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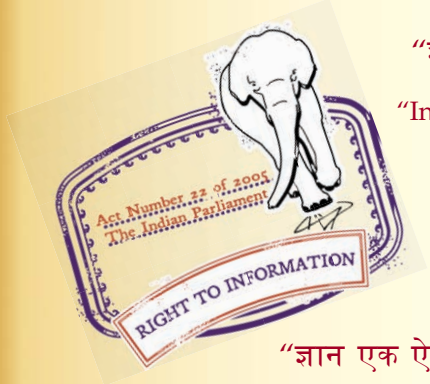
IS 2753-2 (1991): Methods for estimation of preservatives in treated timber and in treating solutions, Part 2: Determination of copper (in copper organic preservative salt) and pentachlorophenol [CED 9: Timber and Timber Stores]



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Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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IS 2753 (Part 2) : 1891
REAFFIRMED 2010

भारतीय मानक

उपचारित लकड़ी और उपचार करने वाले घोल में परिरक्षकों के आकलन की पद्धतियाँ

भाग 2 ताँबा (ताँबे के कार्बनिक परिरक्षक लवणों में) और पेन्टाक्लोरोफेनोल ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

METHODS FOR ESTIMATION OF PRESERVATIVES IN TREATED TIMBER AND IN TREATING SOLUTIONS

PART 2 DETERMINATION OF COPPER (IN COPPER ORGANIC PRESERVATIVE SALT) AND PENTACHLOROPHENOL

(First Revision)

UDC 674·048 : 543

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

June 1991

Price Group 2

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TO
IS 2753 (Part 2) : 1991 METHODS FOR ESTIMATION OF SERVATIVES IN TREATED TIMBER AND IN TREATING SOLUTIONS

PAR DETERMINATION OF COPPER (IN COPPER ORGANIC RESERVATIVE SALT) AND PENTACHLOROPHENOL

(First Revision)

(*Page* 2, *clauses* **3.3.1.1** *and* **3.3.2.1**) — Substitute '0.063 5' *for* '0.635' in the formula.

(CED 9)

Reprography Unit, BIS, New DElhi, India

Timber Sectional Committee, CED 9

FOREWORD

This Indian Standard (Part 2) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Timber Sectional Committee had been approved by the Civil Engineering Division Council.

IS 401 : 1982 'Code of practice for preservation of timber (*third revision*)' covers details of preservatives, methods of preservative treatment of timber, etc. This standard (Parts 1 and 2) details the methods of detection and estimation of the preservatives in such treated timber and treating solutions. While Part 1 covers determination of copper, arsenic, chromium, zinc, boron, creosote and fuel oil, Part 2 of this standard covers the determination of copper (in copper organic preservative salts) and pentachlorophenol.

This standard (Part 2) was first published in 1968. Among other things, it then provided for determination for copper in copper naphthenate only. In this revision the procedure for determination of copper has been extended to other copper organic preservative salts. Further, the formulae for calculation of percentage of pentachlorophenol in treating solution and treated timber have been modified.

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 FOR ESTIMATION OF PRESERVATIVES IN TREATED TIMBER AND IN TREATING SOLUTIONS

PART 2 DETERMINATION OF COPPER (IN COPPER ORGANIC PRESERVATIVE SALT) AND PENTACHLOROPHENOL

(First Revision)

1 SCOPE

1.1 This standard (Part 2) lays down methods for the quantitative estimation of the following in treated timber and in treating solutions:

a) Copper in copper organic preservative salt solution and in timber treated with copper organic salt, and

b) Pentachlorophenol in pentachlorophenol solution in oil and in timber treated with pentachlorophenol.

1.2 These methods are intended for the chemical analysis of oils containing the required preservative and wood treated with the same preservative.

2 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water [*see* IS 1070 : 1977 Specification for water for general laboratory use (*second revision*)] shall be employed in the tests.

3 QUANTITATIVE ESTIMATION OF COPPER IN COPPER ORGANIC PRESERVATIVE SALT AND IN TIMBER TREATED WITH COPPER ORGANIC PRESERVATIVE SALT

3.1 The sample in dilute or concentrated filtered solution should contain approximately 0·02 g of copper.

3.2 Reagents

3.2.1 *Ammonium Hydroxide, Concentrated*

3.2.2 *Sodium Thiosulphate Solution* — Dissolve 2·482 1 g of sodium thiosulphate in water and make up to one litre.

