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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Seedlac — Specification

MODIFICATION TO FOREWORD *(Inside front cover)*

The ISO member body for the United Kingdom has withdrawn its approval of this International Standard. The United Kingdom should therefore be indicated as having disapproved the document.

H-09-25

INTERNATIONAL STANDARD ISO 55

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Seedlac — Specification

Gomme laque en grains — Spécification

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 55 was developed by Technical Committee ISO/TC 50, *Lac,* and was circulated to the member bodies in February 1976.

It has been approved by the member bodies of the following countries :

Austria
Czechoslovakia
Egypt, Arab Rep. of
India
Mexico
Netherlands
Romania
Sweden
Thailand
Turkey
United Kingdom

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 55-1957, of which it constitutes a technical revision.

Acknowledgement is due for the assistance that has been derived from the specifications and publications of the American Society for Testing and Materials, the American Bleached Shellac Manufacturer's Association, the United States Shellac Importer's Association, the British Standards Institution, the Agricultural Marketing Adviser to the Government of India, Messrs. Angelo Brothers, Ltd., Calcutta, and the Indian Lac Research Institute. Considerable assistance has been derived also from *A Handbook of Shellac Analysis,* by M. Rangaswami and H. K. Sen, issued by the Indian Lac Research Institute.

Printed in Switzerland

CONTENTS

Annexes

Seedlac – Specification

0 INTRODUCTION

0.1 The usual trade descriptions of seedlac are based on the Indian names of the host trees, the season of cropping the sticklac, visual differences or a combination of any of these. The use of these grade designations led to confusion and some marketing difficulties. When ISO/R 55 was prepared in 1957, it was decided to adopt only seven grades which were independent of the names of host trees or seasons. However, the expectation that the ISO grades for seedlac would be increasingly adopted in trade and ultimately replace the traditional grade designations has not come about. A new system has therefore been adopted in this International Standard so that seedlac can now be completely identified by a combination of the ISO grade and the trade grade. In this revision, it has been found possible to reduce the number of ISO grades from seven to five.

0.2 For matter insoluble in hot alcohol, two limits are prescribed, in line with trade practice : a basic limit and a relaxed limit. The relaxed limit shall be the limit for rejection.

0.3 The requirement for non-volatile matter soluble in cold alcohol has not been retained as this requirement is applied in practice to waste products of lac only.

0.4 One of the requirements for seedlac, namely that for matter insoluble in hot alcohol, is included in this International Standard as an essential clause. The remainder, namely those for volatile matter (moisture), colour, bleach index and bleachability, matter soluble in water, wax and ash, are optional.

0.5 For determination of bleach index and bleachability, two methods are practised. One has been developed in India and the other in the U.S.A. The method to be followed for the determination of bleach index and bleachability shall be subject to agreement between the purchaser and the supplier.

0.6 For the purpose of deciding whether a particular requirement of this International Standard is complied with, it is necessary for the final value, observed or calculated, expressing the result of test or analysis to be rounded off to the same number of places as that in the specified value, it being always understood that the analyst will carry out his determination to at least one place more than in the specified value.

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies requirements and methods of test for seedlac.

1.2 This International Standard is intended chiefly to cover the technical provisions for guiding the purchase of the material, but does not include all the necessary provisions of a contract.

1.3 The limits specified in this International Standard are limits for rejection (see 0.2).

2 DEFINITIONS

For the purposes of this International Standard, the following definitions apply :

2.1 **sticklac :** The natural product of lac insects.

2.2 **seedlac :** The product obtained by washing crushed sticklac.

2.3 **approved sample :** The sample agreed upon between the purchaser and the vendor as the standard for colour and appearance.

3 FORM AND CONDITION

The form and condition of seedlac are subject to agreement between the purchaser and the supplier.

4 GRADES

Five grades of seedlac, namely Special, A, B, C and D are specified. Further, if required by the purchaser, the names of the grades as prevalent in trade shall be indicated in addition, in parentheses, as shown below :

- Grade Special (Golden Kusmi);
- Grade B (Kusmi No. 2).

The correspondence between ISO grades and trade grades is shown in table 1.

TABLE 1

ISO grade	Trade grade
Special	i) Golden Kusmi ii) Golden Bysacki iii) Grade I, Class A
A	i) Kusmi No. 1 ii) Grade II, Class A
B	i) Kusmi No. 2 ii) Manbhum Fine Bysacki iii) Grade I, Class B
C	i) Fine Bysacki ii) Grade II, Class B
D	i) Ordinary Bysacki

5 MANDATORY REQUIREMENT

Matter insoluble in hot alcohol

Seedlac shall not contain more than the basic limits of matter insoluble in hot alcohol specified in table 2, when determined by either of the methods described in annex A, as agreed between the purchaser and the supplier. By agreement between the purchaser and the supplier, the basic limit may be relaxed but it shall not in any case exceed the relaxed limit as shown in table 2.

TABLE 2

Grade	Basic limit % *(m/m)*	Relaxed limit % *(m/m)*
Special	2,0	3,0
A	3,0	4,0
B	3,0	4,0
C	3,0	5,0
D	5,0	7,0

6 OPTIONAL REQUIREMENTS

6.1 Volatile matter (moisture)

Seedlac shall not contain more than 2,5 % *(m/m)* of volatile matter (moisture) as determined by the method described in annex B.

6.2 Colour index

The colour index of seedlac, as determined by the method described in annex C, shall not exceed the limits given in table 3.

