INTERNATIONAL STANDARD

ISO 56-1

First edition
1979-04-01

AMENDMENT 1
1996-12-15

Shellac — Specification —

Part 1:
Hand-made shellac

AMENDMENT 1

Gomme laque en feuilles — Spécification —

Partie 1: Gomme laque en feuilles de fabrication manuelle

AMENDEMENT 1

Reference number
ISO 56-1:1979/Amd.1:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Amendment 1 to International Standard ISO 56-1:1979 was prepared by Technical Committee ISO/TC 50, *Lac.*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Shellac — Specification —

Part 1:
Hand-made shellac

AMENDMENT 1

Page 9, subclause B.1.5

Add the following note after B.1.5:

NOTE — The solution should preferably be stored in amber-coloured bottles.

Page 13, subclause D.3.1.7

In the title, replace the word "tint" with the word "tin".

Page 17, Annex F

Replace the text of the existing annex with the following:

F.1 General

The colour index of shellac can be determined by either of the two methods described below. However, in case of dispute, method B may be used as the referee method for determination of colour index.

F.2 Method A

Renumber clauses F.1 to F.5.2 as F.2.1, F.2.2, F.2.2.1, F.2.2.2, F.2.3, F.2.3.1, F.2.3.2, F.2.4, F.2.4.1, F.2.4.2, F.2.5, F.2.5.1 and F.2.5.2 respectively and delete the number of subclause F.4.2.1.

F.3 Method B

F.3.1 Principle

The optical density of an alcoholic shellac solution (concentration 1,0 g/l) is measured at a particular wavelength in the visible range, which, after multiplication by 136,9, gives the value of the colour index.

F.3.2 Apparatus

F.3.2.1 Spectrometer.

Any spectrometer/colorimeter (grating type) capable of measuring absorption in the visible range (400 nm to 700 nm).

F.3.2.2 Volumetric flasks, with ground-joint stoppers, of capacity 10 ml and 100 ml.

F.3.2.3 Pipette, of capacity 1 ml.

F.3.3 Reagents

F.3.3.1 Alcohol.

Ethanol (absolute) or 95 % volume fraction rectified spirit or denatured spirit, provided that it is colourless.

F.3.4 Procedure

F.3.4.1 Preparation of test solution

Weigh accurately 1 g of the prepared test sample (see S.3.1 in annex S) of shellac and transfer the material to the 100-ml volumetric flask (F.3.2.2). Add 60 ml to 70 ml of alcohol (F.3.3.1) and shake the flask vigorously as soon as the alcohol is added until the shellac is completely dissolved. Add more solvent, and finally make up the volume to the mark of the volumetric flask. Filter the solution in an ordinary funnel using medium-grade filter paper (preferably Whatman No. 1[1)]) and keeping the funnel covered (best results are obtained if the filtration is carried out under saturated vapour pressure of the solvent). Discard the first 15 ml of the clear filtrate.

Transfer 1 ml of the filtrate by means of a pipette (F.3.2.3) to the 10 ml volumetric flask. Add alcohol to it and make up to the mark of the flask.

F.3.4.2 Measurement of optical density

Switch on the spectrometer/colorimeter. After the warming-up period of the instrument, set the wavelength at 425 nm, match the cuvettes with the alcohol used for the preparation of the solution. Transfer a portion of the diluted test solution to one of the cuvettes. Record the value of the optical density registered by the instrument.

F.3.5 Calculation

Colour index = optical density × 136,9

Page 20, subclause H.1.1

Replace the last sentence with the following:

Alternatively, heat in a muffle furnace at 650 °C to 700 °C until constant mass is obtained.

1) Whatman No. 1 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 56 and does not constitute an endorsement by ISO of this product.

ICS 87.060.20

Descriptors: shellac, specifications, materials specifications, chemical analysis, determination of content.

Price based on 2 pages

H-09-25

Published 1979-06-01

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

Shellac — Specification — Part I : Hand-made shellac

ERRATUM

MODIFICATION TO FOREWORD *(Inside front cover)*

The following sentence is to be added at the end of the foreword :

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

H-09-25

INTERNATIONAL STANDARD ISO 56/I

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

Shellac — Specification — Part I : Hand-made shellac

Gomme laque en feuilles — Spécification — Partie I : Gomme laque en feuilles de fabrication manuelle

First edition — 1979-04-01

UDC 668.447.31 **Ref. No. ISO 56/I-1979 (E)**

Descriptors : shellac, materials specifications, chemical analysis, determination of content.

Price based on 33 pages

FOREWORD

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

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

International Standard ISO 56/I was developed by Technical Committee ISO/TC 50, *Lac*, and was circulated to the member bodies in March 1977.

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

Austria	Egypt, Arab Rep. of	Sweden
Belgium	India	Turkey
Czechoslovakia	Netherlands	Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

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 Indian Lac Research Institute.

CONTENTS

Shellac — Specification — Part I : Hand-made shellac

0 INTRODUCTION

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

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

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

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 hand-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, and

q) iodine value,

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 hand-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 inserts.

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

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

4 GRADES

4.1 Six grades of hand-made shellac, namely Special, A, B, C, D and E, are specified. Further, if required by the purchaser, the names of the grades as prevalent in trade shall be indicated in addition, in parentheses, as in the following examples :

Grade Special (Golden Kusmi),

Grade B (Lemon No. 2).

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

TABLE 1

ISO grade	Trade grade
Grade Special	Kusmi buttonlac Kusmi lemon shellac
Grade A	Lemon No. 1 shellac Pure 1 buttonlac Golden shellac Superior lemon shellac Light pure buttonlac
Grade B	Lemon No. 2 shellac
Grade C	FO superfine shellac Standard 1 shellac Yellow orange shellac
Grade D	Pure TN shellac
Grade E	1 TN shellac

5 MANDATORY REQUIREMENTS

5.1 Matter insoluble in hot alcohol

Hand-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 2. By agreement between the purchaser and the supplier, the basic limit may be relaxed but it shall in any case not exceed the relaxed limit prescribed in table 2.

TABLE 2

Grade	Basic limit % (*m/m*)	Relaxed limit % (*m/m*)
Special	0,75	1,0
A	1,0	1,5
B	1,25	2,0
C	1,5	2,5
D	2,5	3,5
E	3,0	5,0

5.2 Rosin

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

5.3 Orpiment

5.3.1 Hand-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)

Hand-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 hand-made shellac, as determined by the method specified in annex F, shall not exceed the limits given in table 3.

TABLE 3

Grade	Colour index (max.)
Special	6
A	12
B	15
C	18
D	25
E	30

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

Hand-made shellac shall not contain more than 5,5 % (*m/m*) of wax when tested in accordance with the method specified in annex G.

6.4 Ash

Hand-made shellac shall not leave, on incineration, ash in excess of the limits given in table 4 when tested as described in annex H.

TABLE 4

Grade	Limit % (*m/m*) max.
Special	0,5
A	0,5
B	0,8
C	1,0
D	1,0
E	1,0

6.5 Matter soluble in water

Hand-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

Hand-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

Hand-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 hand-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.

7 SAMPLING

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

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).

Dimensions in millimetres
All dimensions are approximate.

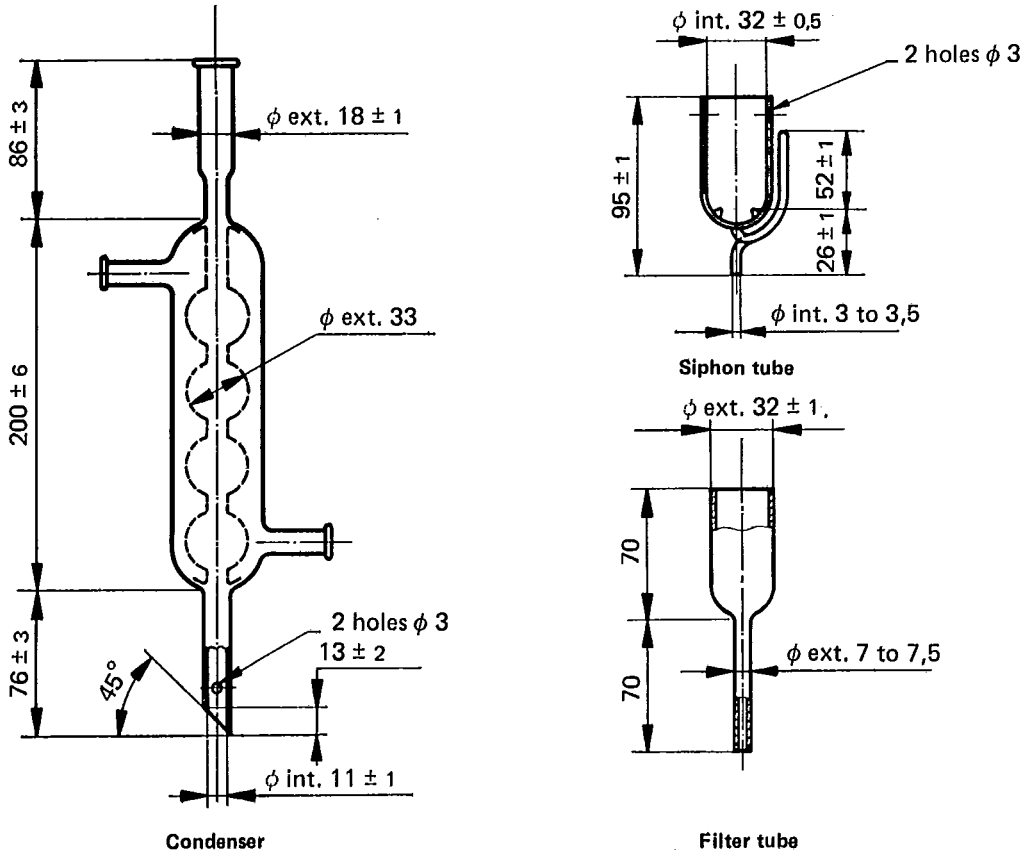


FIGURE 1 – Extraction apparatus for determination of matter insoluble in hot alcohol (method I)

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 or 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 R.3.1 of annex R) by rolling on paper, at least ten times, to ensure uniformity of that sample. Weigh, directly from the paper, 4.5 to 5,5 g of the sample, to an accuracy of 0,01 g.

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 hot alcohol from a wash-bottle, washing down the sides of the beaker.

