

इंटरनेट

मानक

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“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 101-8-5 (1993): Methods of sampling and test for paints, varnishes and related products, Part 8: Tests for pigments and other solids, Section 5: Lead restriction test [CHD 20: Paints, Varnishes and Related Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bharṭṛhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 101 (Part 8/Sec 5) : 1993

(Reaffirmed 1999)
REAFFIRMED

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भारतीय मानक

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

भाग 8 वर्णकों और अन्य ठोसों के परीक्षण

अनुभाग 5 सीसा प्रतिबंध परीक्षण

(तीसरा पुनरीक्षण)

Indian Standard

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

PART 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 5 Lead Restriction Test

(Third Revision)

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BUREAU OF INDIAN STANDARDS
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October 1993

Price Group 2

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.

This standard (Part 8/Sec 5) is one of a series dealing with methods of sampling and test for paints, varnishes and related products. Three methods have been given. Any one method can be used but in case of dispute Method A will be treated as the referee method. This standard supersedes clauses 27 and 28 of IS 101 : 1964 'Methods of test for ready mixed paints and enamels (*second revision*)'.

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*)'.

AMENDMENT NO. 1 OCTOBER 1994
TO
IS 101 (Part 8/Sec 5) : 1993 METHODS OF SAMPLING
AND TEST FOR PAINTS, VARNISHES AND RELATED
PRODUCTS

PART 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 5 Lead Restriction Test

(Third Revision)

(Foreword, second cover, para 1, line 1) — Add the words '(Third Revision)' after the word 'Standard'.

(Para 2) — Substitute the existing text with the following:

"This standard (Part 8/Sec 5) is one of a series dealing with methods of sampling and test for paints, varnishes and related products. In this third revision three test methods have been prescribed for determination of lead in lead restricted paints. Any of the three methods can be used, but in case of dispute, 'Electrolysis Method' being the referee method shall be followed. This standard supersedes clauses 28 and 29 of IS 101 : 1964 Method of test for ready mixed paints and enamels (*second revision*)."

(Para 3) — Delete.

(Page 1, clause 1, para 1) — Substitute the existing text with the following:

'This standard (Part 8/Sec 5) prescribes methods of test for lead restriction and freedom from lead in paint and allied products. For lead restriction test any one of the following three methods may be used:'

(Page 1, clause 1) — Insert the following new clause 1.1 after clause 1:

'1.1 Electrolysis method shall be used as a referee method in case of any dispute.'

(Pages 1 and 2, clauses 2 and 3) — Renumber clause '2' as '3' and clause '3' as '2'. Also renumber the subsequent clauses.

(CHD 020)

Indian Standard

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

PART 8 TESTS FOR PIGMENTS AND OTHER SOLIDS

Section 5 Lead Restriction Test

(Third Revision)

1 SCOPE

This standard (Part 8/Sec 5) prescribes freedom from lead and the following three methods to determine lead restriction in paints and allied products:

- a) Electrolysis method;
- b) Molybdate method; and
- c) Sulphide method.

2 FREEDOM FROM LEAD

2.0 Outline of the Method

Determination of lead in lead-free paints is carried out by treating the ash of the paint with nitric acid and then with ammonium acetate solution, and precipitating lead as lead sulphide and finally oxidizing to lead sulphate.

2.1 Reagents

2.1.1 Dilute Nitric Acid — Lead-free, approximately 4 N.

2.1.2 Ammonium Acetate Solution — Lead-free, 10 percent *m/v*.

2.1.3 Concentrated Sulphuric Acid — Lead-free, sp gr 1.84.

2.1.4 Dilute Sulphuric Acid — 2 percent, obtained by dissolving 11 ml of concentrated sulphuric acid in one litre of water.

2.1.5 Ammonium Acetate — Solid, lead-free.

2.1.6 Hydrogen Sulphide — Gas, from Kipp's apparatus.

2.1.7 Hydrogen Sulphide Water — Saturated solution.

2.2 Procedure

Weigh 50 g of the material in a silica basin, and ash it carefully until only slight traces of carbon

remain. The temperature of the basin shall not be allowed to rise above faint red heat, as at higher temperatures some lead may be lost by volatilization. Treat the ash so obtained with dilute nitric acid. The quantity of acid is immaterial provided it is sufficient to extract the soluble matter, but avoid too great an excess since it has to be evaporated off. Allow the basin to stand on a boiling water-bath for at least three hours. In case a large quantity of insoluble residue is left, heat the basin on the water-bath overnight. Decant off the supernatant liquid through a filter paper and extract the insoluble residue again on a boiling water-bath for one hour with dilute nitric acid. Filter through the same filter paper and wash the residue thoroughly on the filter paper with hot water. Treat the residue on the filter paper with 10 ml of ammonium acetate solution, filter and wash again. Mix the filtrate and washings in a 500-ml evaporating basin, add 2 ml of concentrated sulphuric acid and evaporate the contents of the basin on a sand-bath till fumes appear. Add 100 ml of water to the basin and allow to stand on the boiling water-bath for 15 minutes. Then dilute the contents to about 150 ml and allow to stand overnight at room temperature. Filter the insoluble matter on a No. 42 Whatman filter paper (9 cm) and wash thoroughly with dilute sulphuric acid. Transfer the filter paper and residue to a small beaker, cover with 20 ml of water, and add 1 to 2 g of ammonium acetate. Heat the beaker on the water-bath for not less than half an hour, stirring the contents occasionally. Decant the liquid through No. 42 Whatman filter paper (9 cm). Repeat the extraction with water and ammonium acetate. Transfer all the insoluble matter including the filter pulp to the filter and wash throughout with warm water collecting the filtrate and washings in a 150-ml beaker. Pass hydrogen sulphide through the liquid for 10 to 15

