INTERNATIONAL STANDARD

ISO
57

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1975-02-01

AMENDMENT 1
1996-11-15

Bleached lac — Specification

AMENDMENT 1

Gomme laque blanche — Spécification

AMENDEMENT 1

Reference number
ISO 57:1975/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 57:1975 was prepared by Technical Committee ISO/TC 50, *Lac.*

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Bleached lac — Specification

AMENDMENT 1

Page 8, subclause B.2.4.4.1, penultimate line

Correct the spelling of "draugths" to "draughts".

Page 14, subclause E.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.

Page 14, subclause F.1.5

Add the following note after F.1.5:

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

Page 18, clause L.2

Add a new paragraph at the end of clause L.2:

Alternatively, a potentiometric method may be used.

Page 21, Annex P

Replace the text of the existing annex with the following:

P.1 General

The lead content of bleached lac 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 lead.

P.2 Method A

Renumber clauses P.1 to P.4 as P.2.1, P.2.2, P.2.2.1, P.2.2.2, P.2.2.3, P.2.2.4, P.2.2.5, P.2.2.6, P.2.2.7, P.2.2.8, P.2.2.9, P.2.2.9.1, P.2.2.9.2, P.2.2.10, P.2.2.11, P.2.3, P.2.3.1, P.2.3.2, P.2.3.3, P.2.3.4, P.2.3.5 and P.2.4 respectively.

P.3 Method B — Atomic absorption spectrometric method

P.3.1 Principle

The sample is brought into solution by suitable treatment with acid or acid combinations, diluted with distilled water, filtered and suitable dilutions made for aspiration into the air + acetylene flame of an atomic absorption spectrometer. A standard solution is prepared in the same way, for calibration purposes. The wavelength most sensitive to lead absorption is 217,0 nm. However other lines, more suitable for high concentrations, can also be used.

P.3.2 Apparatus

P.3.2.1 Atomic absorption spectrometer, provided with a background corrector and having the following characteristics:

a) Lamp current: depending on the lamp and instrument used
b) Support: air
c) Fuel: acetylene
d) Flame stoichiometry: oxidizing
e) Wavelength and working range:

Wavelength nm	**Bandpass** nm	**Working range** g/ml
217,0	1,0	5 to 20
283,3	0,2	10 to 40
261,4	0,2	200 to 800
202,2	0,2	250 to 1 000
205,3	0,2	2 000 to 8 000

NOTE — In the case of a multielement hollow-cathode lamp containing copper, the second most sensitive wavelength for lead, that is 283,3 nm, may be used to avoid interference of copper absorption.

P.3.3 Reagents

P.3.3.1 Lead metal, 99,99 % pure.

P.3.3.2 Concentrated nitric acid.

P.3.3.3 Concentrated hydrochloric acid.

P.3.3.4 Standard lead solution.

Dissolve 1,0 g of lead (P.3.3.1) in 1:1 nitric acid (P.3.3.2), dilute to one litre with distilled water.

P.3.4 Sample preparation

A suitable quantity of bleached lac sample is dissolved in hydrochloric acid or a mixture of hydrochloric acid and nitric acid, evaporated to dryness, again dissolved in hydrochloric acid, diluted, filtered and made up to known volume. A suitable dilution is made for the determination of lead before aspirating in the atomic absorption flame. The sample solution should be concentrated by ion exchange or by solvent extraction if lead is expected to be present in very low quantities.

P.3.5 Procedure

Optimize the response of the spectrometer by adjusting the burner height and flame. Aspirate water to determine zero absorption; when a stable response is observed, aspirate standard solutions (at least four) and record the absorption.

Aspirate sample to determine the absorption of the sample. Prepare a calibration curve by plotting the net absorption values of the standards against their concentrations, in grams of lead per millilitre of solution. Locate the sample absorption on the prepared curve and calculate the concentration of lead in the sample.

P.3.6 Calculation

Lead, expressed as percent mass fraction = $\frac{c \times V}{10^6} \times \frac{100}{m}$

where

c is the concentration of lead, in grams per millilitre, in the final solution;

V is the volume, in millilitres, of final solution;

m is the mass, in grams, of sample in the final solution.

ICS 87.060.20

Descriptors: shellac, specifications, materials specifications, chemical analysis, determination of content, volatile matter, insoluble matter, chlorine, inorganic acids, arsenic, lead, ash determination.

Price based on 3 pages

H-09-25

INTERNATIONAL STANDARD 57

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

Bleached lac — Specification

Gomme laque blanche — Spécifications

First edition — 1975-02-01

UDC 668.447.3.004.1 **Ref. No. ISO 57-1975 (E)**

Descriptors : shellac, materials specifications, chemical analysis, determination of content, volatile matter, insoluble matter, chlorine, inorganic acids, ashes, arsenic, lead.

Price based on 24 pages

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 57 was drawn up by Technical Committee ISO/TC 50, *Lac,* and circulated to the Member Bodies in February 1973.

It has been approved by the Member Bodies of the following countries :

Bulgaria
Egypt, Arab Rep. of
India
Netherlands
Poland
Portugal
Thailand
United Kingdom
U.S.A.

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 57-1957, of which it constitutes a technical revision.

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Bleached lac — Specification

0 INTRODUCTION

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

0.2 No limit has been specified for the chlorine content of bleached lac. Normally the chlorine content of bleached lac varies between 1,5 and 4,5 % in moisture-free material, but ISO/TC 50 has decided to place it on record that it is desirable to maintain it at a low value (say at 3 %, maximum).

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

0.4 Similarly the mineral acid content of bleached lac should also be as low as possible, requiring for neutralization under the conditions specified in annex K not more than 82 ml of 0,1 N sodium hydroxide per 100 g of the moisture-free material which is equivalent to 0,4 % of mineral acid calculated as sulphuric acid (H_2SO_4).

0.5 The maximum limit for the volatile matter (moisture) in the bone-dry material has been specified as a mandatory requirement, i.e. a maximum of 6 % (see clause 5). In so far as surface-dry (air-dry) and wet bleached lacs are concerned, the actual limits have been made the subject of agreement between purchaser and vendor. The normal moisture content of the surface-dry (air-dry) material does not exceed 12 %; that of wet bleached lac does not exceed 30 %.

0.6 The mesh sizes of sieves given in this International Standard have been indicated in terms of aperture dimensions, and a note, giving number designations of approximately equivalent sieves according to ISO 565 and according to the national standards of the U.S.A., the United Kingdom, France, Germany and India, has been added for the sake of convenience.

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 test or analysis, shall be rounded off to the same number of places as that in the specified value; it being always understood that the analyst will carry out his determination to at least one place more than in the specified value.

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies the requirements and methods of test for bleached lac in any form that may be agreed upon between the purchaser and the vendor.

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

1.3 The limits specified in this International Standard are not to be exceeded.

2 DEFINITIONS

For the purposes 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 processes or by both heat and solvent processes.

2.4 **bleached lac** : The product obtained by subjecting seedlac or shellac in solution to a process of bleaching and then recovering the product in a solid form.

2.5 regular (cloudy or waxy) **bleached lac :** The ordinary type of bleached lac from which wax has not been removed.

2.6 refined (transparent or wax-free) **bleached lac :** The type of bleached lac from which wax has been removed during the process of manufacture.

2.7 approved sample : The sample agreed upon between the purchaser and the vendor as the standard for colour and appearance.

3 FORM

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

4 CONDITIONS AND TYPES

4.1 Three conditions of bleached lac are specified, namely :

a) bone-dry,

b) surface-dry (air-dry) and

c) wet.

4.2 Two types are specified for each of these conditions, namely :

a) regular (cloudy or waxy), and

b) refined (transparent or wax-free).

5 VOLATILE MATTER (MOISTURE)

5.1 Bone-dry bleached lac shall contain not more than 6 % of volatile matter (moisture) as determined by the method described in annex A.

5.2 The percentage of volatile matter (moisture) present in surface-dry (air-dry) bleached lac shall be subject to agreement between the purchaser and the vendor and shall be determined by the method described in annex A (see 0.5).

5.3 The percentage of volatile matter (moisture) in wet bleached lac shall be subject to agreement between the purchaser and the vendor and shall be determined by the method described in annex A (see 0.5).

6 MATTER INSOLUBLE IN HOT ALCOHOL

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

regular :	1,1 %
refined :	0,2 %

Matter insoluble in hot alcohol shall be determined by either of the methods described in annex B, as agreed between the purchaser and the vendor.

