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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 56

SPECIFICATION FOR SHELLAC

1st EDITION

December 1957

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BRIEF HISTORY

The ISO Recommendation R 56, *Specification for Shellac*, 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 Shellac 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 ISO/TC 50, held in New York, in June 1952. A revised third draft proposal 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. 99) 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	Hungary	Spain
Austria	*Ireland	*Sweden
*Bulgaria	Netherlands	Turkey
*Canada	Pakistan	Union of South Africa
*Denmark	Poland	United Kingdom
France	Portugal	U.S.A.
*Greece	Romania	*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.

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

AMENDMENT 1 - JULY 1966
TO ISO RECOMMENDATION R 56-1957

Amendment 1
to ISO Recommendation R 56-1957

SPECIFICATION FOR SHELLAC

1. **Clause VIII (Foreword), page 5, lines 1 to 13**

Delete

" In pursuance of the resolution ..... for further study."

2. **Section 10, page 9**

Read

" Shellac shall not contain any rosin, as tested by the method prescribed in Appendix F."

3. **Section 14, page 10**

Read

" Shellac shall satisfy a flow test, which is conducted by the method prescribed in Appendix L."

4. **Section 16, page 10**

Read

" The acid value of shellac may be fixed, if desired, by agreement between the purchaser and the vendor. It shall be determined by the method prescribed in Appendix N."

5. **Appendix L, clause L.2.2, page 39, line 4**

Read

" ... for 24 ± 2 hours."

BRIEF HISTORY

At the meeting held in New Delhi in February 1961, Technical Committee ISO/TC 50, *Lac*, decided, after due study, that there was no need to continue the investigation regarding quantitative determination of rosin in shellac, as had been planned in 1957. It was further decided that only the method described in Appendix L should be kept for flow tests. For determination of acid value of shellac, it was decided to retain the method described in Appendix N and not to take up consideration of an alternative method.

Consequently, a Draft Revision of ISO Recommendation R 56-1957 was drawn up and adopted by the Technical Committee.

In November 1963, this Draft Revision (No. 633) was circulated to all the ISO Member Bodies for enquiry. It was approved by 20 Member Bodies. No Member Body opposed the approval of the Draft.

The Draft Revision, entitled *Amendment 1 to ISO Recommendation R 56-1957*, was then submitted by correspondence to the ISO Council, which decided, in July 1966, to accept it.

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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, USA, on June 23, 24 and 25, 1952; and in London, United Kingdom, on October 11, 12 and 13, 1954, respectively.

II. India produces a very large proportion of the world output of lac and lac products, of which a part is consumed in that country for various industrial purposes, but the bulk is exported to America and Europe, so that the manufacture and export of shellac is a matter of great interest to India and to the rest of the world. The need for an agreed international standard specification for this important commodity of international trade is obvious. This need has been hitherto met by the procurement policies adopted by the users of this material outside India and by the standards followed by individual exporting firms in India, the United States Shellac Importers' Association and the London Shellac Trade Association. These have not contributed materially towards evolving a generally acceptable set of specifications; on the contrary, by the introduction of arbitrarily selected names used to describe special brands or makes of shellac, the development of a general standard on shellac has been hindered.

III. Notwithstanding the extensive activities of the Indian Lac Cess Committee concerning the rationalization of production and marketing of shellac 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 Society for Testing Materials, and the London Shellac Trade Association, considerable confusion and disagreement still exist in the various specifications on shellac that have been evolved and are in use in different countries by different agencies.

IV. The International Electrotechnical Commission carried out a considerable amount of preliminary work by way of collection of data on prevalent practices related to shellac and allied products, but this work did not reach a point where an international agreement could be said to have been attained either on the specifications or on the methods of test. The International Organization for Standardization adopted "Lac" among the subjects to be standardized and, in view of the importance to India of such standardization and also considering the extensive experience available in that country, the Indian Standards Institution was appointed as the Secretariat of the ISO Technical Committee on this subject.

V. The usual trade descriptions of shellac, which are based somewhat indiscriminately on the Indian names of the host trees or the season of cropping the sticklac or visual differences or on *ad hoc* descriptions such as fine, superfine and extra fine or a combination of any of these, have failed to distinguish the different grades. The standard of quality

represented by these names has not remained constant but has varied from year to year and from one country to another. For example, London Superfine and American Superfine are recognized in the trade as different qualities. There has also been a tendency to introduce new names to describe slight variants of the commercially known varieties. The distinction between hand-made and machine-made shellac has not been rigidly observed. To remove this confusion, two types, namely hand-made and machine-made have been recognized in this ISO Recommendation and six different grade designations adopted for each of these two types, all independent of the names of host trees and seasons or of any characteristics of shellac. These designations are grades Special, A, B, C, D and E for hand-made shellac and grades D1, D2, I, II, III and IV for machine-made shellac. Machine-made shellac grades D1 and D2 relate to decolourized material and any of the machine-made grades may be supplied in dewaxed or undewaxed condition. In the first meeting of ISO/TC 50 it was agreed that designations of shellac such as 'VSO', 'Double Triangle G', 'Diamond I', 'Button Lac', 'Garnet Lac', be not specified and that the proposed grade designations should be adhered to. There will be no objection to the existing trade designations and private marks being also used for trade purposes, if required by contracting parties. These designations and private marks do not, however, form a part of the international designations. It is felt that although the new nomenclature might take some time to get established, by the time this occurs, the old designations would probably disappear.

VI. Three of the requirements for shellac, namely those for (*a*) matter insoluble in hot alcohol, (*b*) absence of rosin and (*c*) absence of orpiment are included in this ISO Recommendation as essential clauses; the remainder, namely those for (*d*) volatile matter (moisture), (*e*) colour index, (*f*) wax, (*g*) ash, (*h*) matter soluble in water, (*j*) flow test, (*k*) heat polymerization test, (*m*) acid value, (*n*) lead content and (*p*) non-volatile matter soluble in cold alcohol, are optional.

VII. In the first meeting of ISO/TC 50 the clause relating to colour index and the related Appendix D were approved tentatively subject to review after a period not exceeding two years and the requirement made optional subject to agreement between the purchaser and the vendor. In the second meeting of the Committee it was decided that the requirement relating to colour index and the related Appendix D should be approved and the requirement should remain optional. In addition, provision has been made (see 8.2) for the appearance and colour to be judged by visual comparison.

VIII. In pursuance of the resolution passed at the third meeting of ISO/TC 50, the methods of test for the determination of rosin content (see 10) are being studied by ISO/TC 50 and when an agreed method is developed, the ISO will issue a separate ISO Recommendation on the subject. The two methods under study are given in the following documents:

(1) ISO/TC 50 (Secretariat—58) 91 McIlhiney Method for the Determination of Rosin,

(2) ISO/TC 50 (Secretariat—59) 92 Langmuir Method for the Determination of Rosin.

