
INTERNATIONAL STANDARD



56 / II

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

Shellac — Specification — Part II : Machine-made shellac

Gomme laque en feuilles — Spécification — Partie II : Gomme laque en feuilles de fabrication mécanique

First edition — 1979-04-01

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UDC 668.447.31

Ref. No. ISO 56/II-1979 (E)

Descriptors : shellac, materials specifications, chemical analysis, tests, sampling.

FOREWORD

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

International Standard ISO 56/II was developed by Technical Committee ISO/TC 50, *Lac*, and was circulated to the member bodies in November 1976.

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

Australia
Austria
Czechoslovakia
India

Korea, Rep. of
Mexico
Netherlands
Romania

ISO 56-2:1979

Sweden

Turkey

Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

Canada

This International Standard cancels and replaces ISO Recommendation R 56-1957 of which it constitutes a technical revision.

Acknowledgement is due for the assistance that has been derived from the specifications and publications of the American Society for Testing and Materials, the American Bleached Shellac 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 India Lac Research Institute.

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Shellac — Specification — Part II : Machine-made shellac

0 INTRODUCTION

0.1 ISO/R 56, published in 1957, covered shellac, hand-made as well as machine-made. It has been revised into two parts, one for each kind.

0.2 The types and grades of machine-made shellac have been found to require modification in order that the characteristics of a given consignment of this material may be clearly defined. Machine-made shellac has now been divided into four main types, on the basis of the method of manufacture, and then into grades, within each type, on the basis of colour. Requirements for matter insoluble in hot alcohol, colour index and ash have now been prescribed based on the new system of types and grades.

0.3 Whereas in ISO/R 56 the limit for wax was left subject to agreement between the purchaser and the supplier, definite values have now been included. New requirements have been laid down for grit, iodine value, clarity of solution, and alkalinity of the aqueous extract.

0.4 The requirement for non-volatile matter soluble in cold alcohol has not been retained as the requirement is applied in practice to waste products of lac only. The methods for quantitative determination of rosin have also been dropped since this type of adulteration is no longer in evidence. In ISO/R 56, an alternative method (the Westinghouse method) was given for determination of flow. In this International Standard it has been dropped.

0.5 Three of the requirements for machine-made shellac, namely those for

- a) matter insoluble in hot alcohol,
- b) absence of rosin, and
- c) absence of orpiment,

are included in this International Standard as essential clauses.

The remaining requirements, 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,
- p) grit,
- q) iodine value, and
- r) clarity of solution,

are optional.

0.6 The sizes of sieves given in the text of this International Standard have been indicated in terms of aperture dimensions, in accordance with ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures*.

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

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies requirements and corresponding methods of test for machine-made shellac.

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

1.3 The limits prescribed in this International Standard are limits for rejection.

2 DEFINITIONS

For the purpose of this International Standard, the following definitions apply.

- 2.1 sticklac** : The natural product of lac insects.
- 2.2 seedlac** : The product obtained by washing crushed sticklac.
- 2.3 shellac** : The product obtained by refining seedlac by heat process or by solvent process or by both heat and solvent processes.
- 2.4 approved sample** : The sample agreed upon between the purchaser and the supplier as the standard for colour and appearance.

3 FORM AND CONDITION

Machine-made shellac shall be in the form of flakes, sheets, buttons, or any other from agreed between the purchaser and the supplier.

4 TYPES AND GRADES

4.1 Four types of machine-made shellac are specified, namely

- Type I – produced by heat process.
- Type II – produced by hot solvent process.
- Type III – de-waxed.
- Type IV – decolorized and de-waxed.

4.2 Five grades in each of types I and II, namely grades 1, 2, 3, 4 and 5 are specified. Four grades in each of types III and IV, namely grades 1, 2, 3, and 4 are specified.

5 MANDATORY REQUIREMENTS

5.1 Matter insoluble in hot alcohol

Machine-made shellac shall not contain matter insoluble in hot alcohol, determined by either of the methods specified in annex A, as agreed between the purchaser and the supplier, in excess of the limits given in table 1.

TABLE 1

Type	Maximum % (m/m)
I	0,6
II	0,3
III	0,2
IV	0,2

5.2 Rosin

Machine-made shellac shall not contain any rosin, when tested by the method specified in annex B.