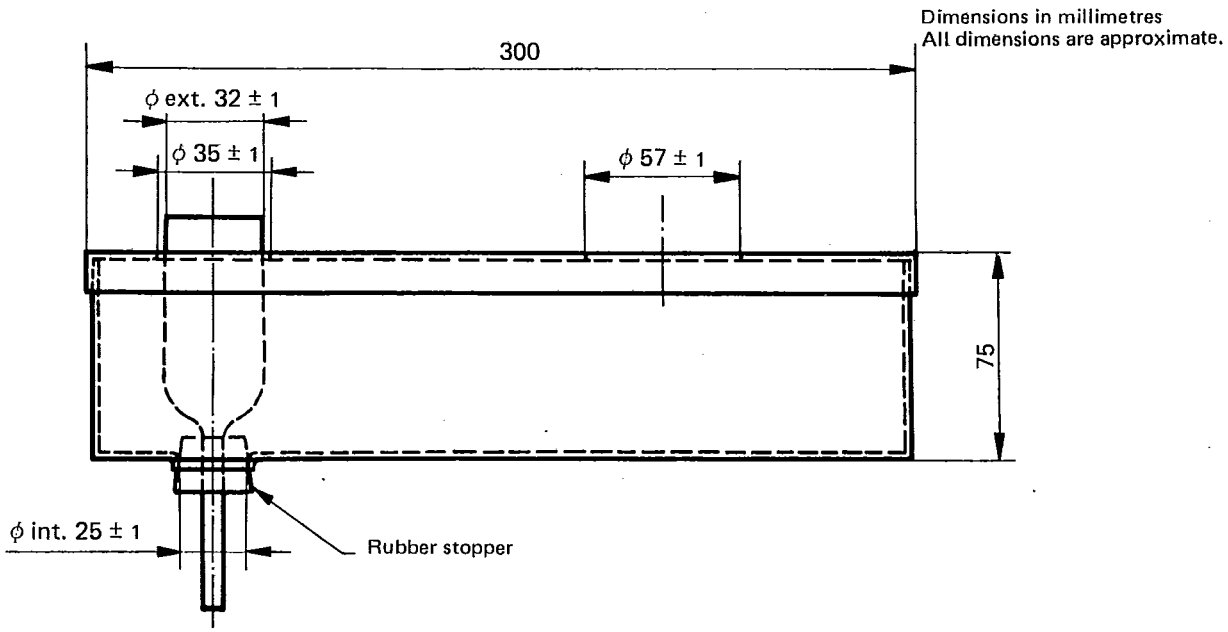


FIGURE 2 — **Hot water bath for determination of matter insoluble in hot alcohol (method I)**

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 the 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 samples 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 the 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 apparatus[1], 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).

Dimensions in millimetres
All dimensions are approximate.

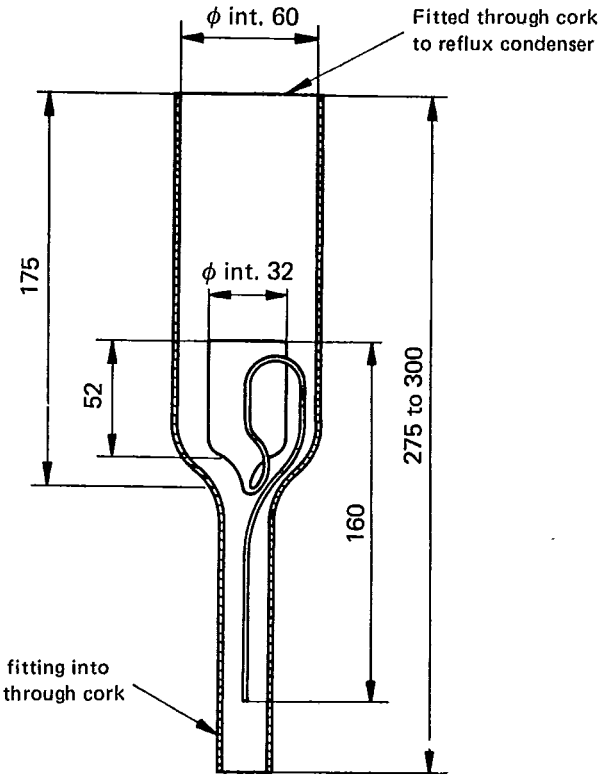


FIGURE 3 – **Siphon tube and adaptor**

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.

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.

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

A.2.3.5 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.2.3.5), 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.

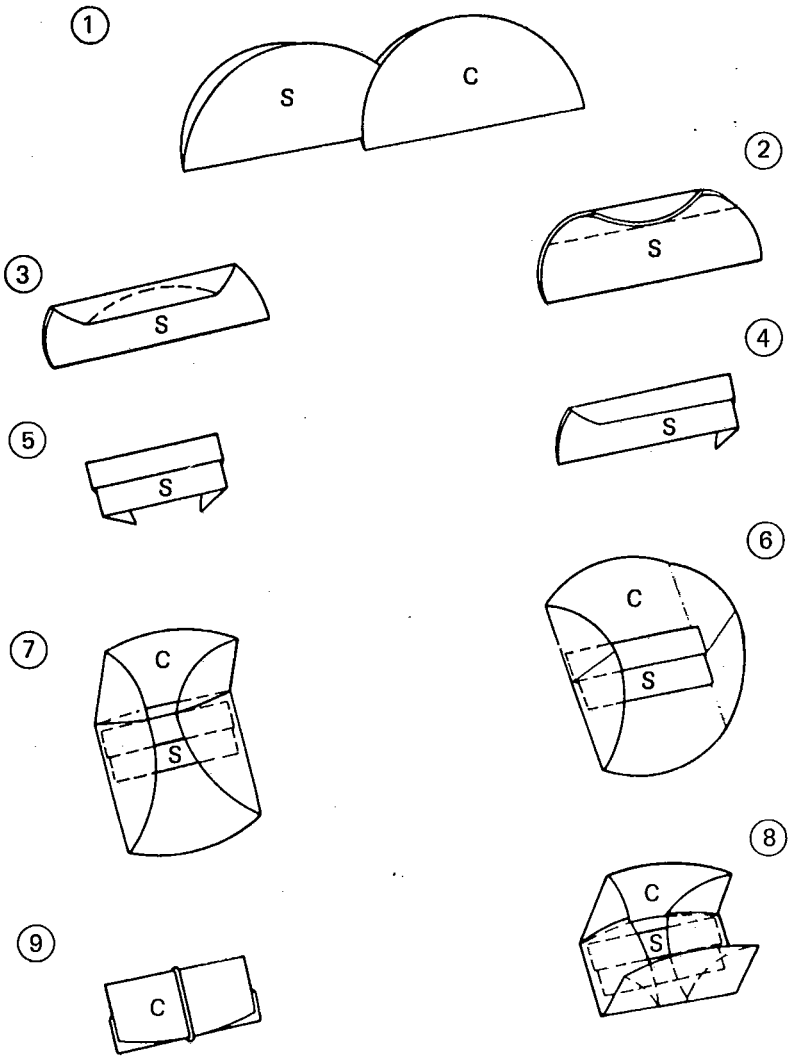


FIGURE 4 — **Folding of filter paper**

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 exact 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 ± 2 °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 ± 2 °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).

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 R.3.1 of annex R) 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 material 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 though 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.

ANNEX C
(See 5.3.1)

DETECTION OF ORPIMENT

C.1 PRINCIPLE

The presence of 0,5 % (*m/m*) 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 ethanol or in aqueous borax if the shellac is free from dirt.

C.2 REAGENTS

C.2.1 Alcohol, 95 % (*V/V*) ethanol or 95 % (*V/V*) denatured spirit.

C.2.2 Borax, 50 g/l solution of sodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$) in distilled water.

C.3 PROCEDURE

C.3.1 Prepare in a small conical flask a 200 g/l solution of the shellac in the alcohol (C.2.1) or borax solution (C.2.2). Allow the solution to stand, to allow any orpiment present to settle in a layer at the bottom. Examine from below.

C.3.2 Yellow particles of orpiment will be visible if it is present; 0,3 % (*m/m*) orpiment gives a continuous layer.

C.3.2.1 This method is most sensitive when the alcohol is cooled to 0 °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.

ANNEX D
(See 5.3.1 and 5.3.2)

DETERMINATION OF ARSENIC CONTENT

D.1 SCOPE

This annex specifies two methods for the determination of arsenic content, one for an appreciable amount of arsenic and the other for traces of arsenic.

D.2 METHOD I (Appreciable amount of arsenic)

D.2.1 Principle

Digestion of the shellac with nitric acid and sulphuric acid, treatment of the solution with a chloride-hydrazine-bromide mixture, distillation, and titration of the arsenic compound obtained by a suitable method.

D.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

D.2.2.1 Sodium hydrogen carbonate.

D.2.2.2 Nitric acid, ρ 1,42 g/ml.

D.2.2.3 Sulphuric acid, ρ 1,84 g/ml.

D.2.2.4 Hydrochloric acid, ρ 1,16 g/ml.

D.2.2.5 Nitric acid, dilute (3 + 7) solution.

D.2.2.6 Sodium hydroxide, approximately 2 N solution.

D.2.2.7 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.

D.2.2.8 Potassium bromate, 0,01 N standard volumetric solution.

D.2.2.9 Iodine, 0,01 N standard volumetric solution.

D.2.2.10 Methyl orange indicator, 0,4 g/l solution in 20 % (*V*/*V*) ethanol.

D.2.2.11 Starch solution.

Make a paste of 0,2 g of soluble starch in cold water and pour into 100 ml of boiling water. Boil for 5 min, cool and bottle.

Prepare a fresh solution every 2 or 3 days.

D.2.3 Apparatus

Ordinary laboratory apparatus, and

D.2.3.1 Kjeldahl flask, made of heat-resistant glass or silica, of capacity 100 or 200 ml.

D.2.3.2 Kjeldahl distillation apparatus, as shown in figure 5.

D.2.4 Test portion

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

D.2.5 Determination

D.2.5.1 Place the test portion (D.2.4) in the Kjeldahl flask (D.2.3.1), add 10 ml of the dilute nitric acid solution (D.2.2.5) and heat the mixture until any initial vigorous reaction subsides and ceases. Cool and add gradually 10 ml of the sulphuric acid (D.2.2.3) at such a rate as to prevent excessive frothing or heating (10 mm are usually required) and continue heating. Add to the hot solution 5 ml of the nitric acid (D.2.2.2) in small portions, and boil until colourless. If necessary, add the nitric acid in further small portions at a time. Note for the purpose of the blank test the total volume of nitric acid added. (The digestion usually takes about 30 min.) Cool, dilute with 50 ml of water and transfer to the flask of the distillation apparatus (D.2.3.2). 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 ml of water. Cool the liquid well, add 5 g of the chloride-hydrazine-bromide mixture (D.2.2.7), followed rapidly by 10 ml of the hydrochloric acid (D.2.2.4).

