INTERNATIONAL STANDARD 57

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Bleached lac — Specification

Gomme laque blanche — Spécifications

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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 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	Netherlands	Thailand
Egypt, Arab Rep. of	Poland	United Kingdom
India	Portugal	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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Annexes

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	6,0	6,0	A
		Other conditions	As agreed between purchaser and vendor		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

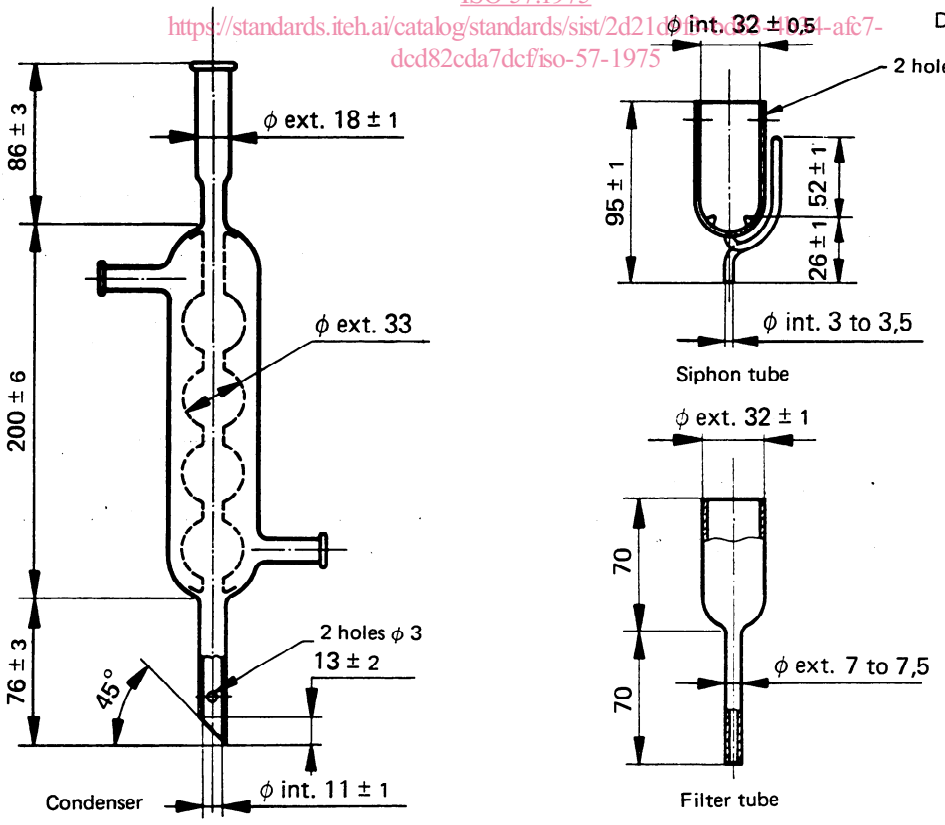

FIGURE 1 — **Extraction apparatus for determining matter insoluble in hot alcohol — Method I**

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

Dimensions in millimetres

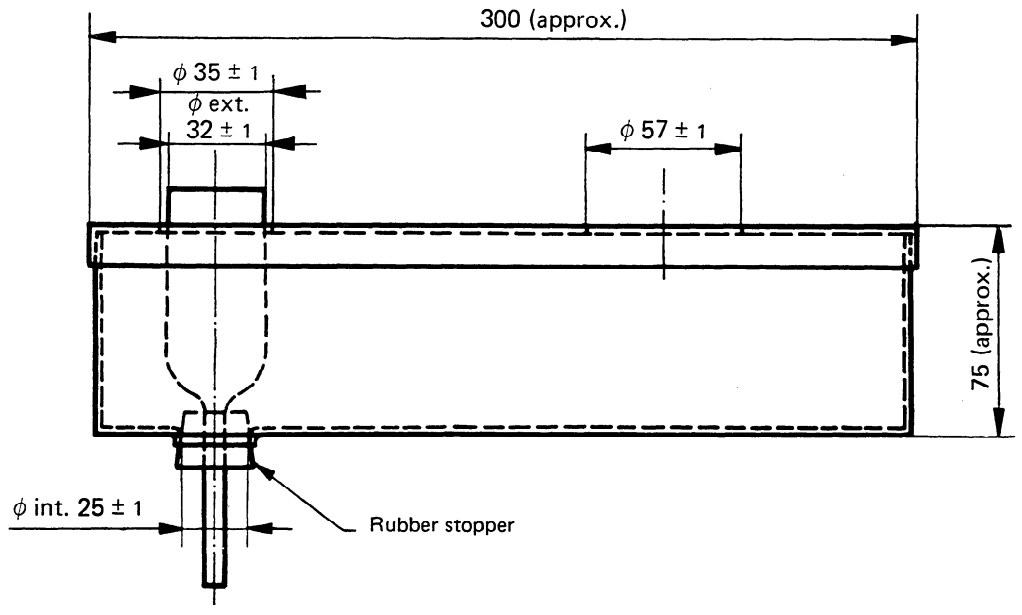

FIGURE 2 — **Hot water bath for insoluble matter test — Method I**

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/m²), 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.