7 COLOUR

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

8 WAX

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

regular :	5,5 %
refined :	0,2 %

9 ASH

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

regular :	1,0 %
refined :	0,5 %

10 ROSIN AND COPALS

10.1 Bleached lac shall contain no rosin, as tested by the method described in annex F.

10.2 Bleached lac shall contain no copals, as tested by the method described in annex G.

11 MATTER SOLUBLE IN WATER

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

regular :	1,0 %
refined :	0,3 %

12 CHLORINE CONTENT

The chlorine content of bleached lac shall be subject to agreement between the purchaser and the vendor and, when specified, it shall be determined by the method described in annex J (see 0.2).

13 ACID VALUE AND MINERAL ACID

13.1 The acid value of bleached lac shall be subject to agreement between the purchaser and the vendor and, when specified, it shall be determined by the method described in annex K (see 0.3).

13.2 The mineral acid content of bleached lac shall be subject to agreement between the purchaser and the vendor and, when specified, it shall be determined by the method described in annex L (see 0.4).

14 FREE CHLORINE OR PEROXIDES

A requirement for the absence of free chlorine or peroxides shall be subject to agreement between the purchaser and the vendor. When specified, the aqueous extract of the material shall not show the presence of free chlorine or peroxides when tested by the method described in annex M.

15 ARSENIC

The arsenic content of bleached lac shall be subject to agreement between the purchaser and the vendor and, when specified, it shall be determined by the method described in annex N.

16 LEAD

The lead content of bleached lac shall be subject to agreement between the purchaser and the vendor and, when specified, it shall be determined by the method described in annex P.

17 REQUIREMENTS

The requirements for bleached lac are given in table 1.

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

TABLE 1 — **Requirements for bleached lac**

Reference	**Character of requirement**	**Characteristic**	**Maximum limits for type**		**Method of testing : reference to annex**
			Regular	**Refined**	
5	Essential	Volatile matter (moisture) % Bone-dry Other conditions	 6,0 As agreed between purchaser and vendor	 6,0	 A A
6	Essential	Matter insoluble in hot alcohol, % 1)	1,1	0,2	B
7	Essential	Colour	Close visual match to the approved sample		C
8	Essential	Wax, % 1)	5,5	0,2	D
9	Essential	Ash, % 1)	1,0	0,5	E
10.1	Essential	Rosin	Nil	Nil	F
10.2	Essential	Copals	Nil	Nil	G
11	Essential	Matter soluble in water, % 1)	1,0	0,3	H
12	Optional	Chlorine content 1)	As agreed between purchaser and vendor (see 0.2)		J
13.1	Optional	Acid value 1)	As agreed between purchaser and vendor (see 0.3)		K
13.2	Optional	Mineral acid 1)	As agreed between purchaser and vendor (see 0.4)		L
14	Optional	Free chlorine or peroxides	Absent	Absent	M
15	Optional	Arsenic 1)	As agreed between purchaser and vendor		N
16	Optional	Lead 1)	As agreed between purchaser and vendor		P

1) To be calculated on a moisture-free basis.

18 TESTS

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

18.2 All analytical work on bleached lac except the determination of volatile matter (moisture) content shall be carried out on the "test sample" obtained as described under Q.3.1 (annex Q).

18.3 The volatile matter (moisture) content of the "test sample" shall be determined by the method described in annex A and other than for volatile matter (moisture), this figure shall be used to correct the analytical results on the basis of the moisture-free material.

NOTE — During the analyses specified in the annexes, use only reagents of recognised analytical grade, and only distilled water or water of equivalent purity.

19 SAMPLING

Samples shall be taken in the manner described in annex Q.

ANNEX A

(See clause 5)

DETERMINATION OF VOLATILE MATTER (MOISTURE)

A.1 PRINCIPLE

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

A.1.2 For the second stage, the partially dried material thus obtained is ground to the specified size, and a portion is dried further by heating it in a well-ventilated oven maintained at a temperature of 41 ± 2 °C for 18 h.

A.2 PROCEDURE

A.2.1 Use a portion of the "sample as received" (see Q.1.5, Q.1.6 and Q.1.7) and crush, if necessary, into granules using a heavy pestle and mortar, keeping the latter covered as far as possible during the process. Weigh a clean, dry, flat-bottomed dish of about 100 mm diameter, provided with a glass cover. Transfer approximately 10 g of the ground sample to the dish as rapidly as possible, cover it with the glass cover and reweigh. The difference gives the mass of test portion.

A.2.2 Transfer the dish and contents to a vacuum desiccator containing concentrated sulphuric acid and remove the cover of the dish. Immediately evacuate the desiccator and keep the sample, uncovered, in vacuo for not less than 12 h and not more than 24 h. Remove the dish, replace the cover and weigh. The difference between this mass and the mass of the dish is the mass of the partially dried test portion. Grind it until it passes a sieve having a nominal aperture of 0,425 mm (see Q.4).

A.2.3 Weigh approximately 5 g of the ground material, to an accuracy of 1 mg, into a covered tared dish of the type described in A.2.1 and transfer to a well-ventilated oven maintained at a temperature of 41 ± 2 °C for 18 h, the cover of the dish being removed during the drying process. At the conclusion of the heating period, replace the cover and transfer the covered dish to a desiccator; weigh when cool. This mass minus the mass of the dish is the mass of the completely dried test portion.

A.3 CALCULATION

A.3.1 The volatile matter (moisture) in the original sample, as a percentage by mass, is given by the formula

$$100\left(1-\frac{m_4\,m_2}{m_3\,m_1}\right)$$

where

m_1 is the mass, in grams, of the test portion taken for drying in vacuo;

m_2 is the mass, in grams, of the partially dried test portion;

m_3 is the mass, in grams, of the part of the partially dried, ground test portion taken for oven drying;

m_4 is the mass, in grams, of the test portion after oven drying.

A.3.2 If the first drying stage (see A.2.2) is omitted, the volatile matter (moisture) in the original sample, as a percentage by mass, is given by the formula

$$100\left(1-\frac{m_2}{m_1}\right)$$

where

m_1 is the mass, in grams, of the test portion taken for oven drying;

m_2 is the mass, in grams, of the test portion after oven drying.

ANNEX B

(See clause 6)

DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

B.1 GENERAL

The matter insoluble in hot alcohol is determined by extracting a known mass of bleached lac with 95 % (*V/V*) ethanol and determining the percentage of the undissolved residue by either of the two methods described below, as may be agreed.

B.2 METHOD I

B.2.1 Apparatus

B.2.1.1 Condenser, all glass, of the type and dimensions shown in figure 1, with the tip cut at an angle of 45°. The condenser has two holes in its tip to hold the siphon tube (B.2.1.2).

B.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.

B.2.1.3 Conical flask, heat resistant, wide-mouthed, conical, preferably of borosilicate glass, 176 ± 3 mm in height and 48 ± 2 mm in inside diameter at the top.

The flask has a tight-fitting cork 25 mm in depth and bored to fit the stem of the condenser. The bottom of the cork is just above the holes for the wire in the condenser. To support the flask a suitable ring support with iron clamp and nickel-chromium or iron gauze is used. The gauze has no asbestos covering.

B.2.1.4 Carbon filter tube of the type and dimensions shown in figure 1, with a light spiral spring at the bottom to hold up the extraction cartridge.

The stem of the filter tube is fitted with a rubber stopper and firmly held in a hot water bath.

B.2.1.5 Extraction cartridges[1], of fat-free paper 26 ± 1 mm in diameter and 60 ± 1 mm in height.

