BRITISH STANDARD

BS 3279:1960

Specification for

Seedlac

Confirmed
February 2011

Co-operating organizations

The Pigments, Paints and Varnishes Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Admiralty
Air Ministry
Association of British Chemical Manufacturers
Board of Trade*
British Colour Makers' Association
British Railways, The British Transport Commission
Building Industry Distributors
Crown Agents for Oversea Governments and Administrations
D.S.I.R. — Building Research Station
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White Lead Convention
Zinc Development Association
Zinc Pigment Development Association

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the Committee entrusted with the preparation of this British Standard:

British Electrical and Allied Industries Research Association
British Electrical and Allied Manufacturers Association
British Hat and Allied Feltmakers Research Association
London Chamber of Commerce, Incorp.
London Shellac Trade Association
Society for Analytical Chemistry
Tropical Products Institute
Individual manufacturers

This British Standard, having been approved by the Pigments, Paints and Varnishes Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 31 October 1960

The following BSI references relate to the work on this standard:
Committee reference PVC/4
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Amendments issued since publication

Amd. No.	Date	Comments

Contents

Foreword

This standard makes reference to the following British Standards:

BS 410, *Test sieves.*

BS 1957, *Presentation of numerical values (fineness of expression; rounding of numbers).*

During recent years, lac has been studied internationally through the International Organization for Standardization (ISO), by various Research Institutions and Trade Organizations in different parts of the world.

It has been decided to split the subject matter of BS 954, the previous British Standard for all types of lac other than bleached lac, into three parts, covering seedlac, hand-made shellac and machine-made shellac, and to publish these as separate documents. Advantage has been taken of the work done at the ISO level, and the Committee responsible for the preparation of this standard desires to acknowledge its indebtedness to the sources of information thus utilized.

The usual trade descriptions of the various grades of seedlac, as recognized in the United Kingdom, have been used throughout. These trade descriptions are well known internationally, and are recognized standards of the London Shellac Trade Association. An attempt has been made, however, to correlate these with the designations of the Recommendations of the International Organization for Standardization: the approximate correspondence for seedlac is as follows:

U.K. grading	Approximate ISO grading
Golden Kusmi	Special
Kusmi No. I	A
Kusmi No. 2	B
Golden Bysacki	C
Fine Bysacki	D
No equivalent	E
Ordinary Bysacki	F

The mandatory requirements included in this specification are those for appearance and colour, volatile matter (moisture) and matter insoluble in hot alcohol, and methods of test are given for these. Also included are optional clauses and methods of test, operative only by agreement between purchaser and vendor, for colour index, matter soluble in water, non-volatile matter soluble in cold alcohol, and wax.

It will be noted that no requirements have been given for a "bleach test" or "bleachability" or for the absence of visible impurities. In the opinion of the Committee, work is not yet in a sufficiently advanced state to allow of the former, and visible impurities are effectively limited by the provisions of Clauses **2** and **5**.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 9 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Mandatory clauses

1 Scope

This British Standard specifies requirements for seedlac, the product obtained by washing crushed sticklac, the natural product of the lac insect (*Laccifer lacca*). Unless otherwise agreed between purchaser and vendor, the seedlac shall be of Indian origin and shall be of the crop implied by the grade name employed.

Six grades are specified, as follows:

Golden Kusmi
Kusmi No. 1
Kusmi No. 2
Golden Bysacki
Fine Bysacki
Ordinary Bysacki

2 Description

The form and condition of the material shall be as agreed between purchaser and vendor. The appearance and colour of the material shall be not inferior to those of an agreed sample, when judged by visual examination.

3 Sample

For the purpose of examination under this specification samples shall be drawn, parted and prepared in the manner described in Appendix A. Only original unopened packages of shellac shall be sampled.

Not less than 10 per cent of the packages, selected at random from each lot of not more than 200 packages, shall be sampled.

4 Volatile matter

The material shall not yield more than 2.5 per cent by weight of volatile matter when tested in the manner described in Appendix B.

5 Matter insoluble in hot alcohol

The material shall not contain more than the following specified limits of matter insoluble in hot alcohol, when tested in the manner described in Appendix C. By agreement between purchaser and vendor this limit may be relaxed but under no circumstances shall it exceed the relaxed limit.