In respect of flow test (see 14), a standard method adopted by the ISO/TC 50 is given in Appendix L, but for specialized requirements, alternative or additional tests may be desired and, for this purpose, the ISO/TC 50 has issued a separate document ISO/TC 50 (Secretariat—52) 85 in which three other tests are described.

Beside the alkalimetric method for the determination of acid value, another alternative method, known as the Kamath-Mainkar method, has been given in document ISO/TC 50 (Secretariat—42) 70 for further study.

Limits and methods of test for non-volatile matter soluble in cold alcohol are also being studied by the ISO/TC 50 and when an agreement is arrived at, the ISO will issue a separate ISO Recommendation on this subject. The two methods for cold alcohol solubles under study by the Committee are contained in the following documents:

(*a*) ISO/TC 50 (Secretariat—50) 83,

(*b*) ISO/TC 50 (Secretariat—51) 84.

IX. 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 USA, United Kingdom, France, India and other countries has been added for the sake of convenience.

X. Acknowledgement is due for the assistance that has been derived from the specifications and publications of the American Society for Testing Materials, the American Bleached Shellac Manufacturers' Association, the United States Shellac Importers' 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, 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, the Standards Association of Australia, the Swiss Association for Standardization, the Association Française de Normalisation and the Indian Standards Institution, which were forwarded for consideration of ISO/TC 50, are also acknowledged.

XI. 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.

XII. 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 56 December 1957

SPECIFICATION FOR SHELLAC

1. SCOPE

1.1 This ISO Recommendation prescribes the requirements and methods of test for shellac, hand-made and machine-made.

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 solvent processes or by both heat and solvent processes.

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

3. SAMPLING

3.1 Samples should be taken in the manner prescribed in Appendix A.

4. FORM AND CONDITION

4.1 Shellac may be in the form of flakes, or sheets, or buttons, or any other form agreed between the purchaser and the vendor.

5. TYPES AND GRADES

5.1 There are two types of shellac, namely:

hand-made and machine-made.

5.2 There shall be six grades in each type of shellac, namely:

(*a*) *hand-made* — Special, A, B, C, D and E,

(*b*) *machine-made* — D1, D2, I, II, III and IV.

6. VOLATILE MATTER (moisture)

6.1 Shellac shall contain not more than 2.0 per cent of volatile matter (moisture) as determined by the method prescribed in Appendix B.

7. MATTER INSOLUBLE IN HOT ALCOHOL

7.1 Shellac shall not contain matter insoluble in hot alcohol, determined by either of the methods prescribed in Appendix C, as agreed between the purchaser and the vendor, in excess of the limits given below:

Hand-made		Machine-made	
Grade	Maximum per cent	Grade	Maximum per cent
Special	1.0	D1	0.5
A	1.0	D2	0.5
B	1.5	I	0.5
C	2.0	II	0.5
D	3.0	III	1.0
E	4.0	IV	1.0

8. COLOUR INDEX

8.1 The colour index of shellac, as determined by the method prescribed in Appendix D, shall not exceed the limits given below:

Hand-made		Machine-made	
Grade	Colour index	Grade	Colour index
Special	6.0	D1	2.0
A	8	D2	5.0
B	10	I	8
C	15	II	12
D	22	III	22
E	50	IV	50

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

9. WAX

9.1 Shellac shall not contain wax in excess of 5.5 per cent, as determined by Method I prescribed in Appendix E.

9.2 *Dewaxed shellac.* When a machine-made grade is offered as dewaxed shellac, the limit of wax is subject to agreement between the purchaser and the vendor and is determined by Method II prescribed in Appendix E.

10. ROSIN

10.1 Shellac shall not contain any rosin, as tested by the method prescribed in Appendix F, except when the presence of a specified percentage is agreed to between the purchaser and the vendor, in which case the determination is carried out as agreed between the purchaser and the vendor (see Foreword, clause VIII).

11. ORPIMENT AND OTHER ARSENICAL IMPURITIES

11.1 Shellac shall not contain any orpiment, as tested by the method prescribed in Appendix G, except when a specified percentage is agreed to between the purchaser and the vendor, in which case the determination is carried out as prescribed in Method I of Appendix H.

11.2 When the material is required for food or drug preparations, the determination of traces of arsenic, too small for titration by Method I of Appendix H, shall be carried out by Method II of Appendix H.

12. ASH

12.1 Shellac shall not leave, on incineration, ash in excess of the limits given below:

Hand-made		Machine-made	
Grade	Maximum per cent	Grade	Maximum per cent
Special	0.5	D1	0.3
A	0.5	D2	0.3
B	0.8	I	0.3
C	1.0	II	0.3
D	1.0	III	0.5
E	1.0	IV	0.5

12.2 The ash is determined by the method prescribed in Appendix J.

13. MATTER SOLUBLE IN WATER

13.1 Shellac shall contain not more than 0.5 per cent 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 prescribed in Appendix K.

14. FLOW TEST

14.1 Shellac shall satisfy a flow test, which is conducted by the method prescribed in Appendix L, unless otherwise agreed between the purchaser and the vendor (see Foreword, clause VIII).

15. HEAT POLYMERIZATION TEST

15.1 If agreed between the purchaser and the vendor, shellac shall satisfy a heat polymerization test. The method shall be that prescribed in Appendix M and unless otherwise agreed, the temperature of test shall be 150 °C.

16. ACID VALUE

16.1 The acid value of shellac may be fixed, if desired, by agreement between the purchaser and the vendor. It is determined by the method prescribed in Appendix N, unless otherwise agreed between the purchaser and the vendor (see Foreword, clause VIII).

17. LEAD CONTENT

17.1 The limit of lead content is subject to agreement between the purchaser and the vendor, and the percentage of lead is determined by the method prescribed in Appendix P.

18. NON-VOLATILE MATTER SOLUBLE IN COLD ALCOHOL

18.1 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).

19. CHARACTER OF REQUIREMENTS

19.1 The character of requirements for shellac is given in table 1, page 11.

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

TABLE 1

Requirements for Shellac

Clause reference	Character of requirement	Characteristic	Maximum limits for grade												Methods of testing Reference to Appendix
			Hand-made						Machine-made						
			Special	A	B	C	D	E	D_1	D_2	I	II	III	IV	
6	Optional	Volatile matter (moisture) per cent	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	B
7	Essential	Matter insoluble in hot alcohol, per cent	1.0	1.0	1.5	2.0	3.0	4.0	0.5	0.5	0.5	0.5	1.0	1.0	C
8.1	Optional	Colour index	6.0	8	10	15	22	50	2.0	5.0	8	12	22	50	D
		or													
8.2	Optional	Appearance and colour	Not inferior to appearance and colour of an approved sample												
9	Optional	Wax, per cent	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	E
		Dewaxed shellac	—	—	—	—	—	—	As agreed between purchaser and vendor						E
10	Essential	Rosin	Nil						Nil						F
11	Essential	Orpiment *	Nil						Nil						G
12	Optional	Ash, per cent	0.5	0.5	0.8	1.0	1.0	1.0	0.3	0.3	0.3	0.3	0.5	0.5	J
13	Optional	Matter soluble in water, per cent	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	K
14	Optional	Flow test	As agreed between purchaser and vendor												L
15	Optional	Heat polymerization test	As agreed between purchaser and vendor												M
16	Optional	Acid value	As agreed between purchaser and vendor												N
17	Optional	Lead content **	As agreed between purchaser and vendor												P
18	Optional	Non-volatile matter soluble in cold alcohol	As agreed between purchaser and vendor (see Foreword, clause VIII).												