minutes and filter the precipitated lead sulphide at once through a No. 40 Whatman filter paper (9 cm). Wash thoroughly but quickly with hydrogen sulphide water keeping the residue on the filter paper, if any, covered with liquid till washing is completed. Transfer the precipitate and filter paper to a tared silica crucible. Dry, carefully ignite to sulphate, cool and weigh.

2.3 Alternatively, determine the quantity of lead by either of the following methods:

a) By electrolysis after dissolving the precipitated lead sulphide in nitric acid, or

b) By polarographic method.

2.4 Calculation — Calculate as lead (Pb) and express the result as percent of the mass of the material taken for test.

3 LEAD RESTRICTION

3.1 Electrolysis Method

3.1.1 Procedure

Transfer about 5 g of well mixed paint to a tared evaporating dish and dry at 105°C to constant mass. Place the exact mass of the dried sample in a muffle furnace and ash it for 20 min, at 315°C, 40 min at 425°C and 1 h at 540°C. Cool in a dessicator. Extract the ash in a 250 ml beaker with 30 ml conc nitric acid and 80-ml. water and heat to boil. Filter into a 400-ml beaker using fine texture paper to prevent manganese dioxide from passing into the filtrate. Wash with water.

Dilute the filtrate to approximately 300 ml, add 20 ml of 20 percent solution of ammonium nitrate and 10 ml of 0.1 percent copper sulphate solution.

Heat nearly to boiling and electrolyse using platinum gauze anode that has been weighed previously.

Electrolyse for 15 min each at 1A, 2A and then at 3A current. Rinse the electrode three times in water with the current still on. Then remove the anodes, rinse in alcohol, dry for 15 min in an oven at 105 ± 5°C. Cool and weigh.

NOTE — The electrodes should be cleaned after each determination. This is best done by placing them in nitric acid solution (1 : 4) that contains a few ml of concentrated hydrogen peroxide (H₂O₂, 30 percent), rinsed with water and dried for next use.

3.1.2 Calculation

$$\text{Lead, percent by mass on non-volatile vehicle} = \frac{M \times 0.86623 \times 100}{W}$$

where

M = mass, in g, of lead oxide PbO₂, and

W = mass of non-volatile vehicle taken.

3.2 Molybdate Method

3.2.0 Outline of the Method

Paint is digested with concentrated sulphuric acid and nitric acid in order to convert lead to lead sulphate followed by extraction with ammonium acetate. Finally lead is precipitated as lead molybdate and weighed as lead molybdate.

3.2.1 Procedure

Transfer about 5 g of well mixed paint to a 400-ml beaker and dry at 105°C. Weigh accurately, add 20 ml of concentrated nitric acid and 15 ml of concentrated sulphuric acid and digest in order to remove all organic material and change lead to lead sulphate. Remove traces of nitric acid by repeated fuming with sulphuric acid. Cool, add 50 ml of water, heat to dissolve the salts and add 50 ml of ethyl alcohol and set aside for several hours. Filter through a paper pulp pad and wash with dilute sulphuric acid (1 : 20 v/v) containing 10 percent of ethyl alcohol. Dissolve the lead sulphate in three 10-ml portions of hot 20 percent ammonium acetate solution, followed by several washings with hot water. Treat the combined filtrate and washings with 2 ml of glacial acetic acid, heat to boil, add 10 ml of 5 percent aqueous ammonium paramolybdate solution and boil for a few minutes until the lead molybdate precipitate has coagulated.

Filter through a porcelain filtering crucible and wash with ammoniacal 2 percent ammonium nitrate solution. Heat to dull redness at 600–650°C to constant mass.

3.2.2 Calculation

$$\text{Lead content, percent by mass on non-volatile matter} = \frac{0.56436 \times W}{S} \times 100$$

where

W = mass of lead molybdate, and

S = mass of dry sample taken for test.

3.3 Sulphide Method

3.3.0 Outline of the Method

Determination of lead in lead restricted paints is carried out by precipitating the lead as sulphide from the separated pigment, which is finally oxidized to lead sulphate.

3.3.1 Procedure

Shake about one gram of the ground pigment obtained after treatment of paint as prescribed in IS 101 (Part 8/Sec 2), accurately weighed, continuously for one hour at room temperature with 1 000 times its weight of an aqueous solution of hydrochloric acid containing 0.25 percent by mass of hydrogen chloride. Allow

the mixture to stand for one hour and then filter. Precipitate the lead salt contained in the clear filtrate as lead sulphide, filter, heat the lead sulphide in air to convert it into lead sulphate, weigh, calculate as lead monoxide (PbO) and express the result as percentage on the dry weight of the material taken for test.

3.3.2 Calculation

$$\text{Lead (as PbO)} = \frac{M_1}{M} \times 100$$

where

M_1 = mass, in g, of the precipitate; and

M = mass, in g, of the sample taken for the test.

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