3.2.3 *Starch Solution* — 1 g soluble starch is made to paste in 5 ml of water. Add 50 ml water and boil for one minute with stirring, cool and add one drop chloroform. Use always a freshly prepared solution of starch.

3.2.4 *Nitric Acid, Concentrated*

3.2.5 *Sulphuric Acid, Concentrated*

3.2.6 *Potassium Iodide Solution* — 20 percent. Dissolve 20 g of potassium iodide in water and dilute to 100 ml.

3.2.7 *Sodium Thiocyanate Solution* — 20 percent Dissolve 20 g sodium thiocyanate in water and dilute to 100 ml.

3.3 Procedure

3.3.1 *In Treating Solution*

Take a known mass of filtered treating solution in a porcelain dish and evaporate the solvent over an electric hot plate under hood. Heat the dish strongly with a burner till all the organic matter is destroyed. Cool it for 5 minutes. Add 2 ml of sulphuric acid and 3 ml of nitric acid. Cover with a watch glass and heat gently until the sample is dissolved. Transfer the solution from the dish into a 300 ml Erlenmeyer flask using *dilute nitric acid.* Heat the flask until fumes of sulphur trioxide appear and a colourless or light green solution remains. Cool and wash down the sides with 5 ml water and repeat heating till fuming. Cool and add 20 ml of water. Wash down the sides of flask with water, boil for one minute, cool and neutralize cautiously with concentrated ammonium hydroxide until a permanent precipitate just forms. Add concentrated sulphuric acid drop by drop until the precipitate just dissolves. Boil down to a volume of 30 ml, cool to below 20°C and dilute to 125 ml.

Add 10 ml of 20 percent potassium iodide solution and 5 ml of 20 percent sodium thiocyanate solution and mix thoroughly by rotating the flask. Titrate with sodium thiosulphate solution, adding 2 ml starch solution just before the brownish colour of the iodine disappears. Stop the titration when no blue colour remains.

3.3.1.1 *Calculation*

$$\text{Percentage of copper} = \frac{V \times 0{\cdot}635}{M}$$

where

V = volume, in ml, of sodium thiosulphate consumed; and

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

3.3.2 *In Treated Timber*

Determine the density, in kilograms per cubic metre, of the wood sample. Grind the sample to sawdust or cut into small pieces with a knife or use increment borings[1]. Weigh accurately into a tared porcelain crucible about 10 g of the sample. Char the material slowly over a Bunsen burner. When the sample is charred heat strongly until all organic matter is destroyed, cool the ash for 5 minutes add 2 ml of sulphuric acid and 3 ml of nitric acid, cover with a watch glass and heat gently until the sample is dissolved. Transfer the solution in 300-ml Erlenmeyer flask using nitric acid and proceed as in **3.3.1**.

3.3.2.1 *Calculations*

$$\text{Kilograms of copper per m}^3 \text{ of wood} = \frac{V \times 0\,635}{M} \times \frac{M_1}{100}$$

where

V = volume, in ml, of sodium thiosulphate consumed;

M_1 = mass, in kg, of one m^3 of wood; and

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

4 QUANTITATIVE ESTIMATION OF PENTACHLOROPHENOL IN PENTACHLOROPHENOL SOLUTION IN OIL AND IN TIMBER TREATED WITH PENTACHLOROPHENOL

4.1 The method is suitable for analysis of one to ten percent pentachlorophenol in oils and up to two percent pentachlorophenol in wood with the limitation of not more than 2 g of organic material.

4.2 Reagents

4.2.1 *Calcium Hydroxide Powder* — having chloride content less than 0·005 percent.

4.2.2 *Potassium Nitrate Powder* — having chloride content less than 0·002 percent, and fineness of 425 micron.

4.2.3 *Lime Nitrate Mixture* — Mix 9 parts of calcium hydroxide with 1 part powdered potassium nitrate.

[1] One increment boring 2 cm long and 0·475 cm in diameter has a volume of 0·354 6 cm^3.