Grade	Limit	Relaxed limit
	per cent by weight	
Golden Kusmi	2.0	4.0
Kusmi No. 1, Kusmi No. 2, Golden Bysacki	3.0	5.0
Fine Bysacki	3.0	6.0
Ordinary Bysacki	5.0	8.0

6 Expression of analytical results

The results of analyses performed in accordance with the appendices to this standard shall be expressed to the number of significant figures indicated in the specification requirements. The principles embodied in BS 1957, "*Presentation of numerical values (fineness of expression; rounding of numbers)*", apply to such results.

Optional clauses

7 Colour index

When agreed between purchaser and vendor, the colour index of the material shall not exceed the following limits when determined in the manner described in Appendix D:

Grade	Limit
Golden Kusmi, Kusmi No. 1	8
Kusmi No. 2	10
Golden Bysacki	15
Fine Bysacki	22
Ordinary Bysacki	50

8 Matter soluble in water

When agreed between purchaser and vendor, the material shall not contain more than 1.0 per cent by weight of matter soluble in water, when determined in the manner described in Appendix F.

9 Non-volatile matter soluble in cold alcohol

When agreed between purchaser and vendor, the material shall contain not less than the following amounts of non-volatile matter soluble in cold alcohol, when tested in the manner described in Appendix E:

Grade	Limit
	per cent by weight
Golden Kusmi	90.0
Kusmi No. 1	89.0
Kusmi No. 2	
Golden Bysacki	
Fine Bysacki	
Ordinary Bysacki	87.0

10 Wax

When agreed between purchaser and vendor, the material shall not contain more than 5.5 per cent by weight of wax, when determined in the manner described in Appendix G.

Appendix A Sampling

Drawing of samples

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 (or 10 lb) of material consisting of approximately equal portions from each package sampled. Thoroughly mix the material and heap and quarter along two diameters which intersect at right angles. Mix two opposite quarters and preserve in air-tight containers, seal and label as the "Original observation sample". If necessary, further sub-divide by quartering to form a number of original observation samples. Part the other two quarters of the material as described below to form the analysis sample.

Blocky or matted seedlac. Take small pieces [not more than 1.2 in (3 cm) across] from different places in each package by chipping or other suitable means so as to yield a total of 5 kg (or 10 lb) of material consisting of approximately equal portions from each package sampled. Thoroughly mix the material and heap and quarter along two diameters which intersect at right angles. Mix two opposite quarters and preserve in air-tight containers, seal and label as the "Original observation sample". If necessary, further sub-divide by quartering to form a number of original observation samples. Part the other two quarters of the material as described below to form the analysis sample.

Preparation of samples

Thoroughly mix the material obtained above, and heap and quarter along two diameters which intersect at right angles.

Mix two opposite quarters and grind the mixture to pass entirely through an 8 mesh BS test sieve[1)]. Thoroughly mix and twice divide by quartering into four samples, each of approximately 300 g (or 11 oz). Place these samples in air-tight containers, seal and label each as "Analysis sample".

a) *Sample for determination of volatile matter*. Grind an adequate amount (40 to 50 g) of the "Analysis sample" to pass entirely through an 18 mesh BS test sieve[1)], taking all precautions to avoid loss of volatile matter. Place in an air-tight container and label "Sample for volatile matter determination".

b) *Prepared sample for all determinations other than volatile matter*. Grind an adequate amount of the "Analysis sample" to pass entirely through a 36 mesh BS test sieve[1)]. Transfer to an air-tight container and label as "Prepared sample for all determinations other than that for volatile matter".

Appendix B Method for the determination of volatile matter

Principle

The volatile matter content is determined by heating a weighed portion of the sample at 41 ± 1 °C for 4 hours and then keeping it over concentrated sulphuric acid *in vacuo* for 18 hours.

Procedure

Weigh, to an accuracy of 0.0005 g, a clean, dry, flat-bottomed dish of about 75 mm diameter, provided with a ground glass cover. Transfer about 2 g of the sample reserved for this purpose (see Appendix A) into the dish and re-weigh to the same accuracy. Place the dish and its cover separately in a well-ventilated oven maintained at 41 ± 1 °C for 4 hours. At the end of this period, transfer the dish and cover to a vacuum desiccator containing concentrated sulphuric acid. Immediately evacuate the desiccator and continue drying *in vacuo* for 18 hours. Remove the dish and immediately replace the cover and weigh to an accuracy of 0.0005 g. Express the loss in weight as a percentage of the weight of the sample taken.

