than twice the number of millilitres of the sulphuric acid solution required in the methyl orange titration, then free boric acid is present. If the number of millilitres of sodium hydroxide solution is less than twice the number of millilitres of sulphuric acid solution, some metaborate is present. If the number of millilitres of sodium hydroxide solution is equal to the number of millilitres of sulphuric acid solution, then all the borax is present as metaborate.

16.3 Method B

16.3.1 *Principle of the Method*

All organic matter is removed by low temperature ashing. The inorganic residue is taken up in water and filtered. An aliquot portion of the solution is passed through cationic and anionic ion-exchange columns in series. Boric acid alone remains in the solution, and is titrated with Decinormal alkali in the presence of mannitol.

16.3.2 *Apparatus*

16.3.2.1 *Ion-exchange columns* — Three glass columns of 1 cm internal diameter, assembled in series in order of ‘first’ column (cationic), ‘second’ column (cationic) and ‘third’ column (anionic).

16.3.2.2 *Beakers squat* — 100 ml and 250 ml capacity.

16.3.2.3 *Filter flask* — 500 ml capacity.

16.3.2.4 *Filter funnel* — with sintered glass plate porosity 4.

16.3.3 *Reagents*

16.3.3.1 *Cation-exchange resin*

16.3.3.2 *Anion-exchange resin*

16.3.3.3 *Mannitol*

16.3.3.4 *Sodium hydroxide solution* — 0.1 N

16.3.3.5 *Sodium hydroxide solution* — 1 N

16.3.3.6 *Hydrochloric acid solution* — 2.0 N

16.3.3.7 *Phenolphthalein indicator solution* — 0.1 percent in rectified spirit

16.3.3.8 *Methyl orange indicator solution* — 0.05 percent solution

16.3.4 *Procedure*

16.3.4.1 *Preparation of ion-exchange column*

Place a small wad of cotton wool in the constriction at the foot of each of the columns. Half fil1 the columns with water and then into the first column introduce the cation-exchange resin in the form of a slurry until a short column 3 cm high is formed. Add quantity of cation-exchange resin to the second column to a height of 85 cm and then fill the third column in exactly the same way to a height of 35 cm with anion-exchange resin. Back wash the columns with water and move them gently to and fro to remove air bubbles and fines.

16.3.4.2 *Regeneration of the resins* — In order to save time, regenerate the resins periodically in bulk. Collect 500 g quantities of the used resin and regenerate as follows:

a) *Cation-exchange resin from column 2* — Introduce the resin into a glass column (2 cm to 3 cm diameter) as described above and pass 500 ml of 2 N Hydrochloric acid solutions through the column at a flow rate of approximately 2 ml per min. Wash the resin with distilled water until it is free from acid. Transfer the regenerated resin into a bottle and keep ready for further use under a layer of distilled water.

b) *Cation-exchange resin from column* 1 *(contaminated with 'insoluble silica')* — Wash the resin continuously with 200 ml lots of 1 N Sodium hydroxide solution, with warming, if necessary, to remove silica. Wash the resin with water and regenerate as described in (a) above.

NOTE — The quantity of resin from column 1 is small and it may be more expedient to discard it rather than regenerate it.

c) *Anion-exchange resin from column* 3 — Regenerate with 1 N sodium hydroxide solution in the same manner as for the regeneration of the cationic column. Wash the resin free from alkali with distilled water, and bottle as described above, ready for future use.

16.3.4.3 *Preparation of solution*

Add 20 g of the sample in a nickel dish by heating over a low Bunsen flame at a low temperature. Powder the mass with a thick glass rod and transfer it to 250 ml beaker with hot water. Cool, filter and transfer the filtrate and washing quantitatively to a 200 ml volumetric flask and dilute to volume with water.

16.3.4.4 *Determination of borate*

Pipette 50 ml of the solution, prepared as described above, on to the top of the first column of the ion-exchange assembly and allow the solution to percolate through the columns until the liquid level falls to the level of the resin in the first column. Elute the columns with 250 ml of water. Collect the eluate in a 500 ml conical flask, boil gently to expel carbon dioxide, cool, add three or four drops of methyl orange indicator and adjust to the first sign of a pale yellow colour by adding 0.1 N sodium hydroxide solution. Add 3 to 4 g of mannitol and a few drops of phenolphthalein indicator. Titrate to a pink end point with 0.1 N sodium hydroxide solution.

16.3.5 *Calculation*

a) Borate (as B_2O_3), percent by mass (on dry basis) = $\frac{348.2\,V\,N}{M(100-M_1)}$

where

V = volume, in ml, of standard sodium hydroxide solution required for the titration using phenolphthalein indicator;
N = normality of standard sodium hydroxide solution;
M = mass, in g, of the sample taken for the test; and
M_1 = percent volatile matter content (as determined in **6**).

b) Borax ($N_2B_4O_7$), percent by mass = Borate (as B_2O_3), percent by mass
(*On dry basis*) (*On dry basis*)×2.7385

17 DETERMINATION OF PROTEIN

17.1 Outline of the Method

The adhesive is oxidized by heating with a mixture of potassium sulphate, copper sulphate and concentrated sulphuric acid, thereby converting nitrogen compounds into ammonium salts from which ammonia is removed by distillation. The distilled ammonia is absorbed in standard sulphuric acid which is then titrated with standard sodium hydroxide solution. The protein content is determined by multiplying the amount of nitrogen by 6.25.

17.2 Apparatus

17.2.1 *Kjeldahl Flasks* — 500 ml capacity.

17.2.2 *Distillation Flask* — 500 ml capacity.

17.3 Reagents

17.3.1 *Copper Sulphate*

17.3.2 *Potassium Sulphate*

17.3.3 *Concentrated Sulphuric Acid* — conforming to IS 266.

17.3.4 *Sodium Hydroxide Solution* — approximately 38 percent by mass.

17.3.5 *Standard Sulphuric Acid* — 0.2 N

17.3.6 *Standard Sodium Hydroxide Solution* — 0.2 N

17.3.7 *Mixed Indicator Solution*

Dissolve 0.1 g of methyl red and 0.05 g of methylene blue in 100 ml of rectified spirit.