5.3 Orpiment

5.3.1 Machine-made shellac shall not contain any orpiment, when tested by the method specified in annex C, except when a specified percentage is agreed to between the purchaser and the supplier, in which case the determination shall be carried out as specified in annex D, method I.

5.3.2 When the material is required for food or drug preparations, the determination of traces of arsenic, too small for titration by method I in annex D, shall be carried out by method II in annex D.

6 OPTIONAL REQUIREMENTS

The optional requirements given below shall be subject to agreement between the purchaser and the supplier.

6.1 Volatile matter (moisture)

Machine-made shellac shall not contain more than 2,0 % (m/m) volatile matter (moisture) as determined by the method specified in annex E.

6.2 Colour index or colour and appearance

6.2.1 The colour index of machine-made shellac, as determined by the method specified in annex F, shall not exceed the limits given in table 2.

TABLE 2

Grade \ Type	A	B	C	D	E
I, II	8	12	22	35	50
III	9	20	35	50	—
IV	0,9	2,0	3,5	5,0	—

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

6.3 Wax

Machine-made shellac of type I and type II shall not contain more than 5,5 % (m/m) of wax, and that of type III and type IV not more than 0,2 % (m/m) of wax, when tested in accordance with method I and method II respectively, specified in annex G.

6.4 Ash

Machine-made shellac shall yield not more than 0,3 % (m/m) of ash, as determined by the method specified in annex H.

6.5 Matter soluble in water

Machine-made shellac shall not contain more than 0,5 % (*m/m*) of matter soluble in water and the aqueous extract shall not be acidic to methyl red or alkaline to bromothymol blue. Matter soluble in water shall be determined by the method specified in annex J.

6.6 Flow test

Machine-made shellac shall have a flow within the range agreed to between the purchaser and the supplier, when tested by the method specified in annex K.

6.7 Heat polymerization test

Machine-made shellac shall have a heat polymerization time within the range agreed to between the purchaser and the supplier, when tested by the method specified in annex L. Unless otherwise agreed, the temperature of test shall be 150 °C.

6.8 Acid value

The acid value of the machine-made shellac shall be fixed, if desired, by agreement between the purchaser and the supplier. It shall be determined by the method specified in annex M.

6.9 Lead content

The maximum limit for lead content shall be subject to agreement between the purchaser and the supplier and the lead content shall be determined by the method specified in annex N.

6.10 Grit content

The maximum limit for grit content shall be as agreed to between the purchaser and the supplier. When required, it shall be determined by the method specified in annex P.

6.11 Iodine value

The maximum limit for the iodine value shall be as agreed to between the purchaser and the supplier. When required, it shall be determined by either of the two methods specified in annex Q.

6.12 Clarity of solution

This requirement is applicable to type III and type IV shellac and shall be subject to agreement between the purchaser and the supplier. The shellac shall show no turbidity when tested by the method specified in annex R.

7 SAMPLING

Samples shall be taken in the manner specified in annex S.

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ANNEX A
(See 5.1)

DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

A.1 PRINCIPLE

Extraction of a test portion with 95 % (V/V) ethanol and weighing of the undissolved residue.

A.2 METHOD I

A.2.1 Reagent

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

A.2.2 Apparatus

Ordinary laboratory apparatus and

A.2.2.1 Extraction apparatus, comprising

A.2.2.1.1 Condenser, all glass, of the type and dimensions shown in figure 1, the tip of which is cut at an angle of 45°. The condenser has two holes in its tip through which passes the wire holding the siphon tube (A.2.2.1.2).

A.2.2.1.2 Siphon tube, of glass, of the type and dimensions shown in figure 1. The siphon tube has two holes near the top for a wire to be fastened to the condenser tip, leaving about 6 mm space between the top of the tube and the condenser tip.

A.2.2.1.3 Conical flask, heat resistant, wide mouthed, conical, preferably of borosilicate glass of height approximately 175 mm and approximately 50 mm inside diameter at the top. The flask has a tight-fitting cork of depth 25 mm, 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 nickel-chromium or iron gauze is used. The gauze has no asbestos covering.

