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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 57

SPECIFICATION FOR BLEACHED LAC

1st EDITION
December 1957

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 57, *Specification for Bleached Lac*, was drawn up by Technical Committee ISO/TC 50, *Lac*, the Secretariat of which is held by the Indian Standards Institution (ISI).

This Technical Committee decided to place on its program of work the question of Bleached Lac and the ISO/TC 50 Secretariat submitted a first draft proposal based on the studies made previously by the Indian Member Body.

This draft proposal was discussed at the first meeting of Technical Committee ISO/TC 50, held in New Delhi, in January 1950. A second draft proposal, which took into account the resolutions adopted at this meeting, was then circulated to the members of the Technical Committee and discussed at the second meeting of Technical Committee ISO/TC 50, held in New York, in June 1952. A major change was made at this meeting; the title of the subject was changed to "Bleached Lac", and a working document submitted by the United Kingdom was taken into consideration. A revised third draft proposal for Bleached Lac was then formulated. Some changes were made in this document at the third meeting of the Technical Committee, held in London, in October 1954 and the Technical Committee adopted the document thus revised, as a Draft ISO Recommendation.

On 12 September 1956, the Draft ISO Recommendation (No. 100) was distributed to all ISO Member Bodies and was approved, subject to modifications, by the following 21 (out of a total of 37) Member Bodies:

*Australia
Austria
*Bulgaria
*Canada
*Denmark
France
*Greece
Hungary
*Ireland
Netherlands
Pakistan
Poland
Portugal
Romania
Spain
*Sweden
Turkey
Union of South Africa
United Kingdom
U.S.A.
*U.S.S.R.

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in December 1957, to accept it as an ISO RECOMMENDATION.

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* These Member Bodies stated that they had no objection to the Draft being approved.

CONTENTS

FOREWORD

I. This ISO Recommendation is based on the decisions arrived at in the first three meetings of the Technical Committee on Lac of the International Organization for Standardization (ISO/TC 50) which were held in New Delhi, India, on January 16, 17, 18 and 19, 1950; in New York, U.S.A., on June 23, 24 and 25, 1952; and in London, United Kingdom, on October 11, 12 and 13, 1954, respectively.

II. Hitherto the material dealt with in this ISO Recommendation has been called bleached shellac. The name is due to the fact that it was manufactured formerly by bleaching shellac, which alone was the common article exported from India. Now, however, bleached lac is manufactured generally by bleaching seedlac or sometimes shellac and the old name is, therefore, incorrect and misleading. The new name introduced in this ISO Recommendation, "Bleached Lac", is therefore not a misnomer and it will, at the same time, avoid confusion between the ordinary shellac and the bleached product.

This ISO Recommendation covers bone-dry bleached lac, an intermediate surface-dry (air-dry) bleached lac, and wet bleached lac, the three differing in their requirements in the moisture content. No distinction is sought to be introduced between bone-dry, kiln-dry or vac-dry bleached lac or between the various designations of the intermediate type covering dried, crushed hanks, or flats in granules or flakes, or between wet bleached lac in hank, bar or any other form. Only three conditions, i.e. bone-dry, surface-dry (air-dry) and wet, and two types in each condition, namely (*a*) regular bleached lac (cloudy or waxy), and (*b*) refined bleached lac (transparent or wax-free), more briefly indicated as "regular" and "refined", are recognized.

III. Notwithstanding the extensive activities of the Indian Lac Cess Committee concerning the rationalization of production and marketing of lac products in India and abroad, through the Indian Lac Research Institute, Namkum, Bihar, India, and the London Shellac Research Bureau, and in spite of the interest taken in this matter by the United States Shellac Importers' Association, the American Bleached Shellac Manufacturers' Association, and the American Society for Testing Materials, considerable confusion and disagreement still exist in the various specifications on seedlac and shellac that have been evolved and are in use in different countries by different agencies. Bleached lac, on the other hand, does not present the same difficulties. There is a considerable degree of uniformity and unanimity of opinion on the requirements for bleached lac, and international acceptance of agreed standards should not, therefore, present any difficulty.

IV. At the second and third meetings of ISO/TC 50, it was decided that the position regarding limits of variation of certain requirements should be indicated in the Foreword for the guidance of the contracting parties. These decisions are given below.

V. No limit has been prescribed for the chlorine content of bleached lac. Normally the chlorine content of bleached lac varies between 1.5 and 4.5 per cent on moisture free material, but ISO/TC 50 has decided to place it on record that it is desirable to maintain it at a low value (say at 3 per cent, maximum).

VI. Likewise, no limit has been prescribed for the acid value of bleached lac. This depends on the technique of bleaching. The acid value of a good bleached lac is normally between 65 and 100 on the moisture-free material. ISO/TC 50 considers it desirable to retain the acid value at as low a value as possible below 105.

VII. Similarly the mineral acid content of bleached lac should also be as low as possible, requiring for neutralization under the conditions specified in Appendix M not more than 82 ml of 0.1 N sodium hydroxide per 100 g of the moisture-free material which is equivalent to 0.4 per cent of mineral acid calculated as sulphuric acid (H_2SO_4).

VIII. The question of requirements and methods of test for non-volatile matter soluble in cold alcohol is under consideration by ISO/TC 50, for which separate ISO Recommendations may be issued in due course. Pending this, the limits and methods of test have been made subject to agreement between the purchaser and the vendor. The two methods for cold alcohol solubles under study by the Committee are contained in the following documents:

(1) ISO/TC 50 (Secretariat—50) 83,

(2) ISO/TC 50 (Secretariat—51) 84.

IX. The maximum limit for the volatile matter (moisture) in the bone-dry material has been specified as a mandatory requirement, i.e. a maximum of 6 per cent (see Section 6.) In so far as surface-dry (air-dry) and wet bleached lacs are concerned, the actual limits have been made the subject of agreement between purchaser and vendor. The normal moisture content of the surface-dry (air-dry) material does not exceed 12 per cent, that of wet bleached lac does not exceed 30 per cent.

X. In pursuance of the resolution passed at the second and third meetings, the mesh sizes of sieves given in the text of this ISO Recommendation have been indicated in terms of aperture dimensions, and a note, giving number designations of approximately equivalent sieves of U.S.A., United Kingdom, France, India and other countries has been added for the sak of convenience.