17.4 Procedure

Weigh accurately about 3 g of the material in a Kjeldahl flask. Add approximately 300 mg of copper sulphate, 3 g of potassium sulphate and 25 ml of concentrated sulphuric acid. Heat the mixture gently, keeping the flask in inclined position and after frothing has ceased, boil briskly until the solution has remained almost colourless for 30 min. Cool and add 200 ml of water. Pour cautiously 50 ml of sodium hydroxide solution in the flask and then add a few pieces of pumice stone. Connect the flask to the distillation apparatus consisting of Kjeldahl connecting bulb and a condenser, the delivery tube of which extends well beneath the surface of standard sulphuric acid contained in a 500 ml flask. Distil until about two-thirds of the solution has been collected in the receiving flask. To the receiving flask add mixed indicator solution and titrate the excess acid with standard sodium hydroxide solution. Carry out a blank determination using the same quantities of reagents and conditions of test.

17.5 Micro-Estimation of Protein

For determining proteins in small amounts of sample, the procedure given in **17.4** is to be followed, except that 0.3 g of the sample shall be taken for digestion with 10 ml of concentrated sulphuric acid, 30 mg of copper sulphate and 0.3 g of potassium sulphate in a 50 ml Kjeldahl flask. The distillation of ammonia shall be done in a micro-distillation apparatus and a micro-burette used for the final titration.

17.6 Calculation

$$\text{Protein, percent by mass } (on\ dry\ basis) = \frac{1.75\ (V_1 - V_2)}{M} \times \frac{100}{(100 - M_1}$$

where

V_1 = volume, in ml, of standard sodium hydroxide solution used for the blank titration;
V_2 = volume, in ml, of standard sodium hydroxide solution used for titration with the material;
M = mass, in g, of the material taken for the test; and
M_1 = percent moisture content of the sample (*see* **6**).

18 DETERMINATION OF ARSENIC

18.1 Reagent

18.1.1 *Isopropyl Alcohol*

18.2 Procedure

Take 1.00 g of the material in a beaker, add 5 ml of isopropyl alcohol and 15 ml of water. Mix it thoroughly and carry out the test as prescribed in IS 2088, using aliquot of standard arsenic trioxide solution for preparing the comparison stains.

19 DETERMINATION OF LEAD

19.1 Outline of the Method

Lead is determined by comparing the colour produced by the material with sodium sulphide solution against that of a standard lead solution in Nessler tubes.

19.2 Apparatus

19.2.1 *Nessler Tubes* — 50 ml capacity

19.3 Reagents

19.3.1 *Isopropyl Alcohol*

19.3.2 *Dilute Ammonium Hydroxide* — approximately 4 N.

19.3.3 *Sodium Sulphide Solution* — 10 percent.

19.3.4 *Dilute Hydrochloric Acid* — approximately 5 N.

19.3.5 *Sodium Potassium Tartrate Solution*

Dissolve 25 g of sodium potassium tartrate in 50 ml of water, make it distinctly ammoniacal and add 10 ml of sodium sulphide solution with stirring. Allow the precipitate to settle and filter the solution. Acidify the filtrate with dilute hydrochloric acid and boil till free from hydrogen sulphide. Cool the solution, make it ammoniacal and dilute to 100 ml.

19.3.6 *Concentrated Nitric Acid*

19.3.7 *Sodium Metabisulphite*

19.3.8 *Potassium cyanide Solution* — 10 percent.

19.3.9 *Concentrated Ammonium Hydroxide* — 20 percent.

19.3.10 *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate in water, add 1 ml of concentrated nitric acid and dilute the solution to 1 000 ml. Pipette out 10 ml of this solution and then dilute to 1 000 ml. One millilitre of the final solution is equivalent to 0.01 mg of lead (as Pb). This solution should be freshly prepared.

19.4 Procedure

Weigh accurately 1.000 g of the material in a beaker, add 5 ml of isopropyl alcohol, 20 ml of water, 10 ml of sodium potassium tartrate solution and 10 ml of dilute hydrochloric acid. Heat the mixture to boiling and add 0.5 g of sodium metabisulphite. Neutralize the free acid with concentrated ammonium hydroxide and add 5 ml in excess, followed by 3 ml of potassium cyanide solution. Heat the mixture until the solution becomes colourless. Cool the solution, transfer to a Nessler tube and make up the volume to 50 ml with water. Add 4 drops of sodium sulphide solution and stir. Carry out a control test in another Nessler tube using aliquot of standard lead solution in place of the material and the same quantities of other reagents. Make up the volume to 50 ml and compare the colour produced in the two Nessler tubes to determine the lead content.

20 DETERMINATION OF STARCH

20.1 Reagents

20.1.1 *Iodine Solution*

Dissolve 2.6 g of iodine and 3 g of potassium iodide in 100 ml of water

20.2 Procedure

Heat 1.0 g of the material along with 100 ml of water to boiling until the material is dispersed. Cool the solution and add a few drops of iodine solution.

20.2.1 The material shall be taken to contain starch if blue colour is produced.

21 DETERMINATION OF REDUCING SUGARS

21.1 Reagents

21.1.1 *Lead Subacetate Solution* — Activate about 200 g of litharge by heating at 650 °C for 3 h (cooled product should be of lemon colour). Boil 43 g of neutral lead acetate and 130 g of freshly activated litharge in 1 litre of water for 30 min. Cool and allow to settle. Dilute the supernatant liquid to density 1.25 with freshly boiled and cooled water.

21.1.2 *Potassium Oxalate* — crystalline.

21.1.3 *Fehling's Solution* (*Soxhlet Modification*) — prepared by mixing immediately before use equal volumes of the following Solution A and Solution B:

> *Solution A* — Dissolve 34.639 g of copper sulphate ($CuSO_4.5H_2O$) in water, and 0.5 ml of concentrated sulphuric acid (analytical reagent) and dilute to 500 ml in a volumetric flask. Filter the solution through prepared asbestos.
>
> *Solution B* — Dissolve 173 g of Rochelle salt [potassium sodium tartrate ($KNaC_4H_4O_6.4H_2O$)] and 50 g of sodium hydroxide (analytical reagent) in water, dilute to 500 ml in a volumetric flask and allow the solution stand for 2 days. Filter the solution through prepared asbestos.