TABLE 3

Grade	Colour index (maximum)
Special	8
A	10
B	12
C	18
D	30

Alternatively, the appearance and colour of seedlac shall not be inferior to those of an approved sample when judged by visual examination.

6.3 Bleach index and bleachability

The bleach index of seedlac shall be determined or the bleachability test for seedlac shall be carried out, if either is agreed between the purchaser and the supplier, in accordance with such methods as may be agreed between them (see 0.5).

6.4 Matter soluble in water

Seedlac shall not contain more than 1 % *(m/m)* of matter soluble in water, and the aqueous extract shall be neutral to methyl red. Matter soluble in water shall be determined by the method described in annex D.

6.5 Ash

The limits and methods of test for ash content of seedlac shall be subject to agreement between the purchaser and the supplier.

6.6 Wax

Seedlac shall not contain more than 5,5 % *(m/m)* of wax when determined by the method described in annex E.

7 SAMPLING

Samples shall be taken in the manner described in annex F.

ANNEX A

(See clause 5)

DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

A.1 PRINCIPLE

The matter insoluble in hot alcohol is determined by extracting a known mass of seedlac with 95 % (*V*/*V*) ethanol and determining the percentage of the undissolved residue by either of the two methods described below, as may be agreed.

A.2 METHOD I

A.2.1 Reagent

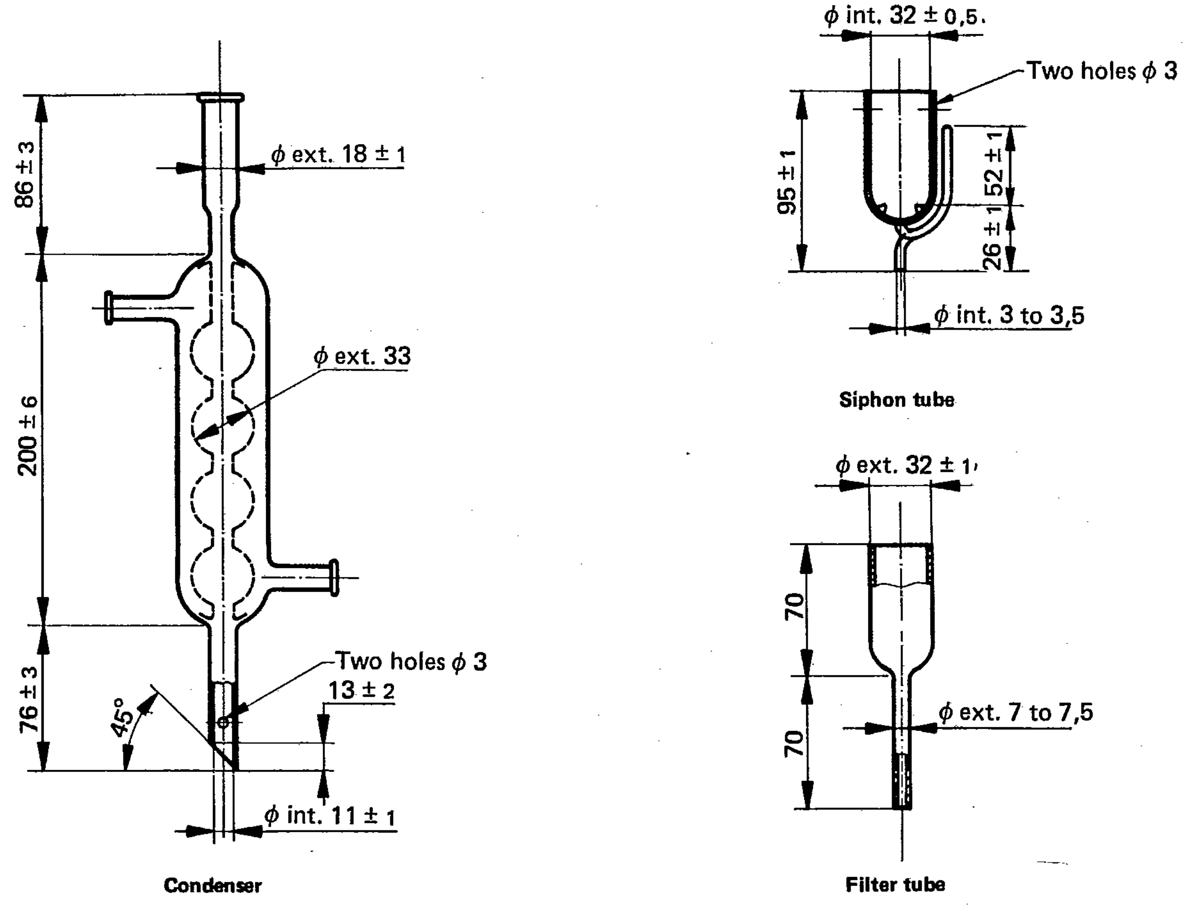
Alcohol : 95 % (*V*/*V*) ethanol, or 95 % (*V*/*V*) denatured spirit.

A.2.2 Apparatus

A.2.2.1 Condenser, all glass, of the type and dimensions shown in figure 1, with the tip cut at an angle of 45°. The condenser shall have two holes in its tip to hold the siphon tube (A.2.2.2).

A.2.2.2 Siphon tube, of glass, of the type and dimensions shown in figure 1. The siphon tube shall have two holes near the top for a wire to be fastened to the condenser tip, leaving about 6 mm space between the top of the tube and the condenser tip.