XI. Acknowledgement is due for the assistance that has been derived from the specifications and publications of the United States Shellac Importers' Association, the American Bleached Shellac Manufacturers' Association, the British Standards Institution, and the Indian Lac Research Institute, more particularly from "A Handbook of Shellac Analysis" by M. Rangaswami and H.K. Sen, issued by the Indian Lac Research Institute, which gives a critical review of the characteristics and methods of tests of lac and lac products and which was designed by the authors to serve as an approach to international agreement on standardization in this field. The comments received from the American Standards Association, the British Standards Institution, Association Française de Normalisation, and the Indian Standards Institution, which were forwarded for the consideration of ISO/TC 50, are also acknowledged.

XII. For the purpose of deciding whether a particular requirement of this ISO Recommendation is complied with, the final value observed or calculated, expressing the result of test or analysis shall 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.

XIII. In order to facilitate cross references to clauses of the different appendices in this ISO Recommendation, the clauses in each appendix bear, besides the serial number, a letter prefix corresponding to the letter number of the appendix. Thus reference to A-2 or B-3 means that clause 2 of Appendix A or clause 3 of Appendix B is under reference.

ISO Recommendation R 57 December 1957

SPECIFICATION FOR BLEACHED LAC

1. SCOPE

1.1 This ISO Recommendation prescribes the requirements and methods of test for Bleached Lac in any form that may be agreed upon between the purchaser and the vendor.

1.2 This ISO Recommendation 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 prescribed in this ISO Recommendation are limits for rejection.

2. TERMINOLOGY

2.1 For the purposes of this ISO Recommendation, the following definitions apply:

(*a*) *Sticklac* is the natural product of lac insects.

(*b*) *Seedlac* is the product obtained by washing crushed sticklac.

(*c*) *Shellac* is the product obtained by refining seedlac by heat processes or by both heat and solvent processes.

(*d*) *Bleached lac* is the product obtained by subjecting seedlac or shellac in solution to a process of bleaching and then recovering the product in a solid form.

(*e*) *Regular (cloudy or waxy) bleached lac* is the ordinary type of bleached lac from which wax has not been removed.

(*f*) *Refined (transparent or wax-free) bleached lac* is the type of bleached lac from which wax has been removed during the process of manufacture.

(*g*) *Approved sample* is the sample agreed upon between the purchaser and the vendor as the standard for colour.

3. SAMPLING

3.1 Samples are taken in the manner prescribed in Appendix A.

4. FORM

4.1 The form of bleached lac is subject to agreement between the purchaser and the vendor.

5. CONDITIONS AND TYPES

5.1 There shall be three conditions of bleached lac, namely:

(*a*) bone-dry,
(*b*) surface-dry (air-dry) and
(*c*) wet.

5.2 There shall be two types under each of these conditions, namely:

(*a*) regular (cloudy or waxy), and
(*b*) refined (transparent or wax-free).

6. VOLATILE MATTER (moisture)

6.1 Bone-dry bleached lac shall contain not more than 6 per cent of volatile matter (moisture), as determined by the method prescribed in Appendix B.

6.2 The percentage of volatile matter (moisture) present in surface-dry (air-dry) bleached lac is subject to agreement between the purchaser and the vendor and is determined by the method prescribed in Appendix B (see Foreword, clause IX).

6.3 The percentage of volatile matter (moisture) in wet bleached lac is subject to agreement between the purchaser and the vendor and is determined by the method prescribed in Appendix B (see Foreword, clause IX).

7. MATTER INSOLUBLE IN HOT ALCOHOL

7.1 Bleached lac shall not contain matter insoluble in hot alcohol, computed on the basis of moisture-free material, in excess of the limits given below:

regular:	1.1 per cent
refined:	0.2 per cent

7.1.1 Matter insoluble in hot alcohol is determined by either of the methods prescribed in Appendix C, as agreed between the purchaser and the vendor.

8. COLOUR

8.1 The appearance and colour of bleached lac shall not be inferior to that of the approved sample when judged by visual examination. Alternatively the colour in solution may be tested by either of the methods prescribed in Appendix D, as agreed between the purchaser and the vendor.

9. WAX

9.1 Bleached lac shall not contain wax, as determined by the appropriate method prescribed in Appendix E and computed on the basis of moisture-free material, in excess of the limits given below:

regular:	5.5 per cent
refined:	0.2 per cent

10. ASH

10.1 Bleached lac shall not leave ash, as determined by the method described in Appendix F and computed on the basis of moisture-free material, in excess of the limits given below:

regular:	1.0 per cent
refined:	0.5 per cent

11. ROSIN AND COPALS

11.1 Bleached lac shall contain no rosin, as tested by the method described in Appendix G.

11.2 Bleached lac shall contain no copals, as tested by the method described in Appendix H.

12. MATTER SOLUBLE IN WATER

12.1 Bleached lac shall not contain matter soluble in water, as determined by the method prescribed in Appendix J and computed on the basis of moisture-free material, in excess of the limits given below:

regular:	1.0 per cent
refined:	0.3 per cent

13. CHLORINE CONTENT

13.1 The chlorine content of bleached lac is subject to agreement between the purchaser and the vendor and, when specified, it shall be determined by the method prescribed in Appendix K (see Foreword, clause V).

14. ACID VALUE AND MINERAL ACID

14.1 The acid value of bleached lac is subject to agreement between the purchaser and the vendor and, when specified, it is determined by the method prescribed in Appendix L (see Foreword, clause VI).

14.2 The mineral acid content of bleached lac is subject to agreement between the purchaser and the vendor and, when specified, it is determined by the method prescribed in Appendix M (see Foreword, clause VII).

15. NON-VOLATILE MATTER SOLUBLE IN COLD ALCOHOL

15.1 The limits and methods of test for non-volatile matter soluble in cold alcohol are subject to agreement between the purchaser and the vendor (see Foreword, clause VIII).

16. CHARACTER OF REQUIREMENTS

16.1 The character of requirements for bleached lac is given in table 1, below.

16.1.1 The optional requirements are subject to agreement between the purchaser and the vendor.

17. TESTS

17.1 Except where otherwise indicated, calculations regarding bleached lac in any form or condition shall be made in terms of the moisture-free materials.

17.2 All analytical work on bleached lac except the determination of volatile matter (moisture) content is carried out on the "prepared sample" obtained as described under clause A-3.1 (Appendix A).

17.2.1 The volatile matter (moisture) content of the "prepared sample" is determined by the method described in Appendix B and this figure is used to correct the analytical results, other than for volatile matter (moisture), to terms of moisture-free material.