Dimensions in millimetres

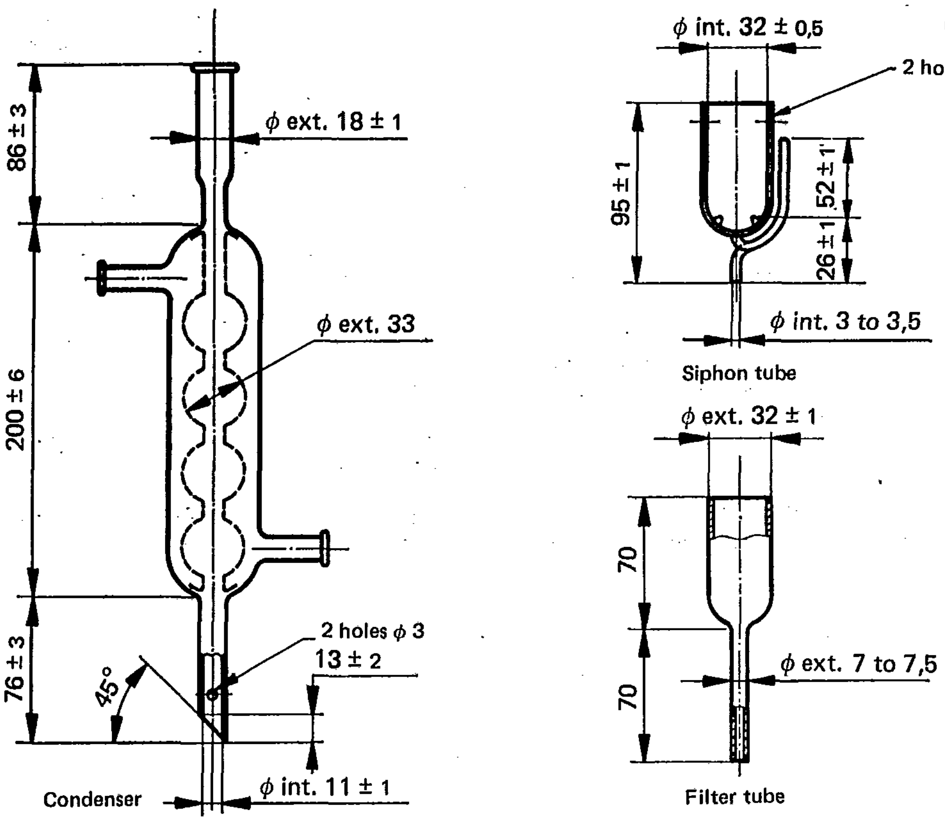

FIGURE 1 – **Extraction apparatus for determining matter insoluble in hot alcohol – Method I**

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[1] Schleicher and Schull No. 603 or equivalent is suitable.

Dimensions in millimetres

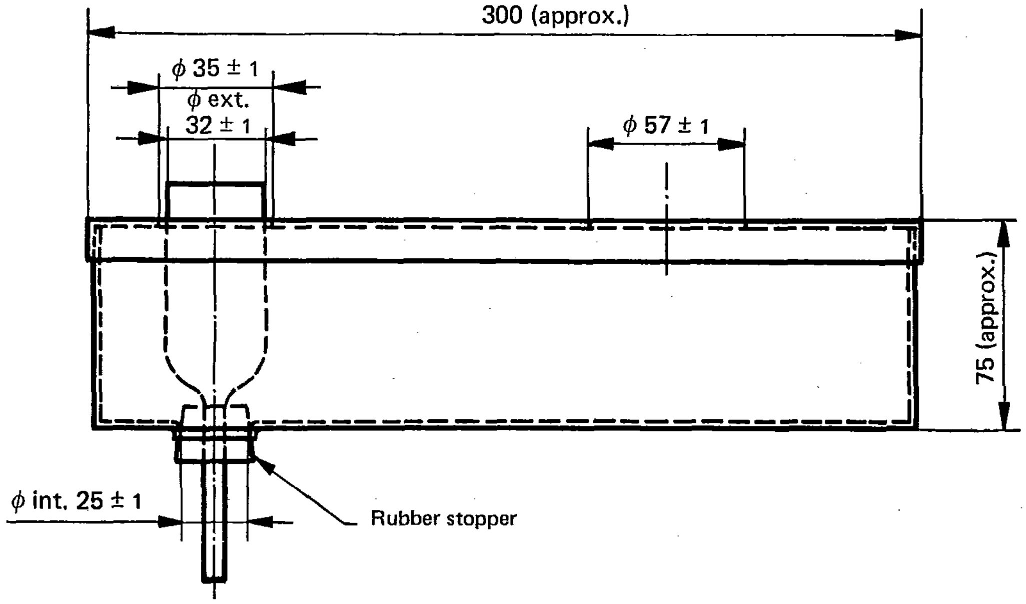

FIGURE 2 – **Hot water bath for insoluble matter test – Method I**

B.2.1.6 Weighing bottle, glass-stoppered, 80 ± 1 mm in height and 40 ± 1 mm in diameter.

B.2.1.7 Hot water bath, made of about 0,9 mm thick (or 21 B.G.) copper (approximately 8 kg/m^2), having a width of approximately 100 mm and other dimensions as given in figure 2.

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

B.2.1.8 Gas burner, low form, adjustable, Bunsen type, carrying a draught shield, or any other suitable heating device.

B.2.2 Reagent

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

B.2.3 Preparation of extraction cartridge

B.2.3.1 Place 125 ml of the alcohol in the flask and a new cartridge in the siphon tube. Introduce the siphon tube into the flask and connect it to the condenser, making sure that there is an ample flow of cold water through the condenser. Adjust the flame of the burner so as to give a cycle of filling and emptying in the siphon tube every 2 min and extract for 30 min. Dry the cartridge in an oven at a temperature of 100 ± 2 °C. At the end of 3 h, weigh it in a tared weighing bottle, which has been kept in a desiccator over sulphuric acid, lifting the stopper of the bottle momentarily before weighing. Continue drying and weigh as before after each hour of drying, until the loss in mass between successive weighings does not exceed 2 mg.

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

B.2.4 Procedure

B.2.4.1 Before analysis, thoroughly mix the "test sample" (see Q.3.1) by rolling on paper, at least ten times, to ensure uniformity of the sample. Weigh, directly from the rolling sheet, 4,5 to 5,5 g of the sample, to an accuracy of 0,01 g, place in a 200 ml tall, lipped beaker, add 125 ml of alcohol, cover with a watch-glass and place on the hot water bath (see figure 2). Boil the solution vigorously for 30 min to ensure complete solution of the bleached lac 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.

B.2.4.2 Meanwhile, place an extracted and weighed cartridge in the filter tube. Maintain the hot water around the tube at a temperature of not less than 90 °C. Wet the cartridge with hot alcohol and decant the boiling solution into the heated cartridge until the beaker is nearly empty.

B.2.4.3 Wash the remaining solution and the insoluble matter into the cartridge, using a "policeman", if necessary, with successive portions of hot alcohol contained in a wash-bottle kept hot on the water bath. Finally, wash the cartridge from the top downwards with a fine stream of hot alcohol. A complete washing and transfer from the original beaker will require at least 75 ml of hot alcohol.

B.2.4.4 Transfer the cartridge containing the insoluble matter to the extraction apparatus, place 125 ml of alcohol in the extraction flask and connect up the apparatus. Start the water flowing through the condenser, making sure that there is an adequate supply for efficient condensation. Light the burner and time the extraction from the first emptying of the siphon, running the extraction for exactly 1 h. Immediately begin to adjust the Bunsen burner so that a complete filling and emptying of the siphon tube takes place every 2 min, as determined by a stop-watch or preferably by a good two-minute sand-glass, one for each extraction apparatus.

B.2.4.4.1 In this way exactly 30 cycles per hour are carried out. If this cycle rate is not meticulously maintained, neither check results on duplicate samples in the same laboratory, nor concordant figures from one laboratory to another can be obtained, even when working on the same standard sample. It is also necessary to guard the apparatus from draugths while in operation, otherwise the proper cycle rate cannot be maintained.

B.2.4.4.2 Occasionally, bleached lacs are encountered which do not yield the required number of 30 siphonings per hour, due to slow filtration. In these instances, continue the extraction until 30 siphonings have been accomplished or repeat the test with a 2 g sample and report the sample as abnormal or slow filtering.

B.2.4.5 Remove the cartridge, drain in an upright position on filter paper and dry in an oven at a temperature of 100 ± 2 °C. After drying for 2 h, place it in the weighing bottle, cool in a desiccator over sulphuric acid (density 1,84 g/ml) and weigh, removing the stopper momentarily just before weighing. Repeat drying and weighing as before after each hour of drying, until the loss in mass between successive weighings does not exceed 2 mg. From the mass of the residue and the mass of the test portion, calculate the percentage of insoluble matter, making allowance for the volatile matter (moisture) content, determined as prescribed in annex A. Use the lowest mass in the calculation.