21.1.3.1 *Standardization of Fehling's Solution*

Pour standard dextrose solution (*see* **21.1.6**) into a 50 ml burette (*see* Note). Find from Table 2 the titre (volume of standard dextrose solution required to reduce all the copper in 10 ml of Fehling's solution) corresponding to the concentration of standard dextrose solution. (For example, if the standard, dextrose solution, contains 167.0 mg of anhydrous dextrose per 100 ml, the corresponding titre amid be 30 ml.) Pipette 10 ml (*see* Note) of Fehling's solution into a 300 ml conical flask and run in from the burette almost the whole of the standard dextrose solution required to effect reduction of all the copper, so that not more than 1 ml will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add, without interrupting boiling, 1 ml of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. [The titration should be completed within 1 min so that the contents of the flask boil altogether for 3 min without interruption (*see*

Note 3).] Note the titre in millilitres of standard dextrose solution. Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose in 1 ml of the standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor given in Table 2 and determine the correction, if any, to be applied to the dextrose factors derived from Table 2.

Example:

Concentration of anhydrous dextrose in standard dextrose solution, mg/100 ml	= 167.0
Titre obtained by direct titration	= 30.1 ml
Dextrose factor for 30.1 ml of standard dextrose Solution (titre in millimetres × number of milligrams of anhydrous dextrose in 1 ml of standard dextrose solution)	= 30.1 × 1.670 = 50.267 0
Dextrose factor for 30.1 ml of standard dextrose solution from Table 2 (calculated by interpolation)	= 50.11
Correction to be applied to the dextrose factors derived from Table 2	= 50.267 0 – 50.11 = +0.157 0

NOTES:

1. In adding dextrose solution to the reaction mixture the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right angles, so that the body of the burette can be kept out of steam while adding sugar solution. Burette with glass taps are unsuitable for this work, as the taps become heated by the steam and are liable to jam.
2. Instead of using 10 ml of Fehling’s solution, a 25 ml portion can also be used for standardization of dextrose solution and also for titration of test solution (**21.2.2**). In that case, Table 3 shall be used in place of Table 2.
3. It should be observed that with both incremental and standard methods of titration the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

TABLE 2 DEXTROSE FACTORS FOR 10 ml OF FEHLING’S SOLUTION

(*Clause* 21.1.3.1)

TITRE ml	*DEXTROS FACTOR*	*ANHYDROU DEXTROSE PER 100 ml OF SOLUTION mg*	*TITRE ml*	*DEXTROS FACTOR*	*ANHYDROU DEXTROSE PER 100 ml OF SOLUTION mg*
(1)	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**
15	49.1	327.0	35	50.4	143.9
16	49.2	307.0	36	50.4	140.0
17	49.3	289.0	37	50.5	136.4
18	49.3	274.0	38	50.5	132.9
19	49.4	260.0	39	50.6	129.6
20	49.5	247.4	40	50.6	126.5
21	49.5	235.8	41	50.7	123.6
22	49.5	225.5	42	50.7	120.8
23	49.7	216.1	43	50.8	118.1

24	49.8	207.4	44	50.8	115.5
25	49.8	199.3	45	50.9	113.0
26	49.9	191.8	46	50.9	110.6
27	49.9	184.9	47	51.0	108.4
28	50.0	178.5	48	51.0	106.2
29	50.0	172.5	49	51.0	104.1
30	50.1	167.0	50	51.1	102.2
31	50.2	161.8			
32	50.2	156.9			
33	50.3	148.0			
34	50.3	148.0			
Milligrams of anhydrous dextrose corresponding to 10 ml of Fehling’s solution					

TABLE 3 DEXTROSE FACTORS FOR 25 ml OF FEHLING’S SOLUTION

[*Clause* 21.1.3.1 (Note) *and* 21.3.1]

TITRE ml	**DEXTROS FACTOR**	**ANHYDROU DEXTROSE PER 100 ml OF SOLUTION mg**	**TITRE ml**	**DEXTROS FACTOR**	**ANHYDROU DEXTROSE PER 100 ml OF SOLUTION mg**
(1)	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**
15	120.2	801.0	35	121.0	345.6
16	120.2	751.0	36	121.0	336.3
17	120.2	707.0	37	121.1	327.4
18	120.2	668.0	38	121.2	318.8
19	120.3	633.0	39	121.2	310.7
20	120.3	601.5	40	121.2	303.1
21	120.3	572.9	41	121.3	295.9
22	120.4	547.3	42	121.4	289.0
23	120.4	523.6	43	121.4	282.4
24	120.5	501.9	44	121.5	276.1
25	120.5	482.0	45	121.5	270.1
26	120.6	463.7	46	121.6	264.3
27	120.6	446.8	47	121.6	258.8
28	120.7	431.1	48	121.7	253.5
29	120.7	416.4	49	121.7	248.4
30	120.8	402.7	50	121.8	243.6
31	120.8	389.7			
32	120.8	377.6			
33	120.9	366.3			
34	120.9	355.6			
Milligrams of anhydrous dextrose corresponding to 25 ml of Fehling’s solution.					

21.1.4 *Methylene Blue Indicator Solution* — Dissolve 0.2 g of methylene blue in water and dilute to 100 ml.

21.1.5 *Stock Solution of Dextrose* — Weigh accurately about 10 g of anhydrous dextrose into a one litre volumetric flask and dissolve it in water. Add to this solution 2.5 g of benzoic acid, shake to dissolve the benzoic acid and make up the volume to the mark with water. This solution should not be used after 48 h.

21.1.6 *Standard dextrose Solution* — Dilute a known aliquot of the stock solution dextrose (**21.1.5**) with water containing 0.25 percent (*m*/*v*) benzoic acid to such a concentration that more than 15 ml but less than 50 ml of it shall be required to reduce all the copper in the Fehling's solution taken for titration in **21.1.3.1**. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml. Prepare this solution fresh every day.

NOTE — When 10 ml of Fehling's solution (*see* also Note under **21.1.3.1)** is taken for titration, a standard dextrose solution containing 0.11 to 0.30 percent (*m*/*v*) of anhydrous dextrose is convenient for use. With 25 ml of Fehling's solution, a standard dextrose solution containing 0.25 to 0.75 percent of anhydrous dextrose should be used.