Dimensions in millimetres
All dimensions are approximate



Condenser

Siphon tube

Filter tube

FIGURE 1 — **Extraction apparatus for determining matter insoluble in hot alcohol — Method I**

A.2.2.3 Conical flask, heat-resistant, wide-mouthed, preferably of borosilicate glass, approximately 175 mm in height and approximately 50 mm in inside diameter at the top.

The flask shall be fitted with a tight-fitting cork 25 mm in depth and bored to fit the stem of the condenser. The bottom of the cork shall be just above the holes for the wire in the condenser. To support the flask, a suitable ring support with iron clamp and nickel-chromium or iron gauze shall be used. The gauze shall not have asbestos covering.

A.2.2.4 Carbon filter tube, of the type and dimensions shown in figure 1, with a light spiral spring at the bottom to hold up the extraction cartridge. The stem of the filter tube shall be fitted with a rubber stopper and shall be firmly supported in a hot water bath.

A.2.2.5 Extraction cartridges, of fat-free paper, approximately 25 mm in diameter and approximately 60 mm in height.

A.2.2.6 Weighing bottle, glass-stoppered, approximately 80 mm in height and approximately 40 mm in diameter.

A.2.2.7 Hot water bath, made of about 0,9 mm thick copper or stainless steel, having a width of approximately 100 mm and other dimensions as given in figure 2.

The cover shall have a flanged hole, 57 ± 1 mm in diameter, for a 200 ml beaker, and also a hole 35 ± 1 mm in diameter through which the top of the filter tube projects. Directly below this hole, in the bottom of the bath, there shall be a flanged hole, 25 ± 1 mm in diameter, to hold the rubber stopper, through which the stem of the filter tube extends, to discharge into the bottle or flask. The hot water bath shall be mounted on a low tripod or stand.

Dimensions in millimetres
All dimensions are approximate

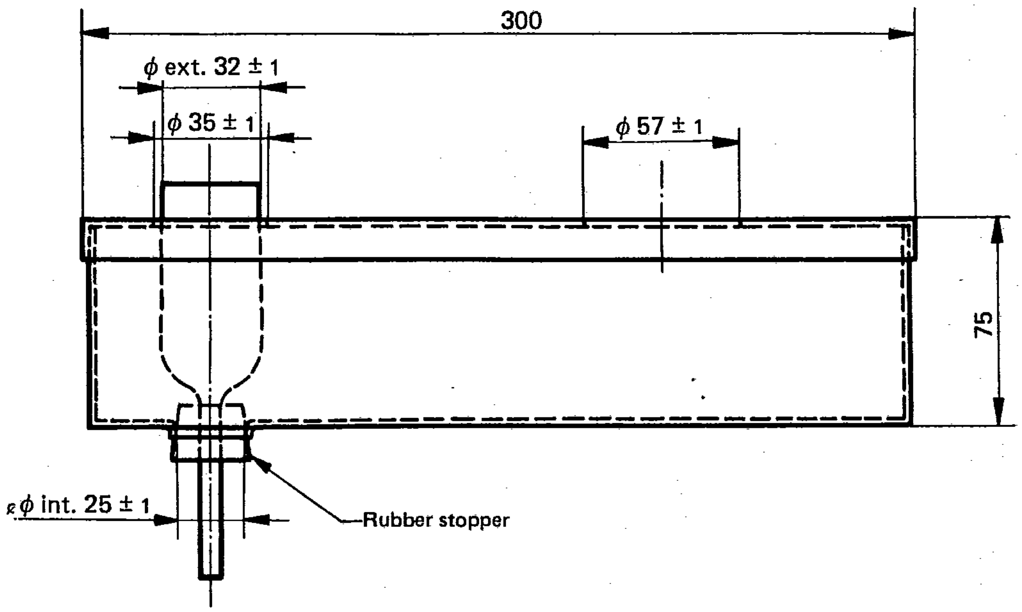


FIGURE 2 — Hot water bath for insoluble matter test — Method I

A.2.2.8 Beaker, tall-form, 200 ml capacity.

A.2.2.9 Desiccator, containing sulphuric acid, ρ 1,84 g/ml, as desiccant.

A.2.2.10 Drying oven, maintained at 100 ± 2 °C.

A.2.2.11 Weighing balance, accurate to 0,1 mg.

A.2.2.12 Gas burner, low form, adjustable, Bunsen type, carrying a draught shield, or any other suitable heating device.

A.2.3 Preparation of extraction cartridge

A.2.3.1 Place 125 ml of the alcohol (A.2.1) in the flask (A.2.2.3) and a new cartridge (A.2.2.5) in the siphon tube (A.2.2.2). Introduce the siphon tube into the flask and connect it to the condenser (A.2.2.1), making sure that there is ample flow of cold water through the condenser. Adjust the rate of heating so as to give a cycle of filling and emptying in the siphon tube every 2 min and extract for 30 min. Dry the cartridge in the oven (A.2.2.10) at 100 ± 2 °C. At the end of 2 h, weigh it in a tared weighing bottle (A.2.2.6), which has been kept in the desiccator over sulphuric acid (A.2.2.9), lifting the stopper of the bottle momentarily before weighing. Repeat the operations of drying, for periods of 1 h, and weighing, until the loss in mass between two successive weighings does not exceed 2 mg.

A.2.3.2 Use only new cartridges. A number of cartridges may be extracted, dried, weighed and kept in weighing bottles in a desiccator until needed for use.