B.2.5 Calculation

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

$$\frac{m_2}{m_1\,(100-M)} \times 10^4$$

where

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

m_2 is the mass, in grams, of the residue;

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

B.3 METHOD II

B.3.1 Apparatus

The apparatus consists 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 pass upwards by way of the adaptor to the condenser. The refluxing solvent runs from the condenser into the cup of the siphon tube.

B.3.1.1 Siphon tube[1], of glass, of the Knoefler type, having minimum internal dimensions of 52 mm in height and 32 mm in diameter, resting in an adaptor tube in such a way that the siphon tube is surrounded by the ascending vapours of the boiling solvent (see figure 3).

B.3.1.2 Condenser, of any convenient pattern.

B.3.1.3 Flask, of any convenient size.

B.3.1.4 Filter paper, 125 mm in diameter, medium grade[2].

B.3.1.5 Weighing bottles, of glass, 80 ± 1 mm in height and 40 ± 1 mm in diameter, with ground glass stoppers.

B.3.1.6 Gas burner, low form, adjustable, Bunsen type, carrying a draught shield, or any other suitable heating device.

B.3.2 Reagent

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

B.3.3 Procedure

B.3.3.1 Fold a filter paper 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 an oven at a temperature of 100 ± 2 °C for 30 min. Rapidly transfer to separate weighing bottles which have been kept in a desiccator over sulphuric acid (density 1,84 g/ml). Place each bottle and its contents back in the desiccator for 20 min, then weigh by counterpoise, preferably using a rapid-weighing balance of the aperiodic type.

B.3.3.2 Weigh 4,5 to 5,5 g of the "test sample" of bleached lac (see 0.3.1) to an accuracy of 0,01 g and place in the filter paper envelope **S**; fold in the original folds, taking care not to leave any channel through which finely divided material might escape. Again enclose in paper **C** and

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[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 described in method I (see B.2), consisting of siphon tube condenser and flask, could be satisfactorily used.

[2] Whatman No. 1 or equivalent is suitable.

Dimensions in millimetres

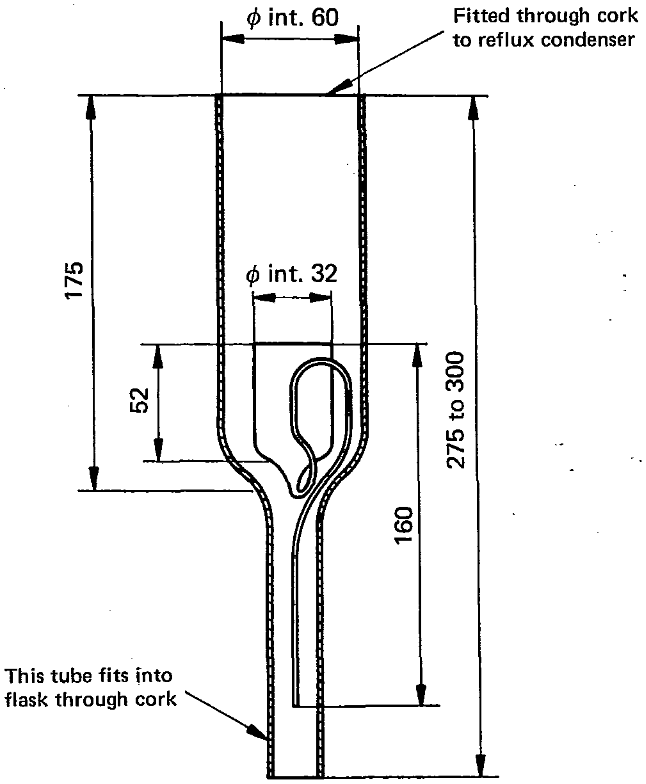

FIGURE 3 – **Siphon tube and adaptor**

secure with thread. Place the resulting envelope, in a 100 ml beaker and cover it with alcohol. Allow to stand overnight at room temperature. Place the envelope in the cup of the siphon tube and extract continuously with hot alcohol for 4 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.

B.3.3.3 At the end of the specified time, remove the paper envelope, allow to drain, separate the two papers, dry each on a glass plate in the steam oven and then for 3 h in a thermostatically controlled oven at a temperature of 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 counterpoise, 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 2 mg, repeat the processes of drying and weighing until the difference between successive weighings is less than 2 mg. Use the lowest mass in the calculation.

B.3.4 Calculation

As for method I (see B.2.5).

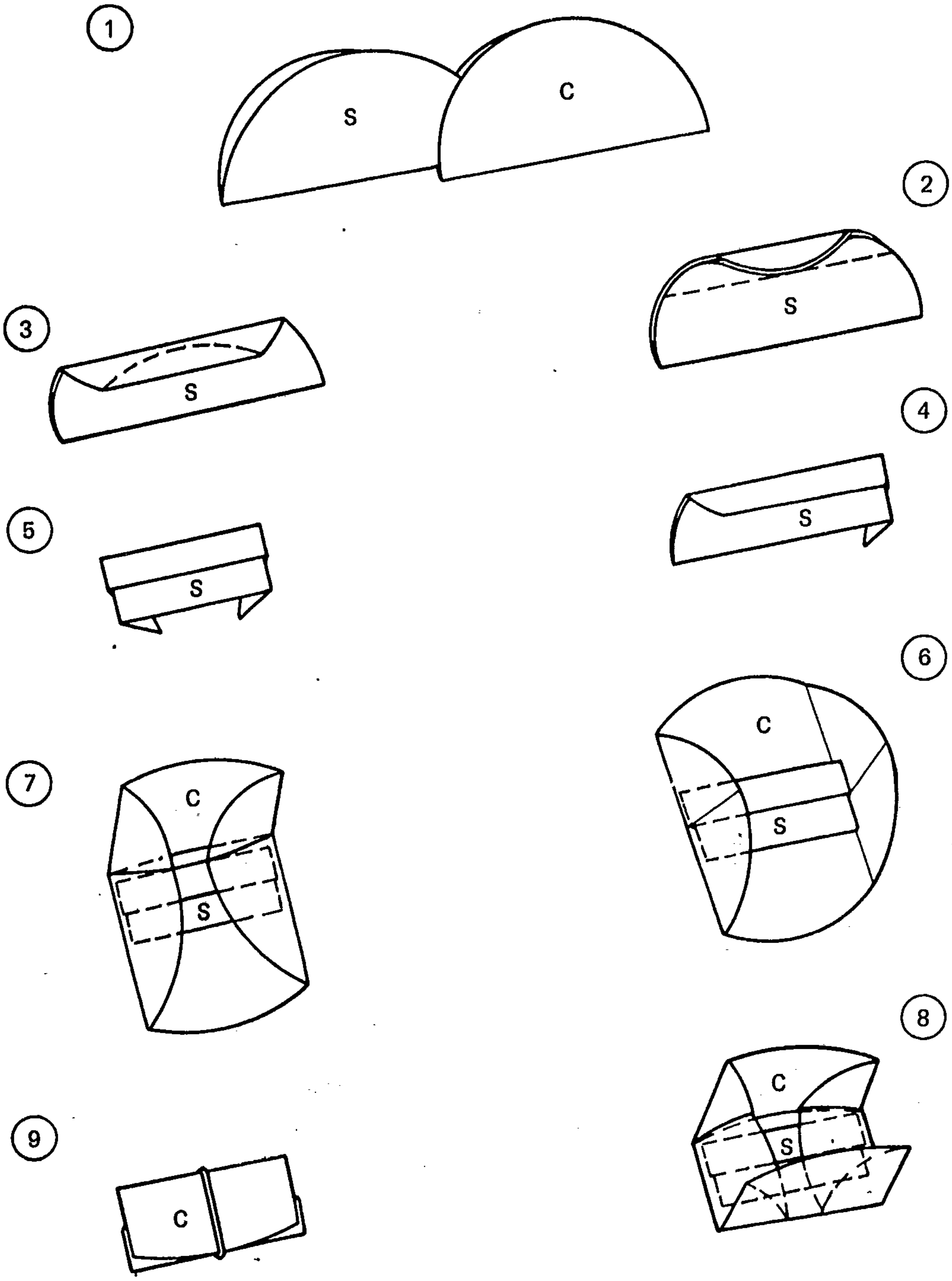

FIGURE 4 — **Folding of filter paper**

ANNEX C

(See clause 7)

COMPARISON OF COLOUR

C.1 GENERAL

Two methods for the comparison of colour are described. One of these is based on the visual comparison of the colour of films prepared from the material and the approved sample; the other is based on the visual comparison of solutions of the material and the approved sample.

