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IS 101-8-4 (1993): Methods of sampling and test for paints, varnishes and related products, Part 8: Tests for pigments and other solids, Section 4: Phthalic anhydride [CHD 20: Paints, Varnishes and Related Products]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

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

भाग 8 वर्णकों और अन्य ठोसों के परीक्षण

अनुभाग 4 थैलिक एनहाईड्राईड

(तीसरा पुनरीक्षण)

Indian Standard

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

PART 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 4 Phthalic Anhydride

(*Third Revision*)

UDC 667-612 : 543 : 547-584

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FOREWORD

This Indian Standard (Part 8/Sec 4) (Third Revision) 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.

This standard is one of series dealing with methods of 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 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 4 Phthalic Anhydride

(Third Revision)

1 SCOPE

This standard (Part 8/Sec 4) prescribes the method to determine the phthalic anhydride content in the paint.

2 APPARATUS

2.1 Flask and Condenser

A 500-ml long-necked flask fitted with water condenser. The joint between the flask and the condenser shall be a taper ground-glass joint.

2.2 Desiccator, with concentrated sulphuric acid as desiccant.

3 REAGENTS

3.1 Toluene

3.2 Alcoholic Potassium Hydroxide Solution

Dissolve 66 g of potassium hydroxide in 1 000 ml of absolute alcohol.

3.3 Ether — Anhydrous.

3.4 Alcohol-Toluene Wash Solution — 1 : 3 (v/v).

3.5 Hydrochloric Acid — 0.1 N.

4 PROCEDURE

4.1 Weigh from a closed container, a sample of the resin or resin solution sufficient to yield 0.8 to 1.2 g of potassium alcohol phthalate into a 500-ml long-necked flask. Add 150 ml of benzene, warm if necessary over a water-bath and bring into solution. Add 60 ml of alcoholic potassium hydroxide solution. Reflux for one hour over a water bath using the condenser. Remove the flask from the water-bath, rinse down the inside of the condenser with a few millilitres of alcohol-toluene wash solution. Remove the condenser and stopper the flask with soda-lime guard tube and cool the flask to below 10°C.

Filter the contents through a weighed sintered G4 glass crucible, when cool. Use alcohol-toluene wash solution to transfer the precipitate completely from the flask to crucible. Wash the precipitate with successive portions of alcohol-toluene wash solution until a few millilitres of wash solution shows no signs of alkalinity to phenolphthalein. Do not allow to draw air through the crystals as they are hygroscopic.

4.2 Finally wash the precipitate with 25 ml of ether. Wipe the outside of the crucible with a clean cloth and place in an oven at 60°C for one hour (see Note). Cool to room temperature in a desiccator, weigh and calculate as follows:

NOTE — The precipitate is alcoholate and alcohol of crystallization may be driven off on prolonged heating. However, it is safe to dry up to 60°C for one hour.

$$\text{Phthalic anhydride, percent by mass} = \frac{M_1 \times 0.5136}{M_2} \times 100$$

where

M_1 = Mass, in g, of the precipitate obtained;
and

M_2 = mass, in g, of the material taken for test.

4.3 Alternately heat the precipitate of alcoholate at 150°C for 2 hour. All alcoholate molecule will be driven off and the precipitate may be weighed as $C_6H_4(COOK)_2$ and phthalic anhydride calculated as follows:

$$\text{Phthalic anhydride, percent by mass} = \frac{M_1 \times 0.6116 \times 100}{M_2}$$

where

M_1 = Mass, in g, of the precipitate obtained;
and

M_2 = Mass, in g, of the material taken for test.

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