* If a specified percentage of orpiment content is agreed to between the purchaser and the vendor, its determination is made by the method prescribed in Appendix II.

** Shellac is not expected to contain any lead, but to guard against inadvertent contamination, when lead-free shellac is required, it shall not contain more than 0.03 percent of lead calculated as lead.

Appendix A

(Section 3)

SAMPLING OF SHELLAC

A-1. DRAWING OF SAMPLES

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

A-1.2 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 SHELLAC IN A FREE FLOWING CONDITION. 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 mixed. One half of the material is labelled as the "original observation sample" and is, if necessary, further sub-divided by the normal process of quartering to form a number of original observation samples (see clause A-1.7). 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 SHELLAC. 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 mixed. One half of the material is labelled as the "original observation sample" and is, if necessary, further sub-divided to form a number of original observation samples (see clause A-1.7). 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 described under clause A-2.1 to form the "analysis sample".

A-1.7 An original observation sample is used for the determination of volatile matter (moisture), when this requirement is agreed upon between the purchaser and the vendor. The original observation sample is placed in an air-tight container, sealed and labelled as "original observation sample", and "sample for the determination of volatile matter (moisture)" when volatile matter (moisture) requirement is agreed upon.

A-2. REDUCTION OF SAMPLES

A-2.1 The material for the analysis sample, as obtained under clause A-1.5 or A-1.6 is mixed thoroughly, and 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 and 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 0.71 mm or is not less than 0.425 mm (see clause A-4.1). This finely ground material is mixed thoroughly and divided into the requisite number of samples for testing, in accordance with the requirements of this ISO Recommendation. These samples are placed in air-tight containers, sealed and labelled "prepared sample".

A-3.2 The original observation sample which is to be used for the determination of volatile matter (moisture), when this requirement is agreed upon between the purchaser and the vendor, is ground to pass entirely through a sieve whose nominal aperture does not exceed 0.71 mm or is not less than 0.425 mm (see clause A-4.1).

A-4. NOTE ON SIEVE SIZES

A-4.1 Table 2 (page 14) gives some of the national sieve designations and mesh apertures corresponding approximately to the sizes specified in the preceding clauses and in Appendix K.

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) Between 0.71 and 0.425 (Woven)	(*a*) No. 25	(*a*) 0.71	(*a*) 22 mesh	(*a*) 0.699	(*a*) —	(*a*) —	(*a*) —	(*a*) —	70	0.708
	(*b*) No. 30	(*b*) 0.59	(*b*) 25 mesh	(*b*) 0.599	(*b*) Test Sieve No. 29	(*b*) 0.63	(*b*) Test Sieve 0.6	(*b*) 0.6	60	0.592
	(*c*) No. 35	(*c*) 0.50	(*c*) 30 mesh	(*c*) 0.500	(*c*) Test Sieve No. 28	(*c*) 0.50	(*c*) Test Sieve 0.5	(*c*) 0.5	50	0.500
	(*d*) No. 40	(*d*) 0.42	(*d*) 36 mesh	(*d*) 0.422	(*d*) Test Sieve No. 27	(*d*) 0.40	(*d*) Test Sieve 0.43	(*d*) 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: Ell—39) ASA No: Z23.1—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 is determined by heating a weighed specimen of the original observation sample at a temperature of 41 °C ± 1 °C for 4 hours and then keeping it over a concentrated sulphuric acid *in vacuo* for 18 hours.

B-2. PROCEDURE

B-2.1 For this test, use the original observation sample ground to specified size (see A-3.2). Weigh a clean, dry, flat-bottomed dish of about 75 mm in diameter, provided with a ground-glass cover. Transfer approximately 2 g of the powdered sample to the dish, cover it with the ground-glass cover and weigh it again. The difference gives the weight of the sample taken.

B-2.2 Keep the dish with the sample, without covering it, in a well ventilated oven maintained at a temperature of 41 °C ± 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 keep the sample uncovered *in vacuo* for 18 hours. Remove the dish, cover it with the ground-glass cover and immediately weigh. Express the loss in weight as a percentage of the weight of the sample taken.

B-3. CALCULATION

B-3.1 Volatile matter (moisture), per cent $= \dfrac{100\ (W-w)}{W}$

where W = weight of sample taken, and
w = weight after drying.

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 shellac with 95 per cent (by volume) ethanol and determining the percentage of the undissolved residue by either (as may be agreed) of the two methods described below.

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.

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

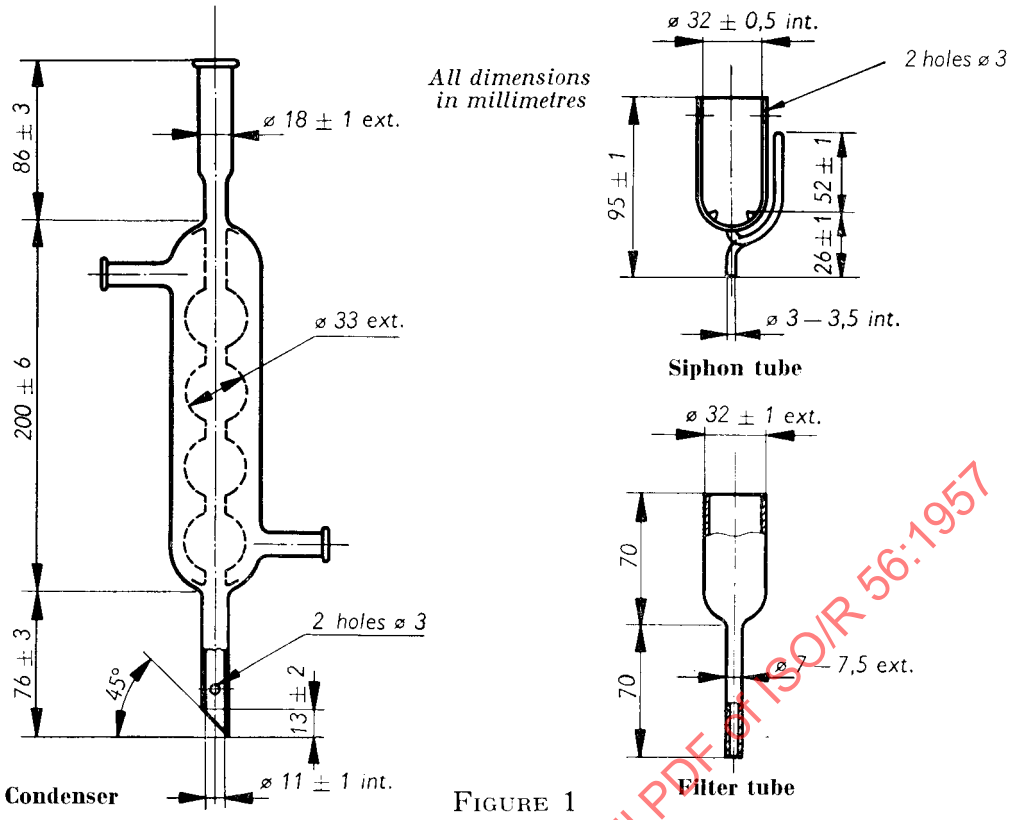

FIGURE 1

Extraction apparatus for determining hot alcohol insolubles, Method I

C/I-1.1.6 *Weighing bottles* — 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.