TABLE 1

Requirements for Bleached Lac

Clause Reference	Character of Requirement	Characteristic	Maximum Limits for Type		Method of Testing Reference to Appendix
			Regular	Refined	
6	Essential	Volatile matter (moisture) per cent			B
		Bone-dry	6.0	6.0	
		Other conditions	As agreed between purchaser and vendor		
7	Essential	Matter insoluble in hot alcohol, per cent*	1.1	0.2	C
8	Essential	Colour	Close visual match to the approved sample		D
9	Essential	Wax, per cent*	5.5	0.2	E
10	Essential	Ash, per cent*	1.0	0.5	F
11.1	Essential	Rosin	Nil	Nil	G
11.2	Essential	Copals	Nil	Nil	H
12	Essential	Matter soluble in water, per cent*	1.0	0.3	J
13	Optional	Chlorine content*	As agreed between purchaser and vendor (*see* Foreword, clause V)		K
14.1	Optional	Acid value*	As agreed between purchaser and vendor (*see* Foreword, clause VI)		L
14.2	Optional	Mineral acid*	As agreed between purchaser and vendor (*see* Foreword, clause VII)		M
15	Optional	Non-volatile matter soluble in cold alcohol*	As agreed between purchaser and vendor (*see* Foreword, clause VIII)		

* To be calculated on a moisture-free basis.

Appendix A

(Section 3)

SAMPLING OF BLEACHED LAC

Note. — It is essential that the operations described for the drawing, reduction and preparation of analysis samples are carried out as expeditiously as possible in order to minimize loss of moisture.

A-1. DRAWING OF SAMPLES

A-1.1 Only original, unopened packages of bleached lac are sampled.

A-1.2 Not less than ten per cent of the packages, selected at random from each lot, are sampled.

A-1.3 For this purpose a lot does not exceed 200 packages.

A-1.4 Unused portions of samples are sent to the purchaser on request.

A-1.5 FREE-FLOWING BLEACHED LAC. Samples are taken 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. The material is then thoroughly mixed and heaped and quartered along two diameters which intersect at right angles, and two opposite quarters are mixed. One half of the material may, if necessary, be further subdivided by the normal process of quartering to form a number of samples which serve separately as "original observation samples" and "samples for the determination of volatile matter (moisture)". These samples are placed in air-tight containers, sealed and labelled accordingly. The samples labelled "for the determination of volatile matter (moisture)" are treated as prescribed in clause A-3.2. The other half of the material is treated as described under clause A-2.1 to form the "analysis sample".

A-1.6 BLOCKY OR MATTED BLEACHED LAC. Samples are taken 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. The material is then thoroughly mixed and heaped and quartered along two diameters which intersect at right angles, and two opposite quarters are mixed. One half of the material may, if necessary, be further subdivided by the normal process of quartering to form a number of samples which serve separately as "original observation samples" and as "samples for the determination of volatile matter (moisture)". These samples are placed in air-tight containers, sealed and labelled accordingly. The samples labelled "For the determination of volatile matter (moisture)" are treated as prescribed in clause A-3.2. The other half of the material is roughly ground so as to pass a sieve having a nominal aperture of 6.3 mm (see clause A-4.1) and is then treated as prescribed under clause A-2.1 to form the "analysis sample".

A-1.7 BLEACHED LAC IN HANKS, BARS OR FLATS. Two hanks, bars or flats are drawn from different places in each package and a large piece is broken from each, by suitable means, so as to yield a total of 5 kg (or 10 lb) of material, consisting of approximately equal proportions from each hank, bar or flat. The composite sample is quickly crushed to lumps of about 25 mm^3 (or 1 in^3). The material is then thoroughly mixed and heaped and quartered along two diameters which intersect at right angles, and two opposite quarters are mixed. One half of the material may, if necessary, be subdivided by the normal process of quartering (without crushing) to form a number of samples to serve separately as "original observation samples" and "samples for the determination of volatile matter (moisture)". These samples are placed in air-tight containers, sealed and labelled accordingly. The samples labelled "for the determination of volatile matter (moisture)" are treated as prescribed in clause A-3.2. The remaining half of the material is roughly ground so as to pass a sieve having a nominal aperture of 6.3 mm (see clause A-4.1) and is then treated as described under clause A-2.1 to form the "analysis sample".

A-2. REDUCTION OF SAMPLES

Note. — If the material at any time during the following operations shows signs of surface moisture, it shall be air dried at room temperature before further mixing and grinding.

A-2.1 The material for the analysis sample, as obtained under clause A-1.5, A-1.6 or A-1.7 is mixed thoroughly, heaped and quartered along two diameters which intersect at right angles. Two opposite quarters are mixed and ground to pass entirely through a sieve having a nominal aperture of about 2 mm (see clause A-4.1). The material is then thoroughly mixed and quartered so as to yield four samples of approximately 250 g (or 0.5 lb) each. These four samples are placed in air-tight containers, sealed labelled "sample for analysis" and sent to the interested parties.

A-2.2 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 are given on a label attached to each sample.

A-3. PREPARATION OF ANALYSIS SAMPLES FOR TESTING

A-3.1 The samples for analysis are ground to pass entirely through a sieve whose nominal aperture does not exceed 0.425 mm (see clause A-4.1). This finely ground material is mixed thoroughly and placed in an air-tight container and labelled "unconditioned sample for analysis". Before this material is used for any analytical work, it is brought to less than 6 per cent volatile matter (moisture) content by exposing it to the atmosphere for at least 24 hours at room temperature and then desiccating overnight over fused calcium chloride. The material is then known as the "prepared sample".

A-3.2 The sample or samples labelled "for the determination of volatile matter (moisture)" (see clauses A-1.5, A-1.6 and A-1.7) are treated according to the method described in Appendix B.

A-4. NOTE ON SIEVE SIZES

A-4.1 Table 2 (page 13) gives some of the national sieve designations and mesh apertures corresponding approximately to the sizes specified in the preceding clauses and in Appendices B and J.

TABLE 2

National Sieve Designations and Mesh Apertures Corresponding to Specified Sizes

Size Specified mm	U.S.A. ASA		UNITED KINGDOM B.S.I.		FRANCE AFNOR		GERMANY DNA		INDIA ISI	
	Designation Standard Sieve	Size mm	Designation Test Sieve	Size mm	Designation	Size mm	Designation	Size mm	Designation	Size mm
(1) 6.3 (Perforated Plate)	$^1/_4$ in No. 3	6.35	Plate $^1/_4$ in	6.35	Plate Sieve No. 38	6.30	Perforated Plate 6.3	6.3	$^1/_4$″ P	6.35
(2) 2.00 (Woven)	No. 10	2.00	8 mesh	2.057	Test Sieve No. 34	2.00	Test Sieve 2.0	2.0	200	2.032
(3) 0.425 (Woven)	No. 40	0.42	36 mesh	0.422	Test Sieve No. 27	0.40	Test Sieve 0.43	0.43	40	0.420
(4) 0.25 (Woven)	No. 60	0.250	60 mesh	0.251	Test Sieve No. 25	0.250	Test Sieve 0.25	0.25	25	0.251

NATIONAL STANDARDS

ASA — American Standards Association Inc.—Standard Specification for Sieves for Testing Purposes,—Wire Cloth Sieves, Round-Hole and Square-Hole, Screens or Sieves.—(ASTM Designation: E 11—39). ASA No: Z 23.1-1939.