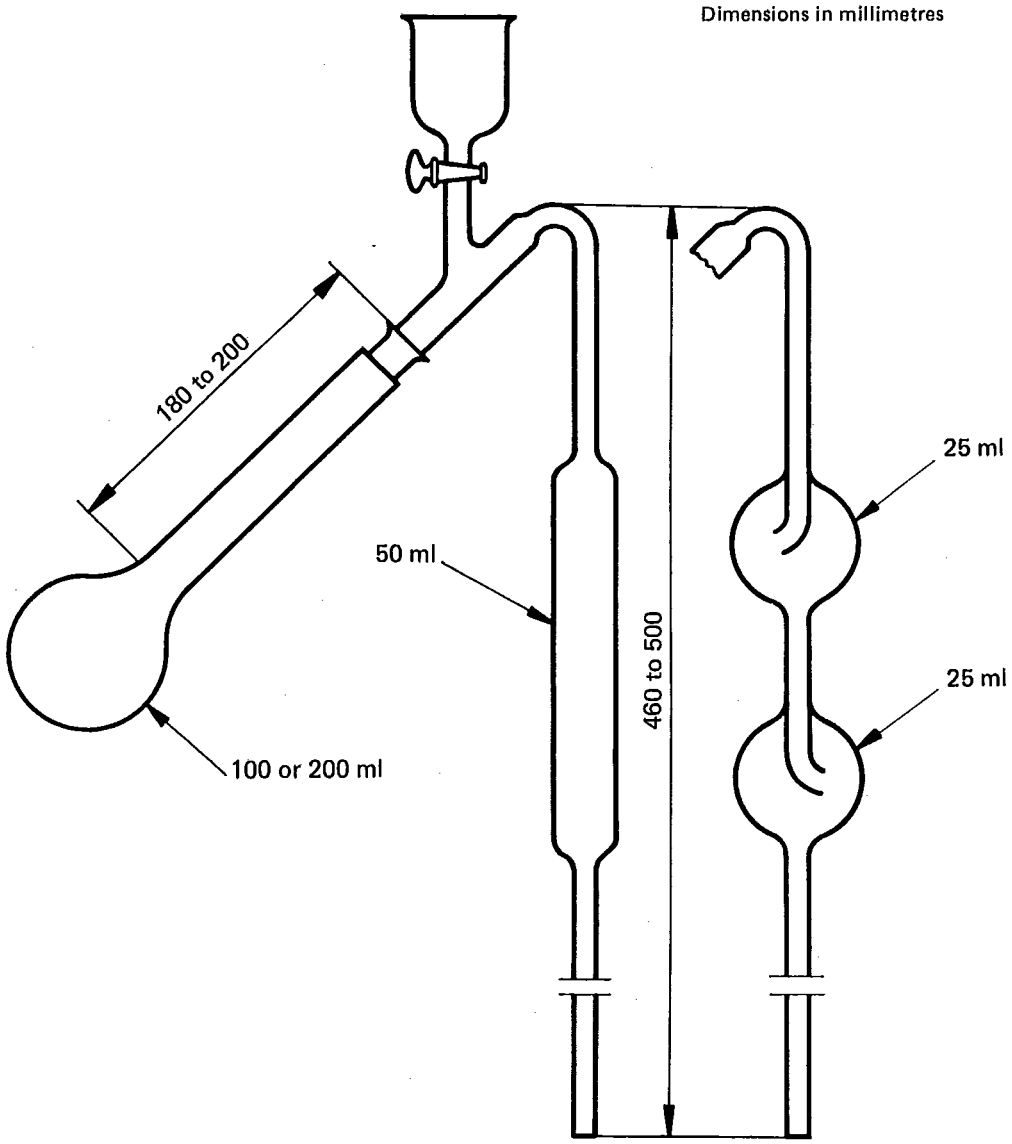


FIGURE 5 – Kjeldahl distillation apparatus for use in the determination of arsenic content (method I)

D.2.5.2 Fit the condenser quickly and distil the liquid into 20 ml of water, the exit tube dipping below the surface of the liquid; cool in ice until about 5 min after the condenser is full of steam. Dilute the distillate to 100 ml, add a few drops of the methyl orange indicator solution (D.2.2.10), heat the solution to 80 °C, and titrate with the standard volumetric potassium bromate solution (D.2.2.8), or, alternatively, nearly neutralize the distillate with the sodium hydroxide solution (D.2.2.6), then add 3 g excess of the sodium hydrogen carbonate (D.2.2.1) and titrate the solution with the standard volumetric iodine solution (D.2.2.9), using the starch solution (D.2.2.11) as indicator.

D.2.5.3 Make sure that no solid material comes in contact with the ground-in portion of the flask.

D.2.6 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same reagents, but omitting the test portion.

D.2.7 Expression of results

The arsenic (As) content is given, expressed as a percentage by mass of arsenic(III) sulphide (As_2S_3), by the formula

$$\frac{6{,}15\ V\ T}{m}$$

where

V is the volume, in millilitres, of the standard volumetric potassium bromate solution (D.2.2.8) or of the standard volumetric iodine solution (D.2.2.9) used for the determination (D.2.5), after applying correction for the blank test;

T is the normality of the standard volumetric potassium bromate solution (D.2.2.8) or of the standard volumetric iodine soluton (D.2.2.9);

m is the mass, in grams, of the test portion (D.2.4).

D.3 METHOD II (Traces of arsenic)

D.3.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. All the reagents, with the exception of (D.3.1.11) and (D.3.1.12), shall be free from traces of arsenic.

D.3.1.1 Potassium iodide, crystals or in the form of powder.

D.3.1.2 Zinc, granulated, complying with the following test :

Take 50 ml of water, 10 ml of the stannated hydrochloric acid solution (D.3.1.8) and 0,1 ml of the dilute solution of arsenic (D.3.1.12) in the wide-mouth bottle (D.3.2.1). Add 1 g of the potassium iodide (D.3.1.1) and 10 ml of zinc. Quickly place the prepared glass tube (D.3.2.2) in position. Allow the reaction to continue for 1 h. A faint but distinct yellow stain shall be produced on the mercury(II) chloride paper (D.3.1.13).

D.3.1.3 Nitric acid, ρ 1,42 g/ml.

D.3.1.4 Sulphuric acid, ρ 1,84 g/ml.

D.3.1.5 Hydrochloric acid, ρ 1,16 g/ml.

D.3.1.6 Nitric acid, dilute (3 + 7) solution.

D.3.1.7 Tint(II) 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.

D.3.1.8 Stannated hydrochloric acid solution.

Mix together 1 ml of the tin(II) chloride solution (D.3.1.7) and 100 ml of the hydrochloric acid (D.3.1.5).

D.3.1.9 Lead acetate, 100 g/l solution in distilled water, recently boiled.

D.3.1.10 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.

D.3.1.11 Arsenic, concentrated solution.

Dissolve 0,132 g of arsenic trioxide in 50 ml of the hydrochloric acid (D.3.1.5) and add sufficient water to produce 100 ml.

D.3.1.12 Arsenic, dilute solution.

Dilute 1 ml of the concentrated solution of arsenic (D.3.1.11) with sufficient water to produce 100 ml.

1 ml of this solution contains 0,01 mg of arsenic (or 0,013 2 mg of As_2O_3).

D.3.1.13 Mercury(II) chloride paper, consisting of smooth white filter paper of width not less than 25 mm, soaked in a saturated solution of mercury(II) chloride in water, pressed to remove superfluous solution, and dried at 60 °C in the dark. The grade of the filter paper shall be such that the grammage is between 65 and 120 g/m^2; the thickness, in millimetres, of 400 papers shall be approximately equal, numerically, to the grammage, in grams per square metre. Mercury(II) chloride paper should be stored in a stoppered bottle in the dark. Papers which have been

exposed to sunlight or to ammonia gas should not be used as they give a lighter coloured stain or no stain at all when employed in the quantitative test for arsenic.

D.3.1.14 Methyl orange indicator, 0,4 g/l solution in 20 % (*V/V*) ethanol.

D.3.2 Apparatus

The following apparatus assembled as shown in figure 6 is required.

D.3.2.1 Wide-mouth bottle, of capacity 120 ml.

D.3.2.2 Glass tube, made from ordinary glass tubing, and having a total length of 200 mm, an internal diameter of exactly 6,5 mm and an external diameter of about 8 mm. It shall be drawn out at one end to a diameter of about 1 mm and a hole not less than 2 mm in diameter shall be blown in the side of the tube, near the constricted part. The upper end of the tube shall be cut off square and shall be either rounded off slightly or ground smooth.

D.3.2.3 Rubber bungs, three.

One shall fit exactly into the mouth of the wide-mouth bottle and shall have a hole bored centrally to take the tube from its constricted end. Each of the other two rubber bungs (about 25 mm x 25 mm) shall have a hole, of diameter 6,5 mm, bored centrally, and shall be fitted with a rubber band or spring clip for holding them tightly together.

D.3.2.4 Preparation of the glass tube

Moisten a small quantity of cotton wool with the lead acetate solution (D.3.1.9) and then dry it in a dust-free atmosphere. Lightly pack the glass tube (D.3.2.2) 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 (D.3.2.3), 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 the mercury(II) chloride paper (D.3.1.11) flat on the top of the bung. Place the other bung over this with its larger end in contact with the piece of mercury(II) chloride paper. Fasten the two bungs by means of the rubber band or the spring clip, in such a manner that the bores of the two bungs (or the upper bung and the glass tube) form a continuous tube of diameter 6,5 mm interrupted by a diaphragm of mercury(II) chloride paper.

Any other method of attaching the mercury(II) chloride paper may be used, provided that

a) the whole of the evolved gas passes through the paper;

b) the portion of the paper in contact with the gas is a circle of diameter 6,5 mm;

c) the paper is protected from sunlight during the test.