21.2 Procedure

21.2.1 Weigh accurately about 10 g of the test sample in a beaker. Mix it thoroughly with 100 ml of water. Transfer to a 250 ml volumetric flask. Add drop by drop lead sub acetate solution till precipitation is complete. Make the solution up to the mark by adding water. Mix thoroughly and allow it to settle down. Filter the supernatant liquid and collect the filtrate in a dry beaker. Add dry potassium oxalate to the filtrate to remove excess of lead. Filter and collect the filtrate (as a clarified solution) in a 250 ml flask. Make it up to the mark for subsequent estimations of reducing sugar (**21.2.2**) and dextrin (**22**).

21.2.2 *Incremental Method of Titration* — Pour the clarified solution (**21.2.1**) into a 50 ml burette (*see* Note under **21.1.3.1**). Pipette 10 ml of Fehling's solution (*see* Note under **21.1.3.1**) into a 300 ml conical flask and run in from the burette 15 ml of the clarified solution. Without further dilution, heat the contents of the flask over a wire gauze, and boil. After the liquid has been boiling for about 15 s, it will be possible to judge if the copper is almost reduced by the bright red colour imparted to the boiling liquid by the suspended cuprous oxide. When it is judged that nearly all the copper is reduced, add 1 ml of methylene blue indicator solution (*see* Note). Continue boiling the contents of the flask for 1 to 2 min from the commencement of ebullition, and then add the clarified solution in small quantities (1 ml or less at a time) allowing the liquid to boil for about 10 s between successive additions, till the blue colour of the indicator just disappears (*see* Note under **21.1.3.1**). In case there appear to be still much unreduced copper after the mixture of Fehling's solution with 15 ml of the clarified solution has been boiling for 15 s, add the clarified solution from the burette in larger increments (more than 1 ml at a time, according to judgement) and allow the mixture to boil 15 s after each addition. Repeat the addition of the clarified solution at intervals of 15 s until it is considered unsafe to add a large increment of the clarified solution. At this stage continue the boiling for an additional 1 to 2 min, add 1 ml of methylene blue indicator solution and complete the titration by adding the clarified solution in small quantities (less than 1 ml at a time) (*see also* Note 2).

NOTES:

1. It is advisable not to add the indicator until the neighbourhood of the end point has been reached, because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.
2. When the operator has had a fair amount of experience with the method, a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration, but for the utmost

degree of accuracy of which the method is capable, a second titration should be carried out by the standard method of titration (*see* **21.2.2.1**).

21.2.2.1 *Standard method of titration* — Pipette 10 ml of Fehling's solution (*see* Note 2 under **21.1.3.1**) into a 300 ml conical flak and run in from the burette almost the whole of the clarified solution required to effect reduction of all the copper (determined under **21.2.2**), so that, if possible, not more than 1 ml shall be required later 10 complete the titration. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add, without interrupting boiling, 1 ml of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. The titration should be completed within 1 min so that the contents of the flask boil altogether for 3 min without interruption (*see* Note 3 under **21.1.3.1**).

NOTE — The indicate is so sensitive that it is possible to determine the end point within one drop of the clarified solution in many cases. The complete decolourization of the methylene blue is usually indicated by the whole reaction liquid in which the cuprous oxide is continuously churned up becoming bright red or orange in colour. In case of doubt, the flame may be removed from the wire gauze for 1 or 2 s and the flask held against a sheet of white paper. (A holder of paper, suitably fixed round the neck of the flask, is very convenient for this purpose as it can be left round the neck of the flask without the risk of overbalancing it.) The top edge of the liquid would appear bluish if the indicator is not completely decolorized. It is advisable not to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access to flask, but there is no danger of this as long as continuous stream steam is issuing from the mouth of the flask

21.3 Calculation

21.3.1 Refer to Table 2 (or Table 3 as appropriate) for the dextrose factor corresponding to the titre (determined as given under **21.2.2.1**) and apply the correction previously determined under **21.1.3.1**. Calculate the dextrose content of the clarified solution (*see* **21.2.1**) as follows:

$$\text{Anhydrous dextrose present in 1 ml of the clarified solution, mg} = \frac{\text{dextrose factor}}{\text{titre}}$$

NOTE — Tables 2 and 3 show for the standard method of titration the values corresponding to integral millilitres of the sugar solutions; values corresponding to intermediate figure shall be obtained by interpolation.

21.3.2 $\text{Reducing sugars, percent by mass } (on\ dry\ basis) = \frac{M\,V}{100\,M_1} \times \frac{100}{100 - M_2}$

where

M = mg, of anhydrous dextrose present in 1 ml of the clarified solution (*see* **21.3.1**);
V = total volume, in ml, of the clarified solution of the material prepared in **21.2.1**;
M_1 = mass, in g, of the material taken for the test in **21.2.1**; and
M_2 = percent volatile matter content of the sample (as determined in **6**).

22 DETERMINATION OF DEXTRIN

22.1 Reagents

In addition to the reagents listed under **21.1**, the reagents listed below are required.

22.1.1 *Hydrochloric Acid* — concentrated.

22.1.2 *Sodium Hydroxide Solution* — 10 percent.

22.2 Procedure

Take 50 ml of the clarified solution (*see* **21.2.1**) in a clean 400 ml flask. Add 20 ml of hydrochloric acid and 100 ml of water. Boil gently using a water-cooled reflux condenser for 2.5 h. Cool to room temperature. Neutralize the solution with sodium hydroxide solution. Transfer the solution to a 500 ml volumetric flask and make up to the mark. Determine the reducing sugar in terms of percent by mass as under **21.2**. Subtract from this the percentage of reducing sugars obtained under **21.3.2** and multiply it by 0.9 to get the percentage of dextrin.

23 DETERMINATION OF GUM CONTENT

23.1 Procedure

The gum content is determined by subtracting the sum of the percentages of moisture, ash, protein and acid insoluble ash from 100.

24 DETERMINATION OF BARK AND OTHER FOREIGN ORGANIC MATTER

24.1 Reagent

24.1.1 *Dilute Hydrochloric Acid* — approximately 4 N.