A.2.2.1.4 Carbon filter tube, of the type and dimensions shown in figure 1, having a light spiral spring at the bottom to hold up the extraction cartridge (A.2.2.2). The stem of the filter tube is fitted with a rubber stopper and firmly held in the hot water bath (A.2.2.4).

A.2.2.2 Extraction cartridges, of fat-free paper, of diameter approximately 25 mm and height approximately 60 mm.

A.2.2.3 Weighing bottle, glass-stoppered, of height approximately 80 mm and diameter approximately 40 mm.

A.2.2.4 Hot water bath, made of copper or stainless steel,

having a width of approximately 100 mm and other dimensions as given in figure 2.

The cover has a flanged hole of diameter 57 ± 1 mm, for a 200 ml beaker, and also a hole of diameter 35 ± 1 mm through which the top of the filter tube (A.2.2.1.4) projects. Directly below this hole, in the bottom of the bath, is a flanged hole, of diameter 25 ± 1 mm, to hold the rubber stopper, through which the stem of the filter tube extends, to discharge into the flask (A.2.2.1.3). The hot water bath is mounted on a low tripod or stand.

A.2.2.5 Gas burner, low form, adjustable, Bunsen type, carrying a draught shield, or any other suitable heating device.

A.2.2.6 Electric oven, capable of being maintained at 100 ± 2 °C.

A.2.2.7 Desiccator, containing sulphuric acid (ρ 1,84 g/ml).

A.2.2.8 Balance, accurate to 0,002 g.

A.2.2.9 Stop watch of good two-minute sand-glass.

A.2.3 Preparation of extraction cartridge

A.2.3.1 Place 125 ml of the alcohol (A.2.1) in the conical flask (A.2.2.1.3) and a new extraction cartridge (A.2.2.2) in the siphon tube (A.2.2.1.2). Introduce the siphon tube into the flask and connect it to the condenser (A.2.2.1.1), making sure that there is an ample flow of cold water through the condenser. Adjust the rate of heating so as to give a cycle of filling and emptying in the siphon tube every 2 min and extract for 30 min. Dry the cartridge in the oven (A.2.2.6), maintained at 100 ± 2 °C. After 2 h, weigh it in the tared weighing bottle (A.2.2.3), which has been kept in the desiccator (A.2.2.7), lifting the stopper of the bottle momentarily before weighing. Repeat the operations of drying, for periods of 1 h, and weighing, until the loss in mass between two successive weighings does not exceed 0,002 g.

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

A.2.4 Test portion

Before analysis, thoroughly mix the "test sample" (see S.3.1 of annex S) by rolling on paper, at least ten times, to ensure uniformity of the test sample. Weigh, directly from the paper, 4,5 to 5,5 g of the sample, to an accuracy of 0,01 g.