4.2.4 *Concentrated Nitric Acid* — vol 1·42, chloride free.

4.2.5 *Standard Silver Nitrate Solution* — 0·1 N. Dissolve 16·989 g of silver nitrate in water and make up the volume to one litre in volumetric flask.

4.2.6 *Ferric Ammonium Sulphate (Indicator)*

Dissolve 10 g of ferric ammonium sulphate in 10 ml of concentrated nitric acid and dilute to 100 ml with water.

4.2.7 *Ammonium Thiocyanate Solution*

0·1 N. Dissolve 7·51 g of ammonium thiocyanate in water and dilute to one litre. Standardize it against 0·1 N silver nitrate solution.

4.3 Procedure

4.3.1 *In Treating Solution*

Take 10 g of the lime nitrate mixture in a 100-ml iron crucible. Drop from a weighed dropping bottle approximately 0·5-1 g of the pentachlorophenol solution on the lime nitrate mixture in such a manner so as to give even distribution. Re-weigh the crucible with reagents and samples and obtain the sample mass. Cover the sample with an additional 20 g of lime nitrate mixture and tap the crucible gently. Heat the crucible on a Bunsen burner at 850°C to 900°C for 20 minutes. Sample should be free of unburnt carbon, if necessary increase the combustion time for 5-10 minutes. Cool the crucible and empty its contents in a 400-ml beaker by gently tapping the crucible. Place a cover glass on beaker and rapidly pour 70 ml of water on the contents of the beaker. Scrub the crucible with water and stirring rod equipped with a rubber policeman to loosen any remaining residue and add it to the beaker containing the sample. If desired 2 percent nitric acid may be used to wash the crucible. The volume of the solution of the beaker should not be more than 100 ml. Place the beaker in cold water and neutralize the lime with concentrated nitric acid with constant stirring. When the ignition mixture is dissolved, the solution should be acid to congo red paper (blue colour), if not add nitric acid dropwise until the solution is acidic. Not more than 2 or 3 ml excess of acid should be present.

Add 15 ml of standard 0·1 N silver nitrate solution from a burette, cover and gently boil for several minutes to coagulate the silver chloride precipitate formed. Cool and filter through Whatman No. 41 filter paper in a clean 400-ml beaker. Wash the beaker and the precipitate thoroughly with water till the wash water does not give any turbidity with ammonium thiocyanate. The volume should not exceed 200-250 ml after washing. Add 5 ml of indicator (**4.2.6**) and back titrate excess silver

nitrate with standard ammonium thiocyanate (**4.2.7**) from a 25-ml burette to a pink end point that lasts at least 5 minutes. Run a blank on all reagents and untreated oil, if possible the oil should be free from chlorides.

4.3.1.1 *Calculations*

Pentachlorophenol, percent by mass

$$= \frac{(A-B) \times 0{\cdot}532\ 7}{C}$$

where

A = (ml silver nitrate for sample) — (ml ammonium thiocyanate for sample) × (ammoium thiocyanate factor);

B = (ml silver nitrate for blank) — (ml ammonium thiyocyanate for blank) × (ammonium thiocyanate factor); and

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

4.3.2 *In Wood Treated with Pentachlorophenol*

Take 10 g of lime nitrate mixture in a 100-ml iron crucible, then add 5-10 increment borings[1], stir gently to ensure complete contact of the lime nitrate mixture with wood and heat the crucible on a Bunsen burner till all the organic matter is destroyed. Then proceed as given in **4.3.1**.

4.3.2.1 *Calculation*

The calculations for treated wood will be made on volume basis (since the moisture content of the wood is often in doubt).

Volume basis kilogram pentachlorophenol per cubic metre of wood $= \frac{(A-B) \times 5{\cdot}327}{D}$

where

A = (ml silver nitrate for sample) − (ml ammonium thiocyanate for sample) × (ammonium thiocyanate factor);

B = (ml silver nitrate for blank) − (ml ammonium thiocyanate for blank) × (ammonium thiocyanate factor); and

D = volume of sample, in cm^3.

[1] One increment boring 2 cm long and 0·475 cm in diameter has a volume of 0·354 6 cm^3.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
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