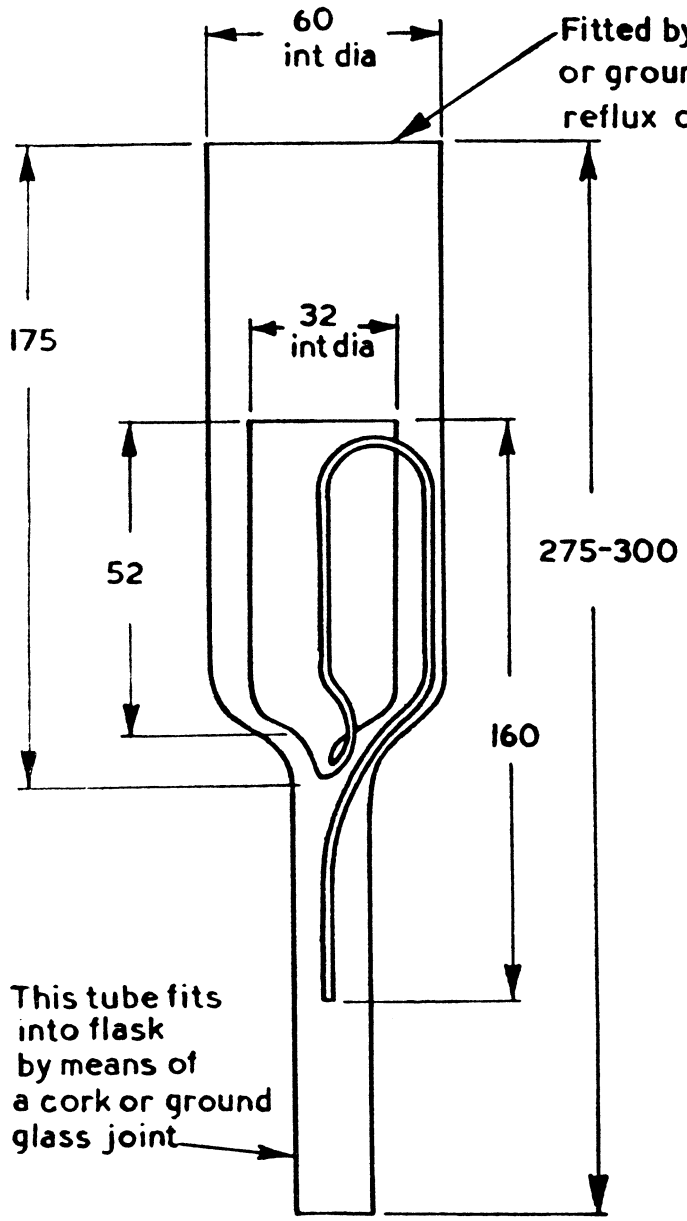
Calculation

$$\text{Volatile water, per cent by weight} = \frac{100(W - W_1)}{W}$$

where W = weight in grammes of sample taken,

and W_1 = weight in grammes of sample after drying.

[1)] BS 410, "*Test sieves*".



Dimensions in millimetres

Figure 1 — Siphon tube and adaptor

Appendix C Method for the determination of matter insoluble in hot alcohol

Apparatus

a) *Siphon tube*[2)], of glass, of the Knoefler type, having minimum internal dimensions of 52 mm height and 32 mm diameter, resting in an adaptor tube in such a way that the siphon tube is surrounded by the ascending vapours of the boiling solvent (Figure 1).

b) *Condenser*, of any convenient pattern.

c) *Flask*, of any convenient size.

d) *Filter paper*, 12.5 cm diameter, medium grade[3)].

e) *Weighing bottles*, of glass, height 80 ± 1 mm, diameter 40 ± 1 mm with ground glass stoppers.

Assembly of apparatus

Assemble the siphon tube, adaptor, condenser and flask, using corks or ground glass joints, so that the solvent can be kept boiling in the flask and its vapour passed upwards, by way of the adaptor, to the condenser and the refluxing solvent run from the condenser into the cup of the siphon tube.

Reagent

Alcohol, 95 per cent (v/v) ethanol or 95 per cent (v/v) industrial methylated spirit[4)].