A.2.4 Procedure

A.2.4.1 Before analysis, thoroughly mix the test sample (see F.3.1) by rolling on paper, at least ten times, to ensure uniformity of the test sample. Weigh, directly from the paper, 4,5 to 5,5 g of the sample, to an accuracy of 0,01 g, place in the beaker (A.2.2.8), add 125 ml of the alcohol (A.2.1), cover with a watch-glass and place on the hot water bath (A.2.2.7) (see figure 2). Boil the solution vigorously for 30 min to ensure complete solution of the seedlac and dispersion of wax. Keep the volume of alcohol constant by adding hot alcohol from a wash bottle, washing down the sides of the beaker.

A.2.4.2 Meanwhile, place an extracted and weighed cartridge in the filter tube. Maintain the hot water around the tube at not less than 90 °C. Wet the cartridge with hot alcohol and decant the boiling solution into the heated cartridge until the beaker is nearly empty.

A.2.4.3 Wash the remaining solution and the insoluble matter into the cartridge, using a "policeman", if necessary, with successive portions of hot alcohol contained in a wash-bottle kept hot on the water bath. Finally, wash the cartridge from the top downwards with a fine stream of hot alcohol. A complete washing and transfer from the original beaker will require at least 75 ml of hot alcohol.

A.2.4.4 Transfer the cartridge containing the insoluble matter to the extraction apparatus, place 125 ml of alcohol in the extraction flask and connect up the apparatus. Start the water flowing through the condenser, making sure that there is an adequate supply for efficient condensation. Light the burner and time the extraction from the first emptying of the siphon, running the extraction for exactly 1 h. Adjust the rate of heating so that a complete filling and emptying of the siphon tube takes place every 2 min, as determined by a stop-watch or preferably by a good two-minute sand-glass, one for each extraction apparatus.

A.2.4.4.1 In this way exactly 30 cycles per hour are accomplished. If this cycle rate is not meticulously maintained, neither check results on duplicate samples in the same laboratory nor concordant figures from one laboratory to another can be obtained, even when working on the same standard sample. It is also necessary to protect the apparatus from draughts while in operation, otherwise the proper cycle rate cannot be maintained.

A.2.4.4.2 Occasionally, seedlacs are encountered which do not yield the required number of 30 siphonings per hour, due to slow filtration, in these cases, continue the extraction until 30 siphonings have been accomplished or repeat the test with a 2 g test portion and report the sample as abnormal or slow filtering.

A.2.4.5 Remove the cartridge, drain in an upright position on filter paper and dry in the oven at 100 ± 2 °C. After drying for 2 h, place it in the weighing bottle, cool in the desiccator over sulphuric acid and weigh, removing the stopper momentarily just before weighing. Repeat the operations of drying, for periods of 1 h, and weighing, until the loss in mass between two successive weighings does not exceed 2 mg. From the mass of the residue and the mass of the sample, calculate the percentage of insoluble matter. Use the lowest mass in the calculation.

A.2.5 Calculation

The matter insoluble in hot alcohol, expressed as a percentage by mass, is given by the formula

$$\frac{100\, m_2}{m_1}$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the residue.

A.3 METHOD II

A.3.1 Reagent

Alcohol : 95 % (*V/V*) ethanol, or 95 % (*V/V*) denatured spirit.

A.3.2 Apparatus[1]

The apparatus consists of siphon tube, adaptor, condenser and flask, assembled with the aid of corks or ground glass joints so that the solvent can be kept boiling in the flask and its vapour pass upwards by way of the adaptor to the condenser. The refluxing solvent runs from the condenser into the cup of the siphon tube.

A.3.2.1 Siphon tube, of glass, of the type shown in figure 3, having approximate internal dimensions of 52 mm in height and 32 mm in diameter, resting in an adaptor tube in such a way that the siphon tube is surrounded by the ascending vapours of the boiling solvent (see figure 3).

A.3.2.2 Condenser, of any convenient pattern.

A.3.2.3 Flask, of any convenient size.

A.3.2.4 Filter paper, 125 mm in diameter, medium grade[2].

A.3.2.5 Weighing bottles, of glass, approximately 80 mm in height and approximately 40 mm in diameter, with ground glass stoppers.

A.3.2.6 Beaker, 100 ml capacity.

A.3.2.7 Drying oven, maintained at 100 ± 2 °C.

A.3.2.8 Desiccator, containing sulphuric acid, ρ 1,84 g/ml, as desiccant.

A.3.2.9 Balance.

A.3.2.10 Gas burner, low form, adjustable, Bunsen type, carrying a draught shield, or any other suitable heating device.