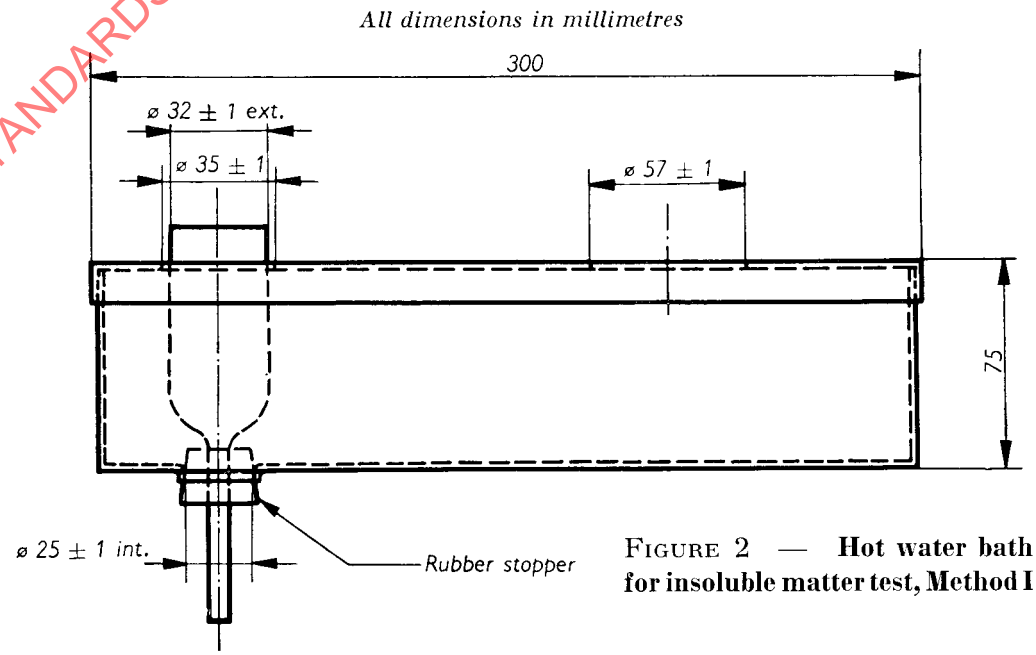

FIGURE 2 — **Hot water bath for insoluble matter test, Method I**

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 shellac 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 hot 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, shellacs 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. Use the lowest weight in the calculation.

C/I-5. CALCULATION

C/I-5.1 Matter insoluble in hot alcohol, per cent $= \dfrac{100\ w}{W}$

where w = weight of residue, and
W = weight of sample taken.

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

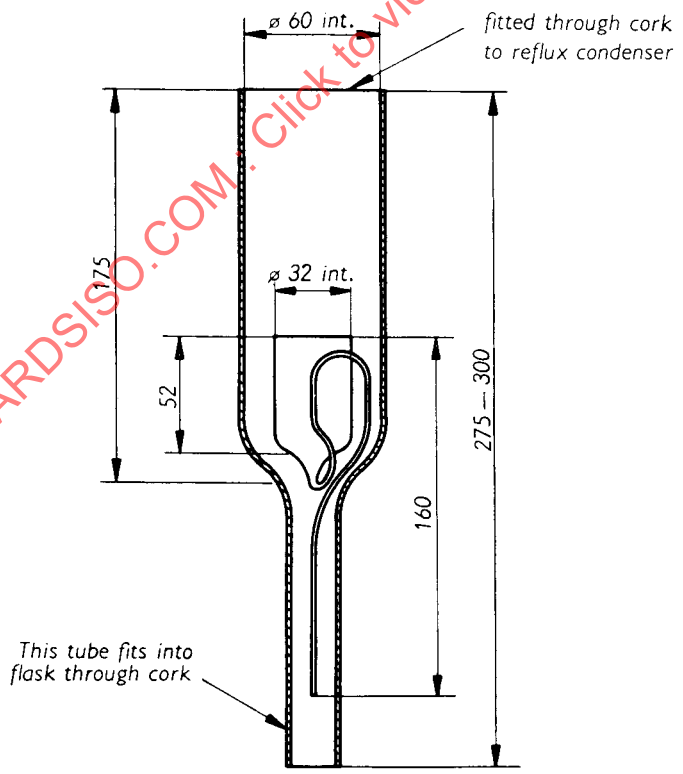

FIGURE 3

Siphon tube and adaptor

* 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.

