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IS 3349 (1989): Perfumery Materials - Resinoid Benzoin, Pure [PCD 18: Natural and Synthetic Fragrance Materials]

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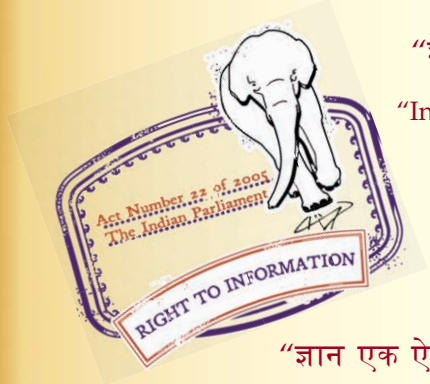

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IS 3349 : 1989
(Reaffirmed 2000)

Indian Standard

PERFUMERY MATERIALS — RESINOID BENZOIN, PURE — SPECIFICATION

(First Revision)

भारतीय मानक

सुगंधकारक सामिग्री — रेजिनायड बेंजोइन, शुद्ध — विशिष्टि

(पहला पुनरीक्षण)

UDC 665·944·22

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1989

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 25 February 1989, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Resinoid benzoin, pure is a product obtained by solvent extraction of the resin exuded by certain species of trees belonging to the family Styraceae. Earlier there were two principal sources of resinoid benzoin, namely, benzoin Siam and benzoin Sumatra. The former was usually derived from *Styrax tonkinensis* Pierre. Craib ex Hartwich and the latter was generally from *Styrax benzoin* Dryand. The main difference between benzoin Siam and benzoin Sumatra is that the former is rich in derivatives of benzoic acid while the latter is rich in those of cinnamic acid.

This standard originally covered resinoid produced from both types of resins, namely, benzoin Siam and benzoin Sumatra. Since resin benzoin Siam is no longer commercially available in perfumery industry, the specification for resinoid produced from this variety which was covered as Type 1 has been deleted in this revision, resinoid produced from *Styrax benzoin* Dryand, previously designated as Type 2 is being retained only. For extraction, the solvents commonly employed are ethyl alcohol, acetone and benzene.

Resinoid benzoin, pure is a very resinous, hard and brittle material. Consequently, it is often used after dilution with a suitable solvent, such as, diethyl phthalate, benzyl benzoate and isopropyl myristate to facilitate the ease of handling. This standard does not cover these diluted varieties as well as the resinoids produced from made up varieties of gum benzoin which contain other natural gums as adulterants.

Resinoid benzoin is used as an ingredient in perfume compounds for soaps, cosmetics and toilet goods, and *AGARBATTIS*.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PERFUMERY MATERIALS — RESINOID BENZOIN, PURE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard (First Revision) prescribes the requirements and the methods of sampling and test for resinoid benzoin, pure.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 323 : 1959	Specification for rectified spirit (*revised*)
IS 326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials (*second revision*)
(Part 7) : 1980	Determination of acid value (*second revision*)
(Part 8) : 1980	Determination of ester value, content of esters and combined alcohols (*second revision*)
IS 336 : 1973	Specification for ether (*second revision*)
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 2284 : 1988	Methods for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
IS 6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

3 TERMINOLOGY

3.1 For the purpose of this standard, definitions given in IS 6597 : 1988 shall apply.

4 REQUIREMENTS

4.1 Description

Resinoid benzoin, pure, shall be obtained by solvent extraction of the resin exuded by the following species of trees belonging to the family Styraceae:

a) *Styrax benzoin* Dryand, and

b) *Styrax benzoides* Craib.

4.1.1 The material shall be a very resinous to hard brittle mass, free from adulterants and diluents.

4.1.2 The material shall be examined for its colour and clarity as prescribed in IS 326 (Part 2) : 1980.

4.2 Identification

The material shall pass the tests prescribed in Annex A.

4.3 Olfactory Assessment

The material shall also be tested olfactorily and specially for by-notes as prescribed in IS 2284 : 1988.

4.3.1 For this test, the material shall be examined in its original form and also after dissolving in diethyl phthalate at a concentration of 10 percent (*m/v*).

4.4 The material shall also comply with the requirements given in Table 1.

5 PACKING

The material shall be supplied in air-tight containers, preferably of aluminium or tin-lined as agreed to between the purchaser and the supplier.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1) : 1984.