BSI — British Standards Institution.—British Standard Specification for Test Sieves.—B.S. 410: 1943.

AFNOR — Association Française de Normalisation.—Analyses Granulométriques par Tamisage (Tamis de Contrôle).—NF X 11-501, Juin 1938.

DNA — Deutscher Normenausschuss—(*a*) Normenentwurf Lochbleche—DIN 24041-1954 Lochbleche, Rundlochung.
(*b*) Drahtgewebe für Prüfsiebe (Abmessungen)—DIN 1171-1934 (Blatt 1).

ISI — Indian Standards Institution.—Indian Standard Specification for Test Sieves—IS: 460-1953.

Appendix B

(Section 6)

DETERMINATION OF VOLATILE MATTER (MOISTURE)

B-1. OUTLINE OF METHOD

B-1.1 The volatile matter (moisture) content of bleached lac is determined in two stages, the first stage being by drying a weighed specimen of the original observation sample marked "for the determination of volatile matter (moisture) content" by keeping it over a concentrated sulphuric acid *in vacuo* for 12 to 24 hours. In the case of bone-dry bleached lacs, this first stage may be omitted.

B-1.2 For the second stage, grind the partially dried material thus obtained to the specified size and further dry a portion by heating it in a well-ventilated oven maintained at a temperature of 41 °C ± 1 °C for 18 hours.

B-2. PROCEDURE

B-2.1 Use a portion of the original observation sample and crush, if necessary, into granules using a heavy pestle and mortar, keeping the latter covered as far as possible during the process. Weigh a clean, dry, flat-bottomed dish of about 100 mm diameter, provided with a glass cover. Transfer approximately 10 g of the ground sample to the dish as rapidly as possible, cover it with the glass cover and reweigh. The difference gives the weight of sample taken.

B-2.2 Transfer the dish and contents to a vacuum desiccator containing concentrated sulphuric acid and remove the cover of the dish. Immediately evacuate the desiccator and keep the sample, uncovered, *in vacuo* for not less than 12 hours and not more than 24 hours. Remove the dish, replace the cover and weigh. The difference between this weight and the weight of the dish is the weight of the partially dried sample. Grind the partially dried sample thus obtained until it passes a sieve having a nominal aperture of 0.425 mm (see A–4.1).

B-2.3 Weigh approximately 2 g of the ground material, to an accuracy of 1 mg, into a covered tared dish of the type described in B-2.1 and transfer to a well-ventilated oven maintained at a temperature of 41 °C ± 1 °C for 18 hours, the cover of the dish being removed during the drying process. At the conclusion of the heating period, replace the cover and transfer the covered dish to a desiccator; weigh when cool. This weight minus the weight of the dish is the weight of the completely dried ground sample.

B-3. CALCULATION

B-3.1 The percentage of volatile matter (moisture) in the original sample is given by the following formula:

$$\text{Volatile matter (moisture), per cent} = 100\left(1 - \frac{W_4\,W_2}{W_3\,W_1}\right)$$

where W_1 = weight of crushed wet sample taken for stage of drying *in vacuo*,
W_2 = weight of partially dried sample,
W_3 = weight of partially dried, ground sample taken for final oven drying and
W_4 = weight of completely dried, ground sample.

B-3.2 If the first drying stage (see B-2.2) is omitted, the percentage of volatile matter (moisture) in the original sample is given by the formula:

$$\text{Volatile matter (moisture), per cent} = 100\left(1 - \frac{W_2}{W_1}\right)$$

where W_1 = weight of sample taken for oven drying and
W_2 = weight of completely dried, ground sample.

Appendix C

(Section 7)

DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

C-1. GENERAL

C-1.1 The matter insoluble in hot alcohol is determined by extracting a known weight of bleached lac with 95 per cent (by volume) ethanol and determining the percentage of the undissolved residue by either of the two methods described below, as may be agreed.

C/I METHOD I

C/I-1. APPARATUS

C/I-1.1 The apparatus consists of the following:

C/I-1.1.1 *Condenser* — all glass, of the type and dimensions shown in figure 1, page 17, with the tip cut at an angle of 45 degrees.

C/I-1.1.2 *Siphon tube* — of glass, of the type and dimensions shown in figure 1. The siphon tube has 2 holes near the top for a wire to be fastened to a condenser tip, leaving about 6 mm space between the top of the tube and the condenser tip.

C/I-1.1.3 *Flask* — heat resistant, wide-mouth, conical, preferably of borosilicate glass, 176 mm ± 3 mm in height and 48 mm ± 2 mm in inside diameter at the top. The flask has a tight-fitting cork 25 mm in depth and bored to fit the stem of the condenser. The bottom of the cork is just above the holes for the wire in the condenser. To support the flask a suitable ring support with iron clamp and nichrome or iron gauze is used. The gauze has no asbestos covering.

C/I-1.1.4 *Filter tube* — a 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 is fitted with a rubber stopper and firmly held in a hot water bath.

C/I-1.1.5 *Extraction cartridges* — fat-free paper extraction cartridges, * 26 mm ± 1 mm in diameter and 60 mm ± 1 mm in height.