Procedure

Fold a filter paper so that it forms a completely closed envelope, as illustrated in Figure 2. 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 an oven at 100 ± 2 °C for 30 minutes. Rapidly transfer to weighing bottles which have been kept in a desiccator over concentrated sulphuric acid. Place each bottle and its contents back in the desiccator for 20 minutes, then weigh by counterpoise, preferably using a rapid weighing balance of the aperiodic type. Weigh 4.5 to 5.5 g of the prepared sample (see Appendix A) to an accuracy of 0.01 g and place in the filter paper envelope, *S*, and fold in the original folds, taking care not to leave any channel through which finely divided material might afterwards escape. Again enclose in the paper, *C*, and secure with thread. Place the resulting package in a 100 ml beaker and cover it with alcohol. Allow to stand overnight at room temperature. Place the package in the cup of the siphon tube and extract continuously with hot alcohol for 4 hours. Keep the package wholly below the surface of the alcohol when the cup is full. The exact time taken for the cycle of filling and emptying the cup of the siphon tube is not critical, but a rapid rate of extraction should be maintained throughout.

At the end of the specified time, remove the package, allow to drain, separate the two papers, dry each on a glass plate in a steam oven and then for 3 hours in a thermostatically controlled oven at 100 ± 2 °C. Place the papers rapidly in their respective weighing bottles, stand in the desiccator for 20 minutes and again weigh by counterpoise, after momentarily removing and replacing the stoppers in the usual manner. Dry the papers for a further period of 1 hour at 100 ± 2 °C and weigh again: if there is a loss in weight exceeding 0.002 g repeat the processes of drying and weighing until the difference between successive weighings is less than 0.002 g. Use the lowest weight in the calculation.

[2)] 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.

[3)] Whatman No. 1 or Green's No. 401 paper is suitable.

[4)] It should be noted that the use of industrial methylated spirit is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230).