Dimensions in millimetres

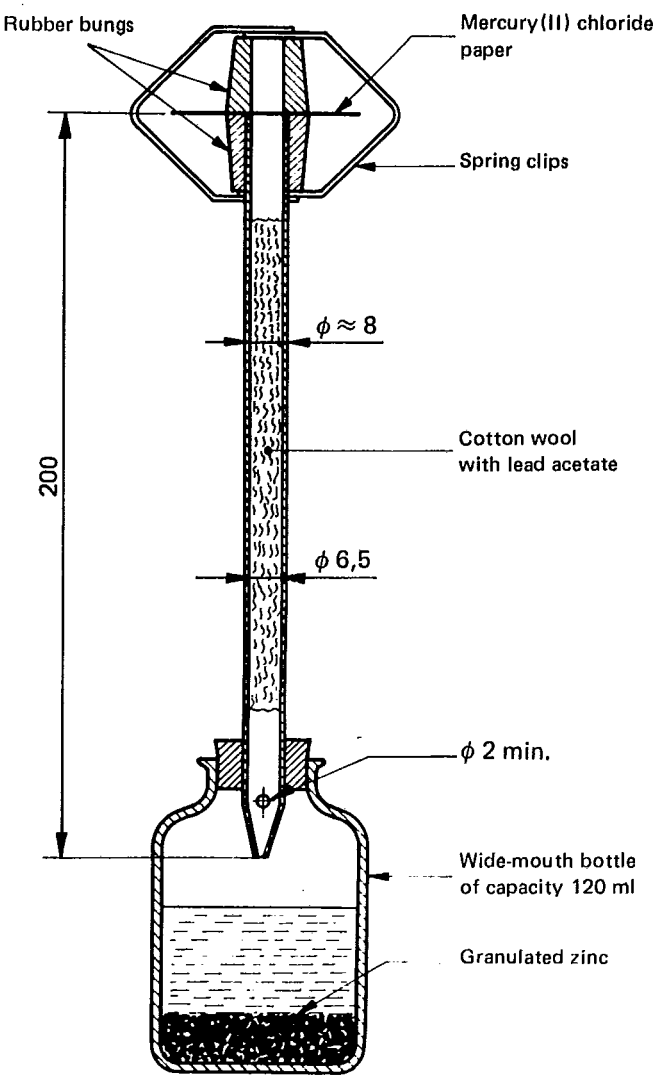


FIGURE 6 — **Apparatus for the determination of arsenic content (method II)**

D.3.3 Procedure

D.3.3.1 Treat 5 g of the "test sample" exactly in the manner prescribed in D.2.5.1 and then proceed as follows.

D.3.3.2 Fit the condenser quickly and distil the liquid into a mixture of 10 ml of water and 2 ml of the nitric acid (D.3.1.3). Then evaporate the distillate to dryness on a water bath and evaporate the residue twice to dryness with 5 ml of water to remove any nitric acid. Dissolve the final residue by warming in 3 ml of the sulphuric acid (D.3.1.4), cool and dilute with water. Transfer the whole of the solution to the wide-mouth bottle (D.3.2.1), add 15 ml of the standard hydrochloric acid solution (D.3.1.8) and 1 g of the potassium iodide (D.3.1.1). Then add 10 g of the zinc (D.3.1.2). Quickly place the prepared glass tube (D.3.2.2) in position. Allow the reaction to continue for 40 min. Remove the piece of mercury(II) 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 D.3.3.4. If the stain in this test exceeds that equivalent to 0,02 mg of arsenic trioxide (As_2O_3), make up the solution to a known volume with dilute sulphuric acid (1 : 8) and take an aliquot portion to produce a stain suitable for matching.

The reaction may be accelerated by placing the apparatus on a warm surface, care being taken that the mercury(II) 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, rinser with water, and dried between successive tests.

D.3.3.3 Compare the stains with freshly prepared standard stains immediately at the completion of the test.

D.3.3.4 Prepare the standard stains as follows :

Mix together 50 ml of water, 10 ml of the stannated hydrochloric acid solution and appropriate volumes of the dilute solution of arsenic (D.3.1.12). Treat the resulting solutions as described under D.3.3.2 to prepare the standard stains.

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

D.3.4 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents, but omitting the test portion. No visible stain should be produced on the mercury(II) chloride paper.

D.3.5 Expression of results

Express the arsenic content of the shellac sample as parts of arsenic (As) or arsenic trioxide (As_2O_3) per million parts of shellac.

ANNEX E
(See 6.1)

DETERMINATION OF VOLATILE MATTER (MOISTURE) CONTENT

E.1 PRINCIPLE

Heating of a weighed test portion of the "sample as received" at 40 ± 2 °C for 4 h followed by storage over concentrated sulphuric acid *in vacuo* for 18 h.

E.2 APPARATUS

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

E.2.2 Oven, well ventilated, capable of being controlled at 40 ± 2 °C.

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

E.3 PROCEDURE

E.3.1 Use the "sample as received" ground to specified size (see R.3.2 of annex E) for this test. Weigh the clean, dry, flat-bottomed dish with ground glass cover (E.2.1). 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 mass of the test portion.

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

E.4 EXPRESSION OF RESULTS

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

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

where

m_0 is the mass, in grams, of the test portion before drying;

m_1 is the mass, in grams, of the test portion after drying.

ANNEX F
(See 6.2.1)

DETERMINATION OF COLOUR INDEX

F.1 PRINCIPLE

Comparison of the colour of a standard solution of iodine with a solution of the shellac in ethanol, by diluting the latter solution progressively until a close match is obtained.

F.2 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water of equivalent purity.

F.2.1 Alcohol, 95 % (*V*/*V*) rectified spirit; or 95 % (*V*/*V*) denatured spirit, provided that it is colourless.

F.2.2 Iodine, 0,005 N standard solution, prepared by introducing 5 ml of 0,1 N solution of iodine in potassium iodide, from a burette, into a 100 ml one-mark volumetric flask and making up to 100 ml with water. This solution corresponds to colour index 5. Shake the solution before use.

F.3 APPARATUS

Ordinary laboratory apparatus, and

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

F.3.2 Test tubes, thin-walled, measuring 200 mm × 13 mm.

F.4 PROCEDURE

F.4.1 Add 10,0 g of the "test sample" (see R.3.1 in annex R) to 100 ml of the alcohol (F.2.1) contained in the stoppered flask (F.3.1) and stir for 30 min at 27 ± 2 °C until dissolution is complete. 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.

NOTE — A temperature of 20 ± 2 °C or 23 ± 2 °C may be used in temperate climates (see ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*).

F.4.2 Pipette 5 ml of the filtered shellac solution into a thin-walled test tube (F.3.2). Take 5 ml of the appropriate standard iodine solution (F.2.2), in another test tube (F.3.2) for matching. Compare the colour of the two solutions, holding the test tubes against the light with a piece of moistened filter paper or opal glass interposed in between the light source and the test tubes. Add the alcohol from the burette to the shellac solution with shaking until the colour is the same as that of the standard iodine solution. Note the volume of alcohol added.

F.4.2.1 It will be found advantageous to use as standard type of light source and a viewing cabinet to cut off extraneous light.

F.5 EXPRESSION OF RESULTS

F.5.1 The colour index is given by the formula

$$V + 5$$

where V is the volume, in millilitres, of alcohol added as in F.4.2, or the total volume, in millilitres, of the shellac solution after dilution.

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

ANNEX G
(See 6.3)

DETERMINATION OF WAX

G.1 PRINCIPLE

Dissolution of a test portion in a hot solution of sodium carbonate, separation of the wax by filtering, extraction with chloroform and weighing after drying.

G.2 REAGENTS

During the analysis use only reagents of recongized analytical grade and only distilled water or water of equivalent purity.

G.2.1 Sodium carbonate, anhydrous.

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

G.2.3 Filter-aid, previously extracted with chloroform and dried before use.

G.3 APPARATUS

Ordinary laboratory apparatus, and

G.3.1 Beaker, tall form, of capacity 200 ml.

G.3.2 Water bath.

G.3.3 Filter paper, double acid-washed, retentive, low ash, of diameter 12,5 cm.[1)]

G.3.4 Filter paper, fat free.

G.3.5 Extraction cartridge, of fat-free paper, 25 mm x 60 mm.

G.3.6 Suitable continuous extraction apparatus, such as the standard apparatus for the determination of matter insoluble in hot alcohol (see annex A, method I).

G.3.7 Electric oven, capable of being maintained at 40 ± 2 °C.

G.3.8 Electric oven, capable of being maintained at 100 ± 2 °C.

G.4 PROCEDURE

G.4.1 Test portion

Weigh 9,5 to 10,5 g of the "test sample" (see R.3.1 of annex R) to an accuracy of 0,01 g and dissolve in 150 ml of hot water containing 2,5 g of the sodium carbonate (G.2.1) in the beaker (G.3.1).

G.4.2 Determination

G.4.2.1 Immerse the beaker containing the test portion (G.4.1) in a boiling water bath (G.3.2) and stir until the lac is dissolved. Then cover the beaker with a watch-glass and allow it to remain in the bath for 2 to 3 h more, without agitation. Remove the beaker from the bath and place it in cold water. The wax will now come to the 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 filter paper (G.3.3), by gravity, or use a Buchner funnel with suction.

In the latter case it is necessary to embed the filter paper in the Buchner funnel with filter-aid, by mixing 1 g of the filter-aid (G.2.3) with water and pouring this mixture onto the paper with the suction on. Filtration by this method is also further aided by stirring 0,5 g of the filter-aid into the shellac solution before starting the filtration.

G.4.2.2 If the filtration is carried out under gravity alone, then, after the filtration is completed and all soluble shellac has been washed out of the paper with water, remove the paper from the funnel and, without further folding it, place it in the beaker, resting it against the stirring rod so that the edge of the paper remains level with the top edge of the beaker. Keep the beaker containing the paper in the oven (G.3.7), maintained at a temperature not exceeding 40 ± 2 °C, for several hours, to remove most of the water.

Then remove the paper from the beaker, wrap carefully in a large piece of clean fat-free filter paper (G.3.4), bind with fine wire and place it in an extraction cartridge (G.3.5) which has been previously extracted with the hot chloroform (G.2.2). Place the cartridge containg the wax and the filter paper into the extraction apparatus (G.3.6) and pour into the beaker, which previously contained the filter paper and wax, a portion of the chloroform to be used for the extraction.