Dimensions in millimetres
All dimensions are approximate

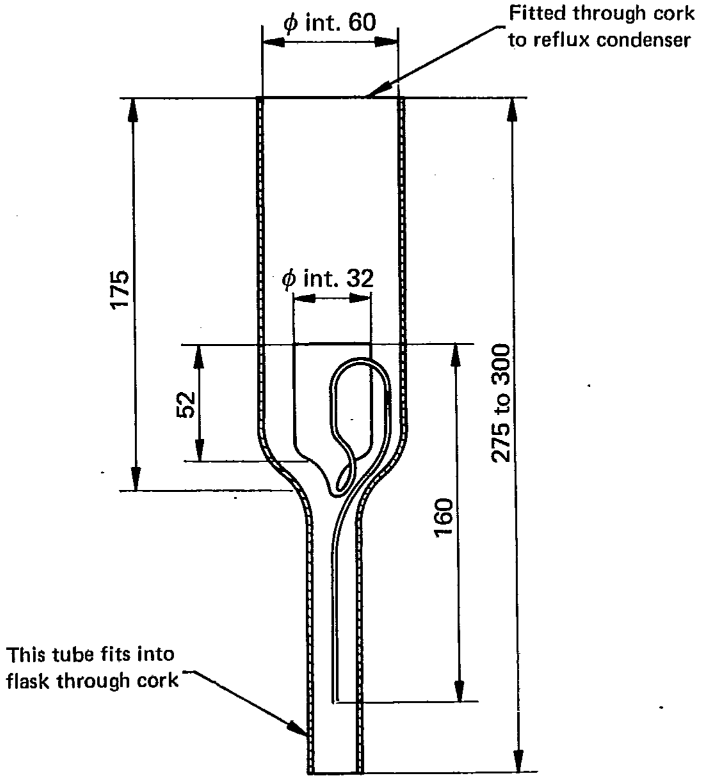


FIGURE 3 — **Siphon tube and adaptor**

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[1] The type of extraction apparatus used is not critical, provided that it is of such a design as to ensure a continuous series of extractions at approximately the boiling temperature of the solvent. If preferred, the apparatus described in Method I (see A.2), consisting of siphon tube, condenser and flask, could be satisfactorily used.

[2] Whatman No. 1 or its equivalent is suitable.

A.3.3 Procedure

A.3.3.1 Fold the filter paper (A.3.2.4) so that it forms a completely closed envelope (see figure 4). Mark this paper S (for "sample"); wrap it closely in a second filter paper marked C (for "counterpoise"). Separate the filter papers and dry in the oven (A.3.2.7) at 100 ± 2 °C for 30 min. Rapidly transfer to separate weighing bottles (A.3.2.5) which have been kept in the desiccator over sulphuric acid (A.3.2.8). Place each bottle and its contents back in the desiccator for 20 min, then weigh by counterbalance, preferably using a rapid-weighing balance of the aperiodic type.