24.2 Procedure

Accurately weigh about 5 g of the material and transfer to a 250 ml conical flask. Add 25 ml of dilute hydrochloric acid and 25 ml of water. Cover the flask with a small watch-glass and boil gently until the mixture loses its viscosity. Shake the flask occasionally. After heating for 10 min, break the lumps with a glass rod. Wash the glass rod with 5 ml of water. Heat the flask for another 20 min with occasional shaking to ensure complete dispersal of the lumps. Filter through a tared filtering crucible and wash the residue with water until the washings are free from chloride ions. Remove the crucible to an air-oven maintained at 105 ± 2 °C, cool in a desiccator and weigh. Then dry to constant mass in the air-oven.

24.3 Calculation

$$\text{Bark and other organic matter, percent by mass } (on\ dry\ basis) = \frac{100 \times 100 \times M}{(100 - M_1) \times M_2}$$

where

M = mass, in g, of the residue,
M_1 = percent volatile matter in the material (*see* **6**); and
M_2 = mass, in g, of the material taken for the test.

25 TEST FOR ANIMAL FILTH

25.1 Apparatus

25.1.1 *Microscope* — with magnification 40 or more.

25.2 Reagents

25.2.1 *Dilute hydrochloric Acid* — 1: 19 (*v*/*v*)

25.2.2 *Petroleum Ether*

5.3 Procedure

25.3.1 *Macroscopic Examination for Insect and Animal Filth*

Take about 100 g of coarse unground gum and examine a small amount at a time in good light and against a white background. Observe if there is embedded insect or animal filth in the pieces of gum. If the gum is finely ground, sieve through an appropriate size sieve to separate filth from gum and examine for the presence of filth.

25.3.2 Microscopic *Examination for Animal Filth*

Take 1 g of the sample, which has been finely powdered in a 500 ml beaker and add 200 ml of dilute hydrochloric acid. Boil for 15 min. Cool the beaker and allow the suspension to settle down. Decant of the liquid so that no solid particles escape. Wash it with additional quantity of water and again pour off the liquid. Observe the residue in the beaker under microscope for rodent excreta and other particles. To confirm further, add 100 ml of petroleum ether to the residue and shake vigorously. Allow it to settle and filter off the petroleum ether layer on a fluted filter paper, taking care that no water drop comes on the filter paper. Observe the filter paper under the microscope for presence of animal hair.

26 DETERMINATION OF SWELLING PROPERTY

26.1 Apparatus

26.1.1 *Graduated Cylinder* — 500 ml capacity, conforming to IS 878.

26.2 Procedure

Collect by means of screening the portion of the material larger than 600 micron and smaller than 2 mm. Weigh 2.00 g of the sieved material and transfer it to the graduated cylinder containing 500 ml of water. Stir any particles rising to the top and allow to stand for 18 h. Read the volume to which the gum swells up. Keep the cylinder with contents aside for test in **27**.

27 DETERMINATION OF WATER ABSORPTION

27.1 Apparatus

27.1.1 *Graduated Cylinder* — 500 ml capacity, conforming to IS 878.

27.2 Procedure

Pour contents of the graduated cylinder (**26.2**) through a bed of moistened glass wool and allow the water to drain into 500 ml graduated cylinder. Read the volume of water collected in graduated cylinder and calculate, by deducting that volume from 500, the number of ml of water retained by the gum.

28 DETERMINATION OF EFFICIENCY OF ADHESIVE JOINTS

28.1 General

Carry out this test on the materials for which the adhesive is intended. The time of drying to be used before the joint is disrupted will vary according to the materials used; allow adequate time for the adhesive to set before continuing the test.

28.2 Procedure

Apply a thin uniform film of the adhesive, approximately 0.9 mm thick, over a length of approximately 20 mm at the end of a strip approximately 20 mm wide cut from one of the materials. The adhesive may be applied by means of a device incorporating shims of appropriate thickness and a slider bar or doctor. Immediately place a similar strip of the second material on the glued surface of the first strip, and press it down firmly by hand. Place the assembled test piece between two lots, each of five layers, of the materials used with the adhesive, and place the whole on a flat, rigid base. Apply a load of 1 kgf, evenly distributed over the surface being tested, and leave until the adhesive is set. Carry out this test at nominal relative humidities of 34 percent and 65 percent, respectively (*see* Note), the materials being kept at the specified relative humidity for not less than 4 h immediately before the joint is tested. Assess the joint strength by attempting to separate the two strips by peeling (*see* Fig. 2). If the two strips can be separated only by continuous disruption of the surfaces of the material, however slowly the peeling is carried out, the efficiency of adhesion is to be deemed satisfactory. If separation occurs at one of the interfaces between adhesive and material, the efficiency of adhesion is to be deemed unsatisfactory.

NOTE — These relative humidities can be obtained in small enclosures containing saturated solutions of suitable salts, provided that there is adequate air circulation. The following salts are suitable:

Temperature Range °C	*Relative Humidity (Percent)*	*Salt Recommended*
5-40	34 ± 2	Magnesium chloride
20-30	65 ± 2	Sodium nitric