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

Bring the solvent to the boil and pour it through the extraction cartridge, collecting it in the extraction flask to be used. Repeat this operation twice more, so as to remove the whole of the residual wax from the beaker. Then connect up the apparatus and extract for at least 2 h. Distil off most of the solvent, transfer the residue to the tared glass basin, wash the extraction flask three times with small lots of 5 ml of chloroform and pour into the basin. Evaporate to dryness, and then dry the residue in the oven (G.3.8), maintained at 100 ± 2 °C, for 30 min, cool in a desiccator and weigh. Repeat the operations of drying for 30 min and weighing until the loss in mass between two successive weighings does not exceed 0,002 g. Use the lowest mass in the calculation.

G.4.2.3 If the Buchner funnel is used, then, after the filtration has been completed and the paper has been well washed with water to take out all soluble shellac, leave the vacuum on for a few minutes so as to suck out as much water as possible. It will then be possible to insert a thin spatula under the edge of the paper and remove it from the funnel, without leaving more than traces of the filter-aid adhering to the funnel walls. Remove such, traces by wiping with pieces of alcohol-moistened paper, combine these with the main paper and wrap the whole, while still damp, in a large piece of filter paper and bind firmly with fine wire.

Dry in the oven (G.3.7), maintained at 40 ± 2 °C. When dry, place it in the extraction cartridge which has been previously extracted with chloroform. Transfer the cartridge and wax to the continuous extraction apparatus, and extract for 2 h with chloroform.

Distil off most of the solvent, transfer the residue to the tared glass basin, wash the extraction flask three times with small lots of 5 ml of the chloroform and pour into the basin. Evaporate to dryness, and then dry the residue in the oven (G.3.8), maintained at 100 ± 2 °C for 30 min, cool in a desiccator and weigh. Repeat the operations of drying for 30 min and weighing, until the loss in mass between two successive weighings does not exceed 0,002 g. Use the lowest mass in the calculation.

G.5 EXPRESSION OF RESULTS

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

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

where

m_0 is the mass, in grams, of the test portion;

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

ANNEX H
(See 6.4)

DETERMINATION OF ASH

H.1 PROCEDURE

H.1.1 Weigh 3 to 5 g of the "test sample" (see R.3.1 of annex R) 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 0,001 g. Use a muffle furnace, if available.

H.1.2 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 0,001 g. Use the lowest mass in the calculation.

H.2 CALCULATION

The ash 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;

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

ANNEX J
(See 6.5)

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

J.1 PRINCIPLE

Digestion of a test portion with water, making up of the solution to a known volume and filtration. Determination of the mass in solution by evaporation of aliquot portion of the filtrate to constant mass and calculation for the whole solution.

J.2 APPARATUS

Ordinary laboratory apparatus, and

J.2.1 Test sieve, having nominal aperture size 0,25 mm.

J.2.2 Filter paper, Whatman No. 1 or equivalent.

J.3 PROCEDURE

J.3.1 Finely grind a sufficient quantity of the "test sample" (See R.3.1 of annex R) to pass a sieve having a nominal aperture size of about 0,25 mm. Weigh 20 to 25 g of the powdered sample to an accuracy of 0,1 g and transfer to a beaker. Add 200 ml of distilled water and stir thoroughly. Cover the beaker with a watch-glass and allow it to stand at a temperature of 27 ± 2 °C for 4 h, with occasional stirring.

NOTE – A temperature of 20 ± 2 °C or 23 ± 2 °C may be used in temperate climates (see ISO 554, *Standard atmospheres for conditioning and/or testing – Specifications*).

J.3.2 Filter by gravity or suction into a 250 ml one-mark volumetric flask. Wash the residual shellac and the filter with distilled water and dilute to the mark. Transfer a measured volume of the filtrate into a weighed evaporating dish and evaporate to dryness. Dry the residue to constant mass (within 0,002 g) in an oven maintained at 100 ± 2 °C.

J.3.3 Test the acidity and alkalinity of another aliquot of the solution with methyl red and bromothymol blue respectively. It shall be neutral to both.

J.4 CALCULATION

J.4.1 The matter soluble in water is given, expressed as a percentage by mass, by the formula

$$\frac{m_1}{V m_0} \times 2{,}5 \times 10^4$$

where

m_0 is the mass, in grams, of the test portion;

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

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

J.4.2 Report the reaction of the aqueous extract to methyl red and bromothymol blue.

ANNEX K
(See 6.6)

FLOW TEST

K.1 PRINCIPLE

Melting of a test portion in either a graduated test tube or a plain test tube. Tilting of the tube, maintained at 100 ± 1 °C, to an angle of 15°, in order to permit the shellac to flow down the tube and measurement of either the time required for the shellac to flow to the various graduations along the test tube or the total distance the shellac flows along the test tube in a specified time.

K.2 TEST PORTIONS

K.2.1 Test two portions for each of the methods.

K.2.2 For each test portion, accurately weigh 2,00 g of the test sample (see R.3.1 of annex R). Spread it out in a shallow vessel and place in a desiccator over a saturated solution of sodium dichromate with an excess of the solid salt and leave in this atmosphere at room temperature for at least 24 h. Test the test portions immediately upon removal from the desiccator.

K.3 METHOD A

K.3.1 Apparatus (see figures 7 and 8)

K.3.1.1 Oil bath, consisting of a glass tank A heated by an electric immersion heater and filled with a suitable medium (glycerol or a clear oil having a kinematic viscosity of approximately 31 mm^2/s).

K.3.1.2 Mechanical stirring device B, for maintaining a uniformly distributed temperature.

K.3.1.3 Thermometers C, of the partial-immersion type, graduated in degrees Celsius. A thermometer having a range of −7 °C to +300 °C, with subdivisions at every 1 °C, longer graduation at every 5 °C and numbered graduations at each multiple of 10 °C, is considered satisfactory for the purpose.

K.3.1.4 Two test tubes D, for holding the sample, preferably made of heat-resistant glass, red lined, of length 125 mm, outside diameter 25 mm and wall thickness 1,5 mm, graduated in 5 mm divisions beginning 10 mm from the lowest tip and extending upwards to 100 mm, every 10 mm line being numbered. Stopper the test tubes with tightly fitting corks, with small breather tubes E.

K.3.1.5 Testing fixture F, for holding the glass tubes in the correct position, consisting of two brass disks supported as a pendulum as indicated in figure 7, the disks being free to turn on the supporting shaft H. The disks are held in the desired position by the pin I and the tubes are supported on narrow V-blocks J and held in position by coil sping K attached to the disks.

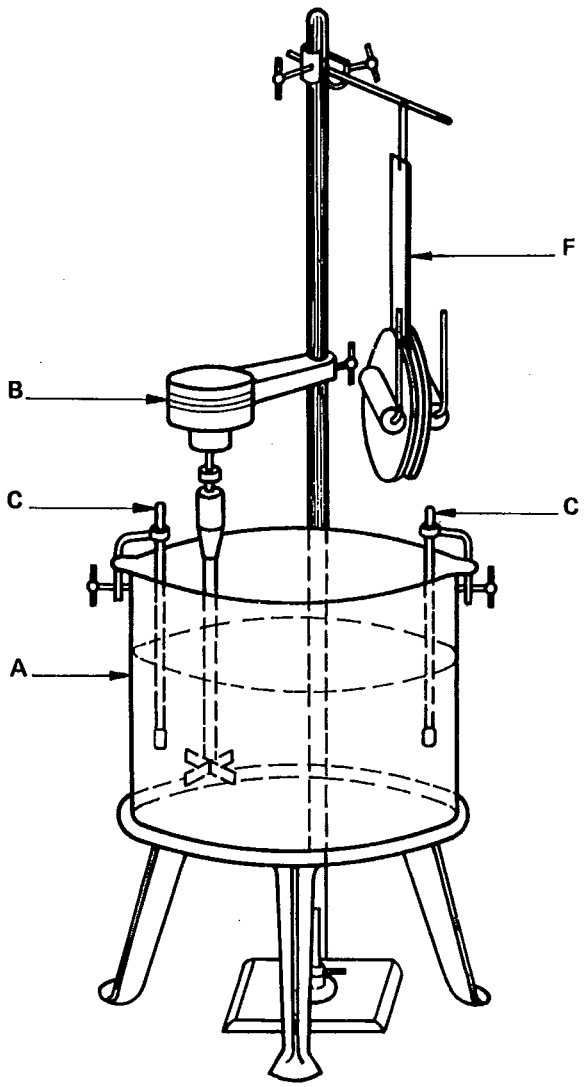


FIGURE 7 – **Apparatus for flow test (the test tubes are held in an inclined position on the testing fixture F, for immersion in the oil bath)**

K.3.2 Procedure

K.3.2.1 Place the two test portions of shellac, each weighing 2,00 g, in separate glass test tubes (K.3.1.4) (see figure 8, letter L), taking care that the top surface of the test portion in each tube is level and at right angles to the walls of the tube and that none of the powdered shellac adheres to the walls. Read the top level of the dry shellac in each tube on the millimetre graduated scale. Clamp the tubes containing the specimens in place in the testing fixture (K.3.1.5) (see figure 8). Insert the testing fixture, with the glass test tubes in a vertical position, in the oil bath (K.3.1.1), maintained at 100 ± 1 °C. Allow the specimen to melt for 3 min.

K.3.2.2 At the end of the 3 min melting period, place each test tube at an angle of 15° to the horizontal, with the corked ends down (see figure 7), and with the breather tube extending above the level of the oil bath. Make the change from the vertical to the flow position as quickly as possible. With the oil bath maintained at 100 ± 1 °C, record the total time required for the shellac in each tube to flow from the initial level of the shellac to each 10 mm marking along the tube. Discontinue the test in each tube when the flow is 90 mm or the total time is 20 min.