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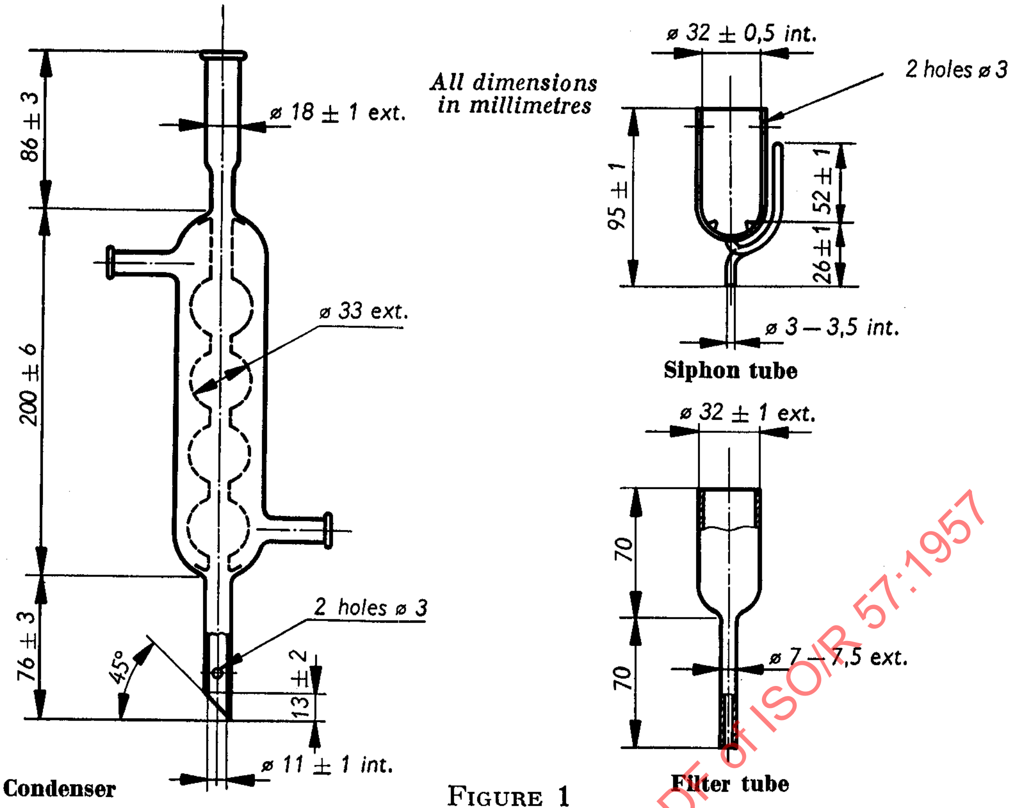
* Schleicher and Schull No. 603 or equivalent is suitable.

All dimensions in millimetres



Condenser

Siphon tube

Filter tube

FIGURE 1

Extraction apparatus for determining hot alcohol insolubles, Method I

C/I-1.1.6 *Weighing bottle* — glass stoppered, 80 mm ± 1 mm in height and 40 mm ± 1 mm in diameter.

C/I-1.1.7 *Hot water bath* — made of about 0.9 mm thick or 21 B.G. copper (approximately 8 kg/m² or 26 oz/ft²), having the dimensions given in figure 2 below. The cover has a flanged hole, 57 mm ± 1 mm in diameter, for a 200 ml beaker, and also a hole, 35 mm ± 1 mm in diameter through which the top of the filter tube projects. Directly below this hole, in the bottom of the bath, is a flanged hole, 25 mm ± 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 is mounted on a low tripod or stand.

All dimensions in millimetres

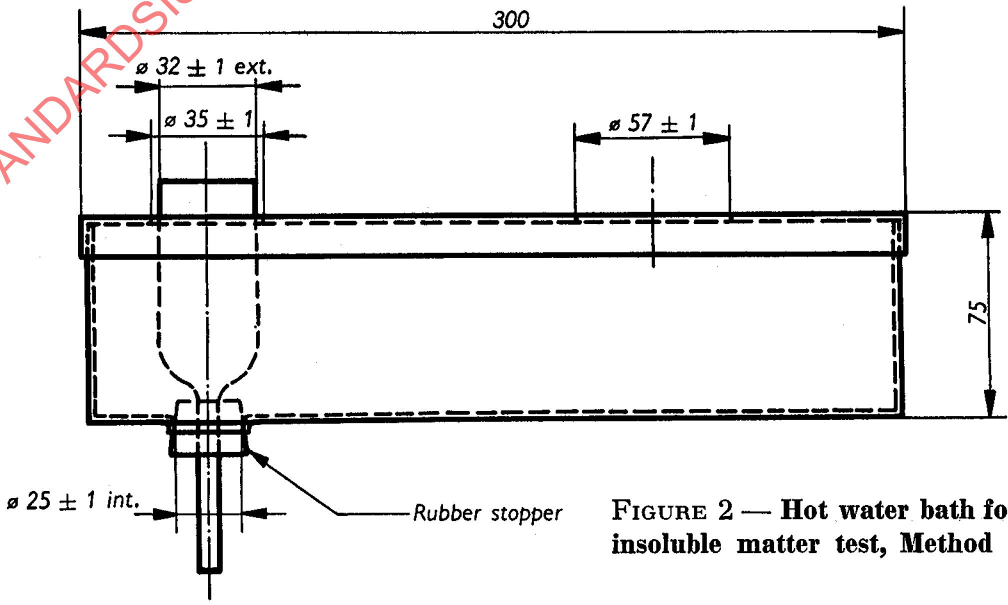


FIGURE 2 — **Hot water bath for insoluble matter test, Method I**

C/I-1.1.8 *Gas burner* — low form, adjustable, Bunsen type, carrying a draught shield.

C/I-2. REAGENT

C/I-2.1 The following reagent is required:

Alcohol — 95 per cent (by volume) ethanol; or 95 per cent (by volume) denatured spirit.

C/I-3. PREPARATION OF EXTRACTION CARTRIDGE

C/I-3.1 Place 125 ml of the alcohol in the flask and a new cartridge in the siphon tube. Introduce the siphon tube into the flask and connect it to the condenser, making sure that there is an ample flow of cold water through the condenser. Adjust the flame of the burner so as to give a cycle of filling and emptying in the siphon tube every 2 minutes and extract for 30 minutes. Dry the cartridge in an oven at a temperature of 100 °C ± 2 °C. At the end of 3 hours, weigh it in a tared weighing bottle, which has been kept in a desiccator over sulphuric acid, lifting the stopper of the bottle momentarily before weighing. Continue drying and weigh as before after each hour of drying, until the loss in weight between successive weighings does not exceed 2 mg.

C/I-3.1.1 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.

C/I-4. PROCEDURE

C/I-4.1 Before analysis, thoroughly mix the "prepared sample" (see A-3.1) by rolling on paper, at least a 100 times, to ensure uniformity of the analytical sample. Weigh, directly from the rolling sheet, 4.5 g to 5.5 g of the sample to an accuracy of 0.01 g, place in a 200 ml, tall, lipped beaker, add 125 ml of alcohol, cover with a watch-glass and place on the hot water bath (see figure 2, page 17). Boil the solution vigorously for 30 minutes to ensure complete solution of the bleached lac and dispersion of wax. Keep the volume of alcohol constant.

C/I-4.2 Meanwhile, place an extracted and weighed cartridge in the filter tube. Maintain the hot water around the tube at a temperature of 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.

C/I-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.