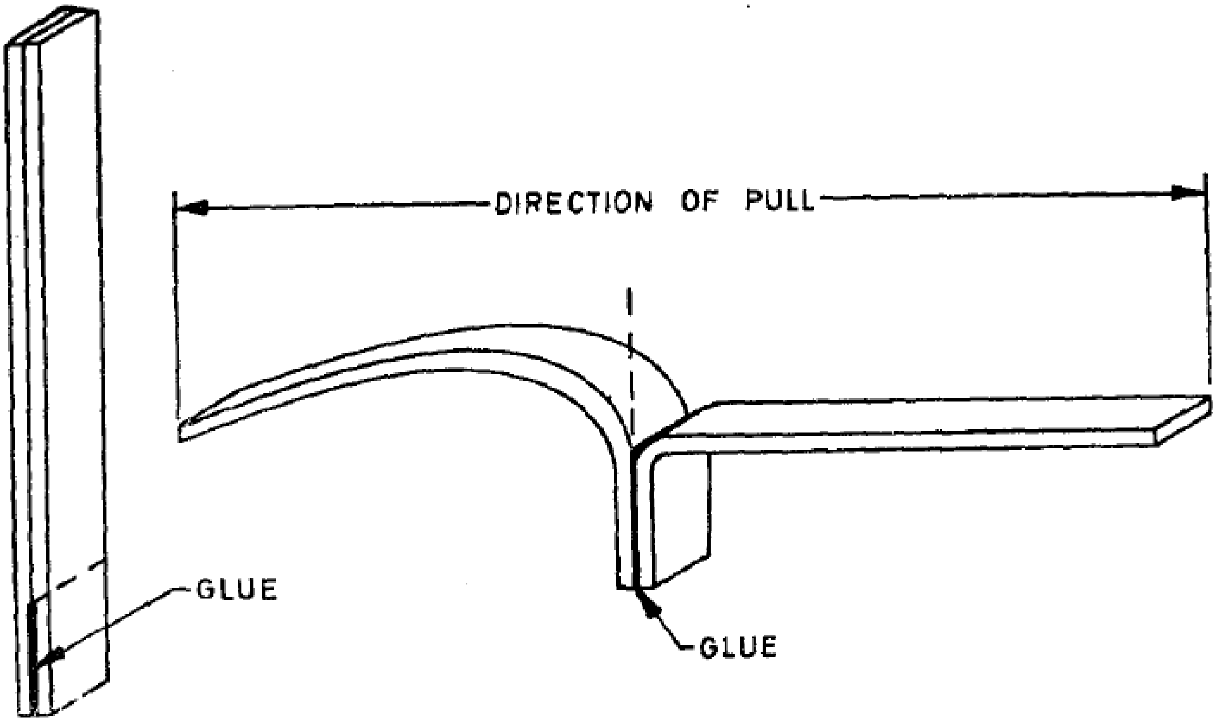

FIG. 2 DIAGRAM OF TEST FOR EFFICIENCY OF ADHESIVE

29 DETERMINATION OF FOAMING TENDENCY

29.1 Application

The results of this test will be useful in assessing the foaming tendencies of adhesives, but it is not possible to simulate, in the test, all the conditions that may arise during application of the adhesives in practice. The results of the test should, therefore, be applied with caution.

29.2 Apparatus

29.2.1 *Stoppered Glass Cylinder* — 100 ml with an air space (above the 100 ml graduation) of 50 ± 2 ml.

29.2.2 *Water-Bath* — 250 mm diameter and 200 mm high

29.3 Procedure

Disperse 5 ± 0.05 g of the adhesive in slightly less than 50 ml of water, transfer the mixture to the glass cylinder and dilute to the 50 ml mark. Place the cylinder in the water-bath, maintained at a temperature of 25 ± 0.2 °C, so that the level of water in the bath is at the 70 ml graduation mark, and leave it for 30 min. At the end of this period, remove the cylinder from the bath and shake it vigorously with a throw of about 300 mm and at the rate of three shakes per second for 1 min. Then replace the cylinder in the water-bath at 25 °C and allow it to remain until the height of the liquid in the cylinder corresponds to 45 ml. Record the time for this 45 ml meniscus to form, together with the volume of the foam above the liquid.

NOTE — It is important that this determination should be carried out in a place free from draughts.

29.4 Reporting

Report both the time in minutes and the volume of foam in milliliters as either figure by itself is not a sufficient indication of the behaviour of the adhesive.

30 DETERMINATION OF KEEPING QUALITY (BIOLOGICAL)

30.1 General

The rate of growth of fungi, yeasts, and bacteria is dependent (Among other factors) upon temperature, and upon water and fungicide concentration of the sample. For this reason, a solid adhesive will not degenerate for many years, provided that it is kept dry. Once the solid is dissolved in water, or all existing liquid adhesive is diluted, more water is available and the fungicide concentration (if any) is decreased. Hence, the diluted adhesive is more prone to growth of fungi which may impair its performance. Therefore, any test of keeping quality should be made at the concentration at which the adhesive will be stored or used.

30.2 Apparatus

Any spatulas, beakers, etc., used to transfer the adhesive and the jars used, should be clean and dry but not necessarily sterile.

30.3 Procedure

30.3.1 *For Adhesives as Stored* — Take sufficient sample from the bulk to half fill a clear glass jar or conical flask. Cover the mouth of the jar with polythene film and secure it with an elastic band to prevent the sample from drying.

30.3.2 *For Adhesives as Used* — Make up the adhesive to its working concentration, following the manufacturer’s instructions. If the adhesive was made up with hot water, allow it to cool to room temperature in the open air before transferring it to a sample jar or flask. Half fill the jar or flask and cover its mouth with polythene film.

30.3.3 Store the sample jars from **30.3.1** and **30.3.2** at 27 ± 2 °C and inspect them daily for the first week, weekly for the first four weeks and then monthly. Biological activity manifests itself in several ways according to the type of adhesive, but the following are the most easily recognized:

a) *Mould growth* — either in colonies or as a turbidity.

b) *Decrease in viscosity* — this may be measured by standard methods or a visual comparison may be made.

c) *Change in odour* — strong, definite rancid odour is sometimes produced.

30.4 Reporting — Report the day, week or month of onset of mould growth, decrease in viscosity and change in odour respectively.

31 DETERMINATION OF PENTOSAN

31.1 General

Pentosans have the general formula $(C_5H_8O_4)_n$ and on hydrolysis by weak acid, change into pentose sugars, $C_5H_{10}O_5$. On boiling with concentrated acid, the pentoses are decomposed with the formation of furfural. The furfural is precipitated by phloroglucinol as a phloroglucide which may be separated by filtration, dried and weighed. Since the precipitation is not complete, a correction is made for the amount remaining in solution, using the factor 0.005 2.

31.2 Reagents

31.2.1 *Phloroglucinol Reagent* — Although phloriglucinol of suitable purity is available, each lot shall be tested by heating nearly to boiling a small quantity, dissolved in a few drops of acetic anhydride, and adding a few drops of concentrated sulphuric acid. A pronounced violet coloration medicates the presence of diresorcin in sufficient amount to vitiate the test. This impurity crystallizes out if the reagent (11 g of phloroglucinol in 1 500 ml of 12 percent hydrochloric acid) is prepared by first adding the phloroglucinol in small amounts with stirring to 300 ml of the hot acid and then pouring this solution into the remainder of the acid which has not been heated, allowing this to stand at least overnight.

