

**Telefax : 011 23236428**  
**E mail : chd@bis.org.in**

DRAFT IN  
WIDE CIRCULATION

DOCUMENT DESPATCH ADVICE

Our Ref: **CHD 20/T- 101**  
Date : 28 06 2010

TECHNICAL COMMITTEE: Paints, Varnishes and Related Products Sectional Committee,  
CHD 20

---

ADDRESSED TO:

- 1. All members of Paints, Varnishes and Related Products Sectional Committee, CHD 20**
- 2. All others interested**

Dear Sir(s),

Please find enclosed the following draft Indian Standard:

- a) CHD 20 (1805) C** IS 101 (Part 8/ Sec 4):1988 Methods of sampling and test for paints, varnishes and related products Part 8 Tests for pigments and other solids  
Sec 4 Phthalic Anhydride (*Fourth Revision*)

Kindly examine the draft Indian Standard and forward your views stating any difficulties which you are likely to experience in your business or profession, if these drafts are finally adopted as National Standard.

**Last date for Comment is 30 09 2010 .**

Comments if any, may please be made in the format as given overleaf and mailed to the undersigned at the above address.

In case no comments are received or comments received are of editorial nature, you will kindly permit us to presume your approval for the above document as finalized. However, in case of comments of technical in nature are received then it may be finalized either in consultation with the Chairman, Sectional Committee or referred to the Sectional Committee for further necessary action if so desired by the Chairman, Sectional Committee.

The document is also hosted on BIS website **www.bis.org.in**.

Thanking you,

Yours faithfully,

**(E. Devendar)**  
**Sc. F & Head (Chemicals)**

**Enclo : as above**

**BUREAU OF INDIAN STANDARDS**

**Draft 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  
( Fourth Revision )**

**(Not to be reproduced without the permission of BIS or used as STANDARD)**

---

**Last date for receipt of comments is 30 09 2010**

---

**0 FOREWORD**

(Formal clause will be added later on)

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

**0.2** This Standard was first published in 1993. In this version method of separation of resin by Centrifuge Method is provided which was not present in the 1993 version of the test method. In earlier version there was confusion about the mass of the sample taken for the test i.e. whether it is of the total paint sample taken for the test or of the non-volatile vehicle. In this version mass of the Resin has been taken into consideration for test. This has been done to remove any confusion about identity of the material being weighed. Non-volatile of the resin solution has been included in the final calculation since phthalic anhydride percent by mass is calculated on non-volatile basis.

**0.3** 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) '.

**1 SCOPE**

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

**2 APPARATUS**

- 2.1** Centrifuge tube, 50 ml, Heavy – walled.
- 2.2** Water Bath
- 2.3** Air Oven, Capable of being maintained at  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .
- 2.4** Analytical Balance, Sensitive upto 0.1 mg.

- 2.5 Centrifugal machine, Capable of swirl at minimum 5000 rpm.
- 2.6 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.
- 2.7 Desiccator : With concentrated Sulphuric Acid as desiccant.
- 2.8 Evaporating Disc, 500 ml capacity
- 2.9 G4 Glass Crucible
- 2.10 Soda Lime Guard Tube.

### 3 REAGENTS

- 3.1 Benzene, See IS 534 : 1992
- 3.2 Methyl Alcohol, See IS 517 : 1986
- 3.3 Acetone, See IS 170 : 2004
- 3.4 Petroleum Ether, See IS:1745 : 1978
- 3.5 Toluene, LR Grade
- 3.6 Alcoholic Potassium Hydroxide Solution : Dissolve 66 gm of Potassium Hydroxide in 1000 ml of Absolute Alcohol.
- 3.7 Ether – Anhydrous, LR Grade
- 3.8 Alcohol–Toluene Wash Solution — 1:3 (V/V)
- 3.9 Hydrochloric Acid — 0.1 (N)

### 4 PROCEDURE

- 4.1 Weigh accurately 15 to 20 gm of the well mixed material into a weighed centrifuge tube. Centrifuge the mixture as per IS:101 (Part-8/Sec.2) : 1990 to isolate the resin part. Collect the resin part in evaporating disc and concentrate, add some boiling chips during this process.
- 4.2 Calculate the non-volatile content of the resin as per IS:101 (Part-8/Sec.2) : 1990.
- 4.3 Weigh the resin solution (M2) sufficient to yield 0.8 to 1.2 gm 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 ml of alcohol-toluene wash solution. Remove the condenser and stopper of the flask with Soda Lime Guard Tube and cool the flask to below 20°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 sign of alkalinity to Phenolphthalein. Do not allow to draw air through the crystals as they are hygroscopic.
- 4.4 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 1 hour. 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 1 hour. Cool to room temperature in a desiccators, weigh (M1) and calculate as follows:  
Phthalic Anhydride contents by Mass:  $(M1 \times 51.36) / (M2 \times \%NVM \text{ of Resin Soln})$

**4.4.1** The result is represented as the % of Phthalic Anhydride in solid resin obtained from the paint.

M1 → Mass in gm. of the precipitate obtained.

M2 → Mass in gm. of the Resin taken for test.

**4.5** Alternately heat the precipitate of alcoholate at 150°C for 2 Hrs. All alcohol molecules will be driven off, weigh the precipitate (M1) which is  $C_6H_4(COOK)_2$  and Phthalic Anhydride % may be calculated as follows:

Phthalic Anhydride present by mass :  $(M1 \times 61.16) / (M2 \times \%NVM \text{ of Resin Soln})$

M1 → Mass in gm. of the precipitate obtained.

M2 → Mass in gm. of the Resin taken for test.

**4.5.1** The result is represented as the percentage of Phthalic Anhydride present in solid resin obtained from the paint.