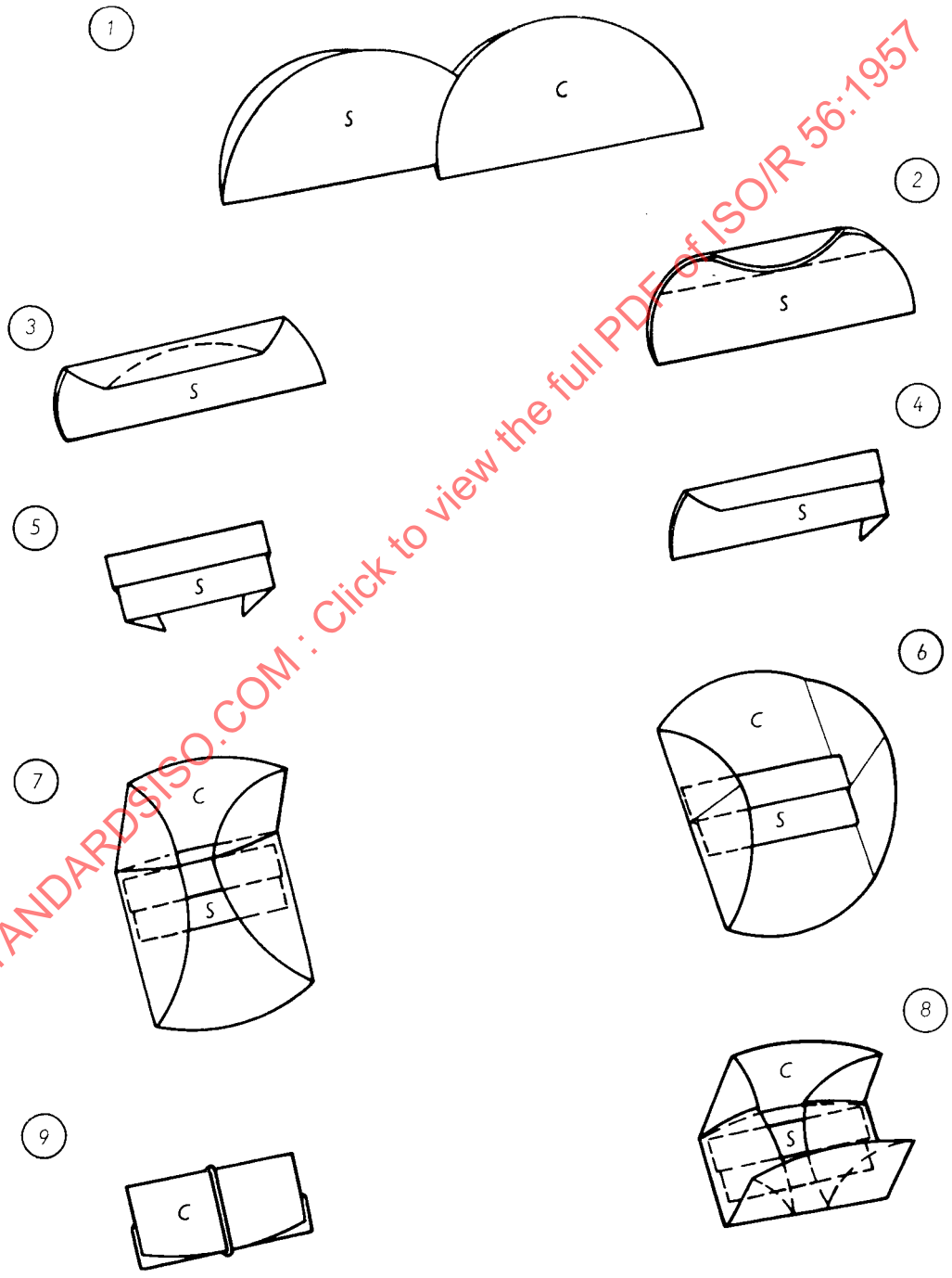

FIGURE 4

Folding of filter paper

C/II-3.2 Weigh 4.5 g to 5.5 g of the "prepared sample" of shellac (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 afterwards 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 filter 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)

DETERMINATION OF COLOUR INDEX

D-1. OUTLINE OF METHOD

D-1.1 The colour index is determined by comparing the colour of a 0.005 N solution of iodine (in potassium iodide solution) with a solution of shellac in ethanol by diluting the latter progressively until a close match is obtained. For machine-made shellac of grades D1 and D2, the modified method as given in D-5 below is used.

D-2. REAGENTS

D-2.1 The following reagents are required:

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

D-2.1.2 *Standard iodine solution* — Prepare the standard 0.005 N solution of iodine by introducing 5 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. This solution corresponds to colour index 5. Shake the solution before use.

D-2.1.3 *Shellac solution* — Dissolve 10.0 g of the "prepared sample" (see A-3.1) in 100 ml of the 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-3. PROCEDURE

D-3.1 Transfer 5 ml of the filtered shellac 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 colours 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 shellac solution, with occasional shaking, until the colour is the same as that of the standard solution. Note the volume of alcohol added.

D-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-4. CALCULATION OF RESULTS

D-4.1 The volume in millilitres of alcohol so added plus five, or the total volume of shellac solution after such dilution, is the colour index of the sample.

D-4.2 The accuracy of this test, including the personal error of different analysts, is about 5 per cent.

D-5. MODIFIED METHOD FOR LIGHTER GRADES OF SHELLAC

D-5.1 For samples having a colour index of less than 5, start with a 0.001 N iodine solution and 10 ml of shellac solution, prepared as described under D-2.1.3. Follow the same procedure as above. The colour index will be the final volume to which the shellac solution has to be diluted for matching the 0.001 N iodine solution, divided by 10.

Appendix E

(Section 9)

DETERMINATION OF WAX

E-1. GENERAL

E-1.1 Two methods for the determination of wax, Method I for shellac containing wax and Method II for dewaxed shellac, are described below.

E/I METHOD I, FOR SHELLAC CONTAINING WAX

E/I-1. OUTLINE OF METHOD

E/I-1.1 A specified quantity of shellac 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 shellac 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 in diameter 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 shellac 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".

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

E/I-3.2 If the filtration is done only under gravity, after the filtration is completed and all soluble shellac 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 shellac, 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 bits 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

$$\text{Wax, per cent} = \frac{100\,w}{W}$$

where

w = weight of wax and
W = weight of sample taken.

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

E/II METHOD II, FOR DEWAXED SHELLAC

E/II-1. OUTLINE OF METHOD

E/II-1.1 A specified quantity of dewaxed shellac 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 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)

DETECTION OF ROSIN (HALPHEN-HICKS METHOD)

F-1. REAGENTS

F-1.1 The following reagents are required:

F-1.1.1 *Ethanol* — absolute.

F-1.1.2 *Acetic acid* — glacial.

F-1.1.3 *Petroleum ether* — boiling point below 80 °C.

F-1.1.4 *Solution A* — one part of phenol by volume dissolved in two parts by volume of carbon tetrachloride.

F-1.1.5 *Solution B* — one part by volume of bromine dissolved in four parts by volume of carbon tetrachloride.

F-2. PROCEDURE

F-2.1 Place about 2 g of the "prepared sample" (see A-3.1) in a 250 ml conical flask, add 10 ml of ethanol or glacial acetic acid and shake until solution of the resinous material is complete. Then add slowly and with continuous agitation 50 ml of petroleum ether. After the addition of petroleum ether, add 50 ml of water in exactly the same manner, transfer to a small separating funnel and allow it to stand until the petroleum ether separates. Draw off the water layer, wash the petroleum ether layer once with water and then filter the petroleum ether extract through a dry filter paper into a round-bottomed evaporating dish. Evaporate to dryness on a steam bath and test the residue as follows.

F-2.2 Add 1 to 2 ml of solution A to the residue left after evaporation of the solution in petroleum ether and pour this mixture into the cavity of an ordinary porcelain colour-reaction plate until it just fills the depression. Immediately fill an adjacent cavity with solution B. Cover the plate with an inverted watch-glass and note the colour, if any, produced in solution A by the action of the bromine vapours from solution B.

F-2.3 A decided purple or deep indigo blue colour is an indication of the presence of rosin.

Appendix G

(Clause 11.1)

DETECTION OF ORPIMENT

G-1. OUTLINE OF METHOD

G-1.1 The presence of 0.5 per cent or more of orpiment in shellac gives the shellac flake a yellow, opaque appearance. Even a trace of orpiment can be detected from a solution in spirit or in aqueous borax if the shellac is free from dirt.

G-2. REAGENTS

G-2.1 The following reagents are required:

G-2.1.1 *Alcohol* — 95 per cent (by volume) ethanol or 95 per cent (by volume) denatured spirit.