C.2 METHOD I : VISUAL COLOUR COMPARISON OF FILMS

C.2.1 Principle

Bleached lac is dissolved in 95 % (*V/V*) ethanol and the colour examined visually against a similarly treated approved sample after forming films.

C.2.2 Procedure

C.2.2.1 Treat a weighed quantity of the "test sample" of bleached lac (see Q.3.1) with twice its mass of cold 95 % (*V/V*) ethanol, shaking at intervals until the lac is entirely dissolved. Compare its colour with the colour of a similar solution of the approved sample by the following method.

NOTE — The insoluble solids content of the approved sample shall be the same as that of the bleached lac solution under test.

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

C.3 METHOD II : VISUAL COLOUR COMPARISON OF ALCOHOLIC SOLUTIONS

C.3.1 Principle

The colour of the solution is compared with that of an approved sample.

C.3.2 Reagents

C.3.2.1 Alcohol, 95 % (*V/V*) ethanol; or 95 % (*V/V*) denatured spirit, provided that it is colourless.

C.3.2.2 Bleached lac solution. Dissolve 10,0 g of the "test sample" (see Q.3.1) in 100 ml of alcohol by shaking for 30 min at a temperature of 27 ± 2 °C. Filter the solution in an ordinary funnel, using a medium grade filter paper. Discard the first 15 ml of the filtrate and then collect 5 ml or more of the clear filtrate for the test. Repeat, using the approved sample in place of the test sample.

C.3.3 Procedure

C.3.3.1 Transfer 5 ml of the filtered bleached lac solution to a thin-walled test tube, measuring about 200 mm X 13 mm, by means of a pipette. Take a similar volume of the solution of the approved sample in another test tube, similar in every respect, for matching. Compare the colours of the two solutions, holding the test tubes against the light, with a piece of moistened filter paper or opal glass interposed between the light source and the test tube.

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

C.3.3.2 The requirement is taken as having been satisfied if the solution prepared from the test sample is not found to be darker than that prepared from the approved sample.

ANNEX D

(See clause 8)

DETERMINATION OF WAX

D.1 GENERAL

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

D.2 METHOD I, FOR REGULAR BLEACHED LAC CONTAINING WAX

D.2.1 Principle

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

D.2.2 Reagents

D.2.2.1 Sodium carbonate, anhydrous, analytical reagent.

D.2.2.2 Chloroform, redistilled, free from non-volatile residue.

D.2.2.3 Filter-aid[1], previously extracted with chloroform and dried before use.

D.2.3 Procedure

D.2.3.1 Weigh 9,5 to 10,5 g of the "test sample" (see Q.3.1) to an accuracy of 0,01 g and dissolve in 150 ml of hot water containing 2,5 g of sodium carbonate in a 200 ml, tall beaker. Immerse the beaker in a steam or boiling water bath and stir until the lac is in solution. Then cover with a watch-glass and allow it to remain in the bath for 2 to 3 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 a 12,5 cm double acid-washed, retentive, low-ash filter paper[2], 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 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 bleached lac solution before starting the filtration.

D.2.3.2 If the filtration is carried out under gravity alone, then, after the filtration is completed and all soluble bleached lac washed out of the paper with water, remove the paper from the funnel and, without further folding it, set it in the beaker, resting 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 an oven maintained at a temperature not exceeding 65 °C, for several hours, to remove most of the water.

Next, remove the paper from the beaker, wrap carefully in a large piece of clean fat-free filter paper, bind with fine wire and place it in a 26 mm X 60 mm fat-free paper extraction cartridge[3] which has been previously extracted with hot chloroform. Put the cartridge containing the wax and paper into a suitable continuous extraction apparatus, such as the standard hot-alcohol-insoluble matter apparatus (see annex B, method I) and pour into the beaker, which previously contained the filter paper and wax, a portion of the chloroform to be used for the extraction.

Bring the solvent to 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 and dry the residue to constant mass (within 10 mg) in a tared vessel, at a temperature of 100 ± 2 °C. Use the lowest mass in the calculation.

D.2.3.3 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 bleached lac, the vacuum is left on for a few minutes so as to suck out as much water as possible. It will then be possible to insert a thin spatula under the edge of the paper and remove it from the funnel, without leaving more than traces of the filter-aid adhering to the funnel walls. Remove such traces by wiping with pieces of alcohol-moistened paper, combine these with the main paper and wrap the whole, while still damp, in a large piece of filter paper and bind firmly with fine wire.

Dry this at a gentle heat for a few hours, for example by placing it on the top of an oven at a temperature of 105 °C. When dry, place it in a 26 mm X 60 mm fat-free paper extraction cartridge, which has been previously extracted with chloroform. Transfer the cartridge and wax to any suitable continuous extraction apparatus, such as the standard apparatus for matter insoluble in hot alcohol (see annex B, method I), and extract for 2 h with chloroform.

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

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

3) Schleicher and Schull No. 603 or equivalent is suitable.

Distil off the solvent and dry the wax at a temperature of 100 ± 2 °C to constant mass (within 10 mg) in a tared vessel. Use the lowest mass in the calculation.

D.2.4 Calculation

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

$$\frac{m_2}{m_1\,(100 - M)} \times 10^4$$

where

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

m_2 is the mass, in grams, of the wax;

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

D.3 METHOD II, FOR REFINED BLEACHED LAC

D.3.1 Principle

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

D.3.2 Reagents

D.3.2.1 Alcohol, 95 % (V/V) ethanol.

D.3.2.2 Oxalic acid, dihydrate, analytical grade.

D.3.2.3 Chloroform, redistilled, free from non-volatile residue.

D.3.2.4 Filter-aid[1], previously extracted with chloroform and dried before use.

D.3.3 Procedure

Weigh 49 to 51 g of the "test sample" (see Q.3.1) to an accuracy of 0,1 g and dissolve in 250 ml of alcohol, add about 1 g of oxalic acid and stir until all is dissolved. Then add about 0,5 g of the filter-aid and allow to flocculate and settle overnight at a temperature of 41 ± 1 °C. Then cool to 0 °C and keep at this temperature for at least 1 h. Pour the clear liquid through a Gooch crucible, prepared with an asbestos mat over which a thin layer of the filter-aid has been applied. Wash the sediment of wax and filter-aid from the beaker into the crucible with cold alcohol at 0 °C. Dry the crucible at a temperature of 41 ± 1 °C. Remove the asbestos mat, wipe out the crucible with small pieces of alcohol-moistened paper and wrap the mat and the wiping carefully in a large piece of fat-free filter paper.

Bind securely with fine wire and transfer to a previously extracted 26 mm X 60 mm fat-free paper extraction cartridge and extract in a suitable continuous-extraction apparatus, such as the standard apparatus for matter insoluble in hot alcohol (see annex B, method I), with chloroform for 2h. Distil off the solvent and dry the wax at a temperature of 100 ± 2 °C in a tared vessel, until the loss in mass between successive weighings does not exceed 1 mg. Use the lowest mass in the calculation.

D.3.4 Calculation

As for method I (see D.2.4).

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

ANNEX E

(See clause 9)

DETERMINATION OF ASH

E.1 PROCEDURE

E.1.1 Weigh 3 to 5 g of the "test sample" (see Q.3.1) to an accuracy of 0,01 g; char in a tared porcelain, silica or platinum crucible and ignite at a low heat, not exceeding dull redness, until free from carbon and until the difference between successive weighings does not exceed 1 mg. Use a muffle furnace, if available.

E.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 1 mg. Use the lowest mass in the calculation.

E.2 CALCULATION

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

$$\frac{m_2}{m_1\,(100 - M)} \times 10^4$$

where

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

m_2 is the mass, in grams, of the ash;

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

ANNEX F

(See 10.1)

DETECTION OF ROSIN (HALPHEN-HICKS METHOD)

F.1 REAGENTS

F.1.1 Ethanol, absolute.

F.1.2 Acetic acid, glacial.

F.1.3 Light petroleum spirit, boiling point below 80 °C.

F.1.4 Solution A : One part of phenol by volume dissolved in two parts by volume of carbon tetrachloride.

F.1.5 Solution B : One part by volume of bromine dissolved in four parts by volume of carbon tetrachloride.

F.2 PROCEDURE

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

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

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

ANNEX G
(See 10.2)

DETECTION OF COPALS

G.1 REAGENTS

G.1.1 Denatured alcohol. Add 5 parts by volume of 99 % methanol to 100 parts by volume of 95 % ethanol.