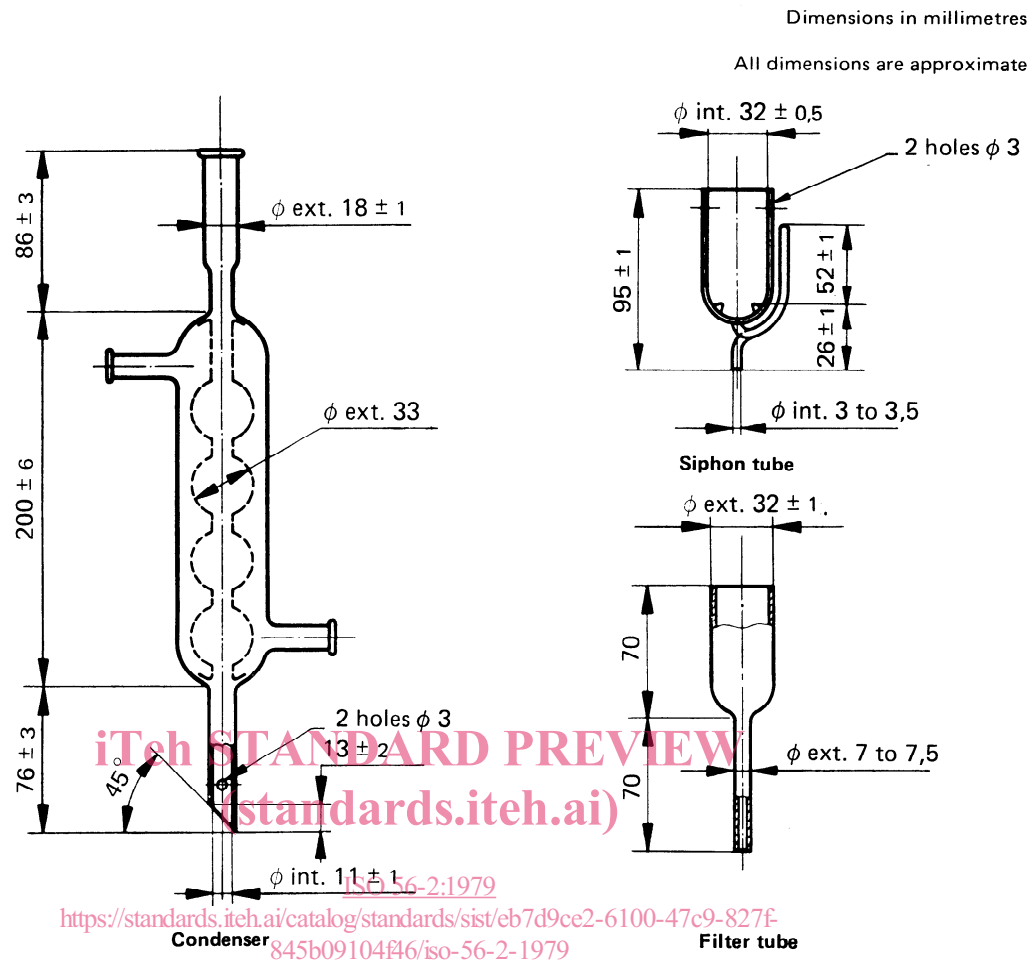


FIGURE 1 – Extraction apparatus for determination of matter insoluble in hot alcohol (method I)

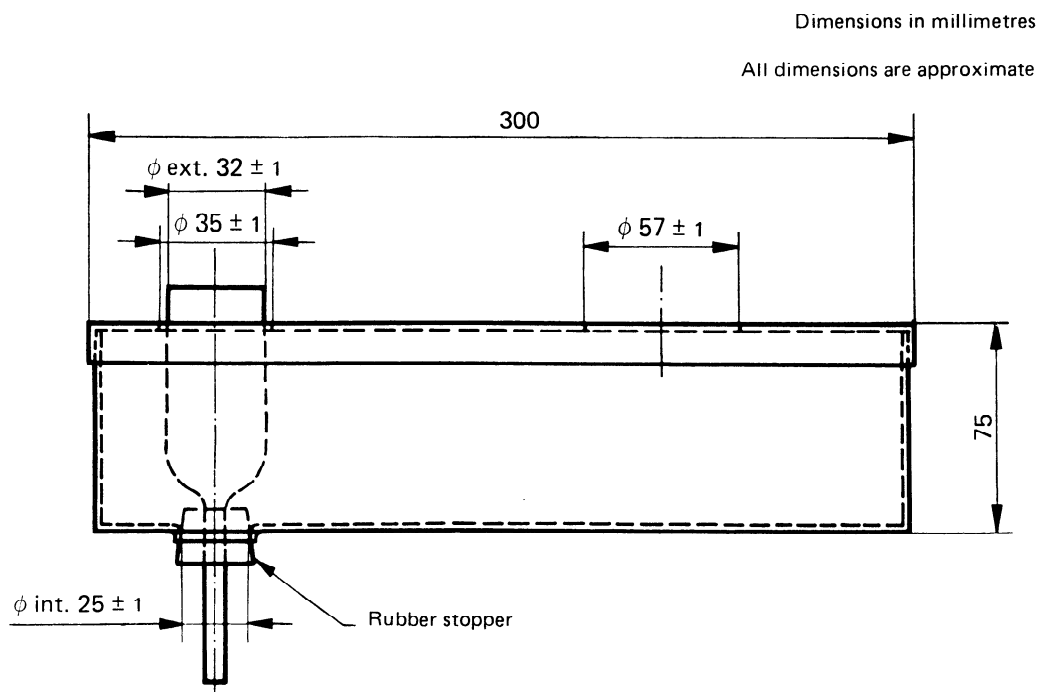


FIGURE 2 – Hot water bath for determination of matter insoluble in hot alcohol (method I)

A.2.5 Determination

Place the test portion (A.2.4) in a 200 ml tall, lipped beaker, add 125 ml of the alcohol (A.2.1), cover with a watch-glass and place on the hot water bath (A.2.2.4) (see figure 2). Boil the solution vigorously for 30 min to ensure complete solution of the shellac and dispersion of wax. Keep the volume of alcohol constant by adding from a wash-bottle, washing down the sides of the beaker.