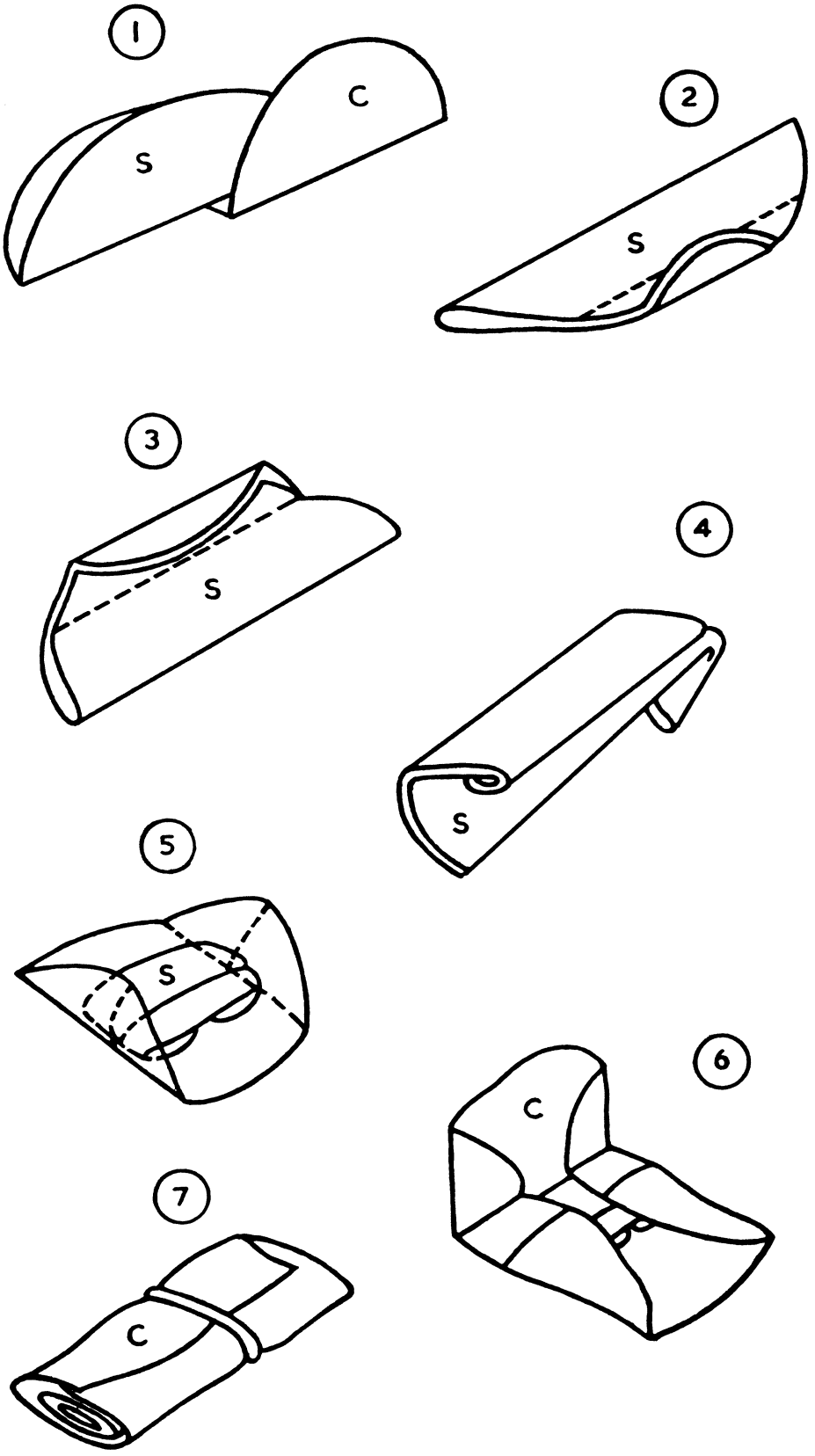


Figure 2 — Folding of filter paper

Calculation

Matter insoluble in hot alcohol, per cent by weight = $\frac{100 W_1}{W}$

where W_1 = weight in grammes of residue,

and W = weight in grammes of sample taken.

Appendix D Method for the determination of colour index

Principle

The colour of a solution of seedlac in alcohol is matched with that of an iodine solution of known concentration. For the purposes of this specification the colour of a freshly made 0.005N iodine solution in aqueous potassium iodide is arbitrarily designated as of colour index 5.

Reagents

a) *Alcohol*, 95 per cent (v/v) ethanol or 95 per cent (v/v) industrial methylated spirit[5)].

b) *Standard iodine solution*, 0.005N. Prepare freshly by appropriate dilution of a 0.1N solution of iodine in potassium iodide with water.

Procedure

Add 100 ml of the alcohol to 10.5 g of the prepared sample (see Appendix A) contained in a stoppered flask. Shake vigorously as soon as the alcohol is added, to prevent coalescence of lac particles, then intermittently over a period of 4 hours. Allow to stand for 16 to 24 hours at 20 ± 5 °C, shake again and allow to settle at 20 ± 2 °C for 2 hours. Filter the solution in an ordinary funnel using a medium grade filter paper and keeping the funnel covered. Discard the first 15 ml of the filtrate and then collect 5 ml or more of the clear filtrate for the test.

By means of a pipette transfer 5.0 ml of the filtered lac solution to a thin-walled 200 × 13 mm test tube. Transfer 5.0 ml of the standard iodine solution to another test tube, similar in every respect, for matching. Compare the colour of the two solutions holding the test tubes against the light with a piece of moistened filter paper or opal glass interposed between the light source and the test tube. It will be found advantageous to use a standard type of light source and a viewing cabinet to cut off extraneous light. Add the alcohol from a burette to the lac solution with mixing, until the colour is the same as that of the standard iodine solution. Note the volume of alcohol added.

When examining dark lacs, of colour index higher than 25, use a flask for the dilutions with alcohol.

Calculation

Colour index = $A + 5$

where A is the volume in millilitres of alcohol added to the lac solution.

NOTE The variability of this test is about ± 5 per cent of the observed figure.

Appendix E Method for the determination of non-volatile matter soluble in cold alcohol

Reagents

a) *Alcohol,* either *ethanol,* redistilled before use to ensure the absence of non-volatile matter and adjusted to 95 per cent (v/v) before use, or *industrial methylated spirit*[5)] free from non-volatile impurities.

b) *Acetone*, of recognized analytical reagent quality, redistilled before use.

Apparatus

a) *Conical flask,* of 150 ml capacity, with a well-fitting ground glass stopper.

b) *Nickel dishes,* flat-bottomed, 70 to 80 mm in diameter and 15 to 25 mm high.

[5)] It should be noted that the use of industrial methylated spirit is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230).

Procedure

Place 10 ± 0.010 g of the prepared sample (see Appendix A) in the flask, and add 90 ± 0.10 g of the alcohol. Shake the mixture vigorously as soon as the alcohol is added, to prevent coalescence of the lac particles, then intermittently over a period of 4 hours. Allow to stand for 16 to 24 hours at 20 ± 5 °C, shake again, then allow to settle at 20 ± 2 °C for 2 hours. Remove a portion of the clear upper layer by means of a pipette and filter it rapidly through a rapid open-texture filter paper[6], keeping the funnel and receiver covered to minimize loss of solvent by evaporation. After rejecting the first 5 to 10 ml of the filtrate, weigh from a weighing pipette[7] 2.5 to 3.0 g, to an accuracy of 0.001 g, into a nickel dish which has been previously dried in an oven at 100 ± 2 °C and cooled and weighed to an accuracy of 0.0001 g, preferably on an aperiodic balance.