31.2.2 *Hydrochloric Acid* — 12 percent *(m/m)*

31.3 Procedure

31.3.1 Weigh out into a flask an amount of the sample sufficient to yield about 0.03 g of furfural in the process, add 100 ml of hydrochloric acid and a few pieces of pumice stone and connect with distillation apparatus by means of a double bored rubber stopper, the second hole carrying a cylindrical separatory funnel with mark at 30 and 60 ml. Heat at first continuously, then adjust the flame so that the distillate passing through a filter paper into a graduated cylinder will fill to the 30 ml mark in about 10 min.

31.3.2 Repeat the distillation, adding 30 ml of hydrochloric acid from the separatory funnel so manipulated as to wash down the sides of the flask, as many times (usually 9 to 12) as are necessary to remove all the furfural as shown by testing with aniline acetate paper. To the joint distillate, add the phloroglucinol reagent in about double the amount required to combine with furfural from the amount of pentosan judged to be present, The colour of the solution changes first to ye1low, then to black. Dilute to 400 ml with hydrochloric acid, allow to stand overnight, filter on a Gooch crucible or sintered crucible, wash with 150 ml of water, keeping the crucible filled with liquid until all has been added. Dry the phloroglucide and weigh.

31.4 Calculation

31.4.1 If the mass of phloroglucide a is between 0.03 and 0.30 g, calculate by the following formula:

$$\text{Pentosans} = (a + 0.005\,2) \times 0.866\,6 \text{ g}$$

31.4.2 For mass of phloroglucide a under 0.03 g:

$$\text{Pentosans} = (a + 0.005\,2) \times 0.899\,4 \text{ g}$$

31.4.3 For mass of phloroglucide *a* over 0.03 g:

$$\text{Pentosans} = (a + 0.005\,2) \times 0.882\,4 \text{ g}$$

32 DETERMINATION OF VISCOSITY

32.1 General

Information on viscosity is usually required to ensure that the adhesive has correct flow characteristics in use. The conditions of test should, therefore, be chosen to approximate to those in actual use. If the adhesive is to be used at ambient temperature, a standard testing temperature of 27 ± 2 °C is recommended.

32.1.1 Methods — Two methods have been recommended:

Method A — by Redwood; and
Method B — by Flow cup viscometers.

32.2 Method A (Redwood No. 1 Viscometer)

32.2.1 *Apparatus* — Redwood viscometer as illustrated in Fig. 3.

32.2.1.1 *Cup*

a) A cylindrical brass vessel of 2 to 3 mm wall thickness, provided with a flange at the base. The upper end of the cup is open, and has a plane rim. The bottom of the cup is concave internally to allow complete drainage of the liquid with which it may be filled. It is preferable for ease of cleaning that the internal junction of the bottom of the cup with the sides should be rounded. The flange is provided with a threaded portion 55 ± 0.2 mm overall diameter by means of which the cup is supported in the water-bath in such a position that the top of the bore of the jet is not less than 5 mm above the upper surface of the bottom of the bath.

b) The base of the cup is further provided with a tapering central hole into which the jet is fitted and cemented with its bore in the axes of the cup and its bottom surface entirely below the adjacent metal. The oil is to be filled into the oil cup up to the level as indicated by a stout wire fixed into the side below the oil level; the sharp end of the wire is 7 ± 1 mm from the inner wall of the cup. Dimensions of the cup are as follows:

Internal diameter of the cup	46.5 ± 0.5 mm
Vertical distance from rim to top of bore	96.0 ± 1.0 mm
Height of cylindrical portion of the cup	86.0 ± 1.0 mm
Height of filling point above upper end of bore	82.5 ± 2.0 mm

32.2.1.2 *Jet* — The jet is constructed of agate, and the central hole is drilled and polished to the greatest possible precision. The upper end is provided with a concave depression into which fits a ball-valve for starting or stopping the flow of adhesive. The lower end is concave to prevent the outflowing adhesive from creeping along the base of the cup. The lower end of the jet may be ground to a flat surface not exceeding 3 mm in diameter. Dimensions of the jet are as follows:

Internal length of the jet	10.0 ± 0.05 mm
Internal diameter of the jet, Min	1.620 mm

32.2.1.3 *Heating bath*

The cylindrical bath, approximately 140 mm in diameter and 95 mm deep, and spun from sheet copper, surrounds the cup. It is provided with a tap for emptying and with a side-tube for heating the water or other liquid.

32.2.1.4 *Stirrer* — Any suitable stirring device operated either by hand or by mechanical means.

32.2.1.5 *Valve* — The valve for starting and stopping the flow of the liquid from the cup consists of a metal ball. The fit of this ball valve in its socket should be such that there should be no leakage.

32.2.1.6 *Thermometer support* — A spring clip is provided to support the cup thermometer.

32.2.1.7 *Cup cover, stand screen* — These may also be provided.