Meanwhile, place an extracted and weighed cartridge (A.2.2.2) in the filter tube (A.2.2.1.4). 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.

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.

Transfer the cartridge containing the insoluble matter to the siphon tube (A.2.2.1.2), place 125 ml of the alcohol in the conical flask (A.2.2.1.3) and connect up the apparatus. Start the water flowing through the condenser (A.2.2.1.1), making sure that there is an adequate supply for efficient condensation. Light the burner (A.2.2.5) and time the extraction from the first emptying of the siphon, running the extraction for exactly 1 h. Immediately adjust the rate of heating so that a complete filling and emptying of the siphon tube takes place every 2 min, as determined by the stop-watch or preferably by the two-minute sand-glass (A.2.2.9), one for each extraction apparatus.

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

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

Remove the cartridge, drain in an upright position on filter paper and dry in the oven (A.2.2.6), maintained at 100 ± 2 °C. After drying for 2 h, place the cartridge in the weighing bottle (A.2.2.3), cool in the desiccator (A.2.2.7) and weigh, removing the stopper momentarily just before weighing. Repeat the operations of drying, for periods of

1 h, and weighing until the loss in mass, between two successive weighings does not exceed 0,002 g.

From the mass of the residue and the mass of the sample, calculate the percentage of insoluble matter. Use the lowest mass in calculation.

A.2.6 Expression of results

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

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (A.2.4);

m_1 is the mass, in grams, of the residue.

A.3 METHOD II

A.3.1 Reagent

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

A.3.2 Apparatus

Ordinary laboratory apparatus and

A.3.2.1 Extraction apparatus¹⁾, consisting of siphon tube, adaptor, condenser and flask, assembled with the aid of corks or ground glass joints so that the solvent can be kept boiling in the flask and its vapour can pass upwards by way of the adaptor to the condenser. The refluxing solvent runs from the condenser into the cup of the siphon tube.

A.3.2.1.1 Siphon tube, of glass, of the type shown in figure 3, having approximate internal height of 52 mm and a minimum internal diameter of 32 mm, resting in an adaptor tube in such a way that the siphon tube is surrounded by the ascending vapours of the boiling solvent (See figure 3).

A.3.2.1.2 Condenser, of any convenient design.

A.3.2.1.3 Flask, of any convenient size.

A.3.2.2 Filter paper, of diameter 125 mm, medium grade.

A.3.2.3 Weighing bottles, of glass of height approximately 80 mm and diameter approximately 40 mm, with ground glass stoppers.