Remove the alcohol from the solution by cautious evaporation on a water bath; when the alcohol has almost disappeared, add 5 ml of the acetone, and manipulate the dish on the water bath during the final stages of the evaporation in order to obtain the residue in the form of a thin even layer on the bottom of the dish.

Heat the dish with its contents for a period of 4 hours in a well ventilated oven at a temperature of 100 ± 2 °C. Cool in a desiccator and weigh to an accuracy of 0.0001 g.

Calculation

Calculate the percentage of non-volatile matter soluble in cold alcohol in the sample as received, according to the following formula:

$$\text{Non-volatile matter soluble in cold alcohol, per cent by weight in sample as received} = \frac{W_1(900 + M)}{W - W_1}$$

where W = weight in grammes of alcoholic solution evaporated,

W_1 = weight in grammes of residue obtained by the evaporation of W grammes of alcoholic solution,

and M = the percentage of volatile matter in the lac under test, determined as described in Appendix B.

Appendix F Method for the determination of matter soluble in water

Procedure

Weigh 20 to 25 g of the prepared sample (see Appendix A), to an accuracy of 0.1 g, and transfer to a beaker. Add 200 ml of distilled water and stir thoroughly. Cover the beaker with a watch glass and allow it to stand at 20 ± 5 °C for 4 hours with occasional stirring.

Filter into a 250 ml graduated flask. Wash the residual seedlac and the filter paper with distilled water and make up to the graduation mark. Transfer at least 50 ml of the filtrate into an evaporating dish weighed to an accuracy of 0.001 g and evaporate to dryness. Dry the residue to constant weight in an oven maintained at 100 ± 2 °C until the difference between successive weighings at half-hourly intervals does not exceed 0.001 g.

Calculation

$$\text{Matter soluble in water, per cent by weight} = \frac{25\,000\,W_1}{V\,W}$$

where W_1 = weight in grammes of residue,

V = volume in millilitres taken for evaporation,

and W = weight in grammes of sample taken.

[6] A Whatman No. 4 or equivalent paper is suitable.

[7] A suitable type is the Lunge-Rey pipette.

Appendix G Method for the determination of wax

Apparatus

Any suitable extraction apparatus, such as that described in Appendix C.

Reagents

The reagents used shall be of recognized analytical reagent quality. Distilled water shall be used throughout.

a) *Sodium carbonate*, anhydrous.

b) *Sodium carbonate,* 1 per cent (w/v) solution of the anhydrous material.

c) *Chloroform*.

Procedure

Weigh 9.5 to 10.5 g of the prepared sample (see Appendix A), to an accuracy of 0.01 g, into a 200 ml tall beaker containing 2.5 g of sodium carbonate. Add 150 ml of hot water, immerse the beaker in a steam or boiling-water bath and stir intermittently until the seedlac is dissolved. Cover with a watch glass and allow it to remain in the bath for 2 to 3 hours more, without stirring. Remove the beaker from the bath and place it in cold water. As the liquid cools, the wax which comes to the top will solidify as a layer or float as small hard particles.

Add 0.5 g of filter paper pulp to the solution and filter. Use either a 12 cm double acid-washed retentive filter paper[8)] or a Büchner funnel, previously prepared by covering the filter plate with a disk of filter paper and pouring on to it 1 g of filter paper pulp dispersed in water. Wash out soluble seedlac with a little of the sodium carbonate solution followed by boiling water. Dry at a temperature of 40 ± 2 °C, wrap in a filter paper, tie or bind with thin copper wire or cotton or linen thread, place in the extraction apparatus connected to a flask, previously weighed to an accuracy of 0.001 g, and extract with chloroform for 2 hours. Distil off most of the chloroform and complete the evaporation on a water bath. Dry the wax at 100 ± 2 °C until the loss does not exceed 0.002 g in consecutive half-hourly periods of heating. Use the lowest weight in the calculation.

Calculation

Wax, per cent by weight = $\frac{100 W_1}{W}$

where W_1 = weight in grammes of wax,

and W = weight in grammes of sample taken.

[8)] A Whatman No. 40 or Green's No. 800 paper is suitable.

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