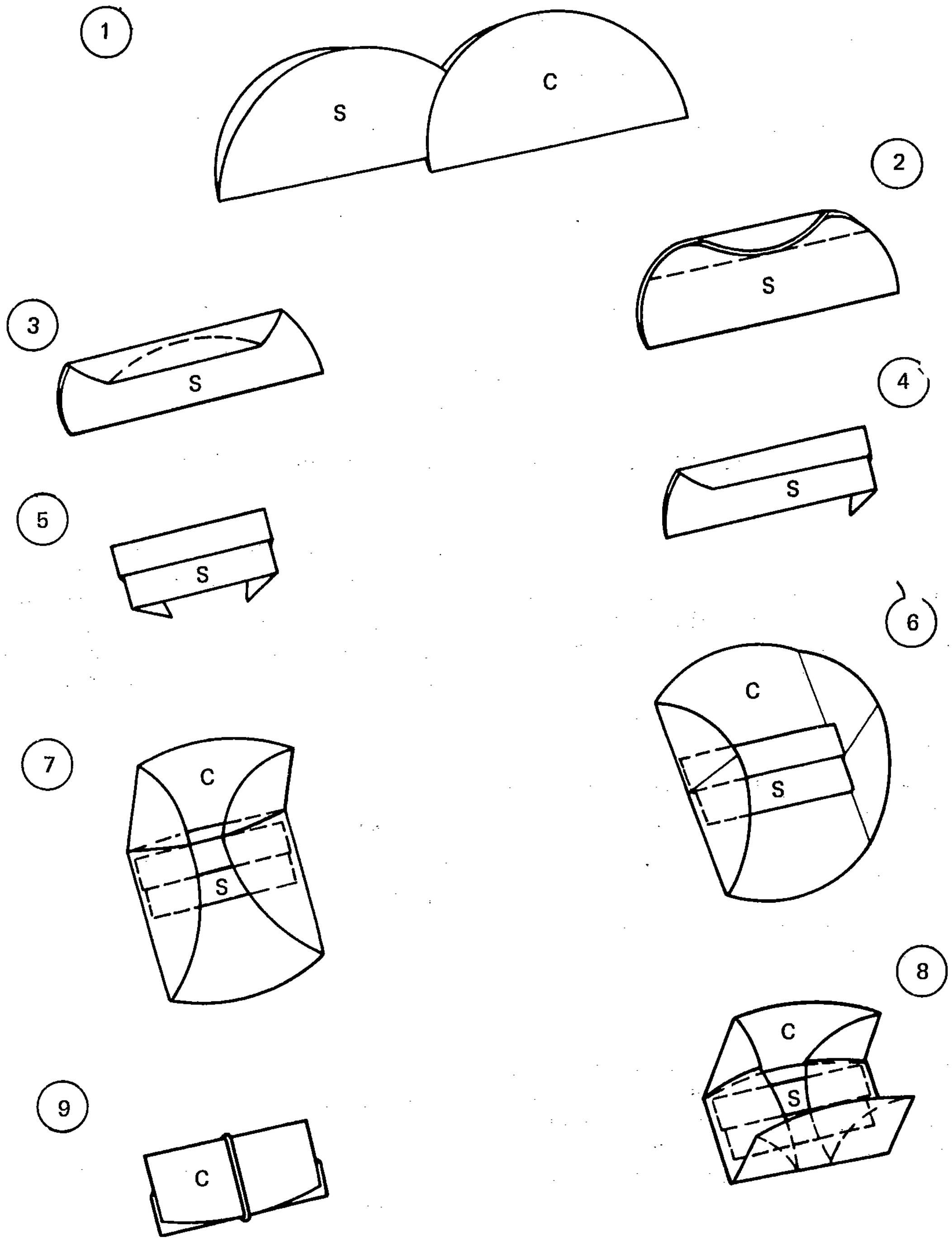

FIGURE 4 — **Folding of filter paper**

A.3.3.2 Weigh 4,5 to 5,5 g of the test sample of seedlac (see F.3.1) to an accuracy of 0,01 g and place in the filter paper envelope S; fold in the original folds, taking care not to leave any channel through which finely divided material might escape. Again enclose in paper C and secure with thread. Place the resulting envelope in the beaker (A.3.2.6) and cover it with the alcohol (A.3.1). Allow to stand overnight at room temperature. Place the envelope in the cup of the siphon tube (A.3.2.1) and extract continuously with hot alcohol for 4 h. Keep the envelope wholly below the surface of the alcohol, when the cup is full. Maintain a rapid rate of extraction throughout, though the exact time taken for the cycle of filling and emptying the cup of the siphon tube is not critical.

A.3.3.3 At the end of the specified time, remove the paper envelope, allow to drain, separate the two papers, dry each on a glass plate in air for 15 min and then for 2 h in the oven (A.3.2.7) at 100 ± 2 °C. Place the papers rapidly in their respective weighing bottles, allow to stand in the desiccator (A.3.2.8) for 20 min and again weigh by counterbalance, after momentarily removing and replacing the stoppers in the usual manner. Dry the papers for a further period of 1 h at 100 ± 2 °C and weigh again. If there is a loss in mass in excess of 2 mg, repeat the operations of drying and weighing until the difference between two successive weighings is less than 2 mg. Use the lowest mass in the calculation.

A.3.4 Calculation

As for method I (see A.2.5).

ANNEX B

(See 6.1)

DETERMINATION OF VOLATILE MATTER (MOISTURE)

B.1 PRINCIPLE

The volatile matter (moisture) content is determined by heating a weighed specimen of the "sample as received" at 40 ± 2 °C for 4 h and then keeping it over concentrated sulphuric acid in vacuo for 18 h.

B.2 APPARATUS

B.2.1 Flat-bottomed dish, of 75 mm diameter, provided with a ground glass cover.

B.2.2 Oven, well-ventilated, maintained at 40 ± 2 °C.

B.2.3 Vacuum desiccator, containing sulphuric acid, ρ 1,84 g/ml, as desiccant.

B.3 PROCEDURE

B.3.1 For this test, use the "sample as received" ground to specified size (see F.3.2). Weigh the clean, dry dish with its cover (B.2.1). Transfer approximately 2 g of the powdered sample to the dish, cover it with its cover and weigh it again. The difference gives the mass of the test portion.

B.3.2 Keep the dish with the test portion, without covering it, in the oven (B.2.2) maintained at 40 ± 2 °C for 4 h. At the end of this period, transfer the dish to the vacuum desiccator containing sulphuric acid (B.2.3). Immediately evacuate the desiccator and keep the sample uncovered in vacuo for 18 h. Remove the dish, cover it with its cover, and immediately weigh.

B.4 CALCULATION

The volatile matter (moisture), expressed as a percentage by mass, is given by the formula

$$\frac{100\,(m_1 - m_2)}{m_1}$$

where

m_1 is the mass, in grams, of the test portion taken for drying;

m_2 is the mass, in grams, of the test portion after drying.

ANNEX C
(See 6.2)

DETERMINATION OF COLOUR INDEX

C.1 PRINCIPLE

The colour index is determined by comparing the colour of a standard iodine solution with the solution of seedlac in ethanol by diluting the latter progressively with ethanol until a close match is obtained.

C.2 REAGENTS

C.2.1 Alcohol : 95 % (*V*/*V*) ethanol, or 95 % (*V*/*V*) denatured spirit, provided that it is colourless.

C.2.2 Iodine, standard solution.

Prepare a 0,005 N standard iodine solution by measuring 5,0 ml of 0,1 N standard iodine solution (in potassium iodide solution), from a burette, into a 100 ml one-mark volumetric flask and diluting to the mark with water. This solution corresponds to colour index 5. Shake the solution before use.

C.3 APPARATUS

C.3.1 Conical flask, stoppered, of capacity 250 ml.

C.3.2 Filter paper, medium grade.

C.3.3 Test tubes, thin-walled, measuring 200 mm x 13 mm.

C.3.4 Burette.

C.4 PROCEDURE

C.4.1 Add 100 ml of the alcohol (C.2.1) to 10,00 g of the test sample (see F.3.1) contained in the flask (C.3.1). Stopper and shake vigorously as soon as alcohol is added, to prevent coalescence of lac particles, and then intermittently over a period of 4 h. Allow to stand for 16 to 24 h at 27 ± 2 °C, shake again, and allow to settle at 27 ± 2 °C for 2 h. Filter the solution in an ordinary funnel using medium grade filter paper (C.3.2) and keeping the funnel covered. Discard the first 15 ml or more of the clear filtrate.

NOTE — A temperature of 20 ± 2 °C or 23 ± 2 °C may be used in temperate climates (see ISO 554).

C.4.2 Pipette 5,0 ml of the filtered seedlac solution into a test tube (C.3.3). Take an adequate volume of the standard iodine solution (C.2.2) in another test tube (C.3.3) for matching. Compare the colours of the two solutions, holding the test tubes against light with a piece of moistened filter paper or opal glass interposed between the light source and the test tubes. Add the alcohol (C.2.1) from the burette (C.3.4) to the seedlac solution, while shaking, until the colour is the same as that of the standard solution. Note the volume of alcohol added.