K.3.3 Test report

The test report shall include the following particulars :

a) the time required for each 10 mm distance of flow for each specimen,

b) a curve showing the data reported in a) above, with time plotted as abscissa and distance of flow as ordinate;

Dimensions in millimetres

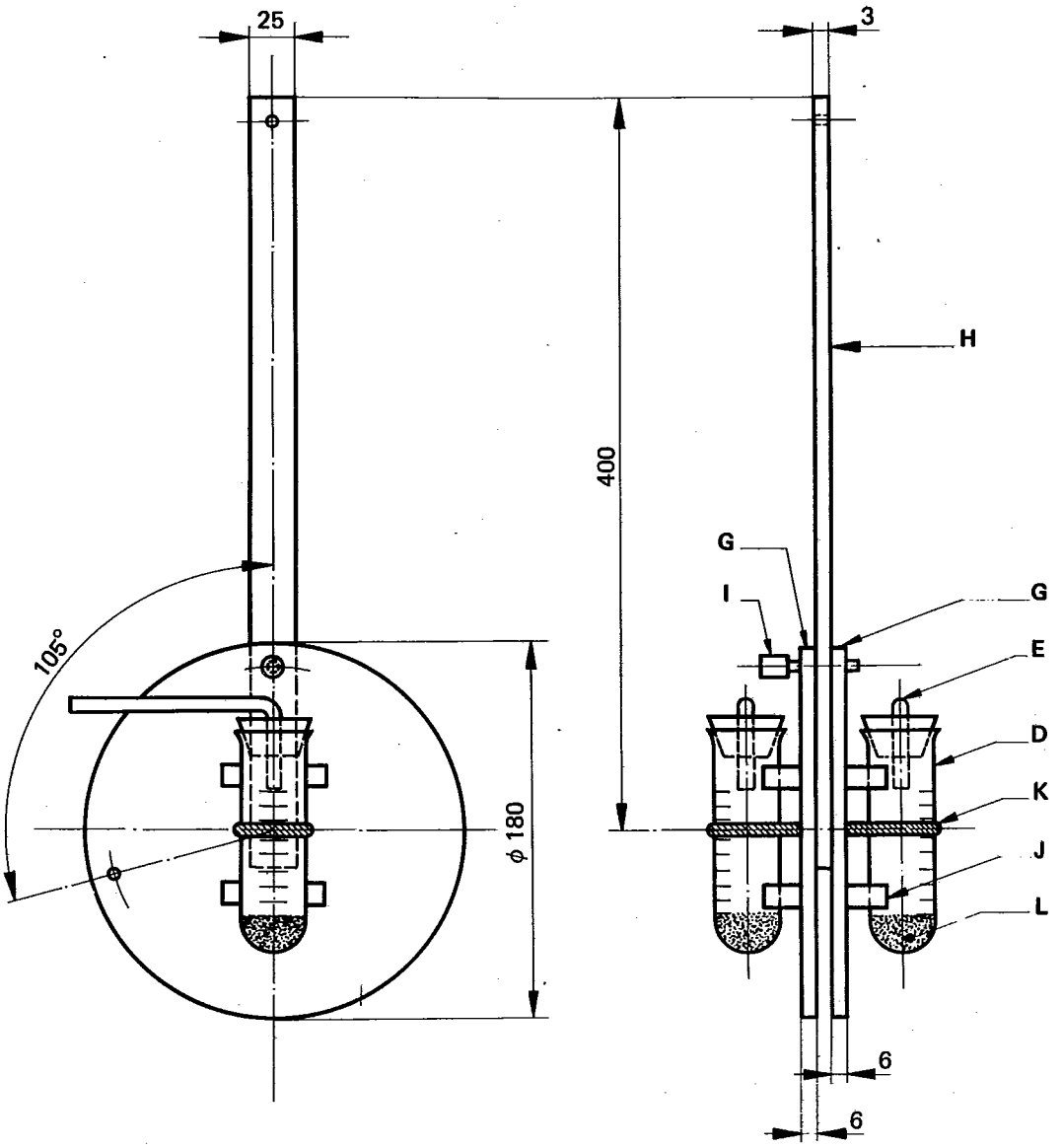


FIGURE 8 — **Essential features of the testing fixture F (the test tubes are shown in the vertical position)**

c) the angle of the test tubes during the flowing period;

d) the atmospheric temperature and humidity of the laboratory.

K.4 METHOD B

K.4.1 Apparatus

Any apparatus that will provide for accurately maintaining the required test temperature and the required positions of the test tubes may be used, but a suitable apparatus (see figures 7 and 8) consists of the following items :

K.4.1.1 Oil bath, as in K.3.1.1, except that a metal or any other suitable container may be used and the heating may be by a Bunsen burner or an electric immersion heater.

K.4.1.2 Thermometers, as in K.3.1.3.

K.4.1.3 Two test tubes D, plain, of length 125 mm and outside diameter 25 mm, made of heat-resistant glass, and stoppered with tightly fitting corks through which small breather tubes E are extended.

K.4.1.4 Testing fixture F, as in K.3.1.5.

K.4.2 Procedure

K.4.2.1 Prepare the apparatus as specified in K.3.2.1.

K.4.2.2 At the end of the 3 min melting period, place the test tubes at an angle of 15° to the horizontal, with the corked ends down (see figure 7), and with the breather tubes extending above the level of the oil bath. Make the change from the vertical position to the flow position as quickly as possible. With the oil bath maintained at the test temperature of 100 ± 1 °C, allow the test tubes to remain in the bath in this position for eactly 12 min. Remove the test tubes immediately from the bath, place in a vertical position, cool, wipe, and measure the flow of shellac in each tube by reading the distance between the initial point and the end of the flow tongue. Disregard the "feather" caused by separation of wax from the shellac, at the very tip of the tongue.

NOTE – The "feather" can be distinguished from the main body of shellac as it is always of a different colour.

K.4.3 Test report

The test report shall include the following particulars :

a) the flow, expressed in millimetres, for each specimen;

b) the average of the values in a) above;

c) the angle of the test tubes during the flowing period;

d) the atmospheric temperature and humidity of the laboratory.

ANNEX L
(See 6.7)

HEAT POLYMERIZATION TEST

L.1 PRINCIPLE

Heating of a test portion under specified conditions in a test tube and observing the time required for it to attain a rubbery state as indicated by the "spring-back" of a glass rod when it is twisted through a full circle.

L.2 APPARATUS (see figure 9)

L.2.1 Flat-bottom dish, of diameter 100 mm.

L.2.2 Flask, with stopper.

L.2.3 Test tube, of length 150 mm and inner diameter 25 mm.

L.2.4 Suitable rack, for holding test tubes in a vertical position in the oil bath (L.2.7).

L.2.5 Glass rod, smooth, of length about 210 mm and diameter 10 mm, having a smaller glass rod, of length 20 mm long and diameter 5 mm attached to it at right angles about 180 mm from bottom end of the main rod. The bottom end of the main rod and the free end of the projection rod shall be smoothly rounded to a hemisphere of the respective rod diameters.

L.2.6 Electric oven, capable of being maintained at 40 ± 1 °C.

L.2.7 Oil bath, provided with means for continuous stirring and of such construction that it can be maintained within ± 1 °C of the specified test temperature which, unless otherwise agreed, shall be 150 °C.

L.2.8 Thermometer, as in K.3.1.3.

L.3 CONDITIONING

Before testing, roll about 12 g of the "test sample" (see R.3.1 of annex R) on a clean filter paper and then carefully dry it, by spreading it out in the flat-bottom dish (L.2.1) and placing the dish in the oven (L.2.6), maintained at 40 ± 1 °C for approximately 16 h. Immediately after removing the dish from the oven, transfer the sample to the clean, dry flask (L.2.2). Stopper the flask tightly and allow the sample of shellac to cool. Do not unstopper the flask except when removing a specimen for test.

L.4 TEST PORTIONS

Test two test portions each of mass 5,0 g for each sample of shellac.

L.5 PROCEDURE

Weigh 5,0 g of the conditioned sample of shellac and transfer to the test tube (L.2.3) along with the glass rod (L.2.5). Insert the tube in the test rack (L.2.4) so that it is held in a vertical position, immersed to a depth of about 100 mm in the oil bath (L.2.7), maintained thermostatically at the specified temperature within ± 1 °C. Record the time when the test tube enters the oil bath. Holding the top of the test tube with one hand, stir the shellac gently with the other hand for the first 3 min of the test, with a rotatory motion of the glass rod, so that the rod moves near the wall of the test tube and the shellac melts in as short a time as possible. At the end of each minute thereafter, turn the rod through a full circle, and standing at arm's length away from the rod, take a line of sight so that when viewed with one eye, one edge of the main rod is in line with one edge of the top of the projection rod (positon A in figure 9). Immediately release the rod and allow it to spring back, keeping the line of sight fixed. If the rod springs back so far as to bring the other edge of the main rod in line with the opposite edge of the top of the projection rod (positon B in figure 9), assume that the end-point has been reached. This amount of angular movement corresponds to about 12° 30′ of spring-back which is arbitrarily fixed for the purpose of controlled observations as indicating the end-point for noting the time for the heat polymerization test. Note the time when the end-point is reached. Note the oil temperature every minute during the test to ensure that it does not vary beyond the specified limit of ± 1 °C.