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

Dimensions in millimetres

All dimensions are approximate

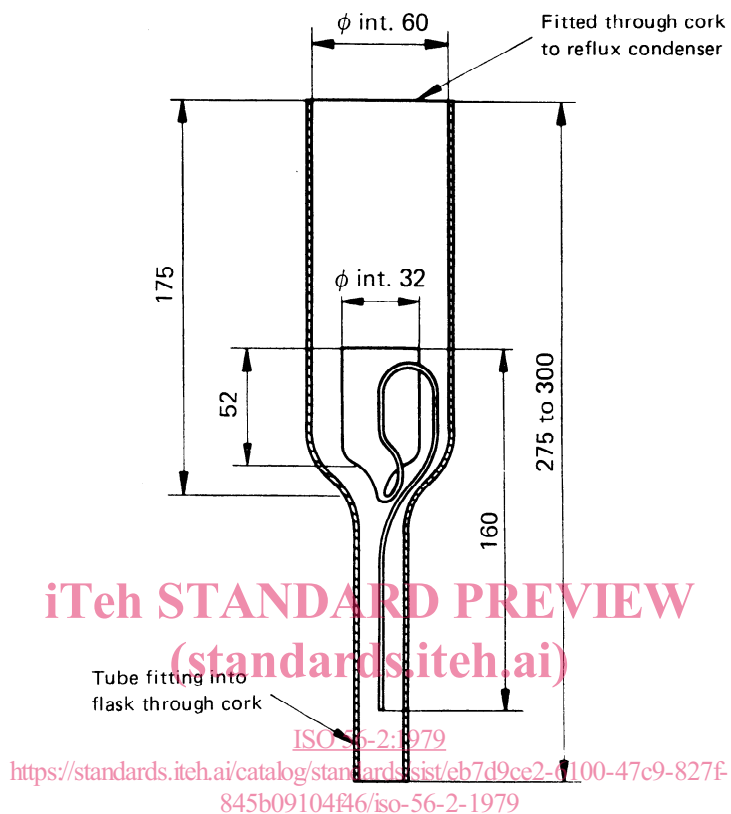


FIGURE 3 – Siphon tube and adaptor

A.3.2.4 Gas burner, low form, adjustable, Bunsen type, carrying a draught shield, or any other suitable heating device.

A.3.2.5 Electric oven, capable of being maintained at 100 ± 2 °C.

A.3.2.6 Desiccator, containing sulphuric acid (ρ 1,84 g/ml).

A.3.2.7 Balance, accurate to 0,002 g.

A.3.3 Test portion

Weigh 4,5 to 5,5 g of the "test sample" of shellac (see S.3.1 of annex S) to an accuracy of 0,01 g.

A.3.4 Determination

Fold the filter paper (A.3.2.2) so that it forms a completely closed envelope (see figure 4). Mark this paper S (for sample); wrap it closely in a second filter paper marked C (for counterpoise). Separate the filter papers and dry in the

oven (A.3.2.5), maintained at 100 ± 2 °C for 30 min. Rapidly transfer to separate weighing bottles (A.3.2.3) which have been kept in the desiccator (A.3.2.6). Place each bottle and its contents back in the desiccator for 20 min, then weigh by counterbalance, preferably using a rapid-weighing balance of the aperiodic type.

Place the test portion (A.3.3) 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 the alcohol (A.3.1). Allow to stand overnight at room temperature. Place the envelope in the cup of the siphon tube (A.3.2.1.1) and extract continuously with hot alcohol for 4 h. Keep the envelope wholly below the surface of the alcohol, when the cup is full. Maintain a rapid rate of extraction throughout, though the extract time taken for the cycle of filling and emptying the cup of the siphon tube is not critical.

At the end of the specified time, remove the paper envelope, allow to drain, separate the two papers, dry each

on a glass plate in air for 15 min and then for 2 h in the oven, maintained at $100 \pm 2^\circ\text{C}$. Place the papers rapidly in their respective weighing bottles, allow to stand in the desiccator for 20 min and again weigh by counterbalance, after momentarily removing and replacing the stoppers in the usual manner. Dry the papers for a further period of 1 h at a temperature of $100 \pm 2^\circ\text{C}$ and weigh again. If there is a loss in mass in excess of 0,002 g, repeat the

operations of drying and weighing until the difference between two successive weighings is less than 0,002 g. Use the lowest mass in the calculation.

A.3.5 Expression of results

As for method I (see A.2.6).