C/I-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 one hour. Immediately begin to adjust the Bunsen burner so that a complete filling and emptying of the siphon tube takes place every 2 minutes, as determined by a stop-watch or preferably by a good two-minute sand-glass, one for each extraction apparatus.

C/I-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 guard the apparatus from draughts while in operation, otherwise the proper cycle rate cannot be maintained.

C/I-4.4.2 Occasionally, bleached lacs are encountered which do not yield the required number of 30 siphonings per hour, due to slow filtration. In these instances, continue the extraction until 30 siphonings have been accomplished or repeat the test with a 2 g sample and report the sample as abnormal or slow filtering.

C/I-4.5 Remove the cartridge, drain in an upright position on filter paper and dry in an oven at a temperature of 100 °C ± 2 °C. After drying for 2 hours, place it in the weighing bottle, cool in a desiccator over sulphuric acid and weigh, removing the stopper momentarily just before weighing. Repeat drying and weighing as before after each hour of drying, until the loss in weight between successive weighings does not exceed 2 mg. From the weight of the residue and the weight of the sample, calculate the percentage of insoluble matter, making allowance for the volatile matter (moisture) content, determined as prescribed in Appendix B. Use the lowest weight in the calculation.

C/I-5. CALCULATION

C/I-5.1 Matter insoluble in hot alcohol, per cent $= \frac{w}{W(100-m)} \times 10^4$

where w = weight of residue,

W = weight of sample taken, and

m = volatile matter (moisture) content per cent (see 17.2.1).

C/II METHOD II

C/II-1. APPARATUS

C/II-1.1 CONSTITUTION OF APPARATUS. The apparatus consists of the following:

C/II-1.1.1 *Siphon tube* * — of glass, of the Knoefler type, having minimum 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 below).

C/II-1.1.2 *Condenser* — of any convenient pattern.

C/II-1.1.3 *Flask* — of any convenient size.

C/II-1.1.4 *Filter paper* — 12.5 cm in diameter, medium grade. **

C/II-1.1.5 *Weighing bottles* — of glass, 80 mm ± 1 mm in height and 40 mm ± 1 mm in diameter, with ground-glass stoppers.

C/II-1.2 ASSEMBLY OF APPARATUS. The siphon tube, adaptor, condenser and flask are 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.

C/II-2. REAGENT

C/II-2.1 The following reagent is required:

Alcohol — 95 per cent (by volume) ethanol; or 95 per cent (by volume) denatured spirit.

All dimensions in millimetres

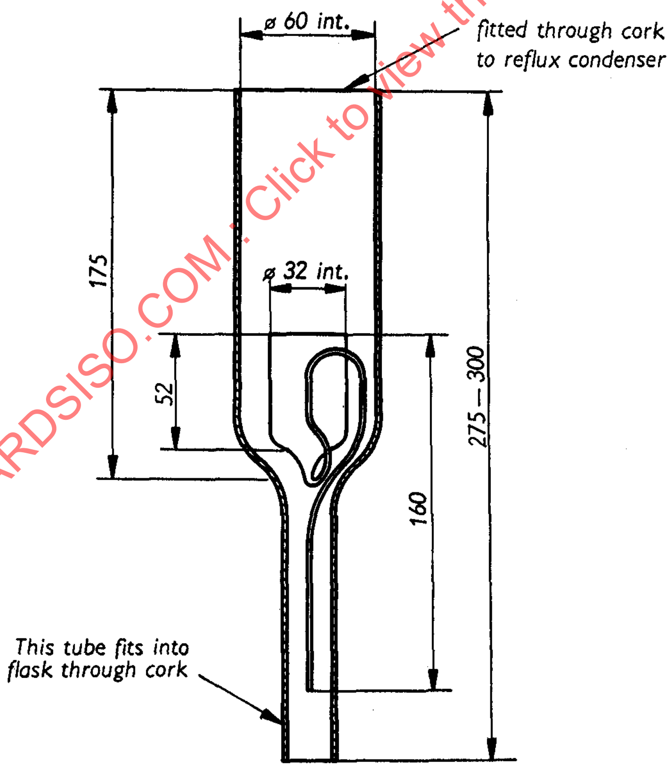


FIGURE 3

Siphon tube and adaptor

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* 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 (Appendix C/I), consisting of siphon tube, condenser and flask, could be satisfactorily used.

** Whatman No. 1 or its equivalent is suitable.

C/II-3. PROCEDURE

C/II-3.1 Fold a filter paper so that it forms a completely closed envelope (see figure 4 below). 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 a temperature of 100 °C ± 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.