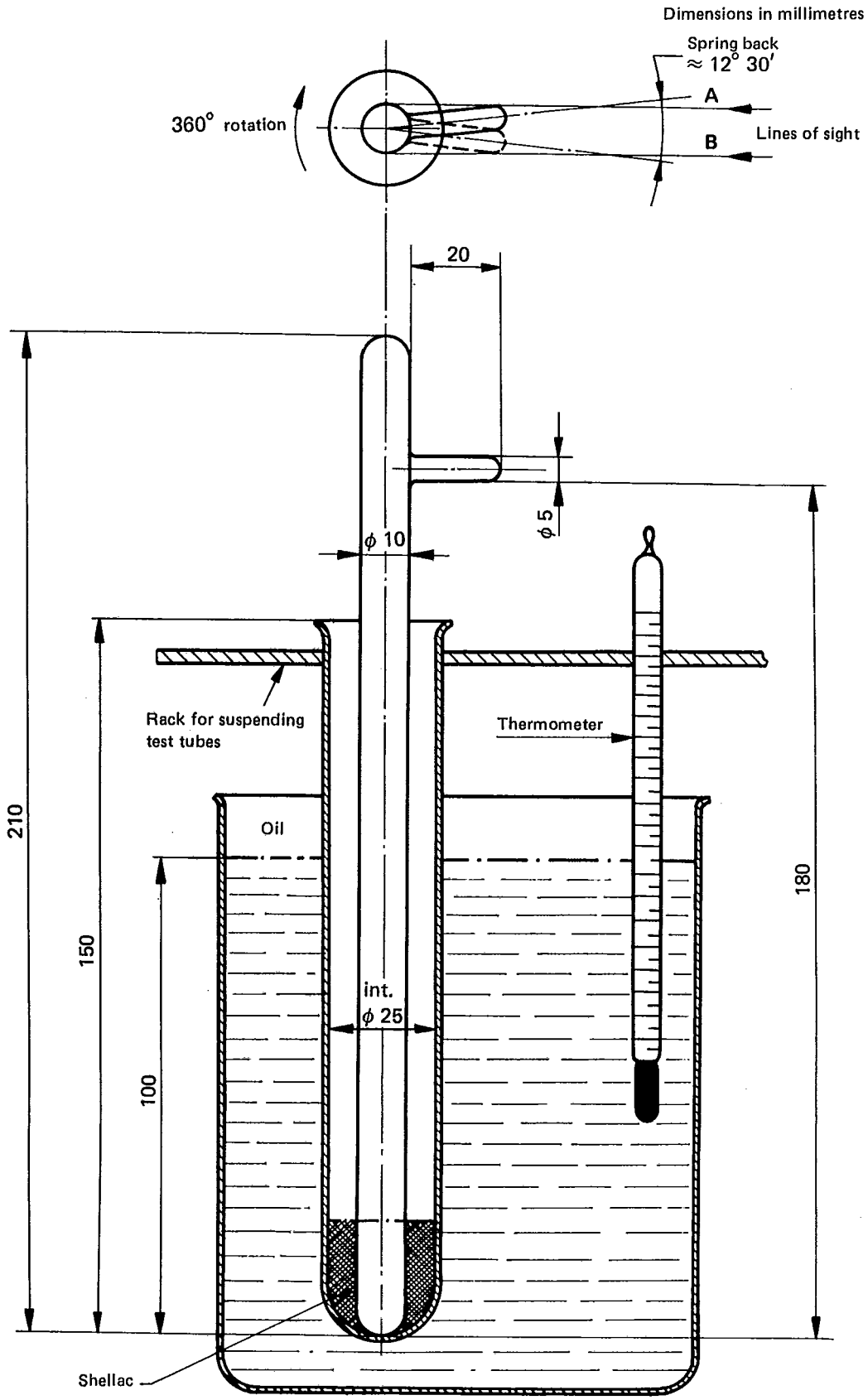
L.6 TEST REPORT

The test report shall include the following particulars :

a) the test temperature;

b) the polymerization time, to the nearest minute, for each specimen;

c) the mean of the readings under b) above.



NOTE – Mechanical stirrer not shown.

FIGURE 9 – **Heat polymerization test apparatus**

ANNEX M
(See 6.8)

DETERMINATION OF ACID VALUE

M.1 PRINCIPLE

Titration of an alcoholic solution of a test portion with a standard volumetric solution of potassium hydroxide, using thymol blue as indicator.

M.2 REAGENTS

M.2.1 Alcohol, 95 % (*V*/*V*) rectified spirit, neutral.

M.2.2 Potassium hydroxide, 0,1 N standard volumetric alcoholic solution.

Check the strength of the solution at intervals to provide for any deterioration in strength.

M.2.3 Thymol blue indicator, 1 g/l alcoholic solution.

M.3 PROCEDURE

Accurately weigh about 2 g of the "test sample" (see R.3.1 of annex R) and dissolve in 50 ml of the alcohol (M.2.1) with slight warming, if necessary. Cool the solution and carry out the titration with the standard volumetric alcoholic potassium hydroxide solution using the thymol blue solution as external indicator.

M.4 CALCULATION

The acid value, expressed as the number of milligrams of potassium hydroxide (mg KOH) equivalent to 1 g of shellac, is given by the formula

$$56{,}1 \times \frac{V\,T}{m}$$

where

V is the volume, in millilitres, of the potassium hydroxide standard volumetric alcoholic solution (M.2.2) required for the titration;

T is the normality of the potassium hydroxide standard volumetric alcoholic solution;

m is the mass, in grams, of the test portion.

ANNEX N
(See 6.9)

DETERMINATION OF LEAD CONTENT

N.1 PRINCIPLE

Colorimetric determination of the lead content of a test portion by matching the colour of the lead sulphide obtained from the material under test with that obtained from a standard lead solution.

N.2 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. All the reagents with the exception of (N.2.11) and (N.2.12) shall be free from traces of lead.

N.2.1 Citric acid, solid.

N.2.2 Ammonium acetate, solid.

N.2.3 Chloroform, redistilled.

N.2.4 Hydrochloric acid, ρ 1,18 g/ml.

N.2.5 Sulphuric acid, ρ 1,84 g/ml.

N.2.6 Ammonium hydroxide, ρ 0,88 g/ml or diluted as required.

N.2.7 Potassium cyanide, 100 g/l aqueous solution.

N.2.8 Sodium sulphide, 100 g/l solution.

N.2.9 Diphenyl thiocarbazone (dithizone), 1 g/l solution in the chloroform (N.2.3), freshly prepared.

N.2.10 Hydrochloric acid, approximately 0,1 N solution.

N.2.11 Lead, concentrated standard solution, corresponding to 1 g of Pb per litre.

Dissolve 0,16 g of lead(II) nitrate [$Pb(NO_3)_2$] in 50 ml of dilute (1 + 3) nitric acid and making up to 100 ml with water.

1 ml of this standard solution contains 1 mg of Pb.

N.2.12 Lead, dilute standard solution corresponding to 0,01 g of Pb per litre.

Dilute 10 ml of the concentrated standard lead solution (N.2.11) to 1 000 ml with water.

Prepare this solution fresh.

1 ml of this standard solution contains 0,01 mg of Pb.

N.3 PROCEDURE

N.3.1 Test portion

Weigh 4,5 to 5,5 g of the "test sample" (see R.3.1 of annex R) into a porcelain or silica basin.

N.3.2 Determination

Char the test portion (N.3.1) at low heat not exceeding 500 °C, until free from carbon, taking care to avoid loss of the light ash. Cool, add 5 ml of water and 10 ml of the hydrochloric acid (N.2.4) and boil gently for 5 min. Cool and transfer the clear solution to a 100 ml one-mark volumetric flask, filtering through a filter paper if necessary. Make up to 100 ml and take an aliquot portion of the solution corresponding to 0,5 g of the test sample. Add 5 ml of the sulphuric acid (N.2.5) and evaporate to fuming. Cool, dilute with about 50 ml of water, add 2 g of the citric acid (N.2.1) and just neutralize with the ammonium hydroxide solution (N.2.6). Add 1 ml of the potassium cyanide solution (N.2.7) and transfer the whole to a separating funnel. The total volume should be 100 to 150 ml.

Extract the liquid with the dithizone solution (N.2.9). Carry out three extractions, using 10, 5 and 5 ml respectively, but if the last extraction gives any indication of a reddish tinge, extract again to ensure complete removal of lead.

Take 10 ml of water in another separating funnel and wash each extract with this water. If suspended matter is present in the chloroform extract, this should be filtered before passing to the separating funnel containing 10 ml of wash water. Transfer the combined chloroform extracts to a separating funnel and extract lead by shaking successively with 50, 20 and 10 ml of the hydrochloric acid solution (N.2.10). Combine the acid extracts in a separating funnel, wash once or twice with 10 ml of the chloroform (N.2.3) and filter through a previously wetted filter paper into a 100 ml graduated flask. Make up the volume of the filtrate to 100 ml with the hydrochloric acid solution (N.2.10) and use this as the test solution.

Transfer a suitable volume of the test solution to a Nessler cylinder. Add 2 g of the ammonium acetate (N.2.2), followed by the ammonium hydroxide solution (N.2.6) until just alkaline, and then 1 ml of the potassium cyanide solution (N.2.7). Dilute to 50 ml with water, add 2 drops of the sodium sulphide solution (N.2.8) and match the colour against a set of standard solutions prepared in the same way using not more than 10 ml of the standard lead solution (N.2.12).

N.3.3 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents but omitting the test portion.

N.4 EXPRESSION OF RESULTS

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

$$\frac{V_1 - V_2}{5A}$$

where

V_1 is the volume, in millilitres, of the standard dilute lead solution present in the matching standard solution for the test sample;

V_2 is the volume, in millilitres, of the standard dilute lead solution present in the matching standard solution for the blank;

A is the volume, in millilitres, of the aliquot of the sample solution taken for colour matching.

ANNEX P
(See 6.10)

DETERMINATION OF GRIT CONTENT

P.1 PRINCIPLE

Drying and weighing of the residue obtained on a 63 µm sieve after alkaline dissolution of shellac and subsequent treatment with aqua regia.

P.2 REAGENTS

During the analysis use only reagents of recognized analytical grade and only distilled water of water of equivalent purity.

P.2.1 Aqua regia.

Add 1 volume of nitric acid (ρ 1,42 g/ml) to 3 volumes of hydrochloric acid (ρ 1,18 g/ml).

P.2.2 Sodium carbonate, anhydrous.

P.2.3 Sodium carbonate, 10 g/l solution of the anhydrous sodium carbonate (P.2.2).

P.3 APPARATUS

Ordinary laboratory apparatus, and

P.3.1 Beaker, tall form, of capacity 600 ml.

P.3.2 Water bath, capable of being maintained at 90 to 95 °C.

P.3.3 Siphon or suction tube, with hooked end.

P.3.4 Sieve, of aperture size 63 µm.

P.3.5 Electric oven, capable of being maintained at 100 ± 2 °C.

P.3.6 Acid-resistant filter paper.

P.3.7 Porcelain crucibles.

P.4 PROCEDURE

P.4.1 To 25 g of the "test sample" (See R.3.1 of annex R) contained in the 600 ml tall beaker (P.2.1), add 400 ml of hot water containing 7 g of the anhydrous sodium carbonate (P.2.2). Immerse the beaker in the hot water bath, maintained at 92,5 ± 2,5 °C, and stir frequently over a period of 1 h to ensure that the material has dissolved as far as possible. Allow the beaker and contents to cool and stand undisturbed for at least 30 min, then carefully remove the turbid supernatant liquor to within 2,5 cm of the bottom of the beaker, using the siphon or suction tube (P.3.3).

P.4.2 Dilute the remaining liquor with 300 ml of the sodium carbonate solution (P.2.3), mix, allow to settle, and remove the supernatant liquor as before. Repeat this process twice more. Strain the remaining contents of the beaker through the sieve (P.3.4), transfer any matter remaining in the beaker to the sieve by means of a jet of water and wash well with water.

