IS 101-9-2 (1993): Methods of sampling and test for paints, varnishes and related products, Part 9: Tests for lacquers and varnish, Section 2: Rosin test [CHD 20: Paints, Varnishes and Related Products]

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IS 101 (Part 9/Sec 2) : 1993
(Reaffirmed 2004)

भारतीय मानक

रोगनों, वार्निशों और सम्बद्ध उत्पादों के नमूने लेने और परीक्षण की पद्धतियां

भाग 9 लैकर तथा वार्निश के परीक्षण

अनुभाग 2 रोज़िन परीक्षण

(तीसरा पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 9 TESTS FOR LACQUERS AND VARNISH

Section 2 Rosin Test

(Third Revision)

UDC 667·61 : 620·1

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

June 1993 Price Group 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Paints (Other than Industrial Paints) and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Methods of sampling and test for varnishes and lacquers were first published in 1952 as IS 197 and subsequently revised in 1969. During the third revision of IS 101, 'Methods of sampling and test for paints, varnishes and related products', the scope of the standard was enlarged to cover the sampling and test of varnishes and lacquers also. The concerned Sectional Committee, therefore, decided to cover this test method under IS 101 so that all the test methods for paints and allied products are covered under one standard. This standard thus supersedes **19** of IS 197.

This standard is one of series dealing with sampling and testing of paints, varnishes and related products.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard
METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 9 TESTS FOR LACQUERS AND VARNISH

Section 2 Rosin Test

(Third Revision)

1 SCOPE

This standard prescribes the method to determine the freedom from rosin in the packaged paint, varnish and related products.

2 DETERMINATION OF FREEDOM FROM ROSIN (ROSIN COLOHONY)

The material shall be tested for freedom from rosin by either of the following two methods:

a) Liebermann-Starch test; and

b) Halphen-Hicks test.

2.1 Liebermann-Starch Test

2.1.0 *General*

This test method covers procedures for the qualitative detection of rosin in varnishes and extracted vehicle from paints by the Liebermann Starch Test. Rosin may be present as either free rosin (abietic acid), esterified rosin or as metal salt.

2.1.1 *Reagents*

2.1.1.1 *Acetic anhydride*

2.1.1.2 *Sulphuric acid reagent*

Add 35·7 ml of concentrated sulphuric acid (H_2SO_4 sp gr 1·84) slowly to 34·7 ml of water and cool to room temperature. Store in a glass-stoppered bottle.

2.1.1.3 *Alcoholic potassium hydroxide*

Dissolve 66 g of AR grade KOH in 1 litre of absolute alcohol in the standard measuring flask.

2.1.1.4 *Alcohol and benzene wash solution*

Mix one part by volume of absolute alcohol and three part by volume of Benzene.

2.1.2 *Procedure*

2.1.2.1 *Liebermann-starch test (for shades other than red and blue of synthetic enamels)*

Place 0·1 to 0·2 g of the sample in a test tube, and add 15 ml of acetic anhydride. Heat gently until the specimen is dissolved or dispersed. Cool and filter with an ashless rapid filter paper, into a clean test tube. Place a few drops of the clear solution in a depression of the spot plate, and add one drop of sulphuric acid reagent, so that the acid will mix slowly with the filtrate. If rosin is present, a fugitive violet colour develops immediately. A pink or brown colouration should be ignored. A control specimen containing rosin should be run simultaneously.

2.1.2.2 *Liebermann-Starch test (for red and blue shades of synthetic enamel)*

2.1.2.2.0 Identification of rosin and rosin derivatives in case of red and dark blue shades of synthetic enamels wherein organic red pigments are used and bleeding of pigments is observed, the following procedure is to be adopted for the removal of red/blue colour of the binder which interferes in the rosin detection, while performing Liebermann-Starch test.

2.1.2.2.1 Take about 15-20 ml vehicle (extracted from the paint using extracting solvent as per IS 101) in a 250-ml round bottom flask. Add 25 ml benzene and 30 ml of alcoholic KOH solution. Reflux the contents for one and half hours over a water bath using air condenser.

2.1.2.2.2 Remove the flask from the water bath and cool the flask by means of running tap water. Filter the contents of the flask through ashless rapid filter paper using Buchner flask. Washing of the precipitate is done with 25 ml alcohol and benzene wash solution.

2.1.2.2.3 Acidify the filtrate collected in the Buchner flask with 25 ml of 5 percent H_2SO_4 (W/V) or with concentrated HCl, 50 ml of Petroleum ether for further dilution. Shake well and transfer the contents of the Buchner flask into a separating funnel. Wash the Buchner flask with 20-30 ml water and transfer the washings into the separating funnel.

2.1.2.2.4 Shake the contents of the separating funnel well and allow it to stand for 15-20 minutes. Discard the lower aqueous layer. The rosin, fatty acids and unsaponifiables pass into

the upper non-aqueous layer. Collect the non-aqueous layer in an evaporating dish. Evaporate the excess of benzene on the hot water bath and use this concentrate for performing Liebermann-Starch test as per **2.1.2.1**.

2.2 Halphen-Hicks Test

2.2.0 *Outline of the Method*

Acetylated and solvent extracted material with rosin as impurity when treated with phenol, develops specific colour with bromine vapours. This reaction is used as basis of detection of rosin.

2.2.1 *Reagents*

2.2.1.1 *Absolute alcohol*

2.2.1.2 *Glacial acetic acid*

2.2.1.3 *Petroleum hydrocarbon solvent*

2.2.1.4 *Solution A*

One part of phenol dissolved in two parts of carbon tetrachloride, volume by volume.

2.2.1.5 *Solution B*

One part of bromine dissolved in four parts of carbon tetrachloride, volume by volume.

2.2.2 *Procedure*

2.2.2.1 Place about 2 g of the material in a 250-ml conical flask, add 10 ml of absolute alcohol or acetic acid and shake until solution is complete. Add slowly and with continuous agitation, 50 ml of petroleum hydrocarbon solvent. Then add 50 ml of water in exactly the same manner, transfer to a small separating funnel, and allow it to stand until the petroleum hydrocarbon solvent separates. Draw off the water layer, wash the petroleum hydrocarbon layer once with water and extract through a paper dry filter into a round-bottom evaporating dish. Evaporate the extract to dryness on steam-bath.

2.2.2.2 Add 1 to 2 ml of solution *A* to the residue left after evaporation (*see* **2.2.2.1**) of the solution in petroleum hydrocarbon solvent 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 bromine vapours from solution *B*.

2.2.2.3 A decidedly purple or deep indigo blue colour indicates the presence of rosin.

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