G-2.1.2 *Borax solution* — 5 per cent (by weight) of borax ($Na_2B_4O_7 \cdot 10\,H_2O$) in distilled water.

G-3. PROCEDURE

G-3.1 Prepare in a small conical flask a solution of shellac (strength 20 per cent *w/v*) in alcohol or borax solution. Allow the solution to stand, when the orpiment settles in a layer at the bottom. Examine from below.

G-3.2 Yellow particles of orpiment will be visible if it is present; 0.3 per cent orpiment gives a continuous layer.

G-3.2.1 This method is most sensitive when the alcohol is cooled to O °C before preparing the solution. The same effect is not obtained by dissolving at room temperature and then cooling because, under these conditions, the agglomerates of wax are liable to hold the orpiment particles in suspension.

Appendix H

(Clauses 11.1 and 11.2)

DETERMINATION OF ARSENIC

H-1. GENERAL

H-1.1 Two methods for the determination of arsenic content, one for appreciable amount of orpiment and the other for traces of arsenic, are described below.

H/I METHOD I, FOR APPRECIABLE AMOUNT OF ORPIMENT

H/I-1. OUTLINE OF METHOD

H/I-1.1 Shellac is digested with nitric acid and sulphuric acid and the arsenic compound, obtained by distillation after treating the solution with the chloride-hydrazine-bromide mixture is titrated by a suitable method.

H/I-2. APPARATUS

H/I-2.1 The apparatus is shown in figure 5 below.

H/I-3. REAGENTS

H/I-3.1 The following reagents are required:

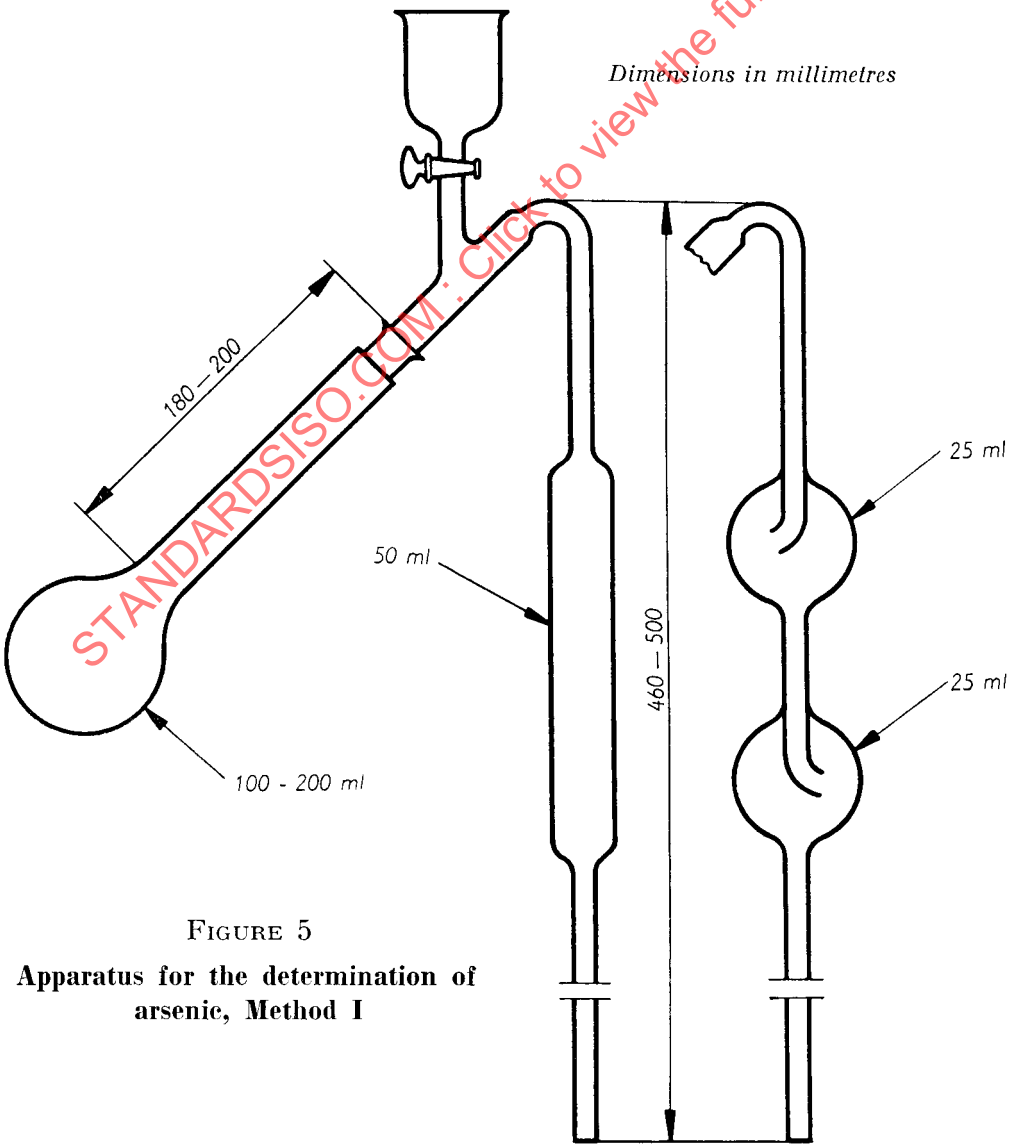

FIGURE 5

Apparatus for the determination of arsenic, Method I

H/I-3.1.1 *Dilute nitric acid* — 30 parts by volume of concentrated nitric acid in 100 parts of solution.

H/I-3.1.2 *Concentrated sulphuric acid* — specific gravity 1.84.

H/I-3.1.3 *Concentrated nitric acid* — specific gravity 1.42.

H/I-3.1.4 *Chloride-hydrazine-bromide mixture* — mix 5 g of sodium chloride, 0.5 g of hydrazine sulphate and 0.02 g of potassium bromide, and store in a tightly stoppered bottle.

H/I-3.1.5 *Concentrated hydrochloric acid* — specific gravity 1.16.

H/I-3.1.6 *Methyl orange indicator* — 0.04 per cent (*w*/*v*) solution in 20 per cent (*v*/*v*) ethanol.

H/I-3.1.7 *Standard potassium bromate solution* — approximately 0.01 N.

H/I-3.1.8 *Sodium hydroxide solution* — approximately 2 N.

H/I-3.1.9 *Sodium bicarbonate* — analytical grade.

H/I-3.1.10 *Standard iodine solution* — approximately 0.01 N.

H/I-3.1.11 *Starch solution* — make a paste of 0.2 g of soluble (potato) starch in cold water and pour into 100 ml of boiling water. Boil for 5 minutes, cool and bottle. The solution should be prepared freshly every two or three days.