G.1.2 Methanol, 99 %.

G.2 PROCEDURE

G.2.1 Dissolve a weighed portion (10 to 20 g) of the "test sample" (see Q.3.1) in twice its mass of denatured alcohol and filter the solution through filter paper. Transfer approximately 10 ml of the clear filtrate to a large test tube, 150 mm X 20 mm. Nearly fill the test tube with methanol, stopper the tube and mix the contents thoroughly.

G.2.2 Bleached lac free from copals remains clear. Immediate formation of a precipitate indicates the presence of copals.

ANNEX H
(See clause 11)

DETERMINATION OF MATTER SOLUBLE IN WATER

H.1 PRINCIPLE

A known mass of powdered bleached lac is digested with water, made up to a known volume and filtered. The mass that goes into solution is determined by evaporating an aliquot portion of the filtrate to constant mass and calculating for the whole solution.

H.2 PROCEDURE

H.2.1 Finely grind a sufficient quantity of the "test sample" (see Q.3.1) to pass a sieve having a nominal aperture of about 0,25 mm (see Q.4.1). 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.

H.2.2 Filter into a 250 ml one-mark volumetric flask. Wash the residual bleached lac and the filter paper with distilled water and make up to the graduation mark. Transfer a measured volume of the filtrate into a weighed evaporating dish and evaporate to dryness. Dry the residue to constant mass in an oven maintained at a temperature of 100 ± 2 °C.

H.3 CALCULATION

The matter soluble in water, as a percentage by mass, is given by the formula

$$\frac{m_2}{V m_1 (100 - M)} \times 2{,}5 \times 10^6$$

where

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

m_2 is the mass, in grams, of the residue;

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

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

ANNEX J

(See clause 12)

DETERMINATION OF CHLORINE

J.1 PRINCIPLE

The bleached lac is treated with metallic sodium to convert the total chlorine into soluble chloride which is estimated volumetrically.

J.2 REAGENTS

J.2.1 Ethanol, 98 to 100 % (*V/V*).

J.2.2 Metallic sodium

J.2.3 Dilute nitric acid, 1 + 4.

J.2.4 Silver nitrate solution, approximately 0,1 N.

J.2.5 Nitrobenzene, redistilled, free from chlorides.

J.2.6 Standard potassium thiocyanate solution, approximately 0,1 N, but of exactly the same normality as the silver nitrate solution above.

J.2.7 Iron(III) ammonium sulphate indicator, saturated solution acidified with nitric acid.

J.3 PROCEDURE

J.3.1 Weigh 0,4 to 0,6 g of the "test sample" (see O.3.1) to an accuracy of 1 mg and dissolve in 50 ml of ethanol in a 250 ml conical flask. Reflux the solution using a condenser with a tube 700 ± 20 mm long and bore diameter 15 ± 1 mm, while heating the flask on a hot-plate. When all the lac is dissolved and the solution is boiling briskly, add slowly, piece by piece, through the top of the condenser about 3 g of sodium freshly cut into 3 mm cubes.

J.3.2 When the reaction is complete and all the sodium is dissolved, allow the solution to cool. Remove the condenser, add 50 ml of water and transfer the solution to a 500 ml conical flask, rinsing two or three times with a further 50 ml of water. Add a few glass beads to prevent bumping and evaporate the solution on a hot-plate to about half the volume to remove ethanol. Cool and make slightly acid with dilute nitric acid. Add from a burette 10 ml of silver nitrate solution and then add 2 ml of nitrobenzene and 1 ml of iron(III) ammonium sulphate solution. Stopper the flask and shake vigorously to coagulate the precipitate. Titrate the residual silver nitrate with standard potassium thiocyanate solution until a permanent faint reddish-brown coloration appears. Carry out a blank determination using all reagents.

J.4 CALCULATION

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

$$\frac{354{,}6\,(V_1 - V_2)\,T}{m\,(100 - M)}$$

where

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

V_1 is the volume, in millilitres, of standard potassium thiocyanate solution used in the blank determination;

V_2 is the volume, in millilitres, of standard potassium thiocyanate solution used with the sample;

T is the normality of the standard potassium thiocyanate solution;

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

ANNEX K

(See 13.1)

DETERMINATION OF ACID VALUE

K.1 REAGENTS

K.1.1 Phenolphthalein indicator. Dissolve 1,0 g of phenolphthalein in 60 ml of 90 % (*V/V*) ethanol and dilute with water to a final volume of 100 ml.

K.1.2 Neutral ethanol, 95 % (*V/V*); or denatured spirit 95 % (*V/V*), neutralized to phenolphthalein indicator.

K.1.3 Standard alcoholic potassium hydroxide solution, approximately 0,1 N in 95 % (*V/V*) ethanol, or alternatively :

K.1.4 Standard aqueous potassium hydroxide or **sodium hydroxide solution,** 0,1 N.

Prepare a stock concentrated solution by dissolving potassium hydroxide or sodium hydroxide in water in the proportion of 112 g of potassium hydroxide or 85 g of sodium hydroxide to 200 ml of water. Allow the solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the carbonate precipitate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off 175 ml and dilute to 10 l with water. Preserve in a stock bottle provided with a guard tube filled with soda-lime.

Determine the normality by titrating against pure potassium hydrogen phthalate solution, using phenolphthalein indicator. This solution will be approximately 0,10 N, but do not attempt to adjust it to any exact value. Determine its normality to ± 0,001 N and make adjustment to the calculation when using it.

K.2 PROCEDURE

Weigh 0,8 to 1,2 g of the "test sample" (see Q.3.1) to an accuracy of 1 mg and dissolve in 50 ml of neutral ethanol, assisting solution by warming in hot water at a temperature of 80 to 90 °C for a few minutes. When solution is complete, cool and add 10 drops of phenolphthalein indicator. Titrate with standard alcoholic potassium hydroxide solution or with standard aqueous potassium hydroxide or sodium hydroxide solution.

K.3 CALCULATION

Report the result in terms of milligrams of potassium hydroxide per gram of moisture-free sample, calculated according to the formula

$$\text{Acid value} = \frac{5\,610\,V\,T}{m\,(100 - M)}$$

where

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

V is the volume, in millilitres, of standard alkali solution used in the titration;

T is the normality of the standard alkali solution;

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

ANNEX L
(See 13.2)

DETERMINATION OF MINERAL ACID

L.1 REAGENTS

L.1.1 Neutral ethanol, 95 % (*V/V*); or denatured spirit, 95 % (*V/V*), neutralized to phenolphthalein indicator.

L.1.2 Sodium chloride solution, 100 g/l solution.

L.1.3 Bromophenol blue indicator. Warm 0,1 g of bromophenol blue with 3,0 ml of 0,05 N sodium hydroxide solution and 5 ml of 90 % (*V/V*) ethanol; dilute the solution with 20 % (*V/V*) ethanol to make the final volume 250 ml.

L.1.4 Standard sodium hydroxide solution, approximately 0,1 N aqueous solution.

L.1.5 Potassium hydrogen phthalate, containing not less than 99,9 % and not more than the equivalent of 100,1 % of potassium hydrogen phthalate ($C_8H_5O_4K$) and impurities not exceeding 0,001 % of chloride (as Cl), 0,01 % of sulphate (as SO_4), 0,1 % of moisture and 0,05 % of sodium (Na).

L.1.6 Buffer solution, pH 4,0. Dissolve 10,21 g of potassium hydrogen phthalate in freshly boiled and cooled water and make up the volume with the water to 1 000 ml.

L.2 PROCEDURE

Weigh 4,5 to 5,5 g of the "test sample" (see Q.3.1) to an accuracy of 0,01 g and dissolve by warming in 50 ml of neutral ethanol in a 500 ml conical flask. Cool the solution and add, with shaking, 200 ml of freshly boiled and cooled water, followed by 50 ml of sodium chloride solution. Shake intermittently over a period of 1 h, so that the lac is well coagulated. Allow to settle. Filter through hardened close-texture filter paper[1], using a Buchner funnel and filter flask, with the aid of a filter pump; wash with two successive quantities of 25 ml of water. Transfer the filtrate and washings to a 400 ml, tall beaker, add 15 drops of bromophenol blue indicator and titrate with standard sodium hydroxide solution, until the colour matches that of a standard solution, consisting of 350 ml of buffer solution, to which 15 drops of the same indicator have been added.