7 TESTS

7.1 Tests shall be conducted as prescribed in **4.1, 4.2, 4.3** and in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Resinoid Benzoin, Pure

(*Clauses* 4.4 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Colour	Orange brown to dark brown	—	IS 326 (Part 2) : 1980
ii)	Odour	Intense, sweet balsamic, slightly fermentative	—	IS 2284 : 1988
iii)	Acid value	122 to 146	—	IS 326 (Part 7) : 1980
iv)	Easter value*	70 to 84	—	IS 326 (Part 8) : 1980
v)	Total aromatic acids (as cinnamic acids percent by mass)	30 to 60	B	—

*One hour refluxing is adequate for complete saponification.

ANNEX A

(*Clause* 4.2)

IDENTIFICATION OF RESINOID BENZOIN, SUMATRA

A-1 REACTION WITH SULPHURIC ACID

A-1.1 Treat about 250 mg of the material with 5 ml of ether, decant about 1 ml of the solution into a porcelain dish, and add to it 2 to 3 drops of concentrated sulphuric acid. Appearance of deep reddish brown colour confirms resinoid benzoin, Sumatra variety.

A-2 REACTION WITH FERRIC CHLORIDE

A-2.1 Triturate about 100 mg of the material with 5 ml of ethyl alcohol. Filter and add to the filtrate 0·5 ml of a 5 percent solution of ferric chloride in alcohol if a yellowish brown colour is developed it confirms resinoid benzoin, Sumatra variety.

A-3 REACTION WITH PERMANGANATE

A-3.1 Take about 500 ml of the material in a test tube, add 10 ml of 0·1 N potassium permanganate solution and heat the odour of benzaldehyde confirms resinoid benzoin, Sumatra variety.

ANNEX B

[*Table* 1, Item (v)]

DETERMINATION OF TOTAL AROMATIC ACIDS

B-1 Outline of the Method

B-1.1 The material is refluxed with alcoholic potassium hydroxide solution, the residue after evaporation of the alcohol is dispersed in hot water and acidified. The liberated aromatic acids are extracted through a process involving extraction with ether, neutralization with sodium bicarbonate and repeated extraction with several portions of chloroform. The purified aromatic acids are dissolved in rectified spirit and estimated by titration with standard sodium hydroxide solution using phenol red as indicator.

B-2 REAGENTS

B-2.1 Alcoholic Potassium Hydroxide Solution, 0·5 N, approximately.

B-2.2 Magnesium Sulphate Solution, 3 per cent (*m/v*) aqueous solution.

B-2.3 Dilute Hydrochloric Acid, 1:1 (by volume).

B-2.4 Ether, solvent grade (conforming to IS 336 : 1973).

B-2.5 Sodium Bicarbonate Solution, 5 percent (*w/v*) aqueous solution.

B-2.6 Chloroform

B-2.7 Ethyl Alcohol, 95 percent by volume or rectified spirit (conforming to IS 323 : 1959); neutral to phenol red.

B-2.8 Phenol Red Indicator Solution

Dissolve 0·1 g of phenol red in 100 ml of rectified spirit (conforming to IS 323 : 1959).

B-2.9 Standard Sodium Hydroxide Solution, 0·1 N.

B-3 PROCEDURE

B-3.1 Weigh accurately about 1·5 g of the material in a 125-ml flask; and 25 ml of the alcoholic potassium hydroxide solution, attach a reflux condenser and boil on a water-bath for one hour, frequently rotating the contents of the flask. Evaporate the alcohol gently, and warm the residue with 50 ml of hot water until it is uniformly dispersed. Cool the mixture and add 80 ml of water and 50 ml of magnesium sulphate solution. Mix thoroughly, set aside for ten minutes, and filter. Wash the residue with 20 ml of water, acidify the mixed filtrate and washings with hydrochloric acid and shake with four 40-ml portions of ether. Separate the ethereal solutions and reject the aqueous portion. Shake the mixed ethereal solutions with two 20-ml portions and three 10-ml portions of sodium bicarbonate solution, separating and washing each aqueous layer with 20 ml of ether. Reject the ethereal layer, acidify the mixed aqueous solution with hydrochloric acid, then shake successively with 30, 20, 20 and 10 ml of chloroform. Separate and filter each chloroform layer through a plug of cotton wool on which a layer of anhydrous sodium sulphate has been placed. Evaporate the chloroform on a water bath in a current of air, stopping the evaporation as soon as the last trace of chloroform has been removed.

B-3.2 Add 10 ml of ethyl alcohol to the residue and warm until it is dissolved. Cool and titrate with standard sodium hydroxide solution using phenol red as indicator.

B-4 CALCULATION

Calculate the total aromatic acids in the material as follows:

Total aromatic acids expressed as cinnamic acid ($C_9H_8O_2$), percent by mass

$$= \frac{14{\cdot}82\ V\ N}{M}$$

where

V = volume in ml of standard sodium hydroxide solution used,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

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