H/I-4. PROCEDURE

H/I-4.1 Weigh 4.5 g to 5.5 g of the "prepared sample" (see A-3.1) to an accuracy of 0.01 g and place with 10 ml of dilute nitric acid in a 100 ml or 200 ml resistance glass or silica Kjeldahl flask and heat the mixture until any initial vigorous reaction subsides and ceases. Cool and add gradually 10 ml of concentrated sulphuric acid at such a rate as to prevent excessive frothing or heating (10 minutes are usually required) and continue the heating. Add to the hot solution 5 ml of concentrated nitric acid in small portions and boil until colourless. If necessary, add concentrated nitric acid in further small portions at a time. Note for the purpose of the blank determination the total amount of concentrated nitric acid added. (The digestion usually takes about 30 minutes.) Cool, dilute with 50 ml of water and transfer to the flask of the distillation apparatus. Boil the solution, without inserting the condensing arm, till the bulk is reduced to about 10 ml or until white fumes appear; cool, dilute and again boil down to 10 ml; cool and add 7.0 ml of water. Cool well the liquid and add 5 g of the chloride-hydrazine-bromide mixture followed rapidly by 10 ml of concentrated hydrochloric acid.

H/I-4.2 Fit the condenser quickly and distil the liquid into 20 ml water, the exit tube dipping below the surface of the liquid; cool in ice until about 5 minutes after the condenser is full of steam. Dilute the distillate to 100 ml, add methyl orange indicator, heat the solution to 80 °C, and titrate with standard potassium bromate solution, or, alternatively, nearly neutralize the distillate with sodium hydroxide solution, then add 3 g excess sodium bicarbonate and titrate the solution with standard iodine solution using starch as indicator.

H/I-4.2.1 Preserve the residue in the distillation flask, if required, for the determination of lead in accordance with Appendix P.

H/I-4.3 Make sure that no solid material comes in contact with the ground-in portion of the flask.

H/I-4.4 See that a blank determination is run under the same conditions, on the same reagents and by the same person, but without using the material.

H/I-5. CALCULATION

H/I-5.1 Express the arsenic content of the shellac sample as percentage by weight of arsenious sulphide (As_2S_3).

$$\text{Arsenious sulphide } (As_2S_3), \text{ per cent} = \frac{6.15\ V\ N}{W}$$

where V = volume of standard iodine solution or standard potassium bromate solution, required in the titration, in millilitres,

N = normality of standard iodine solution or standard potassium bromate solution, employed in the titration and

W = weight, in grammes, of sample taken.

H/II METHOD II, FOR TRACES OF ARSENIC

H/II-1. APPARATUS

H/II-1.1 CONSTITUTION OF APPARATUS. The following apparatus, assembled as shown in Figure 6 below, is required.

H/II-1.1.1 *Wide-mouth bottle* — capacity 120 ml.

H/II-1.1.2 *Glass tube* — made from ordinary glass tubing and having a total length of 200 mm. It has an internal diameter of exactly 6.5 mm and an external diameter of about 8 mm. It is drawn out at one end to a diameter of about 1 mm and a hole not less than 2 mm in diameter is blown in the side of the tube, near the constricted part. The upper end of the tube is cut off square and is either rounded off slightly or ground smooth.

H/II-1.1.3 *Rubber bungs* — three. One fits exactly into the mouth of the wide-mouth bottle and has a hole bored centrally to take the tube from its constricted end. Each of the other two rubber bungs (about 25 mm × 25 mm) has a hole, exactly 6.5 mm in diameter, bored centrally and fitted with a rubber band or spring clip for holding them tightly together.

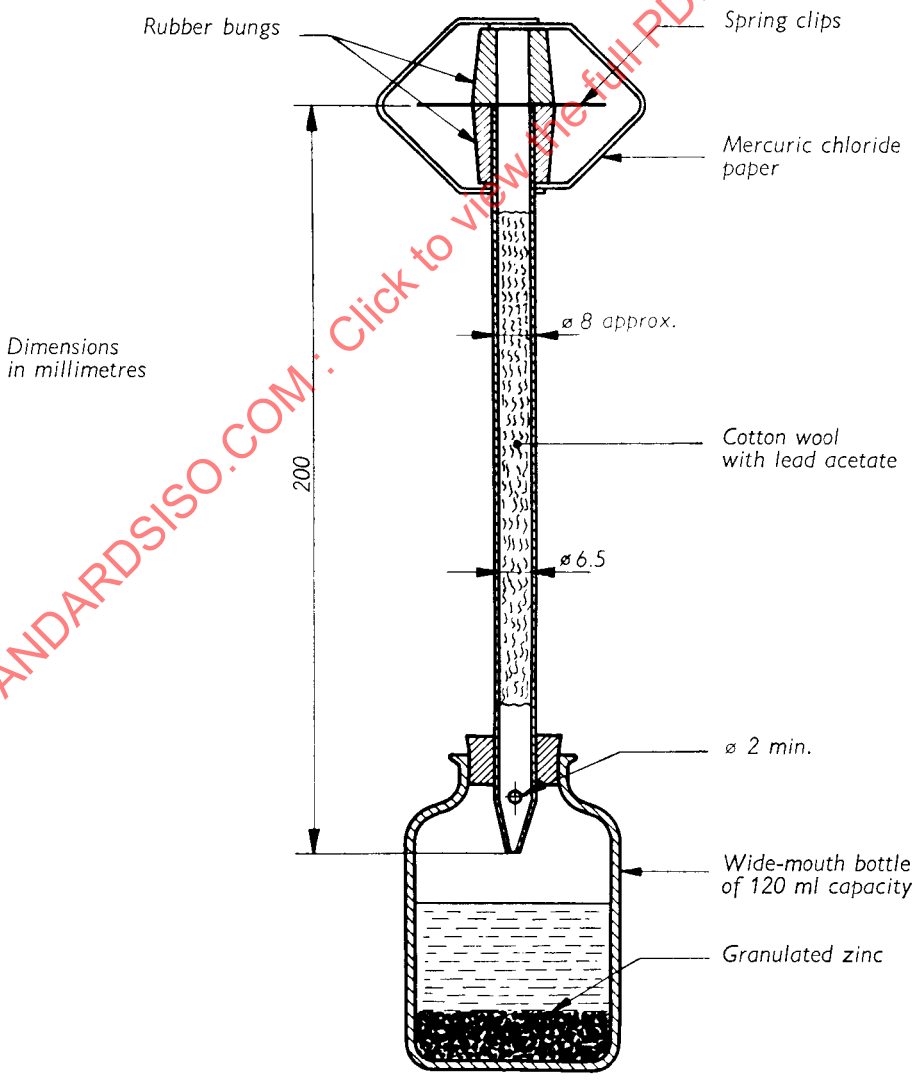

FIGURE 6

Apparatus for the determination of arsenic, Method II

H/II-1.2 PREPARATION OF THE GLASS TUBE. — Moisten a small quantity of cotton wool with lead acetate solution and then dry it in a dust-free atmosphere. Lightly pack the glass tube with this cotton wool so that the upper surface of the cotton wool is not less than 25 mm below the top of the tube. Insert the upper end of the tube into the narrow end of one of the pair of rubber bungs, either to a depth of about 10 mm, when the tube has a rounded-off end, or so that the ground end of the tube is flush with the larger end of the bung. Place a piece of mercuric chloride paper flat on the top of the bung. Place the other bung over this with its larger end in contact with the piece of mercuric chloride paper. Fasten the two bungs by means of the rubber band or the spring clip, in such a manner that the borings of the two bungs (or the upper bung and the glass tube) meet to form a true tube of 6.5 mm diameter interrupted by a diaphragm of mercuric chloride paper.