L.3 CALCULATION

Report the result, to the nearest whole number, as the number of millilitres of 0,1 normal sodium hydroxide solution, required by 100 g of the moisture-free sample, according to the formula

$$\text{Mineral acid} = \frac{VT}{m\,(100 - M)} \times 10^5$$

where

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

V is the volume, in millilitres, of standard sodium hydroxide solution used in the titration;

T is the normality of the standard sodium hydroxide solution;

M is the volatile matter (moisture) content, per cent by mass (see 18.3).

ANNEX M
(see clause 14)

TEST FOR FREE CHLORINE OR PEROXIDES

M.1 REAGENT

Starch-iodide paper.

M.2 PROCEDURE

M.2.1 Grind about 5 g of the "test sample" (see Q.3.1) in a mortar with 20 ml of distilled water. Filter and test the filtrate for free chlorine or peroxides with starch-iodide paper.

M.2.2 If a blue colour does not develop immediately, it shall be taken as indicating absence of free chlorine or peroxides.

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[1] Whatman No. 5 or equivalent is suitable.

ANNEX N

(See clause 15)

DETERMINATION OF ARSENIC

N.1 PRINCIPLE

The sample is taken in a Kjeldahl flask with nitric acid and sulphuric acid. The arsenic present is distilled over and is estimated from the distillate by the intensity of the stain produced on mercuric chloride paper using the Gutzeit apparatus.

N.2 APPARATUS

N.2.1 Distillation apparatus, comprising a Kjeldahl flask, of arsenic-free borosilicate glass, capacity 100 or 200 ml and a condenser.

N.2.2 Gutzeit apparatus. A wide-mouthed bottle, of about 120 ml capacity, is fitted with a rubber bung through which passes a glass tube. The latter, made from ordinary glass tubing, has a total length of 200 mm and an internal diameter of exactly 6,5 mm (external diameter about 8 mm). It is drawn out at one end to a diameter of about 1 mm, and a hole not less than 2 mm in diameter is blown in the side of the tube near the constricted part. The tube is passed through the bung fitting the bottle so that, when inserted in the bottle containing 70 ml of liquid, the constricted end of the tube is above the surface of the liquid and the hole in the side is below the bottom of the bung. The upper end of the tube is cut off square, and is ground smooth.

Two rubber bungs (about 25 mm X 25 mm) each with a hole bored centrally and true, exactly 6,5 mm in diameter, are fitted with a rubber band or spring clip for holding them tightly together. Alternatively, the two bungs may be replaced by any suitable contrivance satisfying the conditions described under the general test.

N.3 REAGENTS

As far as possible, the reagents shall be free from traces of arsenic.

N.3.1 Nitric acid, density 1,42 g/ml.

N.3.2 Dilute nitric acid solution, 3 + 7.

N.3.3 Sulphuric acid, density 1,84 ml.

N.3.4 Dilute sulphuric acid solution, 1 + 8.

N.3.5 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.

N.3.6 Hydrochloric acid, density 1,18 g/ml.

N.3.7 Lead acetate solution, 100 g/l solution in carbon dioxide-free water.

N.3.8 Mercuric chloride paper. Soak pieces of smooth white filter paper, not less than 25 mm in width, in a saturated aqueous solution of mercuric chloride, press to remove superfluous moisture, and dry at about 60° in the dark. The filter paper shall weigh between 65 and 120 g/m^2 and the thickness, in millimetres, of 400 papers shall be approximately equal, numerically, to the substance, in grams per square metre. The mercuric chloride papers should be stored in a stoppered bottle in the dark. Papers which have been exposed to sunlight or to the vapour of ammonia afford a lighter coloured stain or no stain at all, when employed in the arsenic test and shall not be used for the test.

N.3.9 Tin(II) chloride solution, 0,4 % in hydrochloric acid. Dilute 60 ml of the hydrochloric acid (N.3.6) 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 the hydrochloric acid (N.3.6), boil down to the original volume and filter through a fine-grained filter paper. Mix 1 ml of the filtrate with 100 ml of the hydrochloric acid.

N.3.10 Potassium iodide, powder or crystals.

N.3.11 Zinc, granulated.

N.3.12 Standard arsenic solution. Dissolve 0,132 g of arsenic trioxide (As_2O_3) in 50 ml of the hydrochloric acid (N.3.6) and dilute to 100 ml with water. Further dilute, freshly as required, 1 ml of this solution to 100 ml. 1 ml of the resulting solution contains 10 μg of arsenic (as As).

N.4 PROCEDURE

N.4.1 Weigh 4,5 to 5,5 g of the "test sample" (see O.3.1) to an accuracy of 0,01 g, place with 10 ml of the dilute nitric acid solution (N.3.2) in a 100 ml or 200 ml glass or silica Kjeldahl flask, and heat the mixture until any initial vigorous reaction subsides and ceases. Cool and add gradually 10 ml of sulphuric acid (N.3.3), at such a rate as to avoid excessive frothing or heating (10 min are usually required) and continue the heating. Add to the hot solution 5 ml of nitric acid (N.3.1) in small portions, and boil until colourless. If necessary, add more nitric acid in small portions. Note for the purpose of the blank determination the total amount 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.

Boil the solution, without inserting the condensing arm, until the bulk is reduced to about 10 ml or until white fumes appear; cool, dilute and again boil down to 10 ml; cool and add 7,0 ml of water. Thoroughly cool the liquid and add 5 g of the chloride-hydrazine-bromide mixture (N.3.5), followed by 10 ml of the hydrochloric acid.

N.4.2 Fit the condenser quickly and distil the liquid into a mixture of 10 ml of water and 2 ml of nitric acid (N.3.1). Then evaporate the distillate to dryness on the water bath and evaporate the residue twice to dryness with 5 ml of water to remove nitric acid. Dissolve the final residue by warming in 3 ml of sulphuric acid (N.3.3), cool and dilute with approximately 50 ml of water.

N.4.3 Lightly pack the glass tube of the Gutzeit apparatus with cotton wool, previously moistened with the lead acetate solution (N.3.7) and dried, 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, so that the ground end of the tube is flush with the larger end of the bung. Place a piece of mercuric chloride paper (N.3.8) flat on the top of the bung and the other bung over it. Secure by means of spring clips in such a manner that the borings of the two bungs meet to form a true tube of 6,5 mm diameter, interrupted by a diaphragm of mercuric chloride paper.

N.4.4 Transfer the whole of the solution to the wide-mouthed bottle of the Gutzeit apparatus, add 15 ml of the tin(II) chloride solution (N.3.9) and 1 g of potassium iodide (N.3.10). Then add 10 g of zinc (N.3.11). Quickly place the prepared glass tube in position. Allow the reaction to continue for 40 min. Remove the piece of mercuric chloride paper at the end of this period.

NOTE — The reaction may be accelerated by placing the apparatus on a warm surface, care being taken that the mercuric chloride paper remains quite dry throughout the test. The most suitable temperature for performing the test is generally about 40 °C, but this temperature should be adjusted to obtain a regular, but not too violent, evolution of gas. The tube should be washed with hydrochloric acid, rinsed with water and dried between sucessive tests.

N.4.5 At the same time, produce standard stains of arsenic by mixing 50 ml portions of water, 10 ml portions of the tin(II) chloride solution and appropriate volumes of standard arsenic solution (N.3.12) and treating the mixtures as described above.

N.4.6 Compare any stain produced by the sample on the mercuric chloride paper by daylight with the standard stains. If the stain produced in the test exceeds that equivalent to 15 μg of arsenic (as As), repeat the determination, making the solution obtained in N.4.2 to a known bulk using the dilute sulphuric acid (N.3.2), and taking a suitable aliquot in the procedure given in N.4.4.

N.4.7 Perform a blank determination under the same conditions and using the same reagents; no visible stain should be produced.

N.5 CALCULATION

Express the arsenic content as parts of arsenic (as As) per million parts of the bleached lac freed from volatile matter.

ANNEX P

(See clause 16)

DETERMINATION OF LEAD

P.1 PRINCIPLE

From the prepared sample the lead content is determined colorimetrically by matching the colour of lead sulphide obtained from the sample with that obtained from standard lead solution.

P.2 REAGENTS

As far as possible, the reagents shall be free from traces of lead.

P.2.1 Hydrochloric acid, density 1,18 g/ml.