P.4.3 Dry any residue on the sieve in the oven (P.3.5), maintained at 100 ± 2 °C, and transfer the residue to a porcelain crucible (P.3.7), ignite at a dull red heat and allow to cool. Heat the residue with 5 ml of the aqua regia (P.2.1) in a boiling water bath for 30 min. Dilute the acid mixture with water and filter through the acid-resistant filter paper (P.3.6). Transfer any solid matter to the filter by means of a jet of hot water and wash until free from acid. Carefully fold and dry the washed filter paper and ignite in a porcelain crucible at dull read heat. Sieve the residue through the 63 µm sieve and weigh any solid matter retained.

P.5 EXPRESSION OF RESULTS

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

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

where

m_0 is the mass, in grams, of material retained on the sieves,

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

ANNEX Q
(See 6.11)

DETERMINATION OF IODINE VALUE

Q.1 SCOPE

This annex specifies two methods for the determination of the iodine value of hand-made shellac, namely

- Method I : Hubl method
- Method II : Wijs-Langmuir method.

The choice of the method to be used shall be the subject of agreement between the purchaser and the supplier.

Q.2 METHOD I (Hubl method)

Q.2.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Q.2.1.1 Rectified spirit.

Q.2.1.2 Hubl iodine solution.

Mix together equal volumes of a 50 g/l solution of iodine in rectified spirit and a 60 g/l solution of mercury(II) chloride in rectified spirit. Allow to stand for at least 6 h before use, but discard if more than 48 h old.

Q.2.1.3 Potassium iodide, 100 g/l aqueous solution.

Q.2.1.4 Sodium thiosulphate, 0,1 N standard volumetric solution.

Q.2.1.5 Starch, 2 g/l indicator solution, freshly prepared.

Q.2.2 Procedure

Q.2.2.1 *Test portion*

Weigh accurately about 0,6 g of the "test sample" (see R.3.1 of annex R) into a 250 ml heat-resistant glass flask provided with a ground-glass stopper.

Q.2.2.2 *Determination*

Dissolve the test portion (Q.2.2.1) in 10 ml of the rectified spirit (Q.2.1.1) with the aid of gentle heat. Cool and add from a pipette 20 ml of the Hubl iodine solution (Q.2.1.2). In a similar flask place 10 ml of rectified spirit and 20 ml of Hubl iodine solution to serve as a blank.

Place the flasks in a cool, dark cupboard and leave for 16 to 18 h (preferably overnight). Add 10 ml of the potassium iodide solution (Q.2.1.3) to the contents of each flask, rinsing the stopper and neck of the flask to dissolve any iodine which may have tended to sublime. Dilute with 100 ml of water and titrate the excess iodine with the standard volumetric sodium thiosulphate solution (Q.2.1.4), using the starch solution (Q.2.1.5) as indicator, vigorously shaking the contents of the flask before and during the titration so as to break up the clots formed by the addition of water.

Q.2.3 Expression of results

The Hubl iodine value is given by the formula

$$\frac{12{,}69\,(V_1 - V_0)\,T}{m}$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (Q.2.1.4) required for titration of the test solution;

V_1 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (Q.2.1.4) required for titration of the blank test solution;

T is the normality of the standard sodium thiosulphate solution (Q.2.1.4);

m is the mass, in grams, of the test portion.

Q.3 METHOD II (Wijs-Langmuir method)

Q.3.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Q.3.1.1 Acetic acid, glacial, 99 %, having a melting point of 14,8 ± 0,05 °C, and free from reducing impurities, as indicated by the following test :

Dilute 2 ml of acetic acid with 10 ml of water, add 0,1 ml of 0,1 N potassium permanganate solution and maintain at room temperature. At the end of 2 h, the pink colour shall not be discharged.

Q.3.1.2 Chloroform.

Q.3.1.3 Potassium iodide, 100 g/l aqueous solution.

Q.3.1.4 Wijs-Langmuir iodine monochloride solution.

Dissolve 8 g of iodine trichloride in 500 ml of the glacial acetic acid (Q.3.1.1). Place 5 ml of the solution, accurately measured, in a glass stoppered flask containing 10 ml of the

potassium iodide solution (Q.3.1.3). Add 100 ml of distilled water and titrate with the standard volumetric sodium thiosulphate solution (Q.3.1.5) using the starch solution (Q.3.1.6) as indicator. Calculate the exact quantity of iodine trichloride present in the solution from the relationship

1 ml of 0,1 N sodium thiosulphate solution is equivalent to 0,005 832 g of iodine trichloride.

Dissolve 9 g of iodine in 500 ml of the glacial acetic acid, heating if necessary. Determine the exact quantity of iodine present by titrating 10 ml of this solution with the standard volumetric sodium thiosulphate solution using the starch solution as indicator.

1 g of iodine trichloride reacts with 1,098 g of iodine to form iodine monochloride. From this relation, calculate the volume of the iodine solution required to be added to the iodine trichloride solution. Add this calculated volume of iodine solution to the iodine trichloride solution and mix thoroughly. Dilute this solution with the glacial acetic acid until 10 ml is equivalent to 20 ml of the standard volumetric sodium thiosulphate solution when it is titrated in the presence of excess of the potassium iodide solution and water using the starch solution as indicator.

Heat the solution thus prepared to 100 °C for 20 min and then allow to cool. During the preparation of the solution, prevent access of water vapour. Keep the solution in an amber-coloured receptacle protected from light.

Q.3.1.5 Sodium thiosulphate, 0,1 N standard volumetric solution.

Q.3.1.6 Starch, 2 g/l indicator solution, freshly prepared.

Q.3.2 Procedure

Q.3.2.1 *Test portion*

Weigh about 0,2 g of the "test sample" (see R.3.1 of annex R) to an accuracy of 0,001 g and introduce into a 250 ml dry, clear glass flask having a ground glass stopper.

Q.3.2.2 *Determination*

Add 20 ml of the acetic acid (Q.3.1.1) to the flask containing the test portion (Q.3.2.1) and place the flask on the top of a hot water-bath maintained at 67,5 ± 2,5 °C, swirling the flask occasionally until dissolution is complete, except for the wax. This should not require more than 15 min. Add 10 ml of the chloroform (Q.3.1.2) and cool the solution to 22 ± 0,5 °C. Before adding the Wijs-Langmuir solution (Q.3.1.4), allow the bottle to stand at a temperature of 22 ± 0,5 °C for at least 30 min, half-immersed in water in a shallow pan of water which is either well insulated or equipped with a suitable thermostat. Add 20 ml of this Wijs-Langmuir solution at a temperature 22 ± 0,5 °C from a standard pipette with a delivery time of approximately 30 s. Immediately stopper the flask, place it again in the pan of water and note the time. Keep the flask half-immersed in water at 22 ± 0,5 °C for 60 min, swirling the flask occasionally during this time. Add 10 ml of the potassium iodide solution (Q.3.1.4) to the flask and wash into it any Wijs-Langmuir solution left on the stopper. Dilute with about 100 ml of water and titrate the solution immediately, running in rapidly 25 to 30 ml of the standard volumetric sodium thiosulphate solution (Q.3.1.5) and shaking vigorously until the solution assumes a straw colour. Add 1,5 ml of the starch indicator solution (Q.3.1.6) and slowly finish the titration. The end-point is sharp. Disregard any colour returning after about 30 s.

If several samples are being tested, allow at least 5 min interval between the additions of Wijs-Langmuir solution to different flasks to allow time for the final titration.

Q.3.3 Blank test

Carry out a blank test in parallel with the analysis, following the same procedure and using the same reagents, but omitting the test portion.

Q.3.4 Expression of results

See Q.2.3.

ANNEX R
(See 7.1)

SAMPLING

R.1 TAKING OF SAMPLES

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

R.1.1 Only original, unopened packages of shellac shall be sampled.

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

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

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

R.1.5 Free-flowing shellac

Samples shall be taken from different places in each package by means of a suitable tryer so as to yield a total of 5 kg of material consisting of approximately equal portions from each package sampled. The material shall then be thoroughly mixed and heaped and quartered along two diameters which intersect at right angles, and two opposite quarters are mixed. One half of the material may, if necessary, be further subdivided by the normal process of quartering to form a number of samples which are known as "samples as received" and shall be used for the determination of volatile matter (moisture), if this requirement is agreed upon between the purchaser and the supplier. These samples shall be placed in airtight containers, sealed and labelled accordingly. The samples shall be treated as described in R.3.2. The other half of the material shall be treated as described under R.2.1 to form the "laboratory sample".

R.1.6 Blocky or matted shellac

Samples shall be taken from different places in each package, by chipping or other suitable means, so as to yield a total of 5 kg of material consisting of approximately equal portions from each package sampled. The material shall then be thoroughly mixed and heaped and quartered along two diameters which intersect at right angles, and two opposite quarters are mixed. One half of the material may, if necessary, be further subdivided by the normal process of quartering to form a number of samples which are known as "samples as received" and shall be used for the determination of volatile matter (moisture), if this requirement is agreed upon between the purchaser and the supplier. These samples shall be placed in airtight containers, sealed and labelled accordingly. The samples shall be treated as described in R.3.2. The other half of the material shall be roughly ground so as to pass a sieve having a nominal aperture of 6.3 and then treated as described under R.2.1 to form the "laboratory sample".

R.2 REDUCTION OF SAMPLES

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

R.2.1 The material for the laboratory sample, as obtained under R.1.5 or R.1.6 shall be mixed thoroughly, heaped and quartered along two diameters which intersect at right angles. Two opposite quarters shall be mixed and ground to pass entirely through a sieve having a nominal aperture of about 2 mm. The material shall then be thoroughly mixed and quartered so as to yield four samples each of mass approximately 250 g. These four samples shall be placed in airtight containers, sealed, labelled "laboratory sample" and sent to the interested parties.

R.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 shall be stated on a label attached to each sample.

R.3 PREPARATION OF TEST SAMPLES

R.3.1 The laboratory samples shall be ground to pass entirely through a sieve whose nominal aperture is between 0,4 and 0,7 mm. This finely ground material shall be mixed thoroughly, placed in an airtight container and labelled as the "test sample".

R.3.2 The "samples as received" (see R.1.5 and R.1.6) which are to be used for determination of volatile matter (moisture), when this requirement is agreed upon between the purchaser and the supplier, shall be ground to pass entirely through a sieve whose nominal aperture is between 0,4 and 0,7 mm. The grinding operation shall be performed with production of as little heat as possible. The finely ground material shall be mixed thoroughly, immediately placed in airthight container and labelled as the "sample as received".