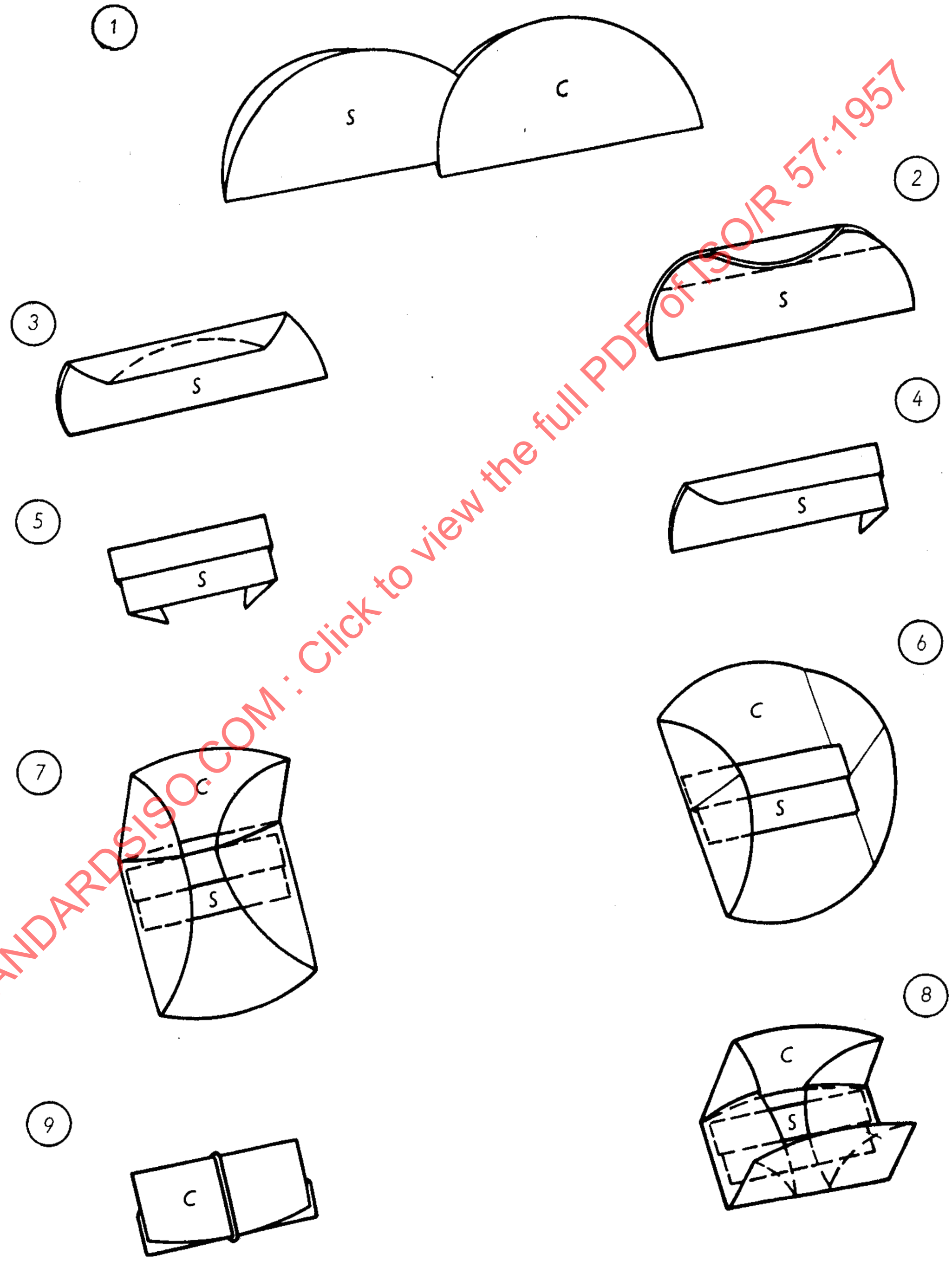

FIGURE 4

Folding of filter paper

C/II-3.2 Weigh 4.5 g to 5.5 g of the "prepared sample" of bleached lac (see A-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 a 100 ml beaker and cover it with alcohol. Allow to stand overnight at room temperature. Place the envelope in the cup of the siphon tube and extract continuously with hot alcohol for 4 hours. 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.

C/II-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 the steam oven and then for 3 hours in a thermostatically controlled oven at a temperature of 100 °C ± 2 °C. Place the papers rapidly in their respective weighing bottles, allow to 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 one hour at a temperature of 100 °C ± 2 °C and weigh again. If there is a loss in weight in excess of 2 mg, repeat the processes of drying and weighing until the difference between successive weighings is less than 2 mg. Use the lowest weight in the calculation.

C/II-4. CALCULATION

C/II-4.1 As under C/I-5.

Appendix D

(Section 8)

COMPARISON OF COLOUR

D-1. GENERAL

D-1.1 Two methods for the determination of colour are described. One of these is based on the visual comparison of solutions or films prepared from the material and the approved sample; the other is based on the determination of the colour index of the material and the approved sample by visual comparison of intensity of colour of a 10 per cent (w/v) solution of the material in 95 per cent (by volume) alcohol with a standard solution of iodine.

D/I METHOD I, VISUAL COLOUR COMPARISON

D/I-1. OUTLINE OF METHOD

D/I-1.1 Bleached lac is dissolved in 95 per cent (by volume) ethanol and the colour examined visually against a similarly treated approved sample either in the solution form or after forming films.

D/I-2. PROCEDURE

D/I-2.1 Treat a weighed portion of bleached lac with twice its weight of cold 95 per cent (by volume) ethanol, shaking at intervals until the lac is entirely dissolved. Compare its colour with the colour of a similar solution of the approved sample by any of the following methods, as agreed between the purchaser and the vendor.

D/I-2.1.1 It should be noted that the insoluble solids content of the approved sample shall be the same as that of the bleached lac solution under test.

D/I-2.2 COMPARISON OF SOLUTIONS. Shake both solutions well and then place equal portions of each in separate clear-glass tubes of the same diameter. Place the tubes together and compare the colour visually.

D/I-2.3 COMPARISON OF FILMS. Shake the solutions well and then allow them to stand for 30 minutes. Flow approximately equal portions of the two bleached lac solutions on separate opal glass plates or porcelain plates and allow the films to dry in a vertical position. Make the colour comparison upon dried films visually.

D/II METHOD II, DETERMINATION OF COLOUR INDEX

D/II-1. OUTLINE OF METHOD

D/II-1.1 The colour index is determined by comparing the colour of a 0.001 N solution of iodine (in potassium iodide solution) with a solution of bleached lac in 95 per cent ethanol, by diluting the darker solution progressively until a close visual match is obtained and then repeating the test with a solution of the approved sample.

D/II-2. REAGENTS

D/II-2.1 The following reagents are required:

D/II-2.1.1 *Alcohol* — 95 per cent (by volume) ethanol; or 95 per cent (by volume) denatured spirit, provided that it is colourless.

D/II-2.1.2 *Standard iodine solution.* — Prepare standard 0.001 N solution of iodine by introducing 1 ml of 0.1 N iodine solution (in potassium iodide solution), with a burette, into a measuring flask and making up to 100 ml with water. Shake the solution before use.

D/II-2.1.3 *Bleached lac solutions.* — Dissolve 10.0 g of the "prepared sample" (see A-3.1) in 100 ml of alcohol by shaking for 30 minutes at a temperature of 27 °C ± 2 °C. Filter the solution in an ordinary funnel, using a medium grade filter paper. Discard the first 15 ml of the filtrate and then collect 5 ml or more of the clear filtrate for the test.

D/II-3. PROCEDURE

D/II-3.1 Transfer 5 ml of the filtered bleached lac solution to a thin-walled test tube measuring 200 mm × 13 mm (8 in × 0.5 in) by means of a pipette. Take an adequate volume of the standard iodine solution in 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 in between the light source and the test tube. Add the alcohol from a burette to the darker solution with occasional shaking until the colour of both the solutions is the same. Note the volume of alcohol added. Repeat, using the approved sample in place of the bleached lac.

D/II-3.1.1 It will be found advantageous to use a standard type of light source and a viewing cabinet to cut off extraneous light.

D/II-3.2 The requirement is taken as having been satisfied, if the solution prepared from the sample under test is not found to be darker than that prepared from the approved sample.

Appendix E

(Section 9)

DETERMINATION OF WAX

E-1. GENERAL

E-1.1 Two methods for the determination of wax, Method I for regular bleached lac containing wax and Method II for refined bleached lac, are described below.