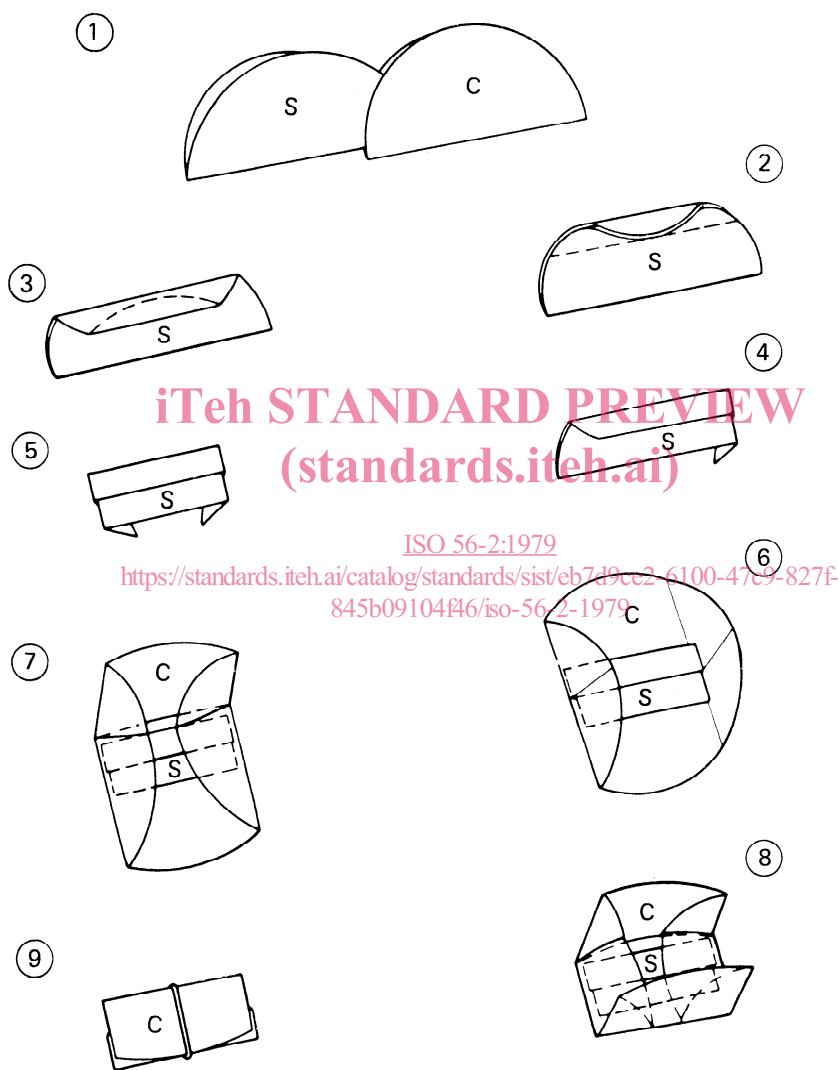


FIGURE 4 – Folding of filter paper

ANNEX B
(See 5.2)

DETECTION OF ROSIN (HALPHEN-HICKS METHOD)

B.1 REAGENTS

B.1.1 Ethanol, absolute.

B.1.2 Acetic acid, glacial.

B.1.3 Petroleum ether, boiling point below 80 °C.

B.1.4 Solution A, comprising 1 part by volume of phenol dissolved in 2 parts by volume of carbon tetrachloride.

B.1.5 Solution B, comprising 1 part by volume of bromine dissolved in 4 parts by volume of carbon tetrachloride.

B.2 APPARATUS

B.2.1 Conical flask, of capacity 250 ml.

B.2.2 Separating funnel.

B.2.3 Filter paper.

B.2.4 Evaporating dish, round bottomed.

B.2.5 Steam bath.

B.2.6 Porcelain colour-reaction plate.

B.2.7 Watch-glass.

B.3 PROCEDURE

B.3.1 Place about 2 g of the "test sample" (see S.3.1 of annex S) in the 250 ml conical flask (B.2.1) add 10 ml of the ethanol (B.1.1) or of the glacial acetic acid (B.1.2) and shake until dissolution of the resinous materials is complete. Then add slowly and with continuous agitation 50 ml of the petroleum ether (B.1.3). After the addition of the petroleum ether, add 50 ml of water in exactly the same manner, transfer to the small separating funnel (B.2.2) 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 (B.2.3) into the evaporating dish (B.2.4). Evaporate to dryness on the steam bath (B.2.5).

B.3.2 Add 1 to 2 ml of solution A (B.1.4) to the residue left after evaporation of the solution in petroleum ether and pour this mixture into the cavity of the porcelain colour-reaction plate (B.2.6) until it just fills the depression. Immediately fill an adjacent cavity with solution B (B.1.5). Cover the plate with the inverted watch-glass (B.2.7) and note the colour, if any, produced in solution A by the action of the bromine vapour from solution B.

B.3.3 A decided purple or deep indigo blue colour is an indication of the presence of rosin.

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