P.2.2 Sulphuric acid, density 1,84 g/ml.

P.2.3 Citric acid, solid.

P.2.4 Ammonia solution, density 0,88 g/ml or diluted as required.

P.2.5 Potassium cyanide solution, 100 g/l aqueous solution.

P.2.6 Diphenyl thiocarbazone (dithizone) solution, 1 g/l solution in chloroform (P.2.8), freshly prepared.

P.2.7 Dilute hydrochloric acid, approximately 0,1 N.

P.2.8 Chloroform, redistilled.

P.2.9 Standard lead solution

Two reference solutions of lead nitrate required in this test are as given below :

P.2.9.1 Standard strong lead solution, obtained by dissolving 0,16 g of lead nitrate [$Pb(NO_3)_2$] in 50 ml of dilute nitric acid (1 + 3) and making up to 100 ml with water.

P.2.9.2 Standard dilute lead solution, prepared freshly, before the test, by diluting 1 ml of standard strong lead solution (P.2.9.1) to 100 ml with water.

P.2.10 Ammonium acetate, solid.

P.2.11 Sodium sulphide solution, 100 g/l.

P.3 PROCEDURE

P.3.1 Weigh 4,5 to 5,5 g of the "test sample" (see Q.3.1) and char in a porcelain or silica basin 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 hydrochloric acid (P.2.1) and boil gently for 5 minutes. 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 sulphuric acid (P.2.2) and evaporate to fuming. Cool, dilute with about 50 ml of water, add 2 g of citric acid (P.2.3) and just neutralize with ammonia (P.2.4). Add 1 ml of potassium cyanide solution (P.2.5) and transfer the whole to a separating funnel. The total volume should be 100 to 150 ml.

P.3.2 Extract the liquid with dithizone solution (P.2.6). 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.

P.3.3 Take 10 ml of water in another separating funnel and wash each extract with this water. If suspended matter is present in the chloroform extracts, 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 dilute hydrochloric acid (P.2.7). Combine the acid extracts in a separating funnel, wash once or twice with 10 ml of chloroform (P.2.8) and filter through a previously wetted filter paper into a 100 ml one-mark graduated flask. Make up the volume of the filtrate to 100 ml with dilute hydrochloric acid (P.2.7) and use this as the test solution.

P.3.4 Transfer a suitable volume of the test solution to a Nessler cylinder. Add 2 g of ammonium acetate (P.2.10), followed by ammonia solution (P.2.4) until just alkaline, and then 1 ml of potassium cyanide solution. Dilute to 50 ml with water, add 2 drops of sodium sulphide solution (P.2.11) and match the colour against a set of standards prepared in the same way using not more than 10 ml of standard solution (P.2.9.2).

P.3.5 A blank determination shall be run under the same conditions, on the same reagents and by the same person but without the sample.

P.4 CALCULATION

Express the lead content as parts of lead (Pb) per million parts of the bleached lac freed from volatile matter.

ANNEX Q
(See clause 19)

SAMPLING

Q.1 DRAWING OF SAMPLES

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

Q.1.1 Only original, unopened packages of bleached lac shall be sampled.

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

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

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

Q.1.5 Free-flowing bleached lac

Take samples 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. Thoroughly mix the material, heap and quarter along two diameters which intersect at right angles, and mix two opposite quarters. 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). Place these samples in airtight containers, and seal and label them accordingly. Treat the samples as described in Q.3.2. Treat the other half of the material as described under Q. 2.1 to form the "laboratory sample".

Q.1.6 Blocky or matted bleached lac

Take samples 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. Thoroughly mix the material, heap and quarter along two diameters which intersect at right angles, and mix two opposite quarters. 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). Place these samples in airtight containers, and seal and label them accordingly. Treat the samples as described in Q.3.2. Roughly grind the other half of the material so as to pass a sieve having a nominal aperture of 6,3 mm (see Q.4) and then treat it as described under Q.2.1 to form the "laboratory sample".

Q.1.7 Bleached lac in hanks, bars or flats

Draw two hanks, bars or flats from different places in each package and break a large piece from each, by suitable means, so as to yield a total of 5 kg of material consisting of approximately equal portions from each hank, bar or flat. Quickly crush the composite sample to lumps of about 25 mm^3. Thoroughly mix the material, heap and quarter along two diameters which intersect at right angles, and mix two opposite quarters. One half of the material may, if necessary, be subdivided by the normal process of quartering (without crushing) to form a number of samples which are known as "samples as received" and shall be used for the determination of volatile matter (moisture). Place these samples in airtight containers, and seal and label them accordingly. Treat the samples as described in Q.3.2. Roughly grind the remaining half of the material so as to pass a sieve having a nominal aperture of 6,3 mm (see Q.4) and then treat it as described under Q.2.1 to form the "laboratory sample".

Q.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.

Q.2.1 Thoroughly mix the material for the laboratory sample, as obtained under Q.1.5, Q.1.6 or Q.1.7, and heap and quarter along two diameters which intersect at right angles. Mix two opposite quarters and grind to pass entirely through a sieve having a nominal aperture of about 2 mm (see Q.4). Then thoroughly mix the material and quarter so as to yield four samples of approximately 250 g each. Place these four samples in airtight containers, seal, label "laboratory sample" and send to the interested parties.

Q.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 given on a label attached to each sample.

Q.3 PREPARATION OF TEST SAMPLES

Q.3.1 Grind the laboratory samples to pass entirely through a sieve whose nominal aperture does not exceed 0,425 mm (see Q.4). Thoroughly mix this finely ground material, place in an airtight container and label "unconditioned test sample". Before this material is used for any analytical work, bring it to less than 6 % volatile matter (moisture) content by exposing it to the atmosphere for at least 24 h at room temperature and then desiccating overnight over fused calcium chloride. The material is then known as the "test sample".

Q.3.2 Treat the "samples as received" (see Q.1.5, Q.1.6 and Q.1.7) according to the method described in annex A.

Q.4 NOTE ON SIEVE SIZES

Table 2 gives the international and some of the national sieve designations and mesh apertures corresponding approximately to the sizes specified in the preceding clauses and in annexes A and H.

TABLE 2 — Sieve designations and mesh apertures corresponding to specified sizes

Size specified mm	ISO		U.S.A. ANSI		UNITED KINGDOM BSI		FRANCE AFNOR		GERMANY DNA		INDIA ISI	
	Designation	Size mm	Designation	Size mm	Designation	Size mm	Designation	Size mm	Designation	Size mm	Designation	Size mm
1) 6,3 (Perforated plate)	6,3 mm	6,3	6,35 mm	6,35	6,30 mm	6,30	6,3 mm	6,3	6,3 mm	6,3	6,3 mm (PS)	6,30
2) 2,00 (Woven)	2,00 mm	2,00	2,00 mm	2,00	2,00 mm	2,00	2 mm	2	2 mm	2	2,00 mm	2,00
3) 0,425 (Woven)	400 µm	0,400	420 µ	0,420	425 µm	0,425	400 µm	0,400	0,4 mm	0,400	425 - micron	0,425
4) 0,25 (Woven)	250 µm	0,250	250 µ	0,250	250 µm	0,250	250 µm	0,250	0,25 mm	0,250	250 - micron	0,250

INTERNATIONAL/NATIONAL STANDARDS

ISO — International Organization for Standardization. *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.* ISO 565-1972.

ANSI — American National Standards Institute. *Sieves for testing purposes — Wire cloth sieves, round-hole and square-hole plate screens or sieves.* ANSI Z 23.1-1961.

BSI — British Standards Institution. *Test sieves.* B.S. 410-1969.

AFNOR — Association Française de Normalisation. *Tamis et tamisage — Toiles métalliques et tôles perforées dans les tamis de contrôle — Dimensions nominales des ouvertures.* ["Sieves and sieving — Woven metal wire cloth and perforated plates in test sieves — Nominal sizes of apertures".] NF X 11-504, December 1970.

DNA — Deutscher Normenausschuss :

a) *Siebböden — Drahtsiebböden für Prüfsiebe — Masse.* ["Screening surfaces — Wire screens for test sieves — Dimensions".] DIN 4 188, Blatt 1, April 1969.

b) *Lochplatten — Rundlochung — Masse.* ["Perforated plates — Round holes — Dimensions".] DIN 24 041, April 1973.

ISI — Indian Standards Institution. *Test sieves.* IS : 460-1962.