E/I METHOD I, FOR REGULAR BLEACHED LAC CONTAINING WAX

E/I-1. OUTLINE OF METHOD

E/I-1.1 A specified quantity of regular bleached lac is dissolved in hot solution of sodium carbonate, the wax separated by filtering suitably, extracted by means of chloroform and weighed after drying.

E/I-2. REAGENTS

E/I-2.1 The following reagents are required:

E/I-2.1.1 *Sodium carbonate* — anhydrous, analytical reagent.

E/I-2.1.2 *Chloroform* — redistilled, free from non-volatile residue.

E/I-2.1.3 *Filter-aid* * — a suitable filter-aid, previously extracted with chloroform and dried before use.

E/I-3. PROCEDURE

E/I-3.1 Weigh 9.5 g to 10.5 g of the "prepared sample" (see A-3.1) to an accuracy of 0.01 g and dissolve in 150 ml of hot water containing 2.5 g of sodium carbonate in a 200 ml tall beaker. Immerse the beaker in a steam or boiling water bath 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 hours more, without agitation. Remove the beaker from the bath and place it in cold water. The wax will now come to the top 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 a 12.5 cm double acid-washed, retentive, low-ash filter paper, * by gravity, or use a Buchner funnel with suction.

E/I-3.1.1 In the latter case it is necessary to embed the filter paper in the Buchner with filter-aid, by mixing 1 g of the filter-aid with water and pouring this mixture on to the paper with the suction on. Filtration by this method is also further aided by stirring 0.5 g of the filter-aid into the bleached lac solution before starting the filtration.

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

E/I-3.2 If the filtration is done only under gravity, after the filtration is completed and all soluble bleached lac washed out of the paper with water, remove the paper from the funnel and, without further folding it, set it in the beaker, resting 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 a temperature not exceeding 65 °C, 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 a 26 mm × 60 mm fat-free paper extraction cartridge ** which has been previously extracted with hot chloroform. Put the cartridge containing the wax and paper into a suitable continuous extraction apparatus, such as the standard hot-alcohol-insoluble matter apparatus (see Method I, Appendix C/I) 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 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 hours. Distil off most of the solvent, and dry the residue to constant weight (within 10 mg) in a tared vessel, at a temperature of 100 °C ± 2 °C. Use the lowest weight in the calculation.

E/I-3.3 If the Buchner funnel is used, after the filtration has been completed and the paper has been well washed with water to take out all soluble bleached lac, the vacuum is left 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 a gentle heat for a few hours, as by placing it on the top of an oven at a temperature of 105 °C. When dry, place it in a 26 mm × 60 mm fat-free paper extraction cartridge, which has been previously extracted with chloroform. Transfer the cartridge and wax to any suitable continuous extraction apparatus, such as the standard apparatus for hot-alcohol-insoluble matter (see Method I, Appendix C/I), and extract for 2 hours with chloroform. Distil off the solvent and dry the wax at a temperature of 100 °C ± 2 °C to constant weight (within 10 mg) in a tared vessel. Use the lowest weight in the calculation.

E/I-4. CALCULATION

E/I-4.1 Wax, per cent $= \dfrac{w}{W\,(100-m)} \times 10^4$

where w = weight of wax,
W = weight of sample taken and
m = volatile matter (moisture) content per cent (see 17.2.1).

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

** Schleicher and Schull No. 603 or equivalent is suitable.

E/II METHOD II, FOR REFINED BLEACHED LAC

E/II-1. OUTLINE OF METHOD

E/II-1.1 A specified quantity of refined bleached lac is dissolved in alcohol, and the wax separated out at a temperature of 0 °C with the use of a filter-aid and oxalic acid; the wax is subsequently extracted from the filter-aid by means of chloroform and weighed after drying.

E/II-2. REAGENTS

E/II-2.1 The following reagents are required:

E/II-2.1.1 *Alcohol* — 95 per cent (by volume) ethanol.

E/II-2.1.2 *Oxalic acid* — dihydrate, analytical grade.

E/II-2.1.3 *Chloroform* — redistilled, free from non-volatile residue.

E/II-2.1.4 *Filter-aid* * — a suitable filter-aid, previously extracted with chloroform and dried before use.

E/II-3. PROCEDURE

E/II-3.1 Weigh 49 g to 51 g of the "prepared sample" (see A-3.1) to an accuracy of 0.1 g and dissolve in 250 ml of alcohol, add about 1 g of oxalic acid and stir until all is dissolved. Then add about 0.5 g of the filter-aid and allow to flocculate and settle overnight at a temperature of 41 °C ± 1 °C. Now cool to 0 °C and keep at this temperature for at least one hour. Pour the clear liquid through a Gooch crucible, prepared with an asbestos mat over which a thin layer of the filter-aid has been applied. Wash the sediment of wax and filter-aid from the beaker into the crucible with cold alcohol at 0 °C. Dry at a temperature of 41 °C ± 1 °C. Remove the asbestos mat, wipe out the crucible with small pieces of alcohol-moistened paper and wrap the mat and the wiping carefully in a large piece of fat-free filter paper. Bind securely with fine wire and transfer to a previously extracted 26 mm × 60 mm fat-free paper extraction cartridge and extract in a suitable continuous-extraction apparatus, such as the hot-alcohol-insoluble matter apparatus (see Method I, Appendix C/I), with chloroform for 2 hours. Distil off the solvent and dry the wax at a temperature of 100 °C ± 2 °C in a tared vessel, until the loss in weight on successive weighings does not exceed 1 mg. Use the lowest weight in the calculation.

E/II-4. CALCULATION

E/II-4.1 As under E/I-4.

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

Appendix F

(Section 10)

DETERMINATION OF ASH

F-1. PROCEDURE

F-1.1 Weigh 3 g to 5 g of the "prepared sample" (see A-3.1) to an accuracy of 0.01 g, char in a tared porcelain, silica or platinum crucible and ignite at a low heat, not exceeding dull redness, until free from carbon and until the difference between successive weighings does not exceed 1 mg. Use a muffle furnace, if available.

F-1.1.1 If a carbon-free ash cannot be obtained in this manner, extract the charred mass with hot water, collect the insoluble residue on an ashless filter paper, wash the filter paper and ignite it until all the carbon is consumed. Then transfer the filtrate and washings to the crucible, evaporate to dryness and heat to dull redness. Cool in a desiccator and weigh. Repeat until the difference between successive weighings does not exceed 1 mg. Use the lowest weight in the calculation.

F-2. CALCULATION

F-2.1 Ash, per cent $= \dfrac{w}{W(100-m)} \times 10^4$

where w = weight of ash,
W = weight of sample taken and
m = volatile matter (moisture) content per cent (see 17.2.1).