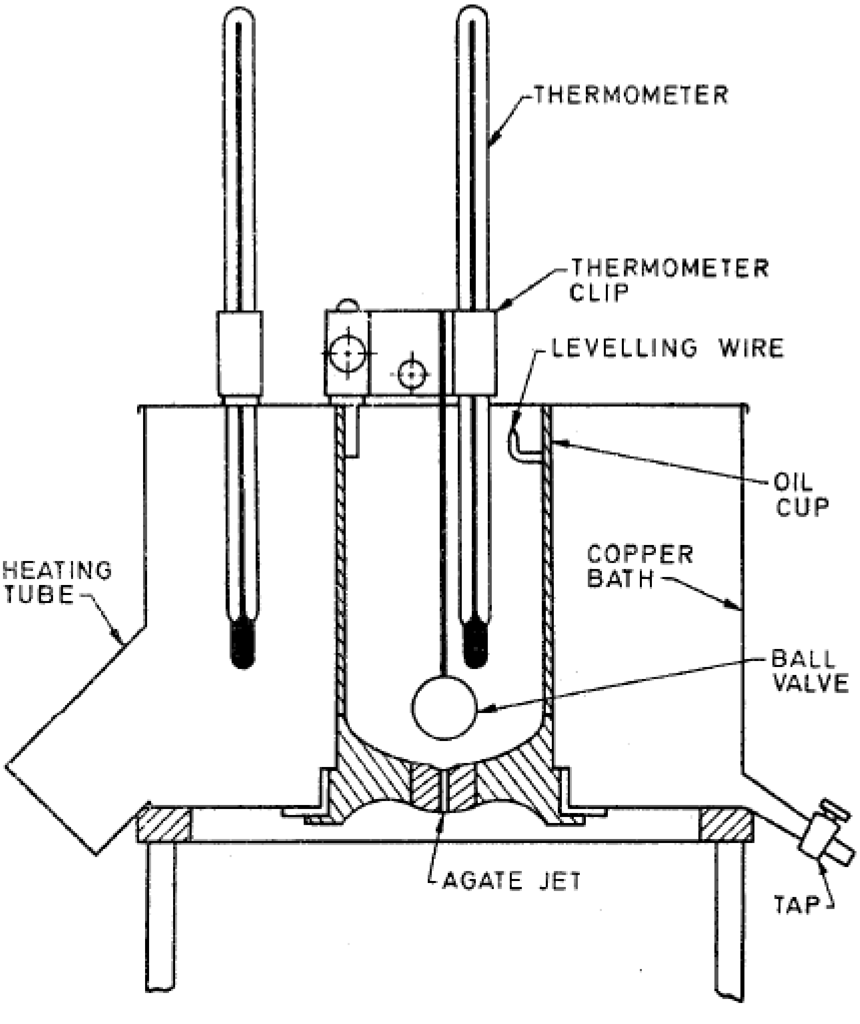

FIG. 3 REDWOOD VISCOMETER

32.2.2 *Procedure*

Heat the sample to a temperature near to that of the test and place it in the cup of the apparatus. Adjust the cup exactly to the temperature of the test and maintain it. Measure the time for 50 ml of the sample to flow through the jet into a receiver below.

32.3 Method B (Flow Cup Viscometer)

32.3.1 *Apparatus*

32.3.1.1 The flow cup generally known as Ford cup viscometer No. 4 shall be essentially of the form and dimensions as shown in Fig. 4. A cup made of any non-ferrous material is suitable. It may be plated. The finish shall be smooth. The jet may be either bored directly or constructed separately of stainless steel and force fitted. Care is essential in order to avoid damage to the lower apex of the cup. A protective skirt which does not interfere with the flow may be provided.

32.3.1.2 *Thermometer* — accurate to within 0.5 ^{o}C.

32.3.1.3 *Stop-watch*

32.3.1.4 *Stand* — A suitable stand, provided with levelling screws.

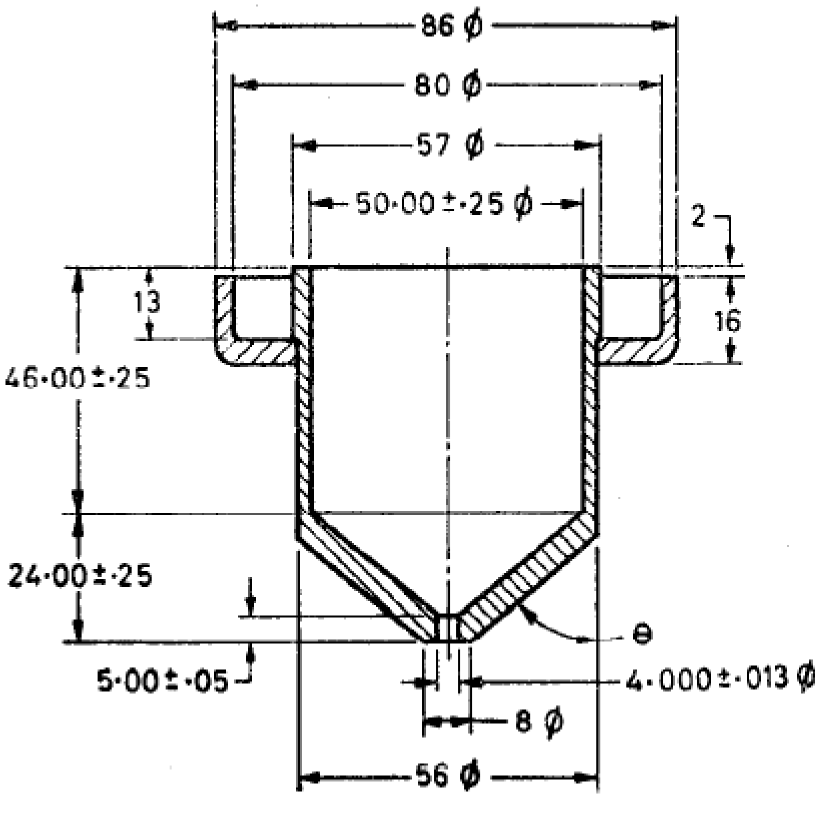

FIG. 4 FLOW CUP

32.3.1.5 *Spirit level*

32.3.1.6 *Scraper* — A straight-edged scraper for the top of the cup.

32.3.2 *Procedure*

32.3.2.1 Place the flow cup on the stand in a place free from draughts, preferably with the air temperature within the range of 27 ± 2 °C and level by using a spirit level placed on the rim.

32.3.2.2 Strain the sample into a clean container and adjust the temperature to meet the requirements as specified in **32.1**. A 150 micron IS Sieve [*see* IS 460 (Part 1 and Part 2)] or finer, is suitable. This and the following operations shall be carried out with minimum delay to avoid loss of solvent.

32.3.2.3 With the orifice closed by the finger, fill the cup with the bubble-free sample until it just begins to overflow into the gallery, pouring slowly to minimize the formation of air bubbles. If bubbles are present, allow them to rise and then remove them from the surface.

32.3.2.4 Check that the temperature of the material in the cup is within 0.5 °C of the test temperature. The cup may be at a temperature different from that of the sample and it is recommended that a minute or so be allowed to elapse before checking the temperature.

32.3.2.5 Place the scraper on the rim of the cup and draw it firmly across until the excess of the sample has flowed into the gallery. Place the receiver under the cup. Remove the finger and simultaneously start the stop-watch. Watch the stream of liquid flowing from the orifice. At the first evidence of a break of the stream into droplets, stop the stop-watch. The time taken is recorded in seconds as time of flow in the flow cup.