NOTE — It will be found advantageous to use a standard type of light source and viewing cabinet to cut off extraneous light.

C.5 CALCULATION

C.5.1 The volume, in millilitres, of alcohol so added plus 5, or the total volume, in millilitres, of the seedlac solution after such dilution, is the colour index of the sample.

C.5.2 The accuracy of this test, including the personal error of different analysts, is about 5 %.

ANNEX D
(See 6.4)

DETERMINATION OF MATTER SOLUBLE IN WATER AND OF NEUTRALITY OF AQUEOUS EXTRACT

D.1 PRINCIPLE

A known mass of powdered seedlac is digested with water, made up to a known volume and filtered. The mass that dissolves is determined by evaporating an aliquot portion of the filtrate to constant mass and calculating for the whole solution.

D.2 REAGENTS

D.2.1 Methyl red indicator solution.

D.3 APPARATUS

D.3.1 Sieve, having a nominal aperture of 0,25 mm[1].

D.3.2 Beaker, of 500 ml capacity, provided with a watch-glass.

D.3.3 Volumetric flask, one-mark, 250 ml capacity.

D.3.4 Evaporating dish.

D.3.5 Drying oven, maintained at 100 ± 2 °C.

D.3.6 Desiccator, containing sulphuric acid, ρ 1,84 g/ml, as desiccant.

D.4 PROCEDURE

D.4.1 Finely grind a sufficient quantity of the test sample (see F.3.1) to pass the sieve (D.3.1). Weigh, to an accuracy of 0,1 g, 20 to 25 g of the powdered sample and transfer to the beaker (D.3.2). Add 200 ml of distilled water and stir thoroughly. Cover the beaker with a watch-glass and allow it to stand at 27 ± 2 °C for 4 h, with occasional stirring.

NOTE — A temperature of 20 ± 2 °C or 23 ± 2 °C may be used in temperate climates (see ISO 554).

D.4.2 Filter by gravity or suction into the volumetric flask (D.3.3). Wash the residual seedlac and then filter with distilled water and dilute to the mark. Transfer a measured volume of the filtrate into the weighed evaporating dish (D.3.4) and evaporate to dryness. Dry the residue in the oven maintained at 100 ± 2 °C (D.3.5), cool in the desiccator (D.3.6) and weigh. Repeat the operations of drying and weighing until the loss in mass between two successive weighings does not exceed 2 mg.

D.4.3 Test for acidity in another aliquot portion of the solution using the methyl red (D.2.1) and observe whether the aqueous extract is neutral to methyl red or not.

D.5 CALCULATION

D.5.1 The matter soluble in water, expressed as a percentage by mass, is given by the formula

$$\frac{m_2}{V \times m_1} \times 2{,}5 \times 10^4$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the residue;

V is the volume, in millilitres, of the filtrate taken for evaporation.

D.5.2 Report the reaction of the aqueous extract to methyl red.

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[1] See ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ANNEX E
(See 6.6)

DETERMINATION OF WAX CONTENT

E.1 PRINCIPLE

A specified quantity of seedlac is dissolved in a hot solution of sodium carbonate, the wax is separated by filtering, extracted with chloroform and weighed after drying.

E.2 REAGENTS

E.2.1 Sodium carbonate, anhydrous, analytical grade.

E.2.2 Chloroform, redistilled, free from non-volatile residue.

E.2.3 Filter-air[1], previously extracted with chloroform and dried before use.

E.3 APPARATUS

E.3.1 Any suitable extraction apparatus, such as that described in annex A, method I, may be used.

E.3.2 Beaker, tall-form, 200 ml capacity, provided with a watch-glass.

E.3.3 Water-bath.

E.3.4 Filter paper, low-ash, retentive, acid-washed, 12,5 cm in size[2].

E.3.5 Drying ovens, maintained at 40 ± 2 °C and 100 ± 2 °C.

E.3.6 Extraction cartridge, made of fat-free paper, 25 mm x 60 mm.

E.3.7 Desiccator, containing sulphuric acid, ρ 1,84 g/ml, as desiccant.

E.4 PROCEDURE

E.4.1 Weigh 9,5 to 10,5 g of the test sample (see F.3.1) to an accuracy of 0,01 g and dissolve in 150 ml of hot water containing 2,5 g of sodium carbonate (E.2.1) in the beaker (E.3.2). Immerse the beaker in the boiling water bath (E.3.3) and stir until the lac is in solution. Then cover with a watch-glass and allow it to remain in the bath for 2 to 3 h more, without agitation. Remove the beaker from the bath and place it in cold water. The wax will now come to the surface and either solidify as a layer or float as small, hard particles, according to the amount of wax present in the sample. Either filter this solution through the filter paper (E.3.4), by gravity, or use a Büchner funnel with suction. In the latter case, it is necessary to embed the filter paper in the Büchner funnel with filter-aid, by mixing 1 g of the filter-air (E.2.3) with water and pouring this mixture onto the paper with the suction on. Filtration by this method is also further aided by stirring 0,5 g of the filter-air into the seedlac solution before starting the filtration.

E.4.2 If the filtration is carried out under gravity alone, then, after the filtration is completed and all soluble seedlac washed out of the paper with water, remove the paper from the funnel and, without further folding it, place it in the beaker, resting it against the stirring rod so that the edge of the paper remains level with the top edge of the beaker. Keep the beaker containing the paper in an oven maintained at 40 ± 2 °C (E.3.5), for several hours, to remove most of the water.