H/II-1.2.1 Instead of this method of attaching the mercuric chloride paper, any other method may be used provided (*a*) that the whole of the evolved gas passes through the paper; (*b*) that the portion of the paper in contact with the gas is a circle of 6.5 mm diameter; and (*c*) that the paper is protected from sunlight during the test.

H/II-2. REAGENTS

H/II-2.1 The following reagents of analytical reagent grade are required. The reagents with the exception of H/II-2.13 and H/II-2.14 should be free from traces of arsenic.

H/II-2.1.1 *Dilute nitric acid* — 30 per cent.

H/II-2.1.2 *Concentrated sulphuric acid* — specific gravity 1.84.

H/II-2.1.3 *Concentrated nitric acid* — specific gravity 1.42.

H/II-2.1.4 *Chloride-hydrazine-bromide mixture* — Mix 5 g of sodium chloride, 0.5 g of hydrazine sulphate and 0.02 g of potassium bromide, and store in a tightly stoppered bottle.

H/II-2.1.5 *Methyl orange indicator* — 0.04 per cent (*w*/*v*) solution in 20 per cent (*v*/*v*) ethanol.

H/II-2.1.6 *Lead acetate solution* — 10.0 per cent (*w*/*v*) in distilled water recently boiled.

H/II-2.1.7 *Mercuric chloride paper* — smooth white filter paper, not less than 25 mm in width, soaked in a saturated solution of mercuric chloride in water, pressed to remove superfluous solution and dried at a temperature of 60 °C in the dark. The grade of the filter paper is such that the weight in grammes per square metre is between 65 and 120; the thickness in millimetres of 400 papers is approximately equal, numerically, to the weight in grammes per square metre. Mercuric chloride paper should be stored in a stoppered bottle in the dark. Paper which have been exposed to sunlight or to the vapour of ammonia should not be used as they give a lighter coloured stain or no stain at all, when employed in the quantitative test for arsenic.

H/II-2.1.8 *Concentrated hydrochloric acid* — specific gravity 1.16.

H/II-2.1.9 *Stannous chloride solution* — Dilute 60 ml of concentrated hydrochloric acid with 20 ml of water, add to it 20 g of tin, heat gently until gas ceases to be evolved and add sufficient water to produce 100 ml, allowing the undissolved tin to remain in the solution. Decant the clear solution, add an equal volume of concentrated hydrochloric acid, boil down to the original volume and filter through a fine-grained filter paper.

H/II-2.1.10 *Stannated hydrochloric acid* — Mix together 1 ml of stannous chloride solution and 100 ml of concentrated hydrochloric acid.

H/II-2.1.11 *Potassium iodide* — crystals or in the form of powder.

H/II-2.1.12 *Zinc* — granulated, complying with the following test: Take 50 ml of water, 10 ml of stannated hydrochloric acid and 0.1 ml of dilute solution of arsenic (see H/II-2.1.14) in the wide-mouth bottle. Add 1 g of potassium iodide and 10 g of zinc. Quickly place the prepared glass tube (see H/II-1.2) in position. Allow the reaction to continue for 1 hour. A faint but distinct yellow stain is produced on the mercuric chloride paper.

H/II-2.1.13 *Strong solution of arsenic* — Dissolve 0.132 g of arsenious trioxide in 50 ml of concentrated hydrochloric acid and add sufficient water to produce 100 ml.

H/II-2.1.14 *Dilute solution of arsenic* — freshly prepared. Dilute 1 ml of strong solution of arsenic with water sufficient to produce 100 ml. This solution contains 0.01 mg of arsenic (or 0.0132 mg of As_2O_3) per millilitre.

H/II-3. PROCEDURE

H/II-3.1 Treat 1 g of the sample exactly in the manner prescribed in H/I-4.1 and then proceed as follows:

H/II-3.2 Fit the condenser quickly and distil the liquid into a mixture of 10 ml of water and 2 ml of concentrated nitric acid. Then evaporate the distillate to dryness on the water bath and evaporate the residue twice to dryness with 5 ml of water to remove nitric acid. Dissolve the final residue by warming in 3 ml of concentrated sulphuric acid, cool and dilute with water. Transfer the whole of the solution to the wide-mouth bottle, add 15 ml of stannated hydrochloric acid and 1 g of potassium iodide. Then add 10 g of zinc. Quickly place the prepared glass tube (see H/II-1.2) in position. Allow the reaction to continue for 40 minutes. Remove the piece of mercuric chloride paper at the end of this period. If arsenic is present in the material, compare the yellow stain produced on the mercuric chloride paper, by daylight, with the standard stains prepared as described under H/II-3.4. If the stain in this test exceeds that equivalent to 0.02 mg of arsenious trioxide (As_2O_3), make the solution to a known bulk with dilute sulphuric acid (1 : 8) and take an aliquot portion to produce a stain suitable for matching.

H/II-3.2.1 The reaction may be accelerated by placing the apparatus on a warm surface, care being taken that the mercuric chloride paper remains quite dry throughout the test. The most suitable temperature for carrying out the test is generally about 40 °C, but because the rate of evolution of the gas varies somewhat with different batches of zinc, the temperature may be adjusted to obtain a regular, but not too violent, evolution of gas. The tube should be washed with concentrated hydrochloric acid, rinsed with water and dried between successive tests.

H/II-3.3 COMPARISON OF STAINS. The comparison of the stains is made with freshly prepared standard stains immediately at the completion of the test.

H/II-3.4 PREPARATION OF STANDARD STAINS. Mix together 50 ml of water, 10 ml of stannated hydrochloric acid and appropriate volume of dilute solution of arsenic. Treat the resulting solutions as described under H/II-3.2 to prepare the standard stains.

H/II-3.5 Make sure that no solid material comes in contact with the ground-in portion of the bottle.

H/II-3.6 A blank determination is carried out under the same conditions, on the same reagents and by the same person but without using the material. The blank shall not produce any visible stain on the mercuric chloride paper.

H/II-4. CALCULATION

H/II-4.1 Express the arsenic content of the original shellac sample as parts of arsenic (As) or arsenious trioxide (As_2O_3) per million parts of shellac.

Appendix J

(Section 12)

DETERMINATION OF ASH

J-1. PROCEDURE

J-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.

J-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.

J-2. CALCULATION

J-2.1 Ash, per cent $= \dfrac{100\ w}{W}$

where w = weight of ash and
W = weight of sample taken.