Next, remove the paper from the beaker, wrap carefully in a large piece of clean, fat-free filter paper, bind with fine wire and place it in the extraction cartridge (E.3.6) which has been previously extracted with hot chloroform (E.2.2). Place the cartridge containing the wax and paper in the extraction apparatus (E.3.1) and pour into the beaker which previously contained the filter paper and wax, a portion of the chloroform to be used for the extraction.

Bring the solvent to the boil and pour it through the extraction cartridge, collecting it in the extraction flask to be used. Repeat this operation twice more, so as to remove the whole of the residual wax from the beaker. Then connect up the apparatus and extract for at least 2 h. Distil off most of the solvent and transfer the residue to a tared glass basin. Wash the extraction flask three times with small portions of chloroform, and pour into the basin. Evaporate to dryness and then dry the residue in the oven (E.3.5) at 100 ± 2 °C for 30 min, cool in the desiccator (E.3.7) and weigh. Repeat the operations of drying for 30 min and weighing until the loss in mass between two successive weighings does not exceed 0,002 g. Use the lowest mass in the calculation.

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1) An example of a suitable filter-aid is a diatomaceous material sold under the name "Filter-Cel".

2) Whatman No. 40 or Munktells No. 2 or equivalent is suitable.

E.4.3 If the Büchner funnel is used, then, after the filtration has been completed and the paper has been well washed with water to take out all soluble seedlac, leave the vacuum on for a few minutes so as to suck out as much water as possible. It will then be possible to insert a thin spatula under the edge of the paper and remove it from the funnel, without leaving more than traces of the filter-aid adhering to the funnel walls. Remove such traces by wiping with pieces of alcohol-moistened paper, combine these with the main paper, and wrap the whole, while still damp, in a large piece of filter paper and bind firmly with fine wire.

Dry this at 40 ± 2 °C. When dry, place it in the extraction cartridge which has been previously extracted with chloroform. Transfer the cartridge and wax to the extraction apparatus, and extract for 2 h with chloroform. Distil off the solvent and transfer the residue to a tared glass basin. Wash the extraction flask three times with small portions of chloroform, and pour into the basin. Evaporate to dryness and then dry the residue in the oven at 100 ± 2 °C for 30 min, cool in the desiccator and weigh. Repeat the operations of drying for 30 min and weighing until the loss in mass between two successive weighings does not exceed 0,002 g. Use the lowest mass in the calculation.

E.5 CALCULATION

The wax content, expressed as a percentage by mass, is given by the formula

$$\frac{m_2}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the wax.

ANNEX F
(See clause 7)

SAMPLING

F.1 DRAWING OF SAMPLES

NOTE — It is essential that the operations described for the drawing, reduction and preparation of laboratory samples be carried out as rapidly as possible in order to minimize loss of moisture.

F.1.1 Prepare samples from original, unopened packages of seedlac.

F.1.2 Not less than 10 % of the packages, selected at random from each lot, shall be sampled.

F.1.3 For this purpose a lot shall not exceed 200 packages.

F.1.4 Unused portions of samples shall be sent to the purchaser on request.

F.1.5 Free-flowing seedlac

Take samples from different places in each package by means of a suitable tryer so as to yield a total of 5 kg of material consisting of approximately equal portions from each package sampled. Thoroughly mix the material, heap and quarter along two diameters which intersect at right angles, and mix two opposite quarters. If necessary, further subdivide one-half of the material by the normal process of quartering to form a number of samples which are known as "samples as received" and shall be used for the determination of volatile matter (moisture). Place these samples in airtight containers and seal and label them accordingly. Treat them as described in F.3.2. Treat the other half of the material as described in F.2.1 to form the "laboratory sample".

F.1.6 Blocky or matted seedlac

Take samples from different places in each package, by chipping or other suitable means, so as to yield a total of 5 kg of material consisting of approximately equal portions from each package sampled. Thoroughly mix the material, heap and quarter along two diameters which intersect at right angles, and mix two opposite quarters. If necessary, further subdivide one-half of the material by the normal process of quartering to form a number of samples which are known as "samples as received" and shall be used for the determination of volatile matter (moisture). Place these samples in airtight containers, and seal and label them accordingly. Treat them as described in F.3.2. Grind roughly the other half of the material so as to pass a sieve having a nominal aperture of 6,3 mm¹⁾ and then treat as described in F.2.1 to form the "laboratory sample".

F.2 REDUCTION OF SAMPLES

NOTE – If the material at any time during the following operations shows signs of surface moisture, air dry it at room temperature before further mixing and grinding.

F.2.1 Mix thoroughly the material for the laboratory sample, as obtained under F.1.5 or F.1.6, and heap and quarter along two diameters which intersect at right angles. Mix two opposite quarters and grind to pass entirely through a sieve having a nominal aperture of about 2 mm[1]. Thoroughly mix the material and quarter so as to yield four samples of approximately 250 g each. Place these four samples in airtight containers, seal and label them as "laboratory sample" and send to the interested parties.

F.2.2 Attach to each sample a label giving the date of sampling, the number of packages sampled, the condition of the packages and contents, and the name and code number of the vendor.

F.3 PREPARATION OF TEST SAMPLES

F.3.1 Grind the laboratory samples to pass entirely through a sieve whose nominal aperture does not exceed 0,425 mm[1], then mix thoroughly, place in an airtight container and label it as "test sample".

F.3.2 Grind the "samples as received" (see F.1.5 and F.1.6) to pass a sieve whose nominal aperture is about 0,850 mm[1]. Grinding shall be done with as little heat production as possible. Place the ground sample immediately in an airtight